

**STUDIES ON OSMOTIC AND CONVECTIVE  
DRYING OF ALOE VERA GEL**

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THESIS

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**CERTIFICATE - I**

This is to certify that **Mr. Pradip Atmaram Borkar** has successfully completed the comprehensive examination held on 18<sup>th</sup> March 2008 as required under the regulations for Ph.D. in Processing and Food Engineering.

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**CERTIFICATE-II**

This is to certify that this thesis entitled “**Studies on osmotic and convective drying of aloe vera gel**” submitted for the degree of **Ph.D.** in Agricultural Engineering in the subject of **Processing and Food Engineering** embodies bonafide research work carried out by **Mr. Pradip Atmaram Borkar** under my guidance and supervision and that no part of this thesis has been submitted for any other degree. The assistance and help received during the course of investigation have been fully acknowledged. The draft of the thesis was also approved by the advisory committee on 18<sup>th</sup> April 2011.

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# CHAPTER 1

## INTRODUCTION

Aloe vera (*Aloe barbadensis* Miller) is one of the important plants used for its medicinal as well as cosmetic value for several thousand of years (Grindlay and Reynolds, 1986; Koga, 1998). It has been recorded to be used in many ancient cultures; from Egypt, Greece, Rome to China and India. In biblical times the Egyptians hailed aloe vera as the plant of immortality, the Chinese called it their elixir of youth (Rolf and Zimmerli, 2000). Aloe vera, though to have originated in tropical Africa, is now being cultivated in warm climatic areas of Asia, Europe and America as well (Bhattacharjee, 2005). Aloe has been extensively grown in the Caribbean island as well as Mexico since the early 1800s. The plant is well known by several names in India such as Ghrit Kumari, Guar Patha and Indian Aloe. However, because of its wide acceptability, it is well distributed in many parts of the country. There are over 300 identified species worldwide (Danhod, 2000). Aloe vera is a perennial plant of Liliaceae family with turgid green leaves joined at the stem in a rosette pattern (Grindlay and Reynolds, 1986; Koga, 1998). Its leaves are 480-655 mm long, 55-115 mm wide at the base and 19-32 mm thick (Chandegara and Varshney, 2007).

Aloe vera is cultivated under approximately 23,600 hectares world wide. According to a report on aloe vera cultivation, published by International Aloe Science Council ([www.iasc.org](http://www.iasc.org)) USA supplies the major bulk of aloe vera in world market having a share of 60-65%, whereas Latin American countries supply 20-25%. Australia, China and India combined have a market share of only 10% (Rath, 2003). The major areas under aloe vera cultivation in India are Alwar in Rajasthan, Satnapalli in Andhra Pradesh and Rajpipla in Gujarat. It is also cultivated in the dry areas of states of Maharashtra and Tamil Nadu. Total production in India is estimated to be about 1, 00,000 tonnes (Anonymous, 2006). The annual consumption of aloe vera extract by the Indian pharmaceutical industries is 200 tonnes which is met from the wild sources from the states of Maharashtra and Tamil Nadu. Ayurvedic pharmacies are using only 1% of the total production from India ([www.ics.trieste.it](http://www.ics.trieste.it)).

The price of dried aloe vera leaves in India ranges from ` 600/- to ` 1000/- per kg depending upon the aloin content and colour of the dried aloe vera. However, the rate of fresh aloe vera leaves varies from ` 4-5 per kg (Chauhan *et al.*, 2007). Present market is flooded with lot of medicinal and cosmetic products containing aloe extract as a functional ingredient. Although aloe vera, is an industrial crop, has long been used in health foods, in the food industry, aloe vera has been utilized as a resource of functional food, especially for the

preparation of health food drinks, beverages like tea, milk, ice-cream, and confectionary and so on (Seoshin *et al.*, 1995). The amount of aloe vera that finds its application in the pharmaceutical industry is not negligible as far as the manufacturing of tropical ointments, gel preparation, tablets and capsules are concerned. Aloe vera gel also finds its application in the cosmetic and toiletry industry, where it is used as a base for the preparation of creams, lotions, soaps, shampoos, and facial cleaners (Grindlay and Reynolds, 1986; Koga, 1998).

Reports credit that aloe has antitumor (Loadman and Christopher, 2001), antidiabetic (Beppu *et al.*, 1993), and antityrosinase properties (Yagi *et al.* 1987) in addition to treatment of gastric ulcers (Maze, *et al.*, 1997). The action of aloe gel as a moisturizing agent is still a popular concept (Briggs, 1995).

According to the literature, the chemical composition of aloe plants is largely dependant on the species analyzed. A prominent feature of Aloe vera fillet is its high water content, ranging from 98.5% to 99.25% of fresh matter, with pH range of 4.4 to 4.7. More than 60% of the remaining solid is made up of polysaccharides (McAnalley, 1993; Femenia *et al.*, 1999). The plant contains many vitamins excluding vitamin D but including the important antioxidant vitamins A, C and E. Vitamin B<sub>1</sub> (thiamine), niacin, vitamin B<sub>2</sub> (riboflavin), choline and folic acid are also present. Some authorities also suggest the presence of vitamin B<sub>12</sub> (cynocobalamin) in trace amount which is normally only available from an animal source (Coats, 1979)

Aloe vera leaves are formed by a thick epidermis (skin) covered with cuticle surrounding the mesophyll, which can be differentiated into chlorenchyma cells and thinner walled cells forming the parenchyma (fillet). The parenchyma cells contain a transparent mucilaginous jelly which is referred to as aloe vera gel consisting of long chain polysaccharides. The gel is a colourless, odourless and hydrocolloid with several natural beneficial substances (Grindlay and Reynolds, 1986).

Aloe vera gel, like most natural juices, from fruit and vegetable, is an unstable product when extracted and is subject to discolouration and spoilage from contamination by microorganisms (Coats, 1979). The great success of aloe vera as a commodity for use in nutritional foods and cosmetics is due to the proper stabilizing procedures that enable processors to store and ship its gel without fear of spoilage throughout the market places of the world.

Unfortunately, because of improper processing procedure, many of these so-called aloe products contain very little or virtually no active ingredients (Ramachandra and Rao, 2006). In view of the known wide spectrum of biological activities possessed by the leaves of the aloe vera plant and its wide spread use, it has become very important to evolve a better

method of preservation for increasing the shelf life and maintaining the quality of aloe vera gel and this can be achieved by some type of processing e.g. heating, dehydration (Chang *et al.*, 2006). The main purpose of drying products is to allow longer periods of storage, minimize packaging requirements and reduce shipping weights (Okos *et al.*, 1992). Controlled drying might also lead to an overall improvement in the quality and economical value of the final products. The challenge of aloe vera drying is retention of nutrients to maximum while reducing the moisture content of product to a level where microbiological growth should not occur.

A faster dehydration that yields a higher quality product is always required. It is generally known that freeze-drying produces highest quality dehydrated products, but this technique is very expensive. Canning is another common method of preservation of foods, but due to high cost of metallic container, bulky and heavy canned products and problems of disintegration and discolouration this technology has also become quite expensive (Shetty and Ranganna, 1972). Hence, a method of convective drying in combination of osmosis (in which partial dehydration of the fruit is brought about by immersing them in osmotic solution using controlled heat transfer applications and then drying) could be a good solution (Jayaraman and Das Gupta, 1992).

Osmotic dehydration is one of the methods of moisture removal where the product is dried by concentration difference or by use of osmotic pressure difference. When two solutions of different concentrations are separated by a semi permeable membrane there is a tendency to establish uniform osmotic pressure throughout the system. The solute molecules being large in size cannot pass through the membrane. Hence, the solvent molecules pass through the membrane from dilute to concentrated solution until a uniform osmotic pressure is established throughout the system. The phenomena of transfer of water from dilute solution to concentrated solution can be made applicable for partial dehydration of fruit slices. The fruit cells contain soluble sugar and other dissolved solids. The cell wall can act as semi permeable membrane and when the fruit/vegetable slices are immersed in concentrated sugar solution, a difference of osmotic pressure is set up with a flow of water starting from the fruit tissue cells to the outside in order to equalize-the-osmotic pressure on both the sides. The rate of osmosis, being proportional to the difference of osmotic pressure, is maximum at the beginning and slowly decreases. About 50% reduction in weight is found to occur within 3h at 45-48°C temperature in most fruits (Barbosa- Canovas and Vega-Mercado, 1996). Direct osmotic dehydration is therefore a simultaneous water and solute diffusion process. Its application to fruits and, to a lesser extent to vegetables has received considerable attention in recent years as a technique for production of intermediate moisture foods (IMF) and shelf

stable products (SSP) or as a pre-concentrating treatment to reduce energy consumption and /or heat damage in other traditional drying processes.

The development of a simple osmotic dehydration process for fruits necessitates the study of the osmotic kinetics and air drying behaviour of osmosed products. Therefore, controlled and regulated osmotic process plays an important role in improving the quality of dehydrated products and this can be achieved by optimization of osmotic process. Similarly, the limitation for large scale production of dehydrated fruit slices using an osmotic concentration process is the quantity of diluted sugar syrup left at the end of the process. The process can become economically feasible, when the sugar syrup is reconcentrated and reused. Also, there is a need to investigate the osmo-air drying process in order to get shelf stable quality dehydrated products. Air drying leads to physical and biochemical changes in food stuffs. These changes are frequently disastrous for the product quality (browning reaction and vitamin destruction). So to improve the product quality, it is important to optimize the drying conditions. The quality of the product needs to be evaluated for the acceptance of the consumer. The functional characteristics of the final dehydrated products such as water activity, rehydration and textural properties are essential to get a better picture about the product regarding its keeping quality. In addition, sensory evaluation has to be undertaken to assess the product characteristics such as taste, colour, flavour and appearance to the level of the consumers' acceptance.

The osmotic parameters like water loss and sugar gains can be correlated with osmosis time as a function of operating conditions. These relationships give only a qualitative information of the process, but the modelling of the process is necessary from the engineering point of view to apply the kinetics data in designing the equipments for the osmotic process and control. This information could be integrated to develop a process technology for commercial production of quality dehydrated fruits/vegetables.

Removal of 40-50% of the water from the fresh ripe fruits/vegetables by osmosis followed by other means of drying provides products with good quality in respect of appearance, taste, flavour and colour. The product could be used as ready to eat snack items or powdered and used for mixes with milk powder or making confectionery items and ice creams. This process could be commercially attractive. The osmotic dehydration process has been studied for many fruits and vegetables, such as apple, banana, carrot, cherry, citrus fruits, grape, guava, mango, *etc.* (Ponting *et al.*, 1966; Flink, 1980; Maguer, 1988; Fito, 1994; Ahmed and Choudhary, 1995 and Chaudhary *et al.*, 2000) and almost negligible information is available on the osmotic dehydration of the aloe vera with regard to the drying kinetics. The development of various processed foods and the high quality dried aloe vera are urgently

needed for enhancement of aloe vera consumption and their competitive values. Therefore, this study will definitely be helpful in development of a good quality consumer acceptable product from aloe vera gel.

Keeping in view the high rising global demand and India's conducive geo-climatic conditions, commercial cultivation and utilization with the application of improved technology can be of great value for aloe vera crop. This will fetch India good foreign reserves as well as domestic economic prosperity.

In light of the above points a study was undertaken with the following objectives:

1. To study osmotic dehydration behaviour of aloe vera gel
2. To optimize the process parameters for osmotic dehydration of aloe vera gel
3. To study convective drying behaviour of osmotically dehydrated aloe vera gel
4. To optimize drying parameters for convective drying of osmotically dehydrated aloe vera gel
5. To evaluate quality of dried product

## CHAPTER II

### REVIEW OF LITERATURE

The drying process is one of the most promising methods for food preservation. Open sun drying alone had been practiced until the turn of the 20<sup>th</sup> century when mechanical drying began to replace the traditional practice. Constant efforts of the researchers have resulted in the development of better techniques and advancement of knowledge in the field of drying. Osmotic and convective dehydration are some of such techniques. This chapter deals with the review of research work carried out by various research workers related to food value of aloe vera, aloe vera processing, osmotic dehydration, effect of process variables on osmotic dehydration and mass transfer during osmotic dehydration. In addition drying of fruits and vegetables including aloe vera and convective drying of osmotically dehydrated fruits are also incorporated in this chapter. The status of the mathematical modelling of convective drying is also reviewed.

#### 2.1 Food Value of Aloe vera

Aloe vera can be used as potential source to develop a wide variety of food products. It can also be incorporated in other food products to enhance their nutritional value. A refreshing juice, ready-to-serve drink, health drink, sport drink, soft drink, diet drink, laxative drink, sherbets etc. can be prepared from aloe. The fleshy portion can also be converted into candies, squash, jam, jellies, bar, munch etc (Fig. 1). Additionally, it can be incorporated to dairy products e.g. yoghurt, curd, lassi, ice-creams etc. as a dairy alternative. The gel can be dried using suitable drying techniques and the dried powder can be used in the development of various products (Chauhan *et al.*, 2007).

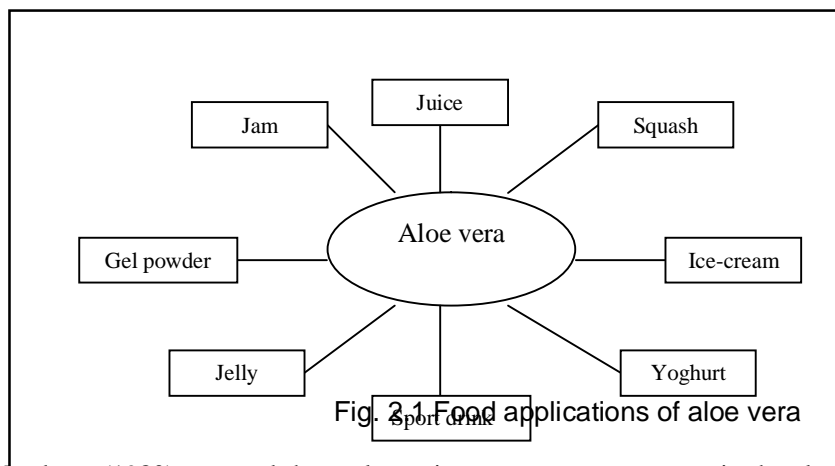


Fig. 2.1 Food applications of aloe vera

Meadows (1980) reported that at least six enzymes were present in the aloe vera gel including braykinase, cellulose, carboxypeptidase, catalase, amylase and oxidase. One important enzyme, carboxypetidase inactivates bradykinase produces and anti-inflammatory

effect. During the inflammatory process, bradykinase produces pain associated with vasodilatation and therefore its hydrolysis reduces these components and produces an analgesic effect.

Wang and Tung (1993) studied the minerals composition of aloe vera juice and reported that potassium and chloride concentrations appear to be excessive for most plant products, whereas the sodium content was less than average. Calcium and magnesium were also found along with manganese, copper, zinc, chromium and iron. Magnesium lactate inhibits histidine decarboxylase and prevents the formation of histamines from the amino acid histidine. Histamine is released in many allergic reaction and cause intense itching and pain. The prevention of its formation may explain the antipyretic effect of aloe vera.

Ishii *et al.* (1994) stated that the anthraquinones present in aloe latex, has direct virucidal effects. It inactivates various enveloped viruses such as herpes simplex, varicella-zoster and influenza. Aloe gel has also been demonstrated to have direct inhibitory effects on both tumor initiation and promotion. Anthraquinone present in aloe latex also functions as laxative stimulation. Aloe latex has been shown to increase intestinal water content, stimulate mucus secretion and increase intestinal peristalsis. Aloe juice is also found to prevent human pulmonary carcinogenesis (lung cancer) as well as it is effective in treating inflammatory bowel diseases. It can rebalance the intestines of diarrhea patients by regulating gastrointestinal pH while improving gastro intestinal motility, increasing stool specific gravity and reducing populations of certain fecal microorganisms, including yeasts. It also helps to detoxify the bowel, neutralize acidity and relieve constipation as well as gastric ulcers

Femenia *et al.* (1999) analysed lyophilized aloe vera plant fractions and reported that lipids represented a minor fraction in all samples ranging from 2.71% to 5.13% of the freeze-dried material. Protein was also a minor fraction, ranging from 6.33% for the skin to 8.92% in the gel. The skin contained 11.22% of soluble sugars, whereas this amount increased to 16.48% in the filet and to 27.81% in the gel, being one of the most important components in this latter fraction. The ash content was relatively high, in particular in the gel fraction where it accounted for up to 23.61% of the dry matter. Ca, K, Na and Mg were the predominant mineral elements in all the aloe fractions. In particular, Ca was the main mineral element in all fractions except in the gel where Na and K were detected in higher amounts.

Lee *et al.* (1999) prepared vinegar from aloe vera juice by using *Acetobactor* sp. Yoghurts were also prepared using aloe vera powder.

Aloe gel is the colourless gel contained in the inner part of the fresh leaves (Reynolds and Dweck, 1999). The gel consists primarily of water (>98%) and polysaccharides (pectines, cellulose, hemicellulose, glucomannan, acemannan and mannose derivatives). Acemannan is

considered the main functional component of aloe vera and is composed of a long chain of acetylated mannose (Djeraba and Quere, 2000; Lee *et al.*, 2001). Aloe gel is often commercialized as powdered concentrate. Traditionally, aloe vera gel is used both, topically (treatment of wounds, minor burns, and skin irritations) and internally to treat constipation, coughs, ulcers, diabetes, headaches, arthritis, immune-system deficiencies (Eshun and He, 2004; Vogler and Ernst, 1999). The Physiological activity of aloe vera polysaccharides has been widely reported. Glucomannan and acemannan were proved to accelerate wound healing, activate macrophages, stimulate the immune system, and have antibacterial and antiviral effects (Davis, *et al.*, 1987; Davis, *et al.* 1988; Djeraba and Quere, 2000; Pugh, *et al.*, 2001; Tan and Vanitha, 2004; Visuthikosol, *et al.*, 1995). Mannose-6-phosphate, a sugar constituent of aloe vera gel, was demonstrated to have wound healing properties (Davis, *et al.*, 1994). A number of glycoproteins present in aloe vera gel have been reported to have antitumor and antiulcer effects and to increase proliferation of normal human dermal cells (Chi *et al.*, 2001; Yagi *et al.*, 1997; Yagi *et al.*, 2003). However, statistically significant clinical studies on the efficacy of aloe vera gel on human health are very limited and often inconclusive (Eshun and He, 2004).

Takeyama *et al.* (2002) studied effect of high pressure and microwave heat treatments on the content of polysaccharide composition of dietary fibre and reported that soluble dietary fibre was more in unheated samples of aloe vera.

Jun *et al.* (2004) studied the effect of processing conditions e.g. temperature, pH, sucrose, vitamin C and citric acid, on the stability of colour and gelatinoids in aloe juice and found that stability was negatively affected by increasing sucrose and citric acid concentration while vitamin C and sodium chloride at low concentrations improved the stability.

Wei *et al.* (2004) prepared a health beverage from fresh aloe vera leaves. The leaves were washed, sterilized, ground, pulped and filtered, then mixed with different concentrations of Dangshen and Maidong juices and Chinese herbs.

Wolf and Danninger (2004) filed patent for making aloe vera gel and powder and their application in manufacture of foods.

As per Chauhan *et al.* (2007) aloe contains more than 200 substances which are responsible for its different types of health and nutritional benefits. He reported that aloe vera extract could be used to treat wounds, stomach complaints, constipation, headaches, oral problems, hair loss, insect bites, kidney ailments and skin irritations. He further claimed that aloe has a long history of use for external and internal applications in the wound healing process. Aloe gel is a mild analgesic relieving itching, swelling and pain. It is also endowed

with antibacterial and antifungal properties besides and facilitating trouble free blood flow to wounded areas and stimulates synthesis of fibroblasts responsible for wound healing.

## 2.2 Aloe vera Processing

Grindly and Reynolds (1986) studied the properties and modern uses of aloe vera leaf parenchyma gel. They reported that aloe vera has been used since early times for a host of curative purpose. Also aloe vera gel has come to play a prominent role as contemporary folk remedy. Numerous optimistic, and in some case extravagant, claims have been made for its medicinal properties. Also through chemical analysis they conclude that gel contain various carbohydrate polymers, notably either glucomannans or pectic acid, along with a range of other organic and inorganic components.

Yaron (1993) has extracted gel from full sized mature leaves and half young leaves picked from the same shrub. After removal of the peel the colourless hydro parenchyma was ground in a blender and centrifuged at 10,000 rpm for 30 min at 4°C to remove the fibres. Leaves weighing 800 g produced about 300 ml of gel.

Shafi *et al.* (2000) developed a commercially viable process for preparing a stable and pharmacological active crystalline substance from the fresh whole leaf meal and tested the product on experimental animal and volunteers for wound healing remedy for all kinds of damaged skin conditions.

Bhargava (2006) suggested that aloe vera gel is chiefly composed of complex sugars (acemannans) and anthraquinone glucosides. As a result aloe vera gel is used as astringent, moisturizer, anti-wrinkle and anti-aging agent, antioxidant, antibiotic, antifungal agent, gut-cleanser and as an immune modulator. As a result the current demand for aloe gel and its planting material has increased million fold.

Chang *et al.* (2006) studied the effects of heat treatment on the stabilities of polysaccharides substances and barbaloin in gel juice from aloe vera. They reported that the polysaccharides from aloe exhibited a maximal stability at 70°C decreasing either at higher or lower temperature and also found that heating promoted a remarkable decrease in barbaloin content depending on the temperature and time as compared to effect on polysaccharides.

Jain *et al.* (2006) standardized the squash and jelly formulated using aloe vera for their ingredients and procedures. These were evaluated by group of 20 panel members for acceptability on nine point hedonic scale. Nutritional analysis was conducted for proximate composition on dry weight basis. Vitamin C was estimated on fresh weight basis. They reported that 100 g of fresh aloe vera contains 750 mg of vitamin C. Dry powder of aloe vera contains 11.64 mg protein, 4.25 mg fat, 63.73 mg carbohydrates 9.35 mg fibre and 6.17 mg ash.

Ramachandra and Rao (2006) compiled the innovative process technologies of aloe vera leaf gel. They reported that aloe vera is highly potential, functional and valuable ingredient that exhibits relatively impressive biological functions of great interest in cosmetic, pharmaceutical and food industries. They also revealed the present processing technologies viz., gel stabilization technique, biological activity of aloe vera leaf gel, spray drying, freeze-drying and traditional hand filleting process. The process technologies like desiccant dehydration, active aloe process, time temperature and sanitation process, whole leaf aloe vera processing and low temperature short time heat treatment process have shown their credibility as innovative process technologies. They also made SWOT analysis to emphasize the scope for opportunities in investment, health benefits out of innovative process technologies.

Chandegara and Varshney (2007) studied the principle of centrifugation for the extraction of gel from aloe vera leaves. The treatment consist of three level of centrifuge temperature i.e. 5, 10 and 32 °C (ambient), with centrifuge speed of 10000 rpm and 30 min centrifuge duration. They reported that 5 °C temperature, 10000 rpm speed and 30 min duration of centrifuge yield in higher viscosity, lower refractive index and optical density with optimum recovery of gel.

Bozzi *et al.* (2007) studied the quality and authenticity of commercial aloe gel powders. They compared and examined nine products from leading international suppliers with fresh aloe vera gel. They reported that only three products out of nine analysed, contained satisfactory amount of acemannan.

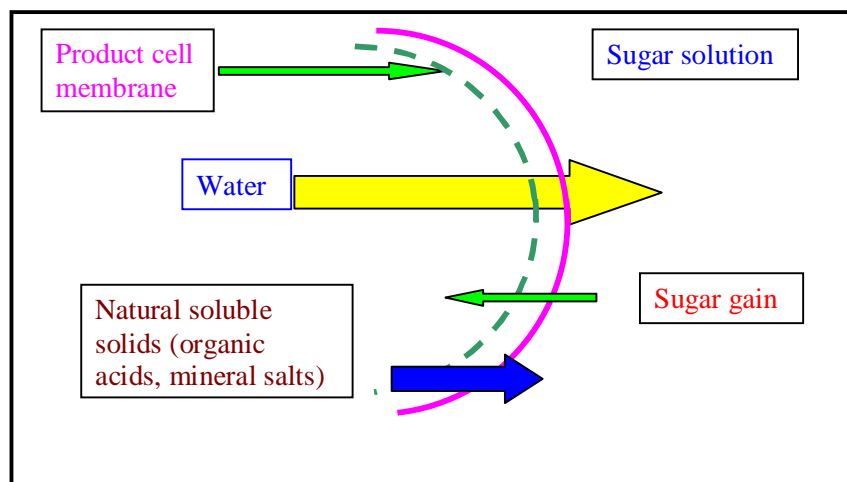
Gautam and Awasthi (2007) studied the process of making aloe vera leaf powder (AVLP). They reported that the recovery rate of AVLP was 3.2%. AVLP contained 18.5% crude fibre, 4.8% crude protein, 2.2% crude fat, 14.0% total ash, 48.0% carbohydrate and 231 kcal energy value. It contained substantial amounts of iron (64.8 mg/100g), ascorbic acid (27.0 mg/100g) and  $\beta$ -carotene (335.8 mg/100g). Dietary fibre was 21.3% while reducing and non-reducing sugars each were 76 mg/10g. The pH of the powder was 4.8, colour of the powder was olive green, and maximum water retention of the powder was obtained on 40 mesh sieve size.

### **2.3 Osmotic Dehydration**

Osmotic dehydration, also termed as “Dewatering and Impregnation Soaking Process (DISP)” (Raoult *et al.*, 1991a) is a water removal process which is based on placing foods, such as fruits and vegetables into concentrated solutions of soluble solutes, having higher osmotic pressure. The driving force for water removal is the concentration gradient between the osmotic solution and the intercellular fluid while the cell wall, acts as a semi permeable

membrane. Since the membrane is only partially selective there is always some solute diffusion (uptake) into the food.

Therefore, osmotic dehydration as shown in Fig. 2.2 has at least two major simultaneous counter current flows: (1) water diffusion out of the food into the solution, at a faster rate initially and slowly afterwards and (2) solute penetration in the opposite direction, at a slow rate initially but increasing with time. Considerable amount of solute penetration takes place if the duration of osmotic dehydration is long. There is also some leakage of solute (sugar, organic acid, minerals, salts, *etc.*) across the membrane. Though quantitatively negligible, it may be essential as far as organoleptic or nutritional qualities are concerned (Ponting *et al.*, 1966; Hawkes and Flink, 1978; Rahman, 1992; Lewicki and Lenart, 1995; Fito *et al.*, 2001). The process generally results into a partially dehydrated product.



**Fig. 2.2 Mass transfer in osmotic dehydration of fruits**

The use of osmosis decreases water activity in the food. As the permeability of the plant tissues is low to high molecular weight compounds such as sugars, salts, *etc.*, therefore, resulting material is impregnated with osmo active substances in the surface layers only. On the other hand water is removed by osmosis and the cell sap is concentrated without a phase transition of the solvent. The water flux is much larger than the counter current flux of osmo-active substance. This is the reason that process is called osmotic dewatering or osmotic dehydration. The dried plums, raisins, candied fruits are some of the examples.

Thus, osmotic dehydration is a low temperature dehydration technique where partial removal of water is achieved by the direct contact of the solid food, in a hypertonic medium *i.e.* highly concentrated sugar or salts solutions, through a semi permeable membrane until equilibrium is reached. The applications of osmosis to food

processing as a means of dehydration have been primarily motivated by economical factors and the quality improvement of the final product. This has been often proposed as a first step followed by various kinds of drying methods such as air, vacuum or freeze-drying.

**There are number of advantages of the osmotic dehydration process. The main advantage is that it minimizes the damage to the tissues and removes water without phase change, as compared to other methods of drying. Some of the merits of the process as described by various researchers (Ponting *et al.*, 1966; Jackson and Mohammed, 1971; Dixon *et al.*, 1976; Islam and Flink, 1982; Lenart and Lewicki, 1988; Rahman and Lamb, 1991; Chaudhary *et al.*, 1993; Karathanos *et al.*, 1995; Vijayanand *et al.*, 1995; Welti *et al.*, 1995; Sankat *et al.*, 1996; Sethi *et al.*, 1999; Rosa and Giroux, 2001; Fito *et al.*, 2001; Jain and Verma, 2003) are as follows:**

- It preserves the wholesomeness of the fruit, as cell damage is minimum.
- Mild heat treatment favours colour and flavour retention resulting in product with superior organoleptic characteristics and textural attributes.
- The use of artificial preservatives may be avoided.
- The process is quite simple, economical and does not require any sophistication. As there is no phase change, the energy requirement is 2-3 times less compared to the conventional drying.
- The process is economically viable for highly perishable fruits as blanching may be eliminated.
- It prevents the enzymatic browning.
- Acid removal and sugar uptake by fruits modifies the composition and improves the taste and acceptability, which is called candying effect.
- Constant immersion of product in osmotic agents avoids the use of CO<sub>2</sub>, thus product retain better colour.
- Salting may improve the acceptability of few vegetables.

Osmotic dehydration has been used as a means for drying fruits and vegetables long back, but it came under scientific investigation with pioneer work of Ponting *et al.*, 1966. It remained in dormant stage after few years of its introduction and again gained sudden interest in last decades.

Osmotic dehydration technique does not reduce the water activity to required level to hinder the proliferation of micro organisms. The technique extends the shelf life of the material to some degree. Hence application of other methods such as freezing, pasteurization, vacuum drying, air-drying or freeze-drying is necessary as shown in the schematic diagram of osmotic dehydration process (Fig. 2.3).

The osmotic dehydration process has been applied to many fruits and vegetables; as listed in Table 2.1 along with process conditions studied. It has been pointed out that this method is not successful in case of frozen cherry, citrus fruits, guava and tomato and moderately successful for blue berry, cherry and grapes (Jackson and Mohammed, 1971; Ponting, 1973).

## **2.4 Parameters Influencing the Osmotic Dehydration Process**

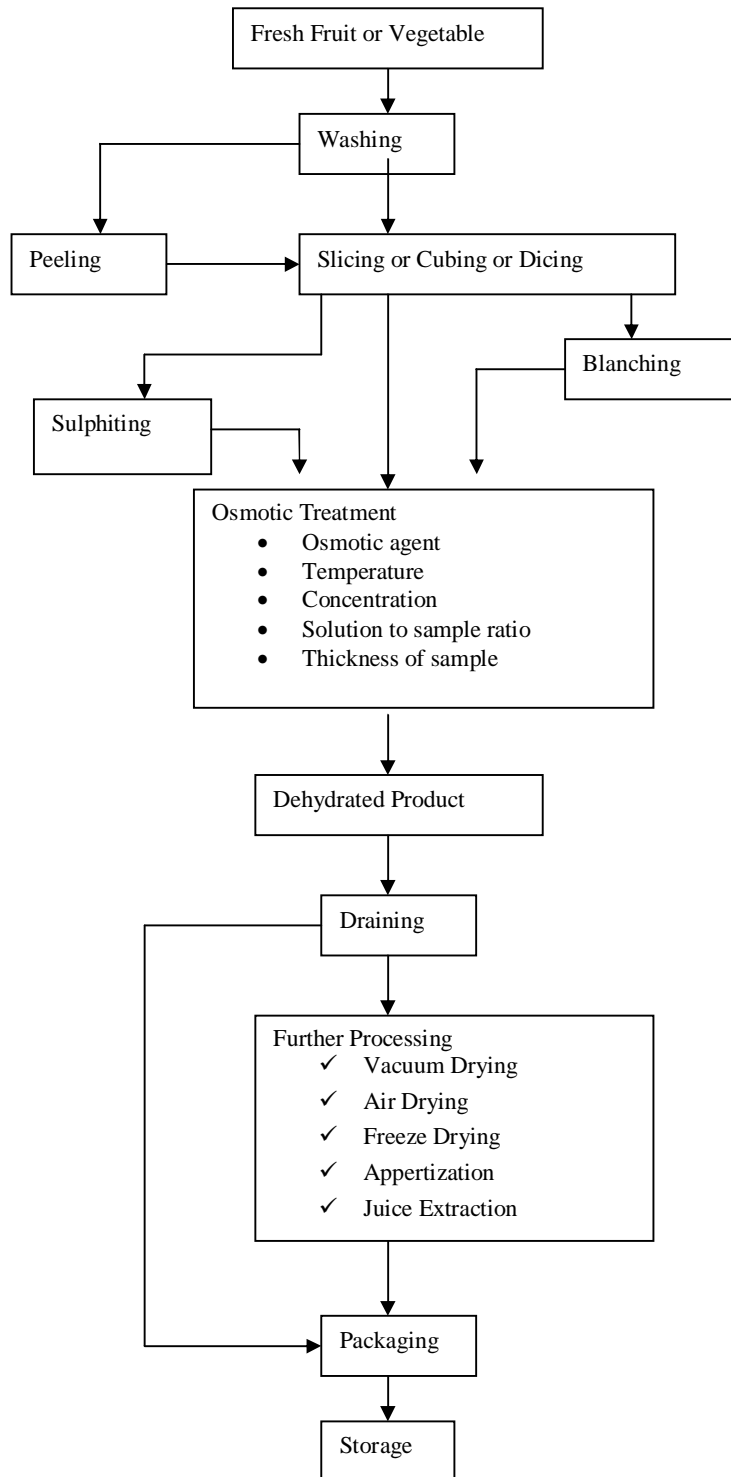
The influence of main process variables like variety, pre-treatment applied to product, concentration and composition of osmotic solution, temperature, process time, solution to sample ratio, *etc.* on the mass transfer mechanism and the product quality has been extensively studied (Ponting *et al.*, 1966; Conway *et al.*, 1983; Yang and Maguer, 1992; Argaiz *et al.*, 1994; Lazarides and Mavourdis, 1996; Kar and Gupta, 2001; Sereno *et al.*, 2001; Lewicki and Lenart, 1995; Fito *et al.*, 2001; Jain *et al.*, 2003a, b and c). Some of the factors considered in various investigations and their effects are presented in the following sections:

### **2.4.1 Variety**

It is a genetic character of fruit, which not only affect size and shape but also to some extent chemical composition, solid content, *etc.* These parameters greatly influence the quality of end product. However, very little work has been reported on this aspect. It is well known that the characteristics of fruits/vegetables depend on the type of soil, variety and agro-climatic conditions. The drying characteristics of the material may therefore, vary considerably. There is a lack of comparative studies on this aspect and some conflicting results for apple and papaya have been found (Jackson and Mohammed, 1971; Ponting, 1973; Mehta and Tomar, 1980; Moy and Kuo, 1985; Levi *et al.*, 1985). It was reported that, the fluctuations might be due to varietals differences.

### **2.4.2 Pre-treatments**

Foods are subjected to a number of preliminary unit operations and pre-treatment before osmo-drying. They include peeling, cutting, blanching freezing/thawing, sulphiting in fruit and vegetables, pressing, kneading, frying, *etc.* Pre-treatment are important in all methods of dehydration to inactivate enzyme and retention of typical characteristics of the product, *etc.* However, researchers are divided on the issue of the selection of an appropriate pre-treatment prior to osmosis. Thermal pre-treatment affects the integrity of natural tissue leading to severe effects on water loss and solid gain. Disruption of structural barriers



**Fig. 2.3 Process flow diagram for osmotic dehydration of fruits and vegetables**

**Table 2.1 Osmotic dehydration of fruits and vegetables**

<i>Product</i>	<i>Variety</i>	<i>Type of osmotic agent and concentration, %</i>	<i>Temperature, °C</i>	<i>Solution to sample ratio</i>	<i>Agitation</i>	<i>Thickness of sample, mm</i>	<i>Reference</i>
Apple	Golden delicious	Dry sugar and syrup	20-49	1 to 4	*	3	Ponting <i>et al.</i> , (1966)
	Mcintosh	Sucrose, 75 and Invert sugar, 50	25	-	-	3-8	Camirand <i>et al.</i> , (1968)
	Golden delicious	Sucrose, 55-75	30-60	25	-	13	Farkas and Lazar (1969)
	Golden delicious	Sucrose, 70	51	4	-	3	Dixon <i>et al.</i> , (1976)
	Golden delicious	Sucrose, 70	50	4	-	3	Dixon and Jen (1977)
	Mcintosh	Sucrose, 25-50 & NaCl, 5-10	23 (room temp)	20	-	3-4	Hawkes and Flink (1978)
	Red delicious	Sucrose, 70	70	4	-	15	Bolin <i>et al.</i> , (1983)
	Mcintosh	Sucrose, 50-70	30-50	4	*	10	Conway <i>et al.</i> , (1983)
	Golden delicious	Sucrose, 59 and Fructose, 60	Room temp	5	-	6-10	Lerici <i>et al.</i> , (1985)
	Golden delicious	Sucrose, 60-75	40-80	26	-	15 and 20	Videv <i>et al.</i> , (1990)
	Golden delicious	Sucrose, 70	50	3	-	13	Sharma <i>et al.</i> , (1991)
	Granny smith	Glucose, 55	45	> 10	*	7	Hough <i>et al.</i> , (1993)
	Mcintosh	Saccharose, 61.5	30-90	4	*	10	Lenart (1994)
	Granny smith	Ethylene glycol, 35-75	30-70	20	*	9	Saurel <i>et al.</i> , (1994a and b)
	Granny smith	Sucrose, 45-65	20-50	30	150rpm	7-8	Lazarides <i>et al.</i> , (1995)
	Golden delicious	Sucrose, 15-45	Room temp	>50	-	10	Karathanos <i>et al.</i> , (1995)
	Golden	Sucrose, 70	30	20	-	20mm dia disc	Azuara <i>et al.</i> , (1996)
	Granny smith	Sucrose, 50	5-40	-	Reys no. 7500	-	Mavourdis <i>et al.</i> , 1998a and b
	Golden	Sucrose, 70	40-70	6	-	10mm cube	Simal <i>et al.</i> , (1998)
	Granny smith	Sucrose, 65	20-50	-	-	20-30	Salvatori <i>et al.</i> , (1999a and b)
	Idared	Sucrose, 61.5	30	4	-	-	Lenart and Dabrowska (1999)
	Granny smith	Sucrose, 35-55	40	>20	-	-	Barat <i>et al.</i> , (1999)
	Granny smith	Sucrose, 65	40-60	-	-	20	Monzo <i>et al.</i> , (2000)
	Granny smith	Sucrose, 25-65	30-50	50	-	10	Barat <i>et al.</i> , (2001)
	Golden delicious	Sucrose, 60	20-70	9	-	-	Erle and Schubert (2001)
	Granny smith	Sucrose, 34-63	34-66	>60	-	12, 17, 20	Nieuwenhuizen <i>et al.</i> , (2001)
	Golden delicious	Sucrose, 40-60	5-60	40	-	8	Sereno <i>et al.</i> , (2001b)
	Idared	Sucrose, 61.5	30	4	-	10	Kowalska and Lenart (2001)
	Golden	Sucrose, 45-65	20-40	62.5	-	10mm cube	Cunha <i>et al.</i> , (2001)
	Fuji	Sucrose + salt	-	100	Rey. No. 300	9	Sacchetti <i>et al.</i> , (2001)
	Granny smith	Sucrose, 34-63	34-66	>60	-	12, 17, 20	Ramaswamy and Nieuwenhuizen (2002)
	Jonagold	Sucrose, 50	40	25	-	8mm slices	Taiwo <i>et al.</i> , (2002)
	Red delicious	Sucrose, 30-70	22-90	26.5	-	25mm cube	Sablani <i>et al.</i> , (2002)

Apricot	-	Corn syrup, 81	49	4	*	-	Ponting (1973)
	Reale	Sucrose, 70	25	5	NK	10mm halves	Giangiocomo <i>et al.</i> , (1987)
	Canino	Sucrose, 65	30-50	-	-	-	Shi and Maupoey (1993)
	Canino	Sucrose, 65	30-50	-	-	15mm cube	Shi <i>et al.</i> , (1995)
	Tonda di costigiole	Sucrose, 65 sorbitol, 65	25	3	-	10mm cube	Forni <i>et al.</i> , (1997)
Banana		Sucrose, 60	NK	NK	NK	NK	Hope and Vitale (1972)
		Sucrose, 60-80	49	4	*	-	Ponting (1973)
		Sucrose, 65	60	NK	NK	NK	Garcia <i>et al.</i> , (1974)
	Dwarf Cavendish	Sucrose, 70	27-60	1-3	*	8-10	Bongirwar and Sreenivasan (1977)
	Dwarf Cavendish	Sucrose, 70	90 for 3 min then 25-30	2.4	-	5-6	Ramanuja and Jayaraman (1980)
	Musa Cavendish	Sucrose, 47.2-72	30-50	20	-	11	Mauro and Menegalli. (1995)
	Cavendish	Sucrose, 35, 50, 65	25	-	-	10	Sankat <i>et al.</i> , (1996)
	Cavendish	Sucrose, 40-70	25-45	20	*	25 mm dia	Rastogi <i>et al.</i> , (1997)
		Sucrose, 32-50	20-60	-	-	-	Panagiotou <i>et al.</i> , (1998)
	Gros michel	Sucrose, 50-70	50-70	6	-	6	Waliszewski <i>et al.</i> , (2002)
Blue berries		Sucrose, 60-80	49	4	*	-	Ponting (1973)
		Sucrose	NK	NK	NK	NK	Kim and Toledo (1987)
		Sucrose, 47-63	37-53	-	*	-	Nsonzi & Ramaswamy (1998a and b)
Carrot		Sucrose, 54 +salt, 10	29	-	-	2	Jackson and Mohammed (1971)
		Sucrose, 40 + salt, 10	Room temp	-	*	-	Flink (1980)
		Sucrose, 10	20	NK	NK	NK	Speck <i>et al.</i> , (1977)
		Salt + ethanol, 0-15	10 and 30	10	NK	10mm dices	Maguer and Biswal (1984)
		Sucrose/salt	30	-	-	-	Lenart and Lewicki (1988)
		Sucrose, 70	30	-	-	-	Chopra and Verma (1994)
		Sucrose, 40-70	30-50	20	-	5	Rastogi and Raghavarao (1997)
	Sucrose, 50-55	23	-	-	-	Qi <i>et al.</i> , (1998)	
Cauliflower	Botrytis	Sodium chloride,5-25	40-90	2-4	-	-	Vijayanand <i>et al.</i> , (1995)
Cherry	Binaca di verona	Corn syrup/ sucrose, 70	25	5	NK	Dia 18 average	Giangiocomo <i>et al.</i> , (1987)
	Vittoria	Mixture/ Sucrose corn syrup, 70	25	5	-	-	Torreggiani <i>et al.</i> , (1987)
	Nefris	Saccharose, 61.5	30-90	4	*	-	Lenart (1994)
	Groniasta	Sucrose, 61.5	25	4	-	-	Lenart and Piotrowski ( 2001)
Citrus fruits		Sucrose, 60-80	49	4	*	-	Ponting (1973)
Coconut		Sucrose, 40-70	25-45	5	*	8	Rastogi and Raghavrao (1994)
		Sucrose, 40-70	25-45	5	*	7	Rastogi and Raghavrao (1995)

Grapes		Sucrose, 60-80	49	4	*	-	Ponting (1973)
	Thompson	Dry sugar	45, 60	1	*	-	Grabowski <i>et al.</i> , (1994)
Green beans	-	Sodium chloride, 17	20	-	-	-	Biswal <i>et al.</i> , (1991)
Kiwifruit	-	Sucrose, 30-50	20-60	-	0-170 rpm	4-20	Panagiotou <i>et al.</i> , (1998)
	Hayward	Sucrose, 70	25	3	-	10mm slices	Torreggiani <i>et al.</i> , (1999)
		Sucrose, 65	25-45	-	-	10mm slices	Escriche <i>et al.</i> , (2000)
	Hayward	Sucrose, 35-65	30	-	-	-	Chiralt <i>et al.</i> , (2001)
	Hayward	Sucrose, 0-65	30	5	-	10mm slices	Gianotti <i>et al.</i> , (2001)
	-	Sucrose, 65	25-45	-	0-170 rpm	10mm slices	Escriche <i>et al.</i> , (2002)
Mango	Green	Salt, 25	29	-	-	10mm slice	Jackson and Mohammed (1971)
	Ripe	Sucrose, 60	29	-	-	10 mm dice	Jackson and Mohammed (1971)
	Ripe	Salt, 25	NK	NK	NK	NK	Hope and Vitale (1972)
	Dashhari	Sucrose, 60	21	3 to 6	NK	10mm slice	Moy <i>et al.</i> , (1978)
	Ataulfo	Sucrose, 40-60	25	2	-	5mm slices	Wolti <i>et al.</i> , (1995)
	Keitt	Cerelese, 22-39.5	25	32	-	4mm slice	Nieto <i>et al.</i> , (2001)
	Kent	Sucrose, 35-65	30	-	-	-	Chiralt <i>et al.</i> , (2001)
	Kent	Sucrose, 50	40	25	NK	8	Tedjo <i>et al.</i> , (2002)
Mushroom	Agarius bisporus	Salt, 5-15 and Sucrose, 60	Room temp & 50	-	-	15	Yang and Maguer (1992)
	Agarius bisporus	Salt, 10-15	20-45	5	-	-	Torringa <i>et al.</i> , (2001)
Muskmelon	Mirado	Sucrose, 60	25	3	-	28	Maestrelli <i>et al.</i> , (2001)
	Mirado	Sucrose, 60	25	3	-	28	Scalzo <i>et al.</i> , (2001)
Onion		Sucrose, 54 + salt, 19	29	-	-	6-7	Jackson and Mohammed (1971)
Papaya		Sucrose, 60-80	49	4	*	-	Ponting (1973)
		Sucrose, 65	NK	NK	NK	NK	Garcia <i>et al.</i> , (1974)
	Solo	Sucrose, 60	21	3 to 6	NK	10mm slice	Moy <i>et al.</i> , (1978)
		Sucrose, 70	Room temp	2	-	-	Mehta and Tomar (1980)
		Sucrose, 60	Room temp and solar dryer	7	-	6	Moy and Kuo (1985)
		Sucrose, 70	Room temp	-	-	-	Levi <i>et al.</i> , (1985)
		Maltodextrin, 50	25	4	*	10	Argaiz <i>et al.</i> , (1994)
		Sucrose, 50- 70	Room temp	4	-	-	Palou <i>et al.</i> , (1994)
		Sucrose, 50- 70	50-70	NK	-	-	Kubiak <i>et al.</i> , (1995)
	Pant 2	Sucrose, 50- 70	35-55	2-5	0-2000	-	Chaudhary, <i>et al.</i> , (2000)
	CO 2	Sucrose, 50-70	32-70	4:1	-	15	Kaleemullah <i>et al.</i> , (2002a)
Peach		Sucrose, 65-80	49	4	*	-	Ponting (1973)
	Baby gold 6	Corn syrup/ sucrose, 70	25	5	NK	20mm slices	Giangiocomo <i>et al.</i> , (1987)
Peas	Araka	Sucrose, 30-60	30	4	200	9mm dia	Ertekin & Cakaloz (1996a and b)

Pepper	Red sweet	Sucrose 54 + salt, 10	29	-	-	6-7 mm dice	Jackson and Mohammed (1971)	
	Red paprika	Sucrose 22 + salt, 2	25-45	25	-	6 mm disc	Omowaye <i>et al.</i> , (2002)	
Pineapple		Sorbitol, 2-10	20-50	3-4.5		10	Ozen <i>et al.</i> , (2002)	
		Sucrose, 65-80	49	4	*	-	Ponting (1973)	
		Sucrose, 65	47	NK	NK	NK	Garcia <i>et al.</i> , (1974)	
	Cayenna		Sucrose, 50-70	30-50	4		100mm dia ring	Beristain <i>et al.</i> , (1990)
			Sucrose, 40-60	25	5		5, 10 & 20	Shahbuddin, <i>et al.</i> , (1990)
			Sucrose, 40-60	20-60	-	-	6.5	Rahman and Lamb (1991)
	Chquita	Sucrose, 65	30-50	NK	-	-	Shi and Maupoey (1993)	
	Queen	Sucrose, 55-70	30-60	5	-	10	Parjoko <i>et al.</i> , (1996)	
	Queen	Sucrose, 50-75	30-60	4	-	12	Silveira <i>et al.</i> , (1996)	
	Queen		Sucrose, 50-70	30-70	10		7	Saputra (2001)
		Smooth cayenne	Sucrose, 50	25	-	-	-	Nicoletti <i>et al.</i> , (2001)
		Sucrose, 30-70	30-70	50	-	5	Tan <i>et al.</i> , (2001)	
		Sucrose, 50-70	50-70	6	-	6	Waliszewski <i>et al.</i> , (2002)	
Potato	Binjee	Sucrose, 45 /salt, 15	Room temp	5	-	5 & 10 mm slices/ cubes	Islam and Flink, (1982)	
	Hansa	Sucrose, 20-60 or salt, 6.5-15	23	1 to 10	-	10mm cubes	Lenart and Flink, (1984a and b)	
		Salt, 0-12	8-35	5	Shaking	10 mm cube	Biswal and Bozorgmehr (1991)	
	Agria	Corn syrup, 55	25-50	30	*	7.5	Lazarides and Mavourdis (1996)	
	Rosita	Sucrose, 45 + salt 15	30	20	-	20mm dia disc	Azuara <i>et al.</i> , (1996)	
	Chandramukhi	Sucrose, 40-50 and salt, 5-15	30-50	-	-	10mm cubes	Ravindra and Chattopadhyay (2000)	
			Sucrose, 50	25	25	-	10mm cubes	Rastogi <i>et al.</i> , (2000)
	Pomea batata	Sucrose, 30-70	26-50	-	100rpm	10mm cubes	Soto <i>et al.</i> , (2001)	
	Spunta	Sucrose, 30-65	22-80	10-22		5, 10, 15	Rahman <i>et al.</i> , (2001)	
			Salt, 10-30	30-70	50	-	5	Tan <i>et al.</i> , (2001)
Strawberry	Senga	Saccharose, 61.5	30-90	4	-	-	Lenart (1994)	
	Sengana	Starch syrup, 67.5	30-50	-	-	-	Shi and Maupoey (1993)	
	Pajaro	Cerelose, 35-51	25	-	-	-	Alvarez <i>et al.</i> , (1995)	
	Dania	Sucrose, 10-60	22	3	1.2 rpm	-	Viberg <i>et al.</i> , (1998)	
	-	Granular sucrose	Room temp	4	-	-	Raghavan, and Silveira (2001)	
	Elsanta	Sucrose, maltose, trehalose, 40	Room temp	-	-	2	Ferrando and Spiess (2001)	
	Camarosa	Sucrose, 35-65	30	-	-	-	Chiralt <i>et al.</i> , (2001)	
Tomato		Salt, 10	29	-	-	10	Jackson and Mohammed (1971)	
		Sucrose, 65-80	49	4	*	-	Ponting (1973)	
	Heinz	Sucrose, 65	25	10	-	30mm dia	Shi <i>et al.</i> , (1997)	

( - : Not mentioned; NK: Not known and \*: Agitation)

improve water and solute diffusivities within the product, resulting in faster equilibrium in favour of high solute uptake (Ponting, 1973; Karel, 1975; Maguer, 1988). Adverse effects of blanching are reported in case of strawberries (Alvarez *et al.*, 1995).

Blanching of fruits and vegetables is invariably practiced in freezing and drying process for inactivating enzymes, which are responsible for enzymatic and oxidative browning. However, it adversely affects the osmotic dehydration process. It causes death of living cells resulting in the loss of semi-permeability of the cell membrane. However, in order to reduce the browning during storage, two methods of inactivating poly phenol oxidase, the enzyme responsible for browning, are practiced and are convenient. They are blanching after the osmotic step and use of sulphur dioxide during or after the osmotic step. Freezing prior to osmotic treatment is also detrimental (Ponting, 1973). Sodium meta-bisulphite can be added in dipping solution at rates up to 8000 ppm of SO<sub>2</sub> (Jackson and Mohammed, 1971).

Non-thermal pre-treatments are found to aid osmosis, especially, for products which have natural waxy coating or any other such barrier. Shi and Maupoey (1993) pre-treated apple and pineapple cubes with 2% ascorbic acid solution to prevent the browning of the fruit samples during handling and subsequent osmotic dehydration. Grabowski *et al.*, (1994) pre-treated grapes before osmosis by two methods. These pre-treatments were (i) dipping of grapes in 3 per cent KMS solution for 1 min at room temperature and (ii) dipping grapes in 2 per cent ethyl oleate in 0.5 per cent KMS solution for 30 second at 80°C. They found that the pre-treatments dissolved the surface wax of the fruits and reduced the stickiness caused by the sugar on the fruit surface, thereby, reducing the drying time to about ½ of the required without pre-treatment. Nabais *et al.*, (1996) subjected carrot slices to a pre-treatment of 0.2 per cent (w/v) solution of KMnO<sub>4</sub> before osmosis under various conditions. This reduced the browning of the final product.

In some materials uptake of solute is not desirable and should be prevented. Therefore, a chemically treated membrane cast around the material is reported to reduce solute uptake and increase osmotic water removal (Camirand *et al.*, 1968). Calcium pectate and pectinate were used for this purpose. Many other membranes such as calcium alginate, methyl or ethyl cellulose, starch derivatives, modified gums or proteins can also be used. They have sufficient selectivity and are edible (Serenó *et al.*, 2001b and Ferrando and Spiess, 2001).

Beaudry *et al.* (2003) examined different skin pre-treatment on cranberries-cutting in halves, surface abrasion of skin and puncturing of skin by needle, and realised that cutting in half is the best possible method for proper osmosis.

Sunjka and Raghavan (2004) tested several techniques of pre-treatment with various levels one against another. The study revealed that chemical pre-treatment parameters (time and temperature of reaction) had no significant influence on mass transfer. Mechanical pre-treatment showed substantial increase in moisture loss and sugar uptake, because the "active" surface area for mass transfer is greater. All three factors of osmotic dehydration (process duration, agent type, and concentration of sugar solution) showed significant influence.

Escobar *et al.*, (2007) used blanching pre-treatment for carrot slices at 100°C for 10, 20 and 30 s before the osmotic dehydration and found that the effective diffusion coefficients of water as well as sucrose increased after blanching treatments which cause the death of cells in the tissue.

The above information suggests that non-thermal pre-treatments are preferable prior to osmosis since they do not affect cellular structure of the material being dehydrated.

### **2.4.3 Temperature of the osmotic treatment**

It is well recognized that diffusion is a temperature dependent phenomena. Higher process temperature seems to promote faster water loss on account of rapid water diffusion within the product and better mass (water) transfer characteristics on the product surface due to lower viscosity of the osmotic medium. At the same time solid diffusion within the product is also promoted by higher temperature, mainly dictated by the molecular size and concentration of the osmotic solution. Many researchers have reported that an increase in temperature up to certain extent increases rate of osmosis. Further increase in temperature affects the semi-permeability of the cell walls and reduces rate of osmosis. Consequently, poor results are obtained in further osmotic water removal process (Lenart and Flink, 1984a and b; Maguer, 1988). Ponting *et al.*, 1966 has, however, reported that enzymatic browning and flavour deterioration take place above 49 °C.

There is an upper temperature limit in case of fruits and vegetables, beyond which there is a negative impact on final product quality due to browning, flavour loss, softening of tissue, *etc.* This limit is specified by the sensitivity of each particular product and is conventionally placed around 50 °C (Ponting *et al.*, 1966; Bongirwar and Sreenivasan, 1977; Lenart and Flink, 1984a and b; and Maguer, 1988).

The effect of temperature in osmotic dehydration has been reported by various researchers. Ponting (1973) conducted experiments on apple chips at 30, 40 and 50°C and concluded that the rate of osmosis increased with temperature up to 50°C. Bongirwar and

Sreenivasan (1977) reported that at 50 °C, 50 per cent loss in weight of banana occurred without any damage to the product quality during osmosis.

Lenart and Flink (1984a) reported that although, the rate increased with the temperature, there was a limit, perhaps 60 °C above which the cell membrane was destroyed and caused internal browning of the fruit pieces on one hand and loss of fruity flavour on the other hand. Consequently, poor results were obtained in further osmotic water removal, while Rahman and Lamb (1990) observed that solid concentration became nearly constant above 60°C, which indicated negligible increase in the rate of sucrose diffusion above 60 °C. They found that it was not normal to find diffusion rates falling with increase in the temperature. The reason might be that the cell wall, which was composed of cellulose and pectin, increased its permeability at higher temperature.

Thus, the temperature has a substantial effect on the course of osmotic dehydration. The rate of mass exchange and chemical reaction and diffusion coefficient of water increases with temperature, but above 49 °C, enzymatic browning and flavour deterioration begins. A high temperature, *i.e.* 60°C, modifies the tissue characteristics thereby favouring impregnation phenomena and thus the solid gain (Ponting *et al.*, 1966; Farkas and Lazar, 1969; Bongirwar and Sreenivasan, 1977; Lenart and Flink, 1984b; Lenart and Lewicki, 1990; Lewicki and Lenart, 1995; Fito *et al.*, 2001).

Panagiotou, *et al.* (1998) studied the mass transfer modelling of the osmotic dehydration of apple, banana and kiwi fruit. Osmotic dehydration was carried at 20, 40 and 60°C using the sugar concentration of 30, 40 and 50 per cent w/w for 0.5, 2, 6 and 16 h. The study showed that of all the osmotic process variables, the process temperature had the most significant effect on the kinetic rate of water loss. The kinetic rate of solid gain did not depend significantly on process temperature.

Pan *et al.* (2003) studied the osmotic dehydration pretreatment in drying of carrot and pumpkin slices at the temperature of 40 and 50 °C. As no significant differences were observed either on the water loss or solute gain for both the materials, the authors concluded that it is better to conduct osmosis at room temperature in view of energy saving. Ade-Omowaye *et al.* (2002) studied the influence of temperature on the osmotic dehydration of bell pepper using the temperature range of 25-55 °C. Increasing the temperature enhanced solids gain and water loss (32-48%). Reduction in vitamin C concentration from 20 to 4 per cent and carotenoids from 80 to 55 per cent was observed with increase in temperature. Processing temperature above 45 °C was undesirable because of unfavourable modifications concerning the residual ascorbic acid content.

Rastogi and Raghavarao, (2004) analysed the mass transfer during osmotic dehydration of pineapple in a temperature range of 30-50 °C and concluded that the value of effective diffusion coefficient of water as well as solute was found to be dependent on the concentration and temperature of osmotic solution. Chenlo *et al.*, (2007) carried out osmotic dehydration of chestnut with sucrose at the temperature of 25, 35 and 45 °C. An increase in the temperature value from 25 up to 45 °C made the dehydration more intense, reaching lower value of moisture content. Water loss and solid gain were affected by the temperature of the osmotic solution.

Denise *et al.* (2005) investigated the process of osmotic dehydration of the acerola fruits using binary (sucrose + water) and ternary (sucrose + salt + water) solutions carried out in an incubator at 25 and 65 °C. Water loss, solid gain and weight reduction were influenced by the temperature for both binary and ternary solutions.

Ramallo and Mascheroni (2005) analysed the effect of the temperature on water loss, solute gain and glucose & fructose leakage during the osmotic dehydration of half-ring pineapples. The process was run at three temperature levels i.e. 30, 40 and 50 °C. As temperature increased from 30 to 50 °C, apparent moisture and sucrose diffusivities ( $D_w$  &  $D_s$ ) increased with temperature. The loss of glucose and fructose also increased with increase in temperature. Pokharkar and Prasad (1998b) studied the effects of temperature on osmotic dehydration of banana slices by conducting the experiments at 30, 40 and 50 °C. The water loss and sugar gain increased with increase in temperature.

Fernandes *et al.* (2006) optimized the osmotic dehydration of the bananas followed by air-drying. The results for air-drying showed that the temperature used in the osmotic dehydration affects the diffusivity of water during the air-drying process elevating the mass transfer rate during air-drying as the temperature in osmotic dehydration increases from 50-70°C. Singh *et al.*, (2006) studied the mass transfer kinetics and effective diffusivity during osmotic dehydration of carrot cubes at temperatures of 35, 45 and 55 °C. There was an increase in rate of water loss and relatively stable rate of solute gain with increase in solution temperature. The effect of increasing temperature of highly concentrated solution might be due to decrease in viscosity of osmotic solution resulting in high diffusion rates of both water and solute. The values of effective diffusivity were high at 55 °C as compared to 35 °C and 45°C. Barat, *et al.*, (2001) analysed the mass transfer and structural changes in apple tissues at the temperature of 30, 40 and 50 °C. The sample impregnation-deformation ratio was affected by the sample temperature. Sample temperature during the experiment affects the characteristics relaxation time of solid matrix; higher the temperature, shorter the relaxation time, and so, the faster is the sample deformation.

Chenlo *et al.* (2007) observed that the osmotic dehydration of chestnut with sucrose at 25, 35 and 45 °C made the dehydration more intense, reaching lower value of moisture content. Water loss and solid gain were affected by the temperature of the osmotic solution.

#### 2.4.4 Duration of osmotic treatment

In a study conducted by Lenart and Flink (1984a), it was shown that equilibrium was characterized by an equality of water activity and soluble solids concentration in the product and the solution. While equilibrium was approached within 20 h, it was found that mass transport data except for solid gain did not change significantly beyond four hours.

In general, as the time of treatment increases the weight loss increases, but the rate at which this occur decreased. A schematic representation of soluble solids content of a sample as a function of treatment time is given in Fig. 2.4 (Maguer, 1988). The treatment time can be selected in such a way that amount of water removal should be maximum with no appreciable uptake of solids. Some examples are given in Table 2.2.

In every case, the practical end point was far ahead of equilibrium and was actually specified by the observed relative changes of the mass transport data in combination with desired final product characteristics. In most non-equilibrium studies, osmotic process was carried out for a period of 3 to 5 h. Various authors have reported that initial period of osmotic process (first 30 min) was the most significant one since the transport phenomena was faster and had a dramatic impact on further evolution of the osmotic process (Hawkes and Flink, 1978; Conway *et al.*, 1983; Biswal and Bozorgmehr, 1991). Thus, an efficient way to limit solute up take and obtain large water loss/ solid gain ratios was to interrupt osmosis early. A fast rinsing, *i.e.* for 20s helped in removing a good portion of solute absorbed by the product during osmosis (Karel, 1975).

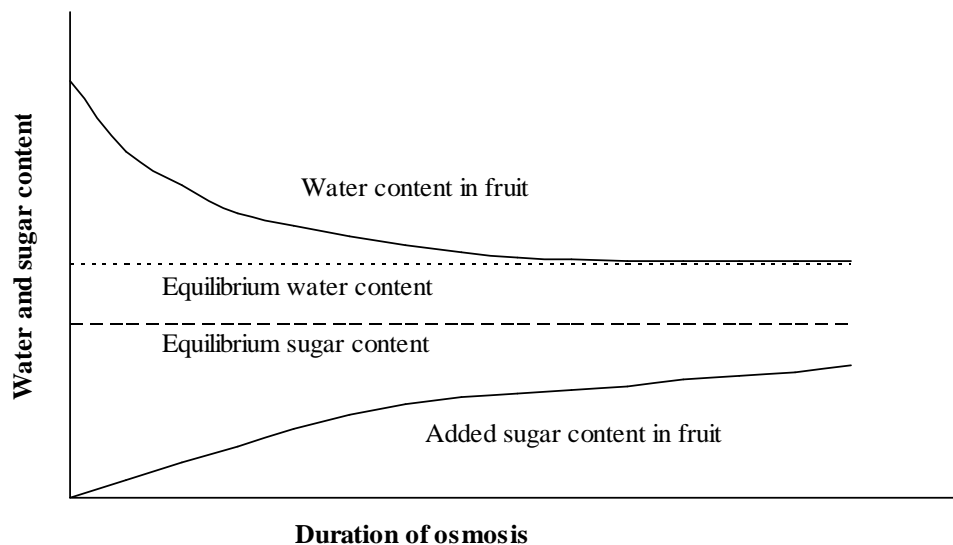


Fig. 2.4 Water and soluble solid content as a function of duration of osmosis

Bogirwar and Sreenivasan (1977) reported that, at 50 °C, bananas lost 50 per cent of their weight within 3 hours without any damage to the quality. While Yang and Maguer (1992) reported 25 per cent moisture loss in button mushroom over a period of 30 minutes in 15 per cent salt solution.

Lazarides *et al.* (1995) reported 25 per cent loss of initial water within the first hour and 40 per cent at the end of 3<sup>rd</sup> hour of osmosis of apple slices subjected to concentration of 45 to 60 per cent. Pokharkar and Prasad (1998b) conducted osmosis of banana slices in concentrated sugar syrup of 40, 50 and 60 °Brix and suggested that a period of at least 5 h was required for the process to approach equilibrium.

The available literature suggests that unless the process of osmosis is expected to reach equilibrium or solute impregnation as one of the objectives of the process as in case of intermediate moisture foods, time period of osmosis should be restricted to 4 h.

The process of osmo-drying (OD) would be very rapid in first few hours because of greater osmotic potentials. Subsequently, a gradual decline in water loss could be noted which is due to decrease in osmotic gradient and formation of a barrier on the surface of food osmosed. Further solid uptake would also increase with longer duration of exposure. Most of the studies on osmotic dehydration were conducted in a batch system with highly concentrated sucrose solutions. According to Ponting *et al.*, (1966); Lenart and Flink (1984b) and Biswal and Bozorgmehr (1991) keeping the concentration of the solution constant, the increase of the contact time resulted in an increase of the weight loss or simply a more effective drying. Although the weight loss was increased as a function of time of osmosis, the rate at which it occurred was decreasing. The water content and sugar concentration changed with time in the fruit pieces dipped or immersed in concentrated sugar solutions. The rate of water removal, which is characterized by the tangent at each point of the curve, was the maximum at the beginning of the process and the difference between the rate of sucrose uptake and the rate of water loss was the maximum at the beginning of the treatment too (Fig. 2.4).

Panagiotou *et al.* (1998) developed an empirical model to predict the water loss and solid gain during osmotic dehydration of apple, banana and kiwi fruit for 0.5, 2, 6 and 16h. Small fruit samples attained equilibrium after approximately 6 hours.

Ramallo and Mascheroni (2005) studied the water loss and sugar uptake during osmotic dehydration of pineapple for 30, 60, 75, 90 and 150 min. The process was then carried out for time more than 3000 min. An increase in sugar content (fructose+glucose) in fruit was observed until 600 min of the process at 30 °C temperature. Variation in natural

sugar content was not detected even for osmosis for more than 3000 min at 40 °C. A slight decrease in natural sugar content was observed at 50 °C.

**Table 2.2 Effect of duration of treatment on weight loss and solid gain**

<b>Material</b>	<b>Remark</b>	<b>Reference</b>
Apple	50% reduction in weight in 2.5 to 3 h	Ponting <i>et al.</i> , (1966)
	50% reduction in 8 h. Rate of weight loss decreased from 10% to 5% per hour after 20 to 30% moisture was removed	Farkas and Lazar (1969)
	Sugar up take was rapid, reaching the maximum level after 0.5 h of treatment, after which it remained constant.	Karel (1976)
	Rapid up take of solute within the first half an hour of osmosis.	Hawkes and Flink, (1978)
	Solute uptake increases as the osmosis solution concentration increased. Rapid water loss for approximately 1 h and slowed thereafter.	Conway <i>et al.</i> , (1983)
	Linear effect of time of treatment on water loss and solute gain. High transfer during first phase (about 30 min), with subsequent decrease at longer processing times. Similar effect was observed for frozen apple tissues	Saurel <i>et al.</i> , (1994a and b)
Apricot	Water loss rates dropped to 20% of the initial rate during the first hour and kept decreasing at much slower rate to nearly levelled off at 10% of the initial rate within 3h. The rate of solid gain dropped to 25% of the initial rate within first hour and levelled of at 15% of initial rate within 3h of dehydration.	Lazarides <i>et al.</i> , 1995)
	Water loss was rapid in first submerged period of 60-90 minutes. Most sugar gain occurred within the first hours and then tended to equilibrate during vacuum osmotic dehydration of fruits.	Shi <i>et al.</i> , (1995)
Banana	50°C was found to be most suitable temperature at which 50% weight loss would occur within 3 h without any damage to quality	Bongirwar and Sreenivasan (1977)
Carrot	Water loss was intensive during first hour	Lenart and Lewicki (1988)
Cauliflower	Maximum water removal of about 40% with a minimum salt pick up of 4.2% was achieved using the optimized conditions, <i>viz.</i> temperature 80°C, brine to material ratio (w/w) of 2, 12% salt concentration and 5 min time	Vijayanand <i>et al.</i> , (1995)
Cherries	Maximum water loss took place within first two hours	Torregiani <i>et al.</i> ,

Papaya	Water loss and solid gain increased with time of treatment up to 4h, as the dextrose equivalent (DE) value of the solution increased. For contact times greater than 24h, water loss decreased and solid gain increased as the DE value increased.	(1987) Argaiz <i>et al.</i> , (1994)
Peas	A significant increase in water loss and sugar gain with first 2h which continued and reached equilibrium between 8-24h.	Ertekin and Cakaloz (1996a and b)
Pineapple	Water loss and sugar gain appeared to increase exponentially with time. Amount of sugar absorbed with increasing initial concentration of sucrose in the syrup	Beristain <i>et al.</i> , 1990
Potato	Water loss remained same from 4 to 20 h but the solid gain is increased Water removal ranged from 50-70% of initial moisture content during first 3h. Based on initial soluble solids, the net solid gain was about 85% at 3h and reached 155% with in 5h causing negative effect on organoleptic quality and nutritional profile.	Lenart and Flink, (1984a) Lazarides and Mavourdis (1996)

Rizzolo *et al.* (2006) studied the influence of time of processing on the volatile compounds of strawberry. Strawberry slices were subjected to osmotic dehydration for 1, 2, 4 and 6 h using sucrose (60% w/w) or sorbitol (60% w/w). The greater changes occurred after 2 h in sucrose with a promotion of fermentative volatiles and a decrease in the others. For the sorbitol solution, major changes in the volatile pattern occurred after 4 h of osmosis and the fermentative process was less important than in sucrose. Nieto *et al.*, (2001) carried out osmotic dehydration of apple slices and concluded that the drying curves exhibited high dehydration rates within first two hours of osmosis and a gradual reduction thereafter. Water loss ranged between 28 and 30 per cent after 4 h besides soluble solid content increased gradually. During first 100-150 minutes, volume changes followed a parallel pattern to water loss. After that time, volume decrease was slightly lower than moisture loss, reaching a final reduction of approximately 27 per cent.

Singh *et al.* (2006) studied the mass transfer kinetics and effectivity during the osmotic dehydration cubes. The time of immersion of the carrot cubes was 0-240 min. With increase in immersion time, increased water loss and solid gain were observed in carrot.

#### 2.4.5 Osmotic agents used for osmosis

A number of osmotic agents can be used in osmotic dehydration. Osmo-active substances used in osmotic dehydration either singly or in combination must comply with

special requirements like edible with accepted taste and flavour, non toxic, inert to food components, if possible, be readily dissolvable to form a high concentrated solution and should be cheap. Sucrose, lactose, glucose, fructose, maltodextrins, starch or corn syrup solids and sodium chloride are commonly used in osmotic dehydration of fruits and vegetables. Honey, glycerol, hydrocolloids were also tested (Ponting *et al.*, 1966; Lerici *et al.*, 1983; Torreggiani, 1993).

Therefore, choice of the solute depends upon several factors. The organoleptic evaluation of the final product was considered as one of the most important character as well as cost of the solute. The solubility of the substance in water was crucial for its effect on the maximum possible concentration in the osmotic solution.

Numerous possibilities of solutes in the osmotic solution allow for its optimization in terms of maximum water removal, minimum solute gain, and optimum organoleptic quality of the product and minimum solute costs. Many salts and saccharides (mono, di and poly) singularly or in conjunction with other solutes have been used as osmotic agents. Among salts, sodium chloride is found to be an excellent osmotic agent (Speck *et al.*, 1977; Lerici *et al.*, 1985), but its application is limited because of the salty taste it imparts to the food (Yang and Maguer, 1992; Vijayanand *et al.*, 1995).

Bolin *et al.* (1983) conducted osmosis using 70°Brix solution of sucrose and high fructose corn syrup on apples, peaches and apricots. They reported a higher solid gain in case of high fructose corn syrup with only marginal increase in water loss in comparison to sucrose. They, therefore, recommended sucrose as an osmotic medium over high fructose corn syrup.

Lazarides *et al.* (1995) conducted experiments using sucrose and corn syrup solids for dehydrating apple slices. They reported that overall mass transfer coefficient for sugar gain was inversely proportional to the molecular size of the osmotic solutes. Therefore, corn syrup solids having high dextrose equivalent was found to be an optimum solute for fruit impregnation processes. Corn syrup solids of medium to low dextrose equivalent were found to be suitable whenever high dewatering rates with minimal sugar uptake was the goal.

Sugar and salt are the most widely used osmotic agents for fruits and vegetables because of their effectiveness, convenience and flavour qualities. Sugar is said to form a thin coating on the surface of food because of poor penetrating ability (2-3 mm), thereby acting as a resistance in diffusion process as compared to salt, which has a good penetrating ability (12-15 mm) thereby avoiding formation of coats on the surface. Combinations of sugar and salt solution with higher levels of salts are reported to result in higher levels of water removal because of increase in osmotic pressure. Further, it was also observed that WL/SG ratio is

affected by molecular weight (Lerici *et al.*, 1985). The effect of various osmotic agents on osmotic dehydration is being discussed below.

El-Aouas *et al.* (2006) studied the influence of two different osmotic agents (sucrose and corn syrup) on the osmotic dehydration of papaya (*Carica papaya* L.) slices. The study was carried out using two factorial experimental designs, with three independent variables whose levels varied from 44 to 56 per cent w/w for concentration, from 34 to 36 °C for temperature and from 120 to 210 min for immersion time. The responses of the experiment designs were the weight reduction (WR), water loss (WL), solids gain (SG) and water activity ( $a_w$ ). The results showed, considering the same osmotic pressure for both osmotic agents, the values obtained for WR, WL and SG for dehydration in sucrose solutions were higher than those obtained in corn syrup solution, due to their high viscosity and polysaccharide content.

#### **2.4.5.1 Sucrose**

Sucrose, among disaccharide, has been widely used as an osmotic agent due to the several advantages it offers. Sucrose prevents food dis-colourization to a large extent by its inhibitive capacity towards poly-phenol oxidase enzyme (Ponting, 1973). Further, it imparts good taste to the food material. Sucrose, being a disaccharide, has lower diffusivity as compared to other osmotic agents (Bolin *et al.*, 1983; Argaiz *et al.*, 1994). Therefore, less amount of sucrose would be absorbed by the food material, making it an ideal choice for an osmotic agent.

Good results were obtained by using the osmo-dehydration in sucrose solution prior to any drying or freezing operation (Ponting *et al.*, 1966; Farkas and Lazar, 1969). Flink and Karel (1970) reported that to a certain extent the immersion of the fruit slices in a sucrose solution acted as a barrier to the atmospheric oxygen. Consequentially enzymatic browning was reduced.

Dixon *et al.* (1976) observed that the substantial increase in the sugar/acid obtained in osmotic dehydration of apples resulted in wide appeal for the product, although the product rehydrated only up to 70% of its original weight.

#### **2.4.5.2 High fructose corn syrup (HFCS)**

Chandrasekaran and Kind (1972) in their study on diffusion characteristics reported that HFCS (high fructose corn syrup) has a diffusion coefficient 32 per cent higher than sucrose. Weight loss was similar for both the syrup, but moisture loss was slightly more rapid for HFCS. However, the water activity of 70 °Brix HFCS was different from the 70 °Brix sucrose with a former being 0.734 while the latter being 0.832. According to Bolin *et al.*,

1983 HFCS produces a sweeter taste than sucrose. Taste panel evaluation indicated that the sucrose solution was preferred as an osmotic medium over HFCS.

Argaiz *et al.* (1994) used maltodextrin and corn syrup solids as the osmotic solution for papaya cylinders at 25 °C. They concluded that the desired water loss and solid gain was obtained using corn syrup solids.

#### **2.4.5.3 Lactose and maltodextrin**

Hawkes and Flink (1978) questioned the economic feasibility of using sucrose and suggested lactose and maltodextrin as substitutes of sucrose for food material requiring less sweetening. Lactose may become available in increasing quantities as cheese whey could be recovered and fractionated to recover proteins, leaving a lactose rich fraction. The solubility limit of the lactose in water (around 25%) could be allowed for the lactose to be only a partial substitute for sucrose, even in dry system.

#### **2.4.5.4 Sodium chloride**

According to Hawkes and Flink (1978) 25 per cent sodium chloride was the best osmotic solution. It could penetrate the tissue very fast, thus it increased the total solids prior to freeze drying in a very short time. However, the ability to use the sodium chloride was limited by its saltiness especially in case of fruits. Flink (1980) reported that the limiting concentration of salt in the osmotic solution, in order to have a satisfactory product from an organoleptic point of view, would probably be around 10 per cent.

Fennema (1975) pointed out that polyphenoloxidase were apparently restricted by chloride ions but the mechanism was not clear. Immersion of the cut fruits in a dilute solution of sodium chloride (10 to 30 %) was a method often used during the interval between cutting and freezing.

Yang and Maguer (1992) conducted osmosis of button mushroom at 5, 10 and 15 % sodium chloride solutions. They concluded that processing of mushroom in a 15 % salt solution resulted in the greatest ratio of rate of moisture change to rate of salt change. It was also the optimum condition for moisture removal and salt loading.

#### **2.4.5.5 Calcium chloride**

Ponting and Jackson (1972) reported calcium treatment of apple in the logical and historical method for increasing firmness. The effect of a calcium dip was effective in preserving texture over an extended period, as well as having a synergistic effect with ascorbic acid or sulphur dioxide in preventing browning. However, they observed that it should be used in small quantity below 0.5 per cent otherwise, it would cause bitterness.

#### **2.4.5.6 Mixtures**

Hawkes and Flink (1978) investigated the possibility of using binary mixtures of solutes with sucrose as a means to reduce solute cost and improve osmotic effectiveness. Binary mixtures of salt and sucrose were found to be more effective than sucrose alone (Islam and Flink, 1982). Lenart and Flink (1984b) reported that the combined properties of both the solutes lowered the water activity for salt and high water removal effect of the sucrose. The penetration of salt in the tissue, which can be organoleptically unfavourable, is limited by the sucrose. The salt hinders the shrinkage at the surface layer occurring mostly with the sucrose solution immersion.

Moy *et al.* (1978) investigated the effect of combining organic acids (acetic, lactic and citric acid) and sucrose on the osmotic concentration of papaya and mango, and found that acidification increased the rate of water removal. The increment was due to changed tissue properties and not from the increase in solute concentration, since acids were added at concentrations (1.0 to 4.0 %), which was low in comparison to the sucrose concentration (60%). The effects of various osmotic agents are reported in Table 2.3.

#### **2.4.6 Concentration of osmotic solution**

The effect of concentration was also studied by number of researchers (Ponting *et al.*, 1966; Beristain *et al.*, 1990; Biswal and Bozorgmehr, 1991 and Sharma *et al.*, 1991; Lewicki and Lenart, 1995; Rastogi and Raghavarao, 1996; Sankat *et al.*, 1996; Kowalska and Lenart, 2001; Fito *et al.*, 2001) and reported that the rate of osmosis increased with the increase of osmotic syrup concentration and this effect is increased with increase in temperature for the same contact time. However, there exists an upper limit beyond which any significant increase in concentration would not affect the water loss/solid gain. Researchers have reported this limit in the range of 65-70 per cent.

Ripe banana slices 19 mm thick were osmotically dehydrated in sugar solution of 35, 50 and 65° Brix concentration for as long as 52 h, at 30 °C by Sankat *et al.* (1992). The rate and extent of changes in weight loss, moisture content and total soluble solids of the slices were reported to be influenced strongly by the strength of the osmotic solutions.

Pokharkar and Prasad (1998b) conducted osmosis of coconut pieces in concentrated sugar syrup of 40, 50 and 60 °Brix for 5 h at 30 °C temperature. They observed that the changes in moisture content of coconut pieces were strongly influenced by the concentration of osmotic solution and concluded that sugar syrup of 50 °Brix was effective for the osmotic dehydration of coconut pieces.

Rahman and Lamb (1990) pointed out that solid gain increased linearly with the increase of sugar concentration and temperature. The rate of sucrose diffusion was also a function of solution concentration and temperature.

Ertekin and Cakaloz (1996b) studied the influence of osmosis on drying behaviour and product quality of peas. Peas were osmotically dehydrated using 60 per cent sucrose solution and 40 per cent sucrose + 20 per cent trisodium citrate solution. Samples osmosed with sucrose + citrate solution had higher moisture diffusivity value compared to that of non-osmosed samples, which could be due to modification of the water permeability of the pea skin prior to drying.

Agnelli *et al.* (2005) studied osmotic dehydration of pears and apples using 69 °Brix sucrose and 47 °Brix glucose solutions. The penetration of glucose was higher than that of sucrose, even though higher concentration of sucrose solution was used. It could be due to lower molecular weight of glucose.

Denise *et al.* (2005) investigated osmotic dehydration of frozen mature acerola fruits using 30 to 60 per cent (w/w) binary (water + sucrose) and 20 to 50 per cent (w/w) ternary (water + sucrose + salt) solutions. The concentration of salt in ternary solution was kept 10 per cent (w/w). The best conditions for osmotic dehydration were observed to be a binary solution with 60 per cent (w/w) of sucrose and a ternary solution with 50 per cent (w/w) of sucrose plus 10 per cent (w/w) salt, both at a temperature of 60 °C.

Rashmi *et al.* (2005) optimized sugar syrup concentration for osmo-air dehydration of 'Giant Kew' variety of pineapple by subjecting pineapple slices to osmosis for 24 h in 50, 60 and 70 °B sugar syrups followed by draining and drying at 60-65 °C. They conclude that highest sensory scores reported for final product which dried by osmo-air dehydration technique.

Oztop *et al.* (2006) optimized the microwave frying of osmotically dehydrated potato slices. The osmotic dehydration treatment was conducted using 20% (w/w) sodium chloride solutions. Sutar and Gupta (2007) studied the mass transfer in osmotic dehydration of onion slices using three salt (NaCl) concentration levels (5%, 12.5% and 20%). It was observed that equilibrium moisture loss and solid gain are related to solution concentration and solution temperature. Jokic *et al.*, (2007) investigated water and solids transfer during osmotic dehydration of sugar beet cassettes in combined aqueous solutions of sucrose (30-70 % w/w) and sodium chloride (0-8% w/w) to find optimum conditions for production of dietary fibres' from whole sugar beet, Chenlo *et al.*, (2007) carried out osmotic dehydration of chestnut (*Castanea sativa* Mill) using aqueous solutions of sucrose of different concentrations (40, 50, 60% w/w).

Singh *et al.* (2006) studied mass transfer kinetics and effective diffusivity during osmotic dehydration of carrot cubes in ternary solution of sucrose, NaCl and water. The osmotic solution concentrations used were 50 °Brix + 5% salt (w/v), 50 °Brix + 10% salt (w/v) and 50 °Brix + 15% salt (w/v). Water loss and solute gain increased with an increase in concentration of salt from 5% to 15% in 50 °Brix sucrose solution, due to the synergistic effect of both sucrose and salt to develop high osmotic potential.

Marani *et al.* (2007) studied the osmotic dehydration of pears, kiwis, strawberries and apples using osmotic solutions (sucrose, glucose, and corn syrup mixtures). They concluded that the water loss was fast with osmotic solutions containing lower molecular weight osmotic agents such as fructose, glucose and sucrose solutions. On the contrary, it was slower with the osmotic solutions containing corn syrup (high molecular weight sugars). However, the equilibrium values of water loss were observed to be similar. Solid gain was significantly higher with glucose solution than with fructose, sucrose and high molecular weight sugars. Thus, sucrose and high molecular weight sugar solution would be recommended osmotic agents as they allow high dehydration with minimum penetration of solids.

**Table 2.3 Effect of osmotic agent on product quality**

<b>Osmotic substance</b>	<b>Remark</b>	<b>Reference</b>
Calcium chloride	Increases the firmness of apple pieces and preserves the texture during storage. Prevents browning because of synergistic effect with ascorbic acid or sulphur di oxide. Imparts bitter taste to the product if used above 0.5% level.	Ponting <i>et al.</i> , (1966) Cipolletti <i>et al.</i> , (1977)
Ethanol	Decreases viscosity and freezing point of the osmotic solution in de-hydro cooling and freezing processes.	Maguer and Biswal (1984)
Fructose	Increases the dry matter content by 50% as compared with sucrose due to higher penetration rate. $A_w$ of the final product is also lower. However, sucrose is preferred over fructose.	Bolin <i>et al.</i> , (1983)
Invert sugar	Theoretically more effective than same concentration sucrose because when completely inverted, it has twice as many molecules per unit volume. Practically little difference in the rate of osmotic dehydration of fruit by sucrose or invert sugar syrups of the same concentration and temperature.	Ponting <i>et al.</i> , (1966)
Lactose	Has much lower level of sweetness than sucrose. Low solubility in aqueous solution.	Hawkes and Flink (1978)
Malto dextrin	Less effective than sucrose at the same concentration.	Hawkes and Flink (1978)
Sodium chloride	Most suitable for vegetables as it retards oxidative and non enzymatic browning. Increases driving force for the drying owing to the lowering capacity of the salt. Sometimes bleaching effect on coloured products can be prevented using mixture of salt and sugar. Hinders shrinkage at the surface layers.	Speck <i>et al.</i> , (1977), Hawkes and Flink (1978), Flink (1980), Lenart and Flink, (1984a and b) Sacchetti <i>et al.</i> , (2001)
Sucrose/ sugar	Dry sugar is unsuitable because of oxidative browning during osmosis. Sugar solution is best as it reduces browning by preventing the entry of oxygen. Sweetness hinders its use in vegetable processing.	Ponting <i>et al.</i> , (1966), Farkas and Lazar (1969), Ponting (1973), Flink (1976)
Ethylene glycol, Polyethylene glycols	At low temperature ( $T < 50^{\circ}\text{C}$ ) and processing times ( $t < 30$ min), osmosis increases as solute molecular weight increases. High molecular weight solutes enhanced osmosis due to marked difference in solute concentrations between the surface and interior of the	Saurel <i>et al.</i> , (1994a,b)

	product. Also solute gain, apparent diffusivity and loss of fruit solutes were reduced, in both fresh and frozen apple.	
Starch/Corn syrup	Favours similar final water content with minimal solid gain as that obtained with sucrose. Dextrose equivalent of the syrup affects strongly the ratio between WL and SG. Corn syrup solids of medium to low DE (<38) give low or negative SG and much larger WL	Argaiz <i>et al.</i> , (1994), Lazarides <i>et al.</i> , (1995), Lazarides and Marvoudis (1996)
Mixtures (invert sugar + sucrose, salt + ethanol, sucrose + salt	More effective than sucrose alone due to combination of properties of both solutes. NaCl, CaCl <sub>2</sub> , lactic and hydrochloric acid in concentration of 1-5% were added to sugar/starch solution to improve osmotic dehydration process by promoting removal of water from the material, Calcium chloride improved the texture of osmosed apples.	Lenart and Flink (1984b) Ferrando and Spiess (2001)

#### 2.4.7 Properties of plant material

There is wide variation in the physical nature of fruits, which is reflected, in osmotically dried products (Ponting, 1973). Physical structure (porosity, arrangement of the cell, fibre orientation and skin) may also affect the osmotic kinetics in the food.

Flink (1976) found that under same conditions of osmosis, the increase in solid concentration in the plant material depends upon the kind of fruits. Lenart and Flink (1984a) have reported the textural difference of tissue after immersion in osmotic solutions. Sugar is known to give firmness to the tissue and salt as softening agent. They observed that the textural difference would be reflected in the tissue mass transport data. They also pointed out that eventual effect of cellular dehydration on transport properties would depend on tissue properties, especially the intercellular space present in the tissue and amount of insoluble solids.

The chemical compositions (proteins, fat, carbohydrates, salts, *etc.*) may also affect the osmotic kinetics in the food. Protein affects the salt penetration in fish (Rahman, 1992).

Islam and Flink (1982) observed reduced water loss and increased solid gain when 4 min steam blanching is done before osmosis. The loss of membrane integrity due to heating was reported to be the cause of poor osmotic concentration behaviour. Any structural changes occurring during osmotic dehydration may also influence the transport properties of the tissue.

#### **2.4.8 Size and shape of food material**

The size and shape has a definite effect on dehydration characteristics of the material. Islam and Flink (1982) reported that the net solid uptake of the sample is more for sample size of 5 mm slice than 10 mm and 10 x 10 mm. Similarly, Lenart and Lewicki (1988) have shown that the thickness of the sample should not exceed 10 mm.

Size and shape also play a significant role in mass exchange since they result in different specific surface area or surface to thickness ratios. Since osmosis is based on diffusion process, which is area specific, increase in contact area between the solution and the product increases osmosis. Lerici *et al.* (1985) conducted osmosis on apples (variety- Golden Delicious) in four different shapes (slices, cube, ring and stick) in glucose solution of 52 °Brix. They concluded that up to a certain A/L ratio (total surface/ half thickness ratio of 160 *i.e.* rings) higher water loss (WL) and solid gain (SG) values were obtained in comparison to slices (A/L=70 and sticks (A/L=90). Beyond this A/L =240 limit favoured solid gain at the expenses of lower water loss resulting in lower weight reduction. The results suggest that although increased surface area increases osmosis there is a limit of A/L ratio for every product beyond which there is no further increase in osmosis process.

Conteras and Smyrl (1981) found that mass reduction increased to about 1.3 times when thickness of apple slices decreased from 10 to 5 mm. Under same osmotic conditions, different size of the fresh fruit sample can give final products with very different characteristics.

Dwivedi and Ilangantileke (1992) reported that the shape of papaya fruit affect the mass transport coefficient (K). The slices gave highest value of K while the rectangular shape gave the lowest K value. The higher surface area of the sample and the lower thickness of the slice could explain it.

Panagiotou *et al.* (1998) studied the effect of different sizes of apple, banana and kiwifruits in the osmotic dehydration process. Banana cylinder of 22 mm average diameter and thickness of 4, 8, 12, 16 and 20 mm were used. It was observed that the size of the fruits samples had negative effect on the water loss and solid gain. Similarly, Agnelli *et al.*, (2005) concluded that the water loss and solid gain during osmotic dehydration were more in apple cube of 1.5 cm as compared to 2 cm in 69 °Brix sucrose and 47 °Brix glucose solutions.

#### **2.4.9 Agitation/ Circulation**

When foods are osmosed for longer periods, possibility of localized dilution causes non-uniform distribution of sugar concentrations in osmotic solution resulting in differences in osmotic gradients. Agitation therefore ensures the uniformity in osmotic solution, there by

resulting in higher degree of water loss. Agitation also ensures a continuous contact of the particle surface with concentrated osmotic solution, securing a large gradient at the product/solution interface. Further, it reveals that solid gain decreases with agitation, because of indirect effect of higher water removal rates (Raoult *et al.*, 1994b).

The agitation may, however, be difficult and may cause damage to the sample. The circulation of syrup with centrifugal pump is simple and quite effective. It was found to be advantageous in case of apple in which little sugar uptake took place when the syrup was circulated and pieces were kept stationary. Sugar up take was rapid, reaching the maximum level after half an hour after which it remained constant (Karel, 1976).

Ponting *et al.* (1966), Bongirwar and Sreenivasan (1977) and Raoult *et al.* (1989) studied the effect of agitated treatments. The analysis of agitation effect was limited only to weight loss and was without quantification. However, it is clear that agitated samples exhibit greater weight loss than non agitated ones.

Hawkes and Flink (1978) conducted osmosis of apple slices. They reported that agitation had little effect on the mass transfer coefficient at 25 and 40 per cent sucrose concentration. However, with increase in concentration above 40 per cent, *i.e.* to 50 and 60 per cent, the mass transfer coefficients of agitated systems were higher than the non-agitated system.

Ertekin and Cakaloz (1996a and b) observed that rate of water loss and solid gain increased with agitation. Similarly, the value of apparent diffusion coefficient for agitated system was reported to be 4 times higher and the equilibrium time decreased significantly compared to non-agitated systems.

Mechanical agitation during the osmotic dehydration reduces the mass transfer resistance at the surface of the fruit or vegetable sample and provides a uniform distribution of osmotic solution around the product. Also Agitation can cause damage to the sample and may be difficult to apply. Few studies showed that the agitation ranging between 100-200 rpm was useful during osmotic dehydration (Pokharkar and Prasad, 1997).

Mavourdis *et al.* (1998a and b) studied the effect of agitation on apple at 20 °C using 50 per cent sucrose solution as the osmotic medium. They concluded that apple showed superior water loss kinetics at higher agitation levels (Reynold's number >16,000) where the turbulent fluid flow was more readily developed .It is well understood that agitation aids in rapid mass transfer during osmosis, but it is not very clear the extent to which it influence.

#### **2.4.10 Solution to sample ratio**

The solution to product mass ratio is another important parameter, which affects osmosis. Most workers used a solution/product ratio of 4:1 in order to monitor mass transfer during osmosis.

In general, as the solution to sample ratio is increased; the rate of osmosis increases up to a certain level and then becomes constant. Ponting *et al.* (1966) observed that increase in dry sugar to fruit ratio above 1:1 neither increased rate nor the extent of osmotic dehydration when sample is agitated. In case of un-agitated process it was 2:1.

Bongirwar and Sreenivasan (1977) reported that 2:1 syrup to sample ratio gave an optimum osmotic dehydration rate in case of banana. However, they experienced the difficulty in draining the syrup from fruit because some un-dissolved sugar was present. With the use of sugar syrup, the rate of osmosis increases with increase in ratio, but the increase is small. Besides larger ratios offer practical difficulties in handling the syrup fruit mixture for processing (Bongirwar and Sreenivasan, 1977; Conway *et al.*, 1983).

Lenart and Flink (1984a and b) carried out experiments on potato with 50 per cent sucrose solution and solution to sample ratio ranging from 1 to 10:1. They concluded that on the basis of dependence of water activity on ratio, the best ratio for osmosis was 10:1. However, when results of water activity and solid gain were considered together, the best osmotic effect was achieved at ratios of 4 to 6. When all aspects were considered, including economics, a ratio of 4 was found to be the optimum. Considering the practical difficulties in handling of large volumes of the solution, a ratio of not more than 10 should be considered for osmosis other than that for some specific study requiring an almost constant solute concentration throughout the process.

Singh *et al.* (2006) Studied the effect of different sample to solution ratio's on the mass transfer kinetics and effective diffusivity during the osmotic dehydration of carrot slices. Sample to solution ratios of 1:4, 1:5 and 1:6 were used. The result showed that there was an increase in water loss and solute gain with increase of fruit to solution ratio. The values of effective diffusivity were high for fruit to solution ratio of 1:5 as compared to 1:4 and 1:6. Nieto *et al.* (2001) studied the kinetics of moisture transfer during the air-drying of blanched and/or osmotically dehydrated mango. For osmotic dehydration at atmospheric pressure, the fruit to syrup ratio used was 1:32 and for vacuum pre-concentration, fruit to sample ratio used was 1:125.

#### **2.4.11 Process pressure**

Most of the osmotic dehydration processes are being carried out under atmospheric pressure. The pressure gradient in the osmotic dehydration process changes the rates of water loss and solid gain. The hydrodynamic mechanism describes the change in the mass transfer

by expansion or compression of internal pore gas of tissue. However, the effect of vacuum on the osmotic dehydration was also studied by few researchers (Fito and Pastor, 1994; Shi *et al.*, 1995).

Mujica-Paz *et al.* (2003) studied the impregnation properties of the some fruits (slices of mango, apple, papaya, banana and peach) at vacuum pressure. Vacuum pressures of 135-675 mbar were applied for 3 and 45 min. Vacuum pressure levels and its application duration had important effect on impregnations of osmotic solutions in the fruits.

Giraldo *et al.* (2006) studied the osmotic dehydration of mango fruits. The process was carried out for 10 min at 50 mbar vacuum, followed by 20 min at atmospheric pressure. The greatest solute gain with more heavily diluted solution observed in the beginning of the process because of vacuum.

#### **2.4.12 Overall effect of various parameters on osmotic dehydration**

Ponting *et al.* (1966) first studied the influence of processing variables on the osmotic dehydration of various fruits such as pre-treatment, temperature, syrup to fruit ratio and additives on the mass transfer and reported 50 per cent reduction in weight of the apples by using sugar solution of 60-70 °Brix. The process resulted in superior quality product and needed no SO<sub>2</sub> treatment to prevent loss of flavour.

Farkas and Lazar (1969) studied the effect of temperature and syrup concentration on rates of osmotic dehydration of Golden Delicious apples. The osmotic drying rates were determined in the temperature range of 30-60°C and syrup concentration of 50-75° Brix. The 50°C and 70°Brix was found to be most favourable condition to reduce 50 per cent of their untreated weight in approximately 8h. The pies made from the products were compared favourably with conventional de-hydro frozen fruit, but their cost would be 25-50 per cent higher.

Ponting (1973) observed that osmotically air dried products though being high in quality, were unstable during storage. The fruits obtained by osmotic dehydration and subsequently vacuum dried were more stable during storage. Applications of osmotic dehydration and optimum operating conditions for apricot, banana and blue berries were also reported.

Karel (1975) reported that intermediate moisture foods (IMF) had received attention since development of new products based on the following technological principles: lowering the water activity by adding a solute, such as glycerol, sucrose, glucose or salt and retarding

microbial growth by adding antimicrobial agents such as ascorbic acid. These intermediate moisture foods were produced with osmotic treatments.

Bongirwar and Sreenivasan (1977) extensively studied the processing temperature and its influence on the quality of the final product and reported a method of partial dehydration of banana by osmosis in sugar solution of 70% concentration and observed that the fruit reduced to about half of its original weight. The temperature also markedly affects the ascorbic acid and chlorophyll retention. They concluded that temperature above 60°C modifies the tissue characteristics favouring impregnation phenomena and thus solid gain.

Theoretical studies carried out by Hawkes and Flink (1978) on osmotic concentration of fruit slices prior to freeze drying, identified that solid gain due to the penetration of soluble substances from the osmotic medium into the fruit and the mass transfer coefficient are the significant factors affecting the water removal.

Canning of apple was not practised commercially due to some inherited problems like presence of high volume of gases in the fruit tissues, difficulty of their removal during exhausting, less drained weight and mushy texture. Sharma *et al.*, (1991) attempted to can apple slices by using firming agents like calcium chloride for improvement in texture and suggested that with the application of this new technology known as osmo-canning, a firm texture, better quality and desired drained weight product could be obtained.

Biswal and Bozorgmehr (1991) reported that partially concentrated fruits and vegetables with osmosis prior to freezing would not only save packaging and distribution cost, but also maintain the product quality comparable to other conventional products. The products obtained by this method were called de-hydro frozen.

Rahman and Lamb (1991) studied osmotic dehydration of pineapple and inferred that water loss and solid gain increased linearly with the increase of sugar concentration and temperature. They reported the rate of sucrose diffusion as a function of solute concentration and temperature, which followed the Magee's model.

Ahmed and Choudhary (1995) prepared the osmotically dehydrated papaya slices by using solution containing sucrose, water and potassium sorbate. The product was subsequently heated in the dry air. It was found that the water loss and solid gain both increased linearly with the increase in sugar concentration. The dried product when packed at 0 °C in high-density polyethylene pouch did not differ in organoleptic quality and was highly

acceptable. The samples kept at room temperature were also found to be acceptable with a little change in colour, flavour and texture.

Vijayanand *et al.* (1995) studied the osmotic dehydration of cauliflower as influenced by temperature, salt concentration, ratio of brine to material and time. They varied the temperature from 40-90 °C, salt concentration from 5-25 per cent, and ratio of brine to material from 2 to 4 weight-to-weight basis and time of osmotic dehydration from 5-180 min. They fitted the weight loss and salt gain to a second order polynomial with multiple regression coefficients in the range of 0.97 and 0.98. They reported maximum water removal of about 40 per cent with a minimum salt gain of 4.2 per cent in the cauliflower at optimum condition of 80 °C temperature, brine to material ratio of 2, 12 per cent salt concentration and 5 min process time.

Ertekin and Cakaloz (1996a and b) studied the influence of solution concentration and the effect of agitation on the mass transfer mechanism for osmotic dehydration of uniformly sized peas at 30 °C temperature using different solutions of sucrose, sodium citrate and their mixtures. In subsequent air-drying of osmosed peas higher drying rates were observed which shortened the air drying time by 55-70 per cent, depending on the osmosis solution and process conditions. It was shown that by initial osmotic dehydration especially in sucrose and citrate mixture, a product with higher allowable moisture content and increased solid density could be obtained. The rehydration characteristics of osmosed samples were improved. The obtained product had a more attractive colour and standard level of organoleptic acceptability.

Pokharkar *et al.* (1997) reported that water loss in banana slices varied with sugar concentration as well as temperature. They observed the mass transfer coefficient to increase with increase in sugar concentration of the solution and developed a model for prediction of water loss during osmotic concentration of banana slices.

Nsonzi and Ramaswamy (1998a) studied the osmotic dehydration kinetics of the blueberries. The kinetics of moisture loss and solid gain during osmotic dehydration of blueberries under different conditions of temperatures (37-60 °C), concentrations of sucrose solution (47-70 °Brix) and contact time between fruit and sucrose solution (0.5-5.5 h) were studied and modelled based on Fick's second law of unsteady state diffusion. The study showed that all factors *viz.* duration of osmosis, concentration and temperature of sugar syrup influenced moisture loss and solid gain ( $p > 0.001$ ), both generally increasing with temperature (T) and sucrose concentration (C). Based on the diffusion model, the calculated effective moisture diffusivity ranged from  $1.98 \times 10^{-10}$  to  $5.10 \times 10^{-10}$  m<sup>2</sup>/s and solid diffusivity ranged from  $2.54 \times 10^{-11}$  to  $2.22 \times 10^{-10}$  m<sup>2</sup>/s. Both the diffusivity values showed

increasing trend with temperature (T) and sucrose concentration (C), and could be modeled as quadratic function of T and C.

Nsonzi and Ramaswamy (1998b) also evaluated the quality of osmo - convective dried blueberries on the basis of colour, texture and rehydration ratio. The total colour difference and the red blue ratio, hardness and stickiness of the osmo-convective dried blueberries were determined. The results showed osmotic dehydration for short contact time minimized the colour losses during convective air-drying. The osmo-convective dried blueberries were not significantly harder ( $p > 0.05$ ) than conventional air-dried blueberries, however, rehydration ratio of the osmo-convective dried blue berries was lesser than that of freeze-dried and air dried blueberries. The process parameters recommended for osmo-convective drying of blueberries was 50 °C syrup temperature, 55 °Brix sugar solution and 4-5 h of osmotic dehydration.

Bhuvanewari *et al.* (1999) studied the osmotic dehydration of peas using solution of sucrose 30 per cent and 40 per cent concentration and their combination with 20 per cent tri-sodium citrate at temperature ranging from 50-70 °C for duration of 30-120 minutes both under static and agitated conditions. They reported the increased rate of osmosis with the increase in solution concentration, temperature and agitation.

Sharma and Lal (1999) studied the effect of partial osmotic dehydration prior to the canning of plums with or without the addition of calcium ion. The fruits were canned in 35 °Brix sugar syrup. They found that the osmotically treated fruits in 70 °Brix sugar solution at 50 °C for 1.0 h prior to canning resulted in desired drained weight with improved texture, colour and taste in comparison to the conventional method of canning.

Sethi *et al.* (1999) reviewed the osmotic dehydration of temperate fruits. The effect of pre-treatment, variety, temperature, osmotic agent, agitation, geometry of the sample, solution to sample ratio and time of treatment on osmotic dehydration were critically reviewed. They also mentioned the other applications of osmotic dehydration with osmo-appertization, dehydro-freezing and extraction of juice.

Lenart and Dabrowska (1999) studied the kinetics of osmotic dehydration of apple slices and reported higher water loss and lower solid gain from apples coated with pectin than uncoated samples. The pectin solution concentration in the range 0.5-4 per cent and drying time in the range 10-40 min as a pre-treatment had significant influence on mass transfer during osmotic dehydration of coated apples. The pectin solution of concentration 2 per cent and 10 min drying time before the osmotic dehydration was found to be best if water loss, solid gain and water loss/solid gain ratios were considered.

Chaudhary *et al.* (2000) studied the effect of concentration of osmotic agent (sucrose), temperature, solution to sample ratio and agitation on osmotic dehydration rate kinetics and quality characteristics of papaya. The water loss per unit weight of sample and amount of water available for osmosis, and the drying rate were determined. It was observed that rate of water loss was high during first two hours. The agitation has the largest effect on the water removal rate, while concentration has the second largest effect. The temperature and solution to sample ratio have almost the same effect. A logarithmic model represents drying behaviour very well. Constant drying rate period was reported to be absent and water removal took place entirely in the falling rate period. Osmotic dehydration for two hours is sufficient to reduce the weight of papaya by about 50 per cent using 70 °Brix agitating sugar solution at 45 °C and solution to sample ratio of 5 and can serve as a pre-treatment prior to further processing. The total solid gain is between 12.8 and 22.18 per cent. Quality evaluation showed that acidity retention varied from 15.9 to 21.3 per cent while vitamin A retention was between 21.0 and 29.7 per cent. The product obtained at high levels of process parameters scores well in sensory evaluation.

Souza *et al.* (2000) evaluated the mass transfer during osmosis in melons processed by hurdle technology. They found that water loss and solid gain increased as the sugar syrup concentration and syrup proportion in relation to fruits were increased. It was also found that the mass transfer was more influenced by the syrup concentration than the fruit to syrup ratios.

Krokida *et al.* (2000a and b) studied the colour and sorption characteristics of osmotically treated and air dried apple and banana. A first order kinetic model was developed and fitted to the experimental data for colour parameters. The sorption data of treated and air-dried product were fitted to GAB model. They concluded that the osmotic dehydration prevented colour damage and decreased the sorption capacity of dehydrated products.

Amitabh *et al.* (2000) studied the effect of osmotic dehydration technique on four mango varieties *viz.* Chausa, Dashehari, Langra and Fazari, keeping the concentration, volume and temperature of sugar syrup constant. Mango slices of Dashehari cultivar dipped for 18 hours in 70 °Brix sugar syrup containing 0.5 per cent citric acid were adjudged the best on drying, having non significant difference with Chausa, but significant difference existed with Fazari and Langra cultivars. The storage studies showed that keeping the osmotic dehydrated mango slices between 64.8 and 75.5 per cent rh at room temperature would be conducive to the retention of colour, flavour and taste and texture of the product.

Rastogi *et al.* (2002) studied the various methods to enhance mass transfer in osmotic dehydration process and suggested that application of high hydrostatic pressure, high

electrical field pulses, ultra sound, vacuum and centrifugal force may increase the rate of mass transfer in osmotic dehydration, but process requires sophisticated techniques to monitor process parameters.

Rodrigues *et al.* (2003) evaluated rheological properties and colour of papaya during osmotic dehydration process in sucrose solution and found increased mass transfer with temperature and soaking solution concentration. They also obtained colour intensification along the process.

Allaeddini and Emam-Djomeh (2004) used osmo-dehydration a pre-treatment for producing dehydrated kiwifruit (cultivar: Hayward or Abbot) slices. The effects of operational variables: solution concentration (30, 40 and 50%), bufferic agent (citric acid) and temperature (20, 40 and 60°C) on dehydration efficiency were studied. Both osmosed and control samples were dried by hot air drying. The dried products were analyzed in order to measure their textural and organoleptic properties. The results showed that using osmotic solution could improve both textural and organoleptic properties of dried product. And the best product was obtained in following operational condition: concentration of sucrose - 40%, concentration buffer - 6%, temperature - 40°C and time - 120 minutes.

Sodhi *et al.* (2006) studied the osmotic dehydration kinetics of carrots using sucrose, glucose and salt solution. The regression analysis was carried out to develop mathematical model for water loss and solid gain involving immersion temperature, solute concentration and immersion duration. They proposed models to determine the osmotic dehydration time in getting the desired level of sucrose, glucose or salt content in carrot slices.

Jokic *et al.* (2007) used response surface methodology for quantitative investigating of water and solids transfer during osmotic dehydration of sugar beet cossettes in combined aqueous solutions of sucrose and sodium chloride. Effects of immersion time (30-240 min), sucrose concentration (30-70%, w/w), sodium chloride concentration (0-8%, w/w), and temperature of the osmotic solution (30-50 °C) were estimated. Quadratic regression equations describing the effects of these factors on the water loss and solids gain were developed. It was found that effects of immersion time and sucrose concentrations were more significant on the water loss than the effects of sodium chloride concentration and temperature. As for solids gain immersion time and sodium chloride concentration were the most significant factors.

Tonon *et al.* (2007) investigated the influence of temperature (20-40 °C), solution composition (0% salt/65% sucrose – 10% salt/55% sucrose) and agitation speed (0-1000 rpm) on the mass transfer kinetics of osmotically dehydrated tomato halves. The mass transfer kinetics were modelled using a first-order kinetic equation, by way of an empirical parameter

(*k*) representing an overall mass transfer coefficient and reported that the temperature and osmotic solution composition showed significant effects on all the responses studied. Increasing temperature and NaCl concentration led to higher water and solute mass transfer coefficients. Agitation speed influenced the water mass transfer coefficient, indicating the presence of a mass transfer resistance in the osmotic solution. The salt and sucrose mass transfer coefficients were not affected by the agitation speed.

Lombard *et al.* (2008) analysed the effect of osmotic dehydration on mass fluxes (water loss, solids gain and weight reduction) was investigated. Pineapple cylinders of 2 cm in diameter and 1 cm thick were immersed in sucrose solutions of 45, 55 and 65 °Brix at 30, 40 and 50 °C for 20, 40, 60, 20, 180 and 240 min. Experiment were conducted at both atmospheric pressure and applying a 200 mbar vacuum pulse during the first 10 min. Water loss and solids gain increased with temperature and concentration. Applying a vacuum pulse facilitated water loss especially at the highest concentration and temperature.

Garcia-Segovia *et al.* (2010) studied the effect of osmotic dehydration (OD) on aloe vera (*Aloe barbadensis* Miller) leaves. Peeled and unpeeled aloe vera slices (15 × 50 mm) were immersed in sucrose solutions at 35, 50 and 65 °Brix at 25 and 40 °C. Moisture, effective diffusion coefficients and mass fluxes (water loss, solids gained and weight reduction) were determined. The best conditions for the OD of aloe slices with the highest effect on diffusivity were obtained using a temperature of 40 °C for peeled samples.

## **2.5 Mass Transfer During Osmosis**

In osmotic dehydration operation, there is a simultaneous counter current mass transfer of water from sample to hypertonic solution and of solute from solution to the sample. Soluble solids of the sample such as organic acid, minerals and vitamins also migrate relatively in small quantity from sample to solution. Fundamental aspect of mass transfer in a cell and in aggregate cell (*i.e.* tissue) has been given in detail by Maguer (1988).

The mass transfer phenomenon is diffusive in such type of processes. The rate of mass transfer can be approximately predicted by appropriate solution of the simplified unsteady state diffusion equation. This requires the knowledge of effective diffusivity. Crank (1975) has given a detailed discussion on theoretical aspect of diffusion process. Analytical solutions are available for the simple geometry of the sample and problems involving either concentration of external solution to be constant or have a fixed volume of solution. Moreover, the resistance to mass transfer is negligible at the surface compared to that of internal resistance in the solids.

Hawkes and Flink (1978) reported so called mass transfer coefficient by plotting the normalized solids content of the apple slices (percent total solids change based on the initial

total solid content) versus the square root of time and calculating slopes for various osmotic solutions and concentrations.

Pokharkar and Prasad (1998a) undertook osmotic dehydration of banana slices in sugar syrup of 50-70 °Brix and constant sugar syrup to fruit ratio of 4:1. They found that about one third of the initial moisture content of the banana slices could be removed by osmotic dehydration. They also reported a linear increase in water diffusivity with increase in sugar syrup concentration.

Cherry tomato samples were osmotically dehydrated in hypertonic NaCl solutions (with or without sucrose) at two different concentrations (Azoubel and Murr, 2004). Mass transfer kinetics were modelled according to Peleg, Fick and Page equations. The Peleg equation presented the best fitting for water loss and Page model showed the best predictive capacity for salt gain data. The effective diffusivity determined using Fick's second law applied to a spherical geometry was found to be in the range of  $0.43 \times 10^{-9}$  -  $1.77 \times 10^{-9}$  m<sup>2</sup>/s for water loss and  $0.04 \times 10^{-9}$  -  $0.54 \times 10^{-9}$  m<sup>2</sup>/s for salt gain.

Falade *et al.* (2007) studied mass transfer during osmotic dehydration of watermelon, and colour changes of fresh, osmosed and osmo-oven dried watermelon. Mass transfer during osmotic dehydration was modelled using Fick's second law of diffusion. Moisture and solid diffusivities were in the range of  $1.030 \times 10^{-8}$  -  $3.549 \times 10^{-9}$  m<sup>2</sup>/s and  $1.117 \times 10^{-8}$  -  $8.540 \times 10^{-9}$  m<sup>2</sup>/s, respectively. Activation energy for moisture and solid diffusivity ranged from 5.09 to 32.77 kJ/mol respectively.

During osmotic dehydration of aloe vera leaves Garcia-Segovia *et al.* (2010) observed water diffusivity values varying between  $0.19 \times 10^{-8}$  and  $1.98 \times 10^{-8}$  m<sup>2</sup>/s, increasing syrup temperature from 25 to 40 °C. The  $D_e$  values in the peeled aloe slices were higher than in unpeeled slices.

## **2.6 Optimization of Osmotic Dehydration Process**

Uddin *et al.* (2004) investigated water and sucrose transfer during osmotic dehydration of carrot slices using response surface methodology with the sucrose concentration (40-60%, w/w), temperature of sucrose solution (40-60 °C) and immersion time (0.5-6.0 h) being the independent process variables. Quadratic regression equations describing the effects of independent process variables on the water loss (WL) and sucrose gain (SG) were developed. It was found that immersion time and concentration of sucrose solution were the most significant factors affecting the WL during osmotic dehydration of carrots followed by temperature. Effect of temperature and time were more pronounced for SG than the concentration of sucrose solution.

Eren and Kaymak-Ertekin (2007) used response surface methodology to determine the optimum processing condition that yield maximum water loss and weight reduction and minimum solid gain and water activity during osmotic dehydration of potatoes. Temperature (20-60 °C), processing time (0.58-8 h), sucrose (40-60% w/w) and salt (0-15% w/w) concentration were the factors investigated with respect to water loss (WL), solid gain (SG), weight reduction (WR) and water activity ( $a_w$ ). Experiments were designed according to Central Composite Rotatable Design with these four factors each at five different levels, including central and axial points. Experiments were conducted in a shaker (Thermoshake-Gerhardt) with constant agitation of 200 rpm and solution to sample ratio of 5:1 (w/w). With respect to water loss, solid gain, weight reduction and water activity, both linear and quadratic effects of four variables were found to be significant. For each response, second order polynomial models were developed using multiple linear regression analysis. The response surfaces and contour maps showing the interaction of process variables were constructed. Applying desirability function method, optimum operating conditions were found to be temperature of 22 °C, sucrose concentration of 54.5 per cent, salt concentration of 14 per cent and treatment time of 329 min. At this optimum point, water loss, solid gain, weight reduction and water activity were found to be 59.1 (g/100 g initial sample), 6.0 (g/100 g initial sample), 52.9 (g/100 g initial sample) and 0.785 respectively.

Singh *et al.* (2007) conducted experiments for optimization of the osmotic dehydration process of carrot cubes in sucrose solution by response surface methodology (RSM). The independent process variables for the osmotic dehydration process were osmotic solution concentrations (45-55 °Brix), temperature (35-55 °C) and process durations (120-240 min). Statistical analysis of results showed that all the process variables had a significant effect on all the responses at 5 % level of significance ( $P < 0.05$ ). The osmotic dehydration process was optimized by RSM for maximum water loss, rehydration ratio, retention of colour, sensory score and minimum solute gain. The optimum process conditions were 52.5 °Brix sucrose syrup concentration, 49 °C osmotic solution temperature and 150 min process duration.

Fresh pineapple samples were osmotically dehydrated by Singh *et al.*, (2008) at different sucrose concentrations, temperature, time and fruit solution ratio. The response variables considered were: water loss, solid gain, ratio of water loss to solid gain (WL/SG) and weight reduction. A central composite rotatable design (CCRD) was used as experimental design. A response surface methodology (RSM) was used to analyse and predict the optimum conditions for pineapple osmotic dehydration. All model terms were significant in water loss except quadratic level of temperature whereas in solid gain, the model terms were significant expect fruit to solution ratio at quadratic level. The optimum dehydration parameters

corresponded to 62°Brix sucrose concentration, 30 °C temperature, 6 h osmosis time and 1: 6 fruit to solution ratio to obtain water loss of 48.41%, solid gain of 10.9%, WL/SG ratio of 4.4 and weight reduction of 37.0%.

Response surface methodology was used by Alam *et al.* (2010) to investigate the effect of sugar concentration (50-70 °Brix), solution temperature (30-61 °C), solution to fruit ratio (4:1-8:1) and immersion time (60-180 min) on the water loss, solute gain, rehydration ratio, vitamin-C loss, colour change and sensory overall acceptability of Indian gooseberry (aonla) slices. The optimum process parameters obtained by computer generated response surfaces, canonical analysis and contour plot interpretation were: sugar concentration, 59 °Brix, solution temperature 51 °C, solution to fruit ratio 4:1 and immersion time of 60 min.

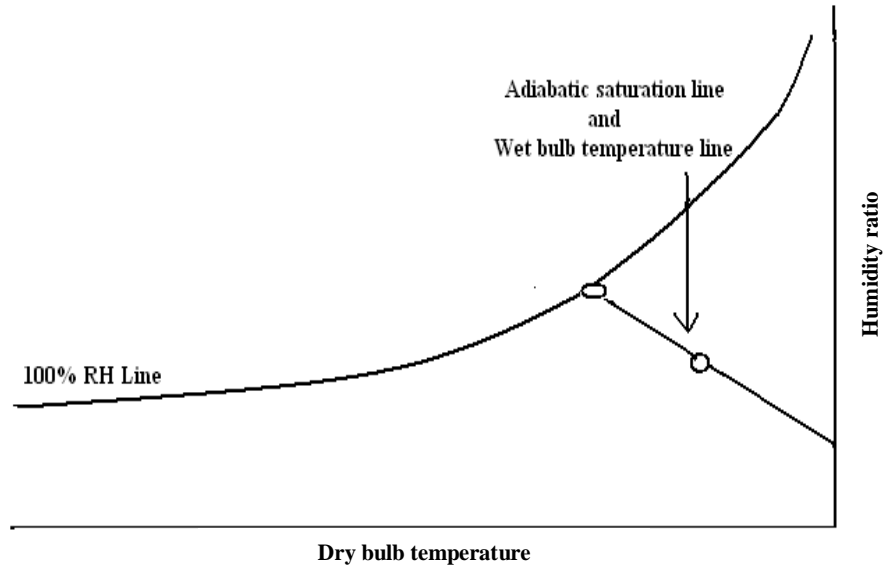
Process temperature (30, 40 and 50 °C), syrup concentration (50, 60 and 70 °Brix) and process time (4, 5 and 6 h) for osmotic dehydration of papaya (*Carica papaya*) cubes were optimized for the maximum water loss and optimum sugar gain by using response surface methodology. The syrup to papaya cubes ratio was 4:1 (w/w). Initial moisture content of papaya samples were 87.5–88.5% (wb), which was reduced to 67.6–81.1% after osmotic dehydration in various experiments showing mass reduction, water loss and sugar gain in the range of 20.6–36.4, 23.2–44.5 and 2.5–8.1%, respectively. The weight reduction, water loss and sugar gain data were statistically analysed and regression equation of second order were found the best fit for all the experimental data. Maximum water loss of 28% with optimum sugar gain of 4% was predicted for the 60°Brix syrup concentration at 37 °C for syrup to fruit ratio as 4:1 in 4.25 h of osmotic dehydration (Jain *et al.*, 2011) .

## **2.7 Drying and Dehydration of Fruits and Vegetables**

Drying is one of the most important methods of preservation and production of wide varieties of the products, and major aim to prolong its storage life. Unfortunately, changes in the physical and biochemical structure are inevitable because the fruits are treated with thermal, chemical and other treatments (Ratti and Mujumdar, 1996).

The techniques of dehydration are probably the oldest method of food preservation applied by mankind. The difference between dehydration and drying – drying is removal of moisture from the materials with phase change, whereas in dehydration (e.g. osmotic dehydration) water mostly stays in liquid form.

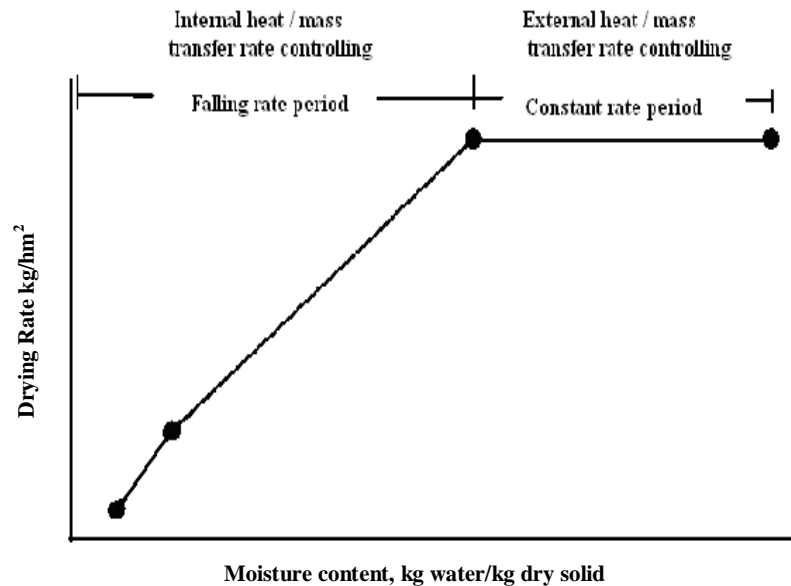
Theoretical concepts of drying deal with air-water mixture properties which consist of moisture content, wet bulb and dry bulb temperature, and adiabatic saturation. The concept of adiabatic saturation line is summarized in Fig. 2.5. It shows no change in the wet bulb temperature but the relative humidity increases due to the absorption of moisture from a drying product.



**Fig. 2.5 Representation of adiabatic saturation in the psychrometric chart**  
(Barbosa- Canovas and Vega-Mercado, 1996)

Generally in mechanisms of drying two processes taking place at the same time, transfer of energy and transfer of mass. Energy transfer can be conductive, convective, radiative or any combination of these three. Mass transfer includes the removal of moisture that moves from the interior of the dried materials toward the object surface under the capillary forces, liquid diffusion due to concentration gradients, surface diffusion, and water vapour diffusion in pores filled with air, flow due to pressure gradient as driving force, and flow owed to a vapourisation-condensation system (Barbosa- Canovas and Vega-Mercado, 1996).

The complete drying process can be divided in three stages. The first stage of drying is when only free moisture at the surface is removed so the drying rate is constant. This is called the “constant rate period”. At the end of constant rate period, dry spots appear on the surface of the material and the drying rate decreases. This is the beginning of the “falling rate period” (Fig. 2.6). Once the surface is completely dried, moisture is transported from inside of the product to the surface by capillary action. The third drying period is called “the second falling rate” and the drying rate is lower than the previous step (Mujumdar and Menon, 1995).



**Fig. 2.6 Typical drying rate curves under constant- drying conditions** (Adapted from Mujumdar and Sirikalaya, 2000)

Several types dryers and drying methods have to be developed and adopted for fruits with each specific situation and commercially application. These dryers can be divided according to many factors, such as pressure (atmospheric, and sub atmospheric), type of unit operation (continuous, batch and semi continuous), the temperature (freeze drying and hot air convective drying) and many others. And several dryers have been developed for the drying of fruits by using different techniques (Somogyi and Luh, 1986; Jayaram and Das Gupta, 1992) as follows:

- i) Sun drying (open sun drying floor).
- ii) Atmospheric drying
  - a) Stationary or batch process (kiln, tower and cabinet dryer).
  - b) Continuous processes (tunnel, continuous belt, belt through, fluidised, explosion puffing, foam mat, spray, drum and microwave- heated dryer).
- iii) Sub atmospheric drying (vacuum shelf, vacuum drum and freeze dryer).

The main advantages adopted in drying of fruits and vegetable are following (Somogyi and Luh, 1986).

- i) Almost getting unlimited shelf life, because inhibition of microbial and enzymatic reaction is high.
- ii) Substantially lower costs of handling, transportation and storage.
- iii) Mineral content is practically unaffected, as well as calorie providing constituent.

- iv) Providing consistent product – seasonal variations are diminished.
- v) Dried fruits are packed in recyclable packages; this is not always done with fresh fruits.
- vi) They can be used in snack products and other processed food.

The main problems in fruit drying are damage to sensory characteristics and loss of nutritional components due to long exposure to high temperatures (Van Arsdel, 1973; Fellows, 1988). These include loss of aroma volatiles, oxidation of pigments and vitamins and case-hardening in certain products.

## 2.8 Convective Drying of Aloe vera

Simal *et al.* (2000) studied the effect of air drying temperatures (30 to 80 °C) on dehydration curves and functional properties (water retention capacity, swelling and fat adsorption capacity) of aloe cubes. They reported that effective diffusivities estimated with the proposed model varied with the air drying temperature according to the Arrhenius law except for the experiment carried out at 80 °C where case hardening took place. They also found that the three studied functional properties exhibited a maximum when a drying temperature was 40 °C decreasing either at lower or higher temperature.

Femenia *et al.* (2003) studied the effects of heat treatment and dehydration at different temperatures (30 to 80 °C) on bioactive polysaccharide acemannan and cell wall polymers from *Aloe barbadensis* Miller. They found that modification of acemannan, a storage polysaccharide, was particularly significant when dehydration was performed above 60 °C.

Ganjyal *et al.* (2003) studied the drying characteristics of sapota (*Achras zapota*, cv, Kalipatti), a tropical fruit crop. Fruits were cut in sizes of half, quarter, and 5 mm slices and dried at temperatures of 55, 60, 65, and 70 °C in convection air and vacuum ovens. Moisture content of the fruit reduced from 72 to 78% (w.b) to 8.5 to 12.5% (w.b.) with drying times of 15 to 35 h in a convection air oven and of 14 to 31 h in a vacuum oven. It was found that with increase in temperature and decrease in sample size, drying was faster. Ganjyal *et al.*, (2005) processed sapota (*Achras Zapota*), a tropical fruit, into a value-added powder and evaluated for use in traditional Indian recipes. Fruits of sapota (cv, kalipatti) were halved, quartered, or sliced 5mm thick; dried at 55, 60, 65 and 70°C in convection and vacuum ovens; then ground to pass through a 105µ sieve. Total soluble solids, acidity, ascorbic acid and overall quality were highest when dried at 65 °C.

Antonio *et al.* (2007) studied and modelled the kinetics of the hot air drying of aloe vera (*Aloe barbadensis* Miller) and evaluated the influence of temperature on the kinetic

parameters for the proposed models. The product was dried at 50, 60, 70, 80 and 90 °C with an air flow of  $2.0 \pm 0.2$  m/s in convection dryer. The sorption isotherm of fresh product was mathematically described by the Guggenheim, Anderson and de Boer (GAB) model, giving as a result monolayer moisture of 0.20 g water/g dm. The mathematical models evaluated in the study included five empirical equations (Newton, Henderson-Pabis, Page, modified Page and Fick's diffusional model). The fit quality of the proposed models was evaluated by using the regression coefficient ( $r^2$ ), sum square error (SSE), root mean square error (RMSE) and Chi-square statistic ( $\chi^2$ ). The diffusivity coefficient increased from  $5.30 \times 10^{-10}$  to  $17.73 \times 10^{-10}$  m<sup>2</sup>/s, for a range of temperatures between 50 and 90 °C, with estimated activation energy of 30.37 kJ/mol. The modified Page model provided the best to fit of the data, showing that this equation correctly simulates the aloe vera drying process and represents an excellent tool for estimating its drying time.

Kingsly and Singh (2007) performed thin layer drying of pomegranate arils in a cabinet drier at drying temperature of 50, 55 and 60°C. the time required to dry pomegranate arils from an initial moisture content of 271.80% (d.b.) to the final moisture content of  $8.70 \pm 1\%$  (d.b.) was 11, 9 and 6 h at 50, 55 and 60°C of drying air temperature, respectively. The Drying time decreased with increase in drying air temperature. Drying of pomegranate arils occurred in the falling rate period.

Swami *et al.* (2007) studied the drying characteristics of bori nuggets, an Indian traditional food, prepared from black gram batter using hot air temperature of 30, 50 and 70°C and air flow rates of 0.6, 1.0 and 1.4 m/s. The drying constant obtained from Lewis equation decreased from 0.457 to 0.139, 0.514 to 0.324 and 0.735 to 0.487 ( $r \geq 0.975$ ;  $MSE \leq 0.0082$ ) for 30, 50 and 70 °C, respectively, while the air velocity decreased from 1.4 to 0.6 m/s in each case. The correlation among drying constant, air velocity and air temperature for the drying was found to best described by Arrhenius equation ( $r = 0.995$ ;  $MSE = 0.0029$ ). Statistical analysis (ANOVA) revealed that effects of both air temperature and air velocity on yellowness index, hardness and cooking time of the nugget as well as on drying time were significant ( $p \leq 0.01$  or 0.05). Response surface methodology was tried for optimization of these two independent variables with respect to the above-mentioned parameters. The superimposed contour plots suggest optimum drying air temperature between 59 and 60 °C and air velocity between 0.9 and 1.05 m/s for attaining minimum values of yellowness index (44), hardness (9.0-9.5 N), cooking time (8.0-8.4 min) and drying time (250-275 min).

Jangam *et al.* (2008) dried the sapota paste in a convective dryer to study the effect of operating parameters such as air temperature and air velocity. In addition, the effect of additives such as oat and wheat fibres and the paste thickness on drying was also studied. The

critical analysis of dehydrated sapota was carried out in terms of water activity, sugar content, colour and rehydration ratio.

Shi *et al.* (2008) employed response surface methodology (RSM) to optimize the drying conditions of horse mackerel dried in a heat pump dehumidifier (HPD). The independent variables were drying air temperature (20-30 °C) drying air velocity (1.5-2.5 m/s) and sodium chloride (NaCl) content in the osmotic solution (5-15%). The coefficient of determination  $R^2$  values for specific moisture evaporation ratio (SMER), drying rate (DR), total colour difference ( $\Delta E$ ) and total volatile base nitrogen (TVBN) were greater than 0.89. The combined effects of these variables on SMER, DR,  $\Delta E$  and TVBN were investigated. The results showed that SMER,  $\Delta E$  and TVBN varied curvilinearly with increasing of temperature, velocity and NaCl content in the osmotic solution and DR increased linearly. Based on response surface and desirability functions, the optimum conditions for horse mackerel drying by HPD were: drying air temperature 30 °C, drying air velocity 1.5 m/s and NaCl content in the osmotic solution 9.9 per cent.

Erbay and Icier (2009) used response surface methodology to optimize operating condition of drying the olive leaves in a tray drier. Optimization factors were air temperature (40-60 °C), air velocity (0.5-1.5 m/s) and process time (240-480min) while investigated responses were total phenolic content (PC) and antioxidant activity loss (AC), final moisture content (MC) and exergetic efficiency ( $\eta$ ). The optimum conditions for drying of olive leaves in a tray drier were determined to obtain the criteria; minimum PC and AC, and maximum  $\eta$  for MC value below 6 per cent and the optimum condition was found to be the temperature of 51.16 °C with the air velocity of 1.01 m/s for the process time of 298.68 min.

Gulia *et al.* (2009) dried aloe vera leaves at different temperatures in hot air oven and powdered. The percent powder yield was found 2.60, 2.55 and 2.52 per cent at 50, 60, 70 and 80 °C respectively. Powder samples had the pH (1% solution) 3.51, 3.53, 3.52 and 3.53 with the rise of drying temperature in the selected range. The "a" values were found 2.028, 2.226, -0.282 and 2.531 for the samples obtained after drying at 50, 60, 70 and 80 °C respectively. Samples obtained at 70 °C showed negative "a" value indicated that the samples were more greenish in colour as compared to other samples. 'L' value (lightness) of different samples were 43.82, 47.82, 59.30 and 54.82 at 50, 60, 70 and 80 °C respectively. The samples obtained at 70 °C had shown the highest value, indicating better colour spectrum in terms of lightness. This work on hot air oven drying of aloe vera leaves at different temperature showed that the aloe powder obtained at 70 °C had better physico-chemical and functional properties than the samples obtained at other different temperatures selected for the study.

Kaya *et al.* (2009) investigated drying kinetics of kiwifruit experimentally and theoretically under varying drying conditions. Experiments were conducted using air temperatures at 35, 45, 55 and 65 °C, mean velocities at 0.3, 0.6 and 0.9 m/s and, relative humidity values at 40, 55, 70 and 85 per cent. The values of the moisture diffusivity,  $D_{\text{eff}}$  were obtained from the Fick's diffusion model. The effects of the governing drying parameters on the vitamin C content as well as on the total drying time were determined. In addition, it is disclosed that increasing drying air temperature causes more loss in vitamin C in the dried fruits. It also found that increasing the temperature or the velocity of the drying air decreases the total drying time, while decreasing the relative humidity decreases the total drying time.

Miranda *et al.* (2009a) investigated the effect of air temperature on the physicochemical and nutritional properties of aloe vera (*Aloe barbadensis* Miller) gel and modeled the drying kinetics of aloe vera gel using the Wang-Singh equation. They reported that the drying temperature of 80 and 90 °C resulted in significant variation in and/or loss of the physicochemical and nutritional properties of the gel. These effects were also observed as a result of a longer drying period even at low temperature (i.e., 810 min at 50 °C). However minor alterations in the physicochemical and nutritional properties of aloe vera gel were produced at drying temperatures of 60-70 °C, resulting in the production of a high quality gel.

Vairat (2009) studied hot air drying of banana. Slice thickness, drying temperature and air velocity were taken as independent variables whereas moisture content, ash content, ascorbic acid, carbohydrates, colours and hardness were considered as dependent variables. The influence of various factors was analysed using analysis of variance (ANOVA) techniques. Second order model was fitted in various responses and independent variables using multiple regression analysis. Multiple regression analysis was done in MINITAB-11.0. The optimization of process parameters (drying temperature, air velocity and sliced thickness) for hot air drying of banana sliced was carried out using numerical optimization techniques with the help of Design Expert 7.1.3 statistical software.

Zielinsk and Markowski (2010) investigated the effects of air temperature on drying kinetics of carrot cubes. Drying was carried out at 60, 70, 80 and 90 °C and the falling drying rate data were used to calculate the effective diffusion coefficients from the Fick's equation. Four mathematical models available in the literature were fitted to the experimental data. The Two-term model gave better prediction than the Henderson and Pabis, Page and Lewis model and satisfactorily described drying characteristics of carrot cubes.

## **2.9 Convective Drying of Osmotically Dehydrated Fruits**

There are various methods of drying such as air, freeze, vacuum *etc.* Many investigators recommended that the quality such as colour, flavour and texture of air or freeze

dried fruits and vegetables were improved by a prior osmotic dehydration step. There are numerous possibilities of subsequent drying of osmosed samples by which the product can be made shelf stable. However, it is beyond doubt that osmosis enhances the quality of the product when it is subjected to subsequent drying process. This makes osmosis a potential pre-treatment method in food preservation. Osmosis is reported to reduce the drying rates during subsequent dehydration in some cases due to choking of pores caused by heavy solute impregnation. An attempt has been made to review the work reported by various researchers.

Jackson and Mohammed (1971) dehydrated banana by osmotic dehydration for period of one minute to 18 hours followed by air drying. They reported that osmotic dehydration reduced the heat damage to colour, flavour and loss of fresh fruit flavour and also prevented enzymatic browning of the fruit.

Nur (1976) made the comparison between the banana slices dehydrated by osmo-air dehydration and air-dehydration. In osmo-air dehydration, moisture of the slices was reduced by 11 to 12.5 per cent by osmotic dehydration with vigorous stirring and further it was reduced to 10.0 per cent by air drying. He reported that osmo-air dehydrated slices rehydrated more slowly than air dehydrated slices and were less hygroscopic. Since the overall quality of osmo-air dehydrated slices was no better than that of air-dehydrated slices, the osmo air dehydration process was not recommended for commercial application. Sulphitation of the banana slices prior to dehydration considerably reduced colour deterioration by 1000 ppm sodium metabisulphite for up to six months and by 500 ppm up to five months storage.

Islam and Flink (1982) investigated the effect of osmotically concentrated and subsequent air drying behaviour of potato slices. From the drying curves, the diffusion coefficient of water (D) was found for the first falling rate period while the activation energy for diffusion was calculated from a plot of  $\ln(D)$  vs  $1/T$ . Result for these tests revealed that for all samples (osmosed and non osmosed) drying rate increased with temperature. They also compared three samples at a given temperature (Table 2.4)

**Table 2.4 Effect of osmosis on drying behaviour\***

Treatment	Water diffusion coefficient, $\text{cm}^2/\text{s}$	Drying time, min**
No osmosis	$8.72 \times 10^{-6}$	325
60% sucrose	$4.48 \times 10^{-6}$	195
45% sucrose and 15% sucrose	$2.31 \times 10^{-6}$	60

\* drying at 65°C, 7 mm slice, 2.5 m/s air flow rate

\*\* Time to obtain moisture content corresponding to 0.7 water activity

Shahabuddin *et al.* (1990) evaluated the drying characteristics of osmosed and fresh pineapple slices. The effect of temperature and sample thickness was evaluated when air drying was carried out under controlled conditions. A mass transfer model was used to calculate the effective diffusivity and was found in order of  $10^{-10}$  m<sup>2</sup>/s. In their study no constant rate drying period was observed at any of the conditions studied.

According to Rahman and Lamb (1990), the drying rates of osmosed pineapples were significantly decreased due to the presence of infused solute. The effective diffusivity values of osmosed and non-osmosed samples were obtained by the method of slope of the drying curve, which varied with moisture content and physical structure of pineapple ( $1.62 \times 10^{-10}$  to  $12.54 \times 10^{-10}$  m<sup>2</sup>/s). The effective diffusivities decreased with increase of solid gain (*i.e.* sucrose) in pineapple slices during the osmosis at the same drying conditions.

Rahman and Lamb (1991) used the Fick's law of diffusion to analyse the air drying behaviour of fresh (non-osmosed) and osmosed pineapple. They reported a significant decrease in the drying rates of osmosed pineapples due to the presence of the infused solute. The effective diffusion coefficient decreased with the increase in solid gain during osmosis.

Sankat *et al.* (1992) conducted convective drying of osmotic treated and fresh slices of banana at 40, 60 and 80 °C. They reported that increase in drying air temperature positively influenced the drying rate of fresh fruits slices. However, the drying rate of osmosed slices reduced at 80 °C, which was attributable to caramelization. Dried, osmotic treated bananas, with added SO<sub>2</sub> were reported to have an attractive yellow colour as compared to the dull, dark brown colour of the conventionally dried fresh fruit slices.

Karathanos *et al.* (1995) studied the air-drying kinetics of fresh and osmotically dehydrated apples using two sugars (glucose and sucrose), three levels of concentration (15, 30 and 45%) and time period of 12 h. They found that the water diffusivity of the sample, infused with significant amount of sugars (*i.e.* apple pre-concentrated in a 45% sucrose solution) was significantly lower than the water diffusivity of untreated apples for all stages of drying process.

Welti *et al.* (1995) evaluated the effect of initial water activity of mango slices obtained by osmotic concentration and air drying behaviour at 50, 60 and 70 °C by calculating the apparent diffusion coefficient using Fick's law. They found that the diffusion coefficient decreased with the increase in solid content during osmosis and increased with the drying temperature. Moreover, the drying time needed in osmotically treated mango was considerably reduced as compared to the process without this pre-treatment.

Sankat *et al.* (1996) carried out osmo-air dehydration of banana slices in sugar solution of 35, 50 and 65 °Brix for 36 h and subsequently, dried them in a cabinet tray dryer at

fixed temperature from 40 to 80 °C. They reported that during drying the moisture diffusivity significantly decreased with the decrease in moisture content and increase in sugar level. Osmo-air dried banana slices showed appealing colour and texture as compared to untreated ones.

Sharma *et al.* (1991) reported that osmo-vac dried slices had good colour, taste, flavour and crunchy texture when treated in sucrose solution for 4-5 h (56-59% water removal and 11.8-14.6% solid penetration), in contrast to the poor taste and objectionable leathery texture of conventionally dried slices. Blanching with steam for 3 min and pre-treatment of slices with antioxidant (0.5%  $K_2S_2O_5$ ) prior to the osmotic dip proved necessary for better retention of ascorbic acid and prevention of browning during drying and storage.

Szymczak *et al.* (1998) dehydrated 8 plum cultivars and evaluated the fruit quality, which showed osmotic dehydration as a pre-treatment before convective drying improved the flavour of product by increasing the soluble solids to acidity ratio.

Krokida *et al.* (2000a and b) conducted osmotic dehydration of apple and banana in glucose solutions (50% w/v) at 30, 40 and 60 °C. After 10 h, in air drying of osmotically dehydrated samples at 70 °C and 15% relative humidity, they observed extensive browning in untreated fruits, while osmotically pre-treated samples did not brown.

Singh (2001) conducted the osmotic dehydration of carrot shreds in sucrose solution (50 °Brix) at room temperature prior to drying in cabinet drier at 55 °C. Drying time required 6 h for osmosed carrot where as 12 h required for un-osmosed carrot shreds. Also moisture sorption isotherm studies revealed that the un-osmosed dehydrated carrot shreds were more hygroscopic as compared to the osmosed dehydrated samples and required a lower RH for safe storage.

The combination of osmotic pre-treatment mainly in sucrose solutions and microwave-vacuum dehydration of straw berries and apples were studied by Erle and Schubert (2001). They reported that as compared to the solely microwave-vacuum dried samples; osmotic pre-treatment improved volume retention from 20 to 50% for strawberries and from approximately 20 to 60% for apples based on fresh volume.

Kaleemullah *et al.* (2002b) studied the osmotic air drying characteristics of papaya cubes. The effect of the solute concentration (50, 60 and 70 °Brix) and temperature (32, 50 and 60 °C) on osmotic dehydration of papaya were studied. The dehydration rate of papaya cubes (6.58 kg water/kg dry matter) were reduced from 2.12 to 0.3, 2.38 to 0.31 and 2.92 to 0.54 kg water /h/kg dry matter during osmosis in first half an hour to fourth hour with the sugar syrup concentration of 50, 60 and 70 °Brix, at 32 °C temperatures. The dehydration rates of papaya cubes were reduced from 2.38 to 0.31, 3.16 to 0.29 and 4.17 to 0.44 kg water/h/kg dry matter

during osmosis in first half an hour to fourth hour at 32, 50 and 60 °C temperatures and 60 °Brix sugar syrup concentrations respectively. The overall drying rate of papaya by osmo convective drying was more (0.528 kg water/h/kg dry matter) than the one dried by convective method (0.318 kg water/h/kg dry matter). The osmotic (60 °Brix, 60 °C) air (60 °C) drying method saved 8 h in drying papaya from 6.58 to 0.24 kg water kg dry matter as compared to the one dried by air drying (60 °C) method.

Noze *et al.* (2002) combined an osmotic drying and air drying process. Banana slices (10 mm thickness) were soaked in two successive sucrose baths with concentrations of 35 and 70 °Brix. A fruit to solution mass ratio was 1:6 and 1:4 used in the respective soaking process. This second treatment (70 °Brix) served to increase the sugar content of the banana slices, extend the degree of water lost and maintain good texture and shape in the product. The osmotic drying process resulted in a weight reduction of 60 per cent and a solute gain of 2 per cent. Slices were then subjected to air drying at 40 °C and relative humidity of 60 per cent for 72 hours. The final dried product had a moisture content of 16 per cent, an average water activity of 0.54 at 25 °C and a highly acceptable colour without any brown pigmentation.

Mandala *et al.* (2005) investigated the influence of different osmotic pre-treatments on apple air-drying kinetics and their physical characteristics during drying. Apple samples were immersed in glucose and sucrose solutions of 30 and 45 per cent (w/w) respectively at different times. Sugar gain (SG) and water loss (WL) were calculated and an immersion time of 12h was selected. Porosity, compressive fracture stress and colour were measured. Apple osmosed in glucose showed a large moisture decline in the early drying periods. Osmosed apples in sucrose showed lower drying rates ascribed to sugars concentration on the outer layers of apple tissue and their crystallization during drying. Samples pre-treated in 45 per cent sugar solutions had greater porosity and better colour retention during drying. In glucose osmosed sample at greater texture hardening rate was observed, in sucrose just the opposite occurred.

Ehabe *et al.* (2006) found that osmotic pre-treatment before air drying improved the figs brightness. Sugar or NaCl solution (at 10 per cent, w/v ) as well as a composite solution (at 5 per cent NaCl +5 per cent sugar) showed improved water binding activity. The composite NaCl and sugar solution improved sensory qualities and more importantly the storage qualities of banana figs.

Fernandes *et al.* (2006) optimized the osmotic dehydration condition of banana followed by air-drying and developed the model for the osmotic dehydration condition of banana followed by air-drying. The results showed that the temperature used in the osmotic

dehydration affects the diffusivity of water during air-drying elevating the mass transfer rate during air drying as the temperature in osmotic dehydration increased from 50-70 °C.

Ghosh *et al.* (2006) osmosed carrot slices of 5 mm thick for 10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 min in an osmotic solution at 30°C at a sample to solution ratio of 1 : 5 and a constant agitation of 150 rpm. The osmotic solution was prepared by 5, 10 and 15% concentration of salt along with 50 °Brix sugar and 0.1% sodium metabisulphite. A cut-off time for osmotic dehydration prior to hot air drying was decided to be 1 h which would result in a moisture loss of around 50% of initial carrot mass and a solid gain of about 8%, Carrots were osmosed to the cut-off time and then hot air dried at 50, 60 and 70 °C with an air velocity of 0.6 m/s. Non-osmosed carrot slices were also dried by conventional hot air drying method-blanching and drying at 70 °C. Osmo-hot air dried carrot slices received higher scores at 5% significance level in both its dried and rehydrated forms, compared to the conventional air dried carrots. Based on the organoleptic quality and the rehydration ratio, the osmo-hot air drying process recommended for carrot was – osmotic dehydration of carrot slices in 50 °Brix sugar solution containing 5% salt concentration and 0.1% sodium metabisulphite for 1 h followed by hot air drying at 50C. These had a rehydration ratio of 3.3.

Garcia *et al.* (2007) investigated kinetics of osmotic dehydration (OD) and effects of sucrose impregnation on thermal air-drying of pumpkin slices. A simplified model based on the solution of Fick's law was used to estimate effective diffusion coefficients during OD and air-drying. In order to take into account shrinkage, average and variable thicknesses were considered. Pumpkin slices were dehydrated in sucrose solutions (40%, 50%, and 60%, w/w, at 27 °C). The effective water diffusion coefficients were higher than the sucrose and low diffusivity dependence with solution concentration was observed. Samples non-treated and pre-treated in 60% osmotic solutions during one hour were dried in a hot-air-dryer at 50 and 70 °C (2 m/s) until equilibrium was achieved. Pre-treatment enhanced mass transfer during air-drying.

Pisalkar (2008) studied osmotic dehydration ( sugar syrup concentration - 30, 40 and 50 °Brix, sugar syrup temperatures - 30 and 50 °C) and drying characteristics (drying air temperature – 50, 60, 70 and 80 °C and drying air velocity - 1.5 m/s) of aloe vera gel under convective drying system . He observed that the aloe vera samples lost water rapidly for the first 2 h of osmosis and subsequently reduced with in increase in duration of osmosis. Osmosis as a pre-treatment prior to convective air drying was able to decrease drying time by approximately 40 percent. Constant rate drying period was also reported to be absent throughout the drying process of aloe vera cubes under all drying air temperatures.

## **2.10 Diffusivity During Convective Drying**

In drying, the diffusion coefficient or diffusivity is used to indicate the flow of moisture or mass out of the material. The diffusion coefficient of the food material varies with its moisture content. Many researchers have tried to correlate effective moisture diffusivity with temperature and moisture content; and some of that have been reviewed hereunder.

Suk and Bhowmik (1995) estimated the effective moisture diffusivity of yoghurt by method of slopes, using isothermal drying data obtained by microwave vacuum dehydration. The use of microwave vacuum drying seemed advantageous for obtaining isothermal data. The method of slopes of the drying curve was found useful for estimating effective moisture diffusivity from the drying data of yoghurt, because the model obtained from this method simulated the drying curve better.

Simal *et al.* (2000) studied the effect of air-drying temperature (from 30 to 80 °C) on dehydration curves and functional properties [water retention capacity (WRC), swelling (SW) and fat adsorption capacity (FAC)] of aloe vera cubes. A diffusional model taking into account sample shrinkage has been proposed and solved by using finite difference method. The effective diffusivities estimated with the proposed model varied with the air drying temperature according to the Arrhenius law except for the experiment carried out at 80 °C, where case-hardening took place. Effective diffusion coefficients ranged from  $5.64 \times 10^{-10}$  m<sup>2</sup>/s at 30 °C to  $18.1 \times 10^{-10}$  m<sup>2</sup>/s at 70 °C. The activation energy in this study for the dehydration of aloe vera cubes with hot air was 24.4 kJ/mole.

Kin *et al.* (2002) investigated the osmotic dehydration kinetics of pears for three different concentrations and three different temperatures of solution. Peleg, Fick, and Page's mathematical models were fitted to the experimental data. Effective diffusivity calculated with Fick's equation varied from  $3.47 \times 10^{-10}$  to  $1.92 \times 10^{-10}$  m<sup>2</sup>/s for water loss and from  $1.99 \times 10^{-10}$  to  $3.60 \times 10^{-9}$  m<sup>2</sup>/s for solid gain. Water loss and solids gain increased as temperature or solute concentrate increased.

Jaruk and Roberts (2006) determined moisture transfer during drying of hygroscopic non-porous materials such as potato, carrot core and carrot cortex. Drying curves and temperature profiles were obtained from cylindrical samples (0.7 and 1.4 cm diameter) under convective hot air drying (40, 50, 60 and 70 °C and 1.5 and 3 m/s air velocity). The effective moisture diffusivity of these materials was in the range of  $10^{-12}$  -  $10^{-08}$ . Temperature dependence of the effective moisture diffusivity was found to follow the Arrhenius type relationship.

Hassini *et al.* (2007) reported moisture diffusivity data determined from experimental convective drying kinetics. Two different methods have been applied to evaluate this coefficient. The first one was based on the analytical solution of a fickian diffusive model

which enabled diffusivity determination by simple linear regression over experimental data. Due to several simplifying hypothesis the first method could only provide approximate results, while the second could be considered as reference.

Vega-Galveg *et al.* (2007) studied hot-air drying of Aloe vera (*Aloe barbadensis* Miller) at 50, 60, 70, 80 and 90 °C with an air flow of 2.0±0.2 m/s. He observed that diffusivity coefficient increased with the temperature from 5.30 to 17.73 × 10<sup>-10</sup> m<sup>2</sup>/s for a range of temperature between 50 and 90 °C, with an estimated activation energy of 30.37 kJ/mol.

## 2.11 Mathematical Modelling

Inadequate control of product characteristics results in major difficulties regarding process modelling and optimization. Raw material variability is probably the most crucial source of deviations. Variety, maturity level, even cultivation procedures drastically affect the composition and barrier properties of a specific fruit or vegetable.

The complex non-homogenous structure of natural tissues seriously complicates any effort to study and understand the mass transport mechanisms of several interacting counter current flows (water, osmotic solute, soluble product solids).

Hawkes and Flink (1978) obtained the mass transfer coefficients (k) by plotting the normalized solid content (NCS) of the apple slices (percent total solid change) based on the initial total solids versus the square root of time (t) and by calculating slopes for various osmotic solutions and their concentrations. They proposed the following equation:

$$NCS = kt^{0.5} \quad \dots 2.5$$

Conway *et al.* (1983) calculated diffusion coefficient (D) and reported as linear function of temperature (T) and concentration (C). Moreover, interaction between 1/T and D was reported as

$$\log(D) = 11.639 - 5878.2 \times \left(\frac{1}{T}\right) - 0.0194C + 58.427 \times \left(\frac{1}{T}\right) \times C \quad \dots 2.6$$

Biswal and Bozorgmehr (1991) conducted experiments on green beans in sodium chloride solution at three concentrations (5, 10 and 15%) and three temperatures (8, 20 and 40 °C) and developed following relationships for the prediction of moisture loss in fraction of initial water and solid gain in kg/kg of the product:

$$\begin{aligned} M &= 0.037C^{0.629}T^{-0.338}t^{0.5} \\ S &= 0.072C^{1.217}T^{-0.213}t^{0.5} \end{aligned} \quad \dots 2.7$$

Where,

M = moisture loss in fraction  
S = solid gain in kg per kg of initial product  
C = concentration of sugar syrup, °Brix  
T = temperature, °C  
t = time, h

Azuara *et al.* (1992a and b) developed two-parameter equation to predict the kinetics of osmotic dehydration and the final equilibrium point from the mass balance considerations. The model was tested using kinetic data from ten different experiments and could predict water loss and solid gain over long periods of drying with deviation in the range of 2.0-7.7 %. The final equilibrium was estimated using data obtained during a relatively short period of time. Simple equations were obtained from the model related to Fick's second law for non-steady one-dimensional diffusion through a thin slab.

Hough *et al.* (1993) developed a model for osmotic dehydration of fruits taking into account the out going water, incoming solute and shrinkage of fruits during the process. The model adequately described experimental results in terms of water loss, solid gain, reduction of water activity and shrinkage during osmosis for apple pieces in 55 °Brix glucose syrup at 45 °C.

Rastogi and Raghavarao (1995) proposed a semi empirical relation for describing the kinetics of osmotic dehydration of coconut, which considers the moisture diffusion as a function of concentration of osmotic solution and its temperature.

Osmotic dehydration of banana slices, at initial ratio of banana slices to sugar solution 1:4, was studied in relation to temperature (30-50 °C) and initial sugar syrup concentration (50-70 °Brix) by Pokharkar *et al.*, (1997). They reported that water loss varied with temperature and concentration, while the mass transfer coefficient increases with increase in syrup concentration and temperature. The developed model could be used for prediction of water loss during osmotic dehydration of banana slices within the range of experimental study.

Panagiotou *et al.* (1998) osmotically dehydrated samples of apple, banana and kiwifruit by immersing in 30, 40 or 50 per cent sucrose solutions for 0.5, 2, 6 and 16 h at 20, 40 and 60 °C. They developed an empirical model to predict the water loss and solid gain based on a first order kinetic equation in which the rate constant is a function of the main process variables (speed of agitation, solute concentration, size of fruit and process temperature). They applied this model to a wide range of experimental data on the osmotic dehydration and estimated the parameters using non-linear regression analysis. Results

showed that all the stated process variables significantly affected the mass transfer phenomena.

Shi and Maupoey (1993) studied the vacuum osmotic dehydration and suggested it as a novel method of partial dehydration of fruits which leads to a special behaviour of mass transfer in fruit-sugar solution systems. Vacuum treatments intensify the capillary flow function and increase the water transfer ratio significantly. A mathematical model is proposed to describe the total water transfer ratio, which results from a combination of traditional Fickian diffusion and vacuum capillary flow of water during vacuum osmotic dehydration.

Rastogi and Raghavarao (1996) studied osmotic dehydration under vacuum. The effect of vacuum application was explained on the basis of the diffusional osmotic transport parameter, the mass transfer coefficient and interfacial area. A mathematical model is proposed to explain the mechanism of water transport during osmotic dehydration based on a more fundamental parameter, the osmotic pressure. Osmotic pressure ratios were calculated for dehydration under atmospheric conditions and under vacuum. The increased osmotic pressure due to vacuum application was also calculated. It was found that the rate of mass transfer during osmotic dehydration of food increases under vacuum.

Pardeshi *et al.* (2001) conducted study to understand the drying behaviour of peas with high levels of moisture content. Three varieties of peas viz. 'Pb-87' 'Pb-88' and 'Matar Ageta-6' were dried in a series of drying experiments in thin-layer within the temperature and relative humidity range of 55 to 75 °C and 24 to 40%, respectively. Each sample was pre-treated before drying. The automatic weighing system was used to note the loss in weight of drying sample to minimize the errors, which otherwise would have been caused by interruptions. A drying air temperature of 60 °C and drying time of 345 min were found to be most suitable for drying the variety 'Pb-87'. Thin-layer drying rates of peas were determined as a function of drying air temperature. The data were fit to several thin-layer drying models. The drying air temperature had a significant effect on drying rate. Page's equation with drying constants as a function of drying air temperature provided the best fit to the data.

Jain and Pathare (2004) studied the infrared radiative and convective drying of onion slices at different operating parameters of drying using mathematical models. Experiments were conducted using three levels of infrared radiation intensity, 2-65, 3-53, 4-42 W /cm<sup>2</sup>, air temperature, 35, 40, 45 °C and air velocity, 1.0, 1.25, 1.5 m/s. Nine mathematical model (Newton, Page, modified Page, Henderson and Pabis, asymptotic, logistic, two-term exponential geometric, and Wang and Singh) describing thin layer drying have been investigated. The results were compared to their goodness of fit in terms of coefficient of correlation, standard error and the mean square of the deviation  $\chi^2$ . The asymptotic regression

model could satisfactorily describe the infrared radiative and convective drying of onion slices with values for the coefficient of correlation of 0.99995, standard error of 0.0064 and  $\chi^2$  of  $4.4 \times 10^{-5}$ .

Mandala *et al.* (2005) studied the air drying of osmotically dehydrated apple. The experiment data were fitted successfully using the Page model:  $MR = \exp(-kt^n)$ .

Vega-Galveg *et al.* (2007) studied hot-air drying of aloe vera (*Aloe barbadensis* Miller) at 50, 60, 70, 80 and 90 °C with an air flow of  $2.0 \pm 0.2$  m/s. The mathematical models evaluated in the kinetic research include five empirical equations (Newton, Henderson-Pabis, Page, modified Page and Fick's diffusional model). When comparing the experimental moisture values with those estimated by the proposed models, the modified Page model provided the best fit of the data.

Kingsly and Singh (2007) performed thin layer drying of pomegranate arils in a cabinet drier at drying temperature of 50, 55 and 60 °C. Drying curves obtained from the data are fitted with different mathematical models for evaluating a suitable thin layer drying model. The Page model satisfactorily represented the drying characteristics of pomegranate arils than other models.

## **2.12 Quality Evaluation of Dehydrated Product**

Consumption of food is directly related to the quality. Important attributes identified with food are colour, rehydration ratio and sensory evaluation. Small and medium sized processing businesses all over the world increasingly have to consider the production of good quality products. Quality commonly thought of as a degree of excellence, is one of the major positioning tool of the producer for marketability and for consumers satisfaction. It is the combination of attributes or characteristics of a product that have significance in determining the degree of acceptability of the product to the user. To retain its quality and nutritive value, it is essential to ensure the integrity and safety of food throughout the food chain. Any measure taken to ensure all above will also add to its value as a commodity and create better demand, both at domestic and international level. Some reviews related to quality evaluations of dehydrated product are already covered in section 2.1, 2.5 and 2.10 and some are presented hereunder.

Mehta *et al.* (1982) reported that the pineapple rings blanched in 50 °Brix sugar syrup, cooled to room temperature and allowed to equilibrate gave the best reconstitution. Dehydrated pineapple rings were dipped in 1:4 ratio of pineapple rings to water, initially at 100 °C, for four hours. Reconstituted rings of this treatment were significantly superior in colour, flavour, texture and taste and compared well with the canned pineapples rings.

Howard *et al.* (1996) observed that lightness of carrot was also affected by processing temperatures with higher temperatures causing darker colour. A similar observation was also reported by Abbatemarco and Ramaswamy (1994). Also, the air dried and microwave vacuum dried carrots would appear darker since the density of these slices is greater than freeze dried product yielding a higher concentration of pigment per volume of tissue. The yellow and red colour of carrot slices is attributed to the presence of carotenes (Wagner and Warthesen, 1995).

Bongirwar (1997) reported that rehydration of osmotically dried fruit is slightly less than conventional dried product. Its reconstitution was about 70 per cent because part of the solid is coated of sugar. Osmo-air dried products were found to be soft in texture and sweet in taste with good retention of flavour. After rehydration in dilute sugar solution (12 °Brix) at room temperature by over night soaking, the pineapple slices were found to give fairly equivalent characteristics similar to canned pineapples slices (Nanjundaswamy *et al.*, 1978).

Lin *et al.* (1998) observed that the air dried carrot slices were darker, with less red and yellow than the freeze dried and microwave vacuum dried samples. They concluded that during the microwave vacuum drying the high internal vapour pressure produced by microwave heating and the low chamber pressure provided by vacuum caused the structure of carrot slices to expand and puff. Due to this puffing effect a less dense structure had higher capacity to absorb water when reconstituted. Also concluded that the darker appearance of the air dried and microwave vacuum dried carrot slices compared to the freeze dried samples may be due to the exposure to heat during drying.

Durance and Wang (2002) also reported that microwave-vacuum dehydration causes puffing of the tomato tissue such that the dry product was less than half as dense as air dried tomatoes and rehydrated more quickly and more completely than the air dried product.

Sacilik (2007) studied quality of dried tomato, dried at 50-70 °C air temperature with constant air velocity 0.8 m/s on the basis of rehydration ratio and colour evaluation. He reported that air temperature had significant effect on the colour and rehydration ratio of dried samples, also 50 °C air temperature had lowest values of lightness (L). He also reported that the rehydration ratio was increased with increase in air temperature.

Velic *et al.* (2007) studied the drying characteristics of “Granny Smith” apples dried using a tunnel drier at different temperatures and different pre-treatments. The effect of the temperatures and pre-treatments on the quality of dried apple samples was determined on the basis of colour, volume changes and reconstitution characteristics. They reported that the non-treated apple samples have better rehydration properties and less colour changes were achieved at the drying temperature of 60 °C at airflow velocity of 2.8 m/s. Blanching in hot

water, steam blanching and blanching in hot 0.6 per cent CaCl<sub>2</sub> solution resulted in higher values of rehydration ratio in comparison to other tested pre-drying methods.

Vega-Galveg *et al.* (2008) dried aloe vera (*Aloe barbadensis* Miller) slabs at 60 °C and further rehydrated at three temperatures (20, 40 and 60 °C) in order to study the influence of temperature on the kinetics of mass transfer during rehydration phenomenon. Fick, Peleg and Weibull models were applied for rehydration modelling, as well as one new proposed model. All kinetic parameters of the models showed positive dependence on temperature, according to the Arrhenius-type equation Weibull model and the new proposed model provided the best fit quality for each rehydration curve based on the statistical tests RMS, SSE and Chi-square. In accordance with the results, both models may be used to estimate the rehydration time of aloe vera. Furthermore, two rehydration indices (RR and WHC) were analysed which decreased as temperature increased. These indices confirm that drying process modifies the cell structure of aloe vera, reducing the rehydration ability due to cellular and structural disruption that takes place during dehydration.

Miranda *et al.* (2009b) dried Aloe vera (*Aloe barbadensis* Miller) gel at five inlet temperatures 50, 60, 70, 80 and 90 °C, in a convective dryer with a constant air flow of 2.0±0.2 m/s. Rehydration ratio, water holding capacity, texture, microstructure and total polysaccharide content were evaluated. Drying kinetics was estimated using the Weibull distribution ( $r^2 > 0.97$  and Chi-square  $< 0.0009$ ). These effects were more evident for the long drying period (e.g. 810 min at 50 °C). However, minor alterations in the structural properties and total polysaccharide content were produced at drying temperatures of 60-70 °C, resulting in a high quality gel.

Mota *et al.* (2009) studied the drying of onions in terms of drying kinetics, which was evaluated at 30, 50 and 60 °C. The experimental data was fitted to different empirical kinetic models from the literature and this kinetic study was then complemented with the modeling for estimation of the diffusion coefficients. On the other hand, the chemical characterization in fresh and dried onions at different temperature (varying from 30 to 70 °C) was analysed, to evaluate the effect of drying and drying temperature on the chemical composition of the product. In this way, the analyses of moisture content, sugar content, crude protein, ash, fat, crude fibre, acidity and vitamin C were made and reported in this paper. From the results obtained it was verified that some chemical components of the onions are not affected by drying (ash, fat, protein and fibre) whereas some others are considerably influenced by drying (sugars, acidity and vitamin C).

From the overall discussion on the osmotic dehydration and the effect of various operating parameters on the osmotic dehydration it is very clear that with proper precautions,

like selection of material and process conditions, osmosis is one of the most promising and economical methods of partial moisture removal prior to any other method of preservation. It not only removes moisture, but it also inactivates enzymes and enhances the final product quality. Although a large number of studies have been conducted, the process still needs to be studied further so as to characterize the process for all the new varieties which are specifically developed for processing.

The literature surveyed in the preceding paragraphs revealed that a limited research work has been carried out on osmotic dehydration of aloe vera. Thus, there is a need for in depth investigation on all aspects of osmotic dehydration as well as convective drying of osmotically dehydrated aloe vera and optimization of process parameters.

## CHAPTER III

### MATERIALS AND METHODS

This chapter deals with the experimental set-up, methods and techniques followed in the production of osmo-convectively dehydrated aloe vera product. The experiments were divided into two parts, viz., osmotic dehydration of aloe vera (gel) cubes in sugar syrup and convective drying of osmotically dehydrated product. The preliminary experiments were planned for fixing the levels of input variables such as concentration and temperature of sugar solution and duration of osmosis. The kinetics of osmotic dehydration and convective drying was also studied. The procedure for optimisation of process parameters for osmotic dehydration followed by convective drying is also discussed in this chapter. The methodology is also discussed for determining the quality of the osmo-convectively dehydrated product.

#### 3.1 Selection of Raw Materials

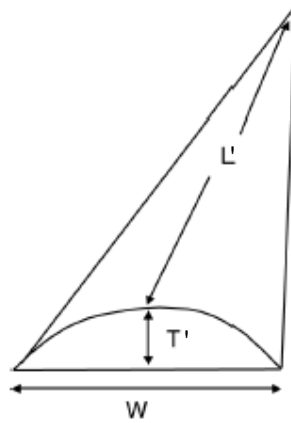
A widely grown '*Aloe barbadensis* Miller' variety of aloe vera was selected for all the experiments. Fresh aloe vera leaves were obtained from Banki Research Farm, Sisarama, Department of Forest, Udaipur (Rajasthan). The two years old and matured aloe vera leaves were selected for the study. The leaves were cut in the early morning every day for experimentation. To avoid bio-degradation of aloe vera leaves, each leaf was cut manually with knife and pulled carefully from the mother plant so as not to break the rind. The leaves were transported to the working place in a covered polyethylene bag to avoid oxidation or contamination. The commercial sugar, being cheap and easily available, was considered as an osmotic agent. A number of laboratory grade chemicals were also used for various analyses described later in this chapter.

#### 3.2 Some Physical Properties of Aloe Vera

##### 3.2.1 Leaf size and shape

The maximum tri-axial dimensions length (L'), width (W) and thickness (T') of aloe vera leaves (Fig. 3.1), were measured in 22 randomly selected leaves (Chandegara and Varshney, 2007) using metallic tape having a least count of 1 mm. Each tri-axial dimension were replicated thrice and reported. The shape was determined by comparing the

longitudinal and lateral cross-section of the aloe vera leaves .



**Fig. 3.1 Diagram of the approximate geometry of aloe vera leaf**

### 3.2.2 Apparent leaf volume

The apparent volume was calculated by considering the geometry of the object similar to the geometrical shape. Knowing the values of length, width and thickness, the volume of the aloe vera leaf was calculated, by considering the geometry as to be a cone with elliptical rather than circular cross section as shown in Figure 1. The volume was calculated by the formula given by Hernandez *et al.*, (2002) as below:

$$V' = (L'/12) \pi W T' \quad \dots 3.1$$

Where,

$V'$  = Volume of the leaf,  $\text{mm}^3$

$L'$  = Length of the leaf, mm

$W$  = Width of the leaf, mm

$T'$  = Thickness of the leaf, mm

### 3.2.3 Leaf weight

For determination of leaf weight, 22 nos. of randomly selected leaves were weighed in a precision balance (Sartorius make, 0.01 g least count) and their weights were recorded.

The relationship between leaf weight and leaf volume, leaf weight and gel recovery as well as leaf volume and gel recovery was established.

### 3.2.4 Moisture content

Moisture content of fresh as well as osmotically dehydrated papaya pieces was determined using method suggested by Ranganna (2000). A brief description of the method is as follows:

1. A thin layer of finely divided asbestos (Gooch grade) powder was spread into a flat bottom moisture box and dried at 110 °C for 1 hour, cooled and weighed.
2. A small sample of aloe vera was kept in a pre-dried and weighed moisture box. The mass of the sample was recorded as  $W_1$ .
3. The box was placed in oven and temperature was maintained at 100 °C for 18 h (Chaudhari *et al.*, 2000).
4. After drying the sample was cooled in a desiccator to a room temperature and then weighed. The mass of the dried sample was recorded as  $W_2$ .

The moisture content of the sample was calculated using following equation:

$$MC (\% db) = \frac{W_1 - W_2}{W_2} \times 100 \quad \dots 3.2$$

Where,  $W_1$  = mass of original sample, g  
 $W_2$  = mass of sample after drying, g

### 3.2.5 Total soluble solids

The total soluble solids of prepared syrup was found out using hand refractometers of various ranges (0-32, 28-62 and 58-92°Brix), which gave the reading directly in Brix (Ranganna, 2000).

### 3.2.6 Density

Density of prepared aloe vera gel sample was determined using Archimedes principle with n-heptane (Yan *et al.*, 2008). Sugar syrups of desired concentrations were prepared by blending the required amount of sugar with tap water. Density of the syrup was determined by filling it into a tared specific gravity bottle of known volume (25 ml) and taking its weight. Density was calculated as the ratio of mass of the syrup and volume (25 ml) and expressed in  $\text{kg/m}^3$  (Lenart and Flink, 1984a).

## 3.3 Development of Aloe Vera Peeler

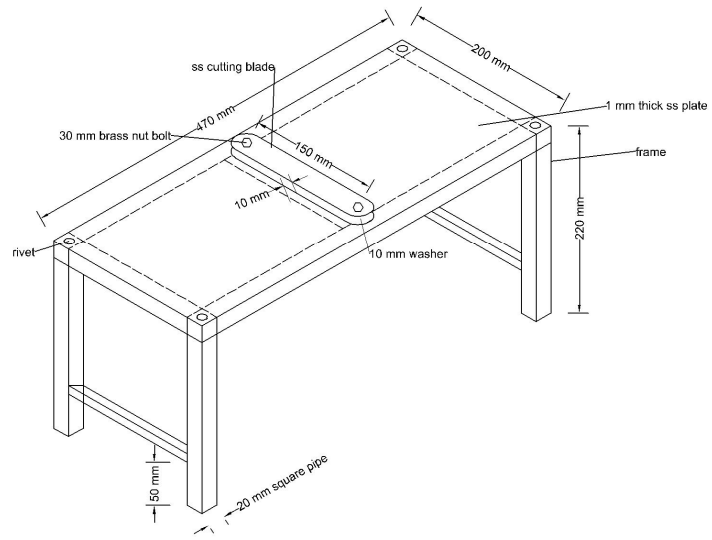
Preparation of aloe vera sample (10x10x10 mm aloe vera gel cubes) was bit difficult particularly with respect to removal of epidermis (skin) from aloe vera slabs. All other operations during preparation of sample such as removing base of leaf (20 to 30 mm), tips

(less than 14 mm thick) , margins (with spikes ), cutting 10 mm with slabs (with epidermis), were possible manually with sharp stainless steel cutter fast and safe but it was very difficult to remove the epidermis from rest two sides of slabs manually with knife, as the hand could not be held firm for each cut and straight cut was not coming proper and also it was taking too much time. Moreover there was possibility to cut the finger and cause drudgery while doing this operation.

Therefore in order to overcome all this, efforts were made to develop aloe vera peeler as discussed below for removal of epidermis from both sides of slabs (with epidermis) of aloe vera leaf.

### **3.3.1 Construction of aloe vera peeler**

A mild steel frame 470 (length), 200 (width) and 220 mm (height) was fabricated with 20 mm square M.S. pipe. On this frame, stainless steel sheet (1 mm thick) was riveted at 6 points as shown in Fig.3.2. A cutting blade (1.25 mm thick) made up of surgical stainless steel with more than 12 per cent chromium and 0.3 per cent carbon, which is required composition for maintaining maximum corrosion resistance and hardness level of the best quality knives, was used for removing the epidermis of the aloe vera leaves. A specific serrated cutting edge which gives a long lasting cutting performance and never needs sharpening was made on cutting blade by grinding with laser technique. Two separate cutting blades were mounted on the cutting platform with nuts and bolts of brass enabling it to dismantle easily for packaging and transportation of this machine. Considering the thickness of the epidermis of aloe vera leaves, the lower blade was mounted 3 mm above the platform. In order to obtain 10 mm thick gel slice upper surgical blade was mounted over the lower blade by placing two 10 mm thick brass washers. However, the spacing between the blades can be varied by varying thickness of the washers (Plate 3.1).



**Fig. 3.2 Schematic diagram of aloe vera peeler**



**Plate 3.1 Aloe vera peeler**

### **3.4 Sample and Solution Preparation**

The aloe vera leaves were thoroughly washed under tap water to remove adhering impurities. The tips of the leaves (not useful for getting 10 mm thickness gel) were removed. Aloin (a yellow colour liquid) which is laxative or purgative if consumed in large amount was extracted by cutting the base of the leaves and allowing them to drain vertically for 1 hour. The spikes, along their margins, were removed before slicing the leaf. The tips and base of leaves not useful for sample preparation were also removed. The leaves were then cut along the length into 10 mm slabs with epidermis. These slabs were then passed through the aloe vera peeler developed for removing epidermis (or skin) from gel (pulp) keeping the required thickness of gel (10 mm). The 10 x 10 mm gel slabs were then cut into 10 x 10 x 10 mm  $\pm$  1 cubes (Miranda *et al.*, 2009) with the help of sharp stainless steel cutter. The cubes were washed extensively with distilled water to remove the exudates from their surfaces. Sugar syrups of various concentrations were prepared by dissolving required amount of sugar in distilled water.

### **3.5 Osmotic Dehydration of Aloe Vera Samples**

In the process of osmotic dehydration, a sample placed in the hypertonic solution and due to concentration difference water comes out from sample to solution. Simultaneously

transport of solids takes place from solution to sample and vice versa. The mass transport in terms of water loss, mass reduction and solid gain were studied as detailed below.

### 3.5.1 Mass Transport Data for Osmotic Dehydration

Lenart and Flink (1984b) were first to define terminologies, for mass transport data during osmotic concentration and same have been used by various researchers such as Shi *et al.*, (1995); Silveira *et al.*, (1996) and Kaleemullah *et al.*, (2002) and in this study also as follows:

#### 3.5.1.1 Water loss (WL)

Water loss was calculated as the net loss of water from food material on an initial mass basis as

$$WL = \frac{W_{si} X_{swi} - W_{s\theta} X_{sw\theta}}{W_{si}} \times 100 \quad \dots 3.3$$

#### 3.5.1.2 Mass reduction (WR)

Mass reduction was calculated as the net mass reduction of the food material on initial mass basis as

$$WR = \frac{W_{si} - W_{s\theta}}{W_{si}} \times 100 \quad \dots 3.4$$

#### 3.5.1.3 Solid gain (SG)

Solid gain was calculated as a net uptake of solids by food material on an initial mass basis as

$$SG = \frac{W_{s\theta}(1 - X_{sw\theta}) - W_{si}(1 - X_{swi})}{W_{si}} \times 100 \quad \dots 3.5$$

From Eqns (3.3) and (3.4), the solid gain (SG) can be correlated with mass reduction (MR) and water loss (WL) as

$$SG = WL - WR \quad \dots 3.6$$

Where,

WL = water loss (g water per 100 g initial mass of sample)

WR = mass reduction (g mass per 100 g initial mass of sample)

SG = solid gain (g solids per 100g initial mass of sample)

$W_{si}$  = initial mass of sample, g

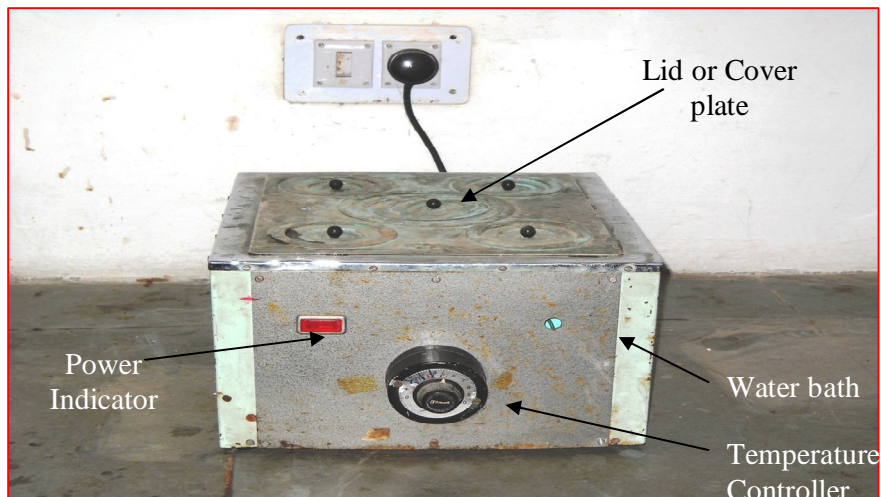
$W_{s\theta}$  = mass of the sample after time  $\theta$ , g

$X_{swi}$  = water content as a fraction of the initial mass of the sample

$X_{sw\theta}$  = water content as a fraction of the syrup at time  $\theta$

### 3.5.2 Experimental set up

A small capacity laboratory temperature controlled water bath of size 35 cm x 30 cm x 25 cm (approximate capacity, 5 litres) was used as osmotic dehydration unit. Temperature controller was used to regulate the required temperature in the investigation (Plate 3.2).



**Plate 3.2 Temperature controller water bath**

### 3.5.3 Preliminary experiment for fixing the range (levels) of input parameters

In order to decide range of input variables for further experimentation (kinetics and optimization of osmotic dehydration) preliminary experiments were performed. After the preparatory steps aloe vera gel was cut into pieces of approximately 1cm size. The cut pieces resembled like a cube, but they have been referred as samples throughout the thesis. Though smaller sample size would normally accelerate the process of osmo-dehydration but deshaping of aloe vera samples of less than 10 mm size was observed in air drying during preliminary experiments. Hence, aloe vera cubes of 10 mm size was used in all the experiments. The investigation was performed with the following fixed parameters.

- Equipment : Temperature controlled water bath
- Aloe vera gel size : 10 mm cube
- Sample size, g : 50
- Sample to sugar syrup ratio : 1:5

In general, following maximum levels were adopted for the four parameters, temperature as 50 °C; concentration as 70°Brix; ratio of sugar syrup to aloe vera as 5:1 and duration of osmosis as 6 h, as suggested by earlier workers (Ponting *et al.*, 1966; Islam and Flink, 1982; Rahman and Lamb, 1990; Jain, 2007 and Pisalkar 2008) for various fruits/vegetables. Keeping ratio of sugar syrup to aloe vera constant at 5:1 (Simal *et al.*, 1997; Kar and Gupta 2001; Pokharkar and Prasad 2002; Azoubel and Murr 2004 and Pisalkar,

2008), three sets of experiments were conducted by varying one factor at a time (Table 3.1) as reported by earlier workers (Pokharkar, 1994; Jain 2007). The response parameters were water loss and sugar gain.

**Table 3.1 Variables and their levels for preliminary experiment**

Variables	Experimental runs		
	A	B	C
Sugar syrup temperature, °C	50	50	30, 40, 50, 60 and 70
Sugar syrup concentration, °Brix	70	10, 20, 30, 40, 50, 60, 70 and 80	70
Duration of osmosis, h	1, 2, 4, 6, 8 and 10	6	6

In every trial aloe vera samples of approximately 50 g were completely immersed in 250 g sugar syrup contained in a 500 ml glass beaker. The beakers were placed inside the constant temperature water bath. The syrup in the beakers was manually stirred at regular intervals to maintain uniform temperature. The beaker was removed from the water bath at pre-decided time (experimental run A of Table 3.1) and the samples were immediately removed and placed on tissue paper to soak the surface moisture. The samples were weighed and their moisture contents were determined.

The overall mass transport data namely, mass reduction, water loss, sugar gain and soluble solid concentration were used to indicate the overall exchange of solute and water between aloe vera samples and sugar syrup as explained earlier. The same procedure was adopted for other experiments (experimental run B and C of Table 3.1) by varying one factor at a time. The observations of the preliminary experiment have been presented and discussed in section 5.3.

From section 5.3, it was observed that by increasing the value of input variables, the response (water loss and sugar gain) was increasing, but the rate of increase was found to be decreased at later stage. The upper level of the input variables (sugar syrup temperature, sugar syrup concentration and duration of osmosis) was decided at that point from which there was negligible change (increase) in response (water loss and sugar gain). The lower and middle level of the input variables was chosen logically on the similar lines as described by earlier researchers (Pokharkar, 1994; Jain, 2007).

The results of these tests, as discussed later in section 5.2 revealed that the levels of the process variables should be:

Process temperature, °C (T)	: 30, 40 and 50
Sugar syrup concentration, °Brix (C)	: 10, 25 and 40
Osmosis time, min ( $\theta$ )	: 60, 150 and 240

### 3.5.4 Product quality

Two responses considered for optimization of osmotic dehydration were water loss and sugar gain. In an osmotic dehydration process, the higher the water loss the better is the dehydration process. However, high solid gain affects the final product quality and sensory characteristics. When high levels of solids are incorporated into the fruit during

the osmotic dehydration, significant sensory alterations can occur and the final product may present a taste that is very different from the overall acceptability.

Thus, considering the importance of the 'sugar gain' in product quality, acceptability and marketability of osmo-convectively dried product, this factor was used as targeted constraint for optimization of the input parameters of osmotic dehydration. For this purpose, it was necessary to fix the level of sugar gain in the final product, so that it is acceptable by the consumers. For this purpose, the experimental run as given in Table 3.1 column B, followed by convective drying was used to evaluate the product quality by sensory evaluation with the following fixed parameters

- Aloe vera gel size : 10 mm cube
- Sample size, g : 250
- Sugar syrup to sample ratio : 5:1
- Sugar syrup temperature, °C : 50
- Duration of osmosis, h : 6
- Equipment : Temperature controlled water bath and tray dryer

#### **Variable**

- Sugar syrup concentration, °Brix: 10, 20, 30, 40, 50, 60, 70 and 80

A sample of 250 g aloe vera cubes each was immersed into a 10, 20, 30, 40, 50, 60, 70 and 80 °Brix sugar syrup with 5:1 sugar syrup to sample ratio, contained each in a 2000 ml glass beaker and maintained at 50 °C. The samples were removed from the solutions after 6 hour of osmosis and blotted. The loss or gain in weight of sample was recorded. Moisture content was measured to calculate the sugar gain and the sample was air dried in a tray dryer at 70 °C with drying air velocity 2 m/s (Miranda *et al.*, 2009a) to reduce the moisture content to below 15 per cent (db). The quality of the osmo-convectively dried aloe vera was evaluated by test panel of ten judges. Evaluation card as per BIS - 1971 (Appendix D) was used. A nine point hedonic scale was employed for the sensory attributes evaluated where 9 denoted "like extremely" and 1 indicated "dislike extremely". The data on the sensory attributes like overall acceptability (w.r.t. sugar gain) was analysed by analysis of variance (ANOVA).

#### **3.5.5 Optimization of input parameters for osmotic dehydration**

The variables of osmotic dehydration process were optimized using RSM technique, since this is useful statistical technique for investigation of complex processes. RSM is a collection of certain statistical techniques for designing experiments, building models, evaluating the effects of the factors and searching for optimal conditions for desirable responses (Cochran and Cox 1957; Box *et al.*, 1978 and Myer and Montgomery 2002). The response surface analysis involves fitting the experimental values of water losses/solute gain

to a general quadratic polynomial equation and subsequently optimizing the values with suitable optimization software or mathematical solutions. The studies of optimization have been carried out by various research workers (Pokharkar, 1994; Vijayanand *et al.*, 1995; Chaudhari *et al.*, 2000; Kar and Gupta, 2001; Liyana-Pathirana and Shahidi, 2005; Eren and Kaymak-Ertekin, 2007; Jain, 2007; Altan *et al.*, 2008; Corzo *et al.*, 2008; Mestdagh *et al.*, 2008; Shi *et al.*, 2008 etc.).

The process parameters (sugar syrup concentration, syrup temperature and duration of osmosis) were optimized for maximum of water loss and optimum (targeted) sugar gain.

### 3.5.5.1 Design of experiments

The method of response surfaces deals with the problem of seeking the conditions of an experiment, which are optimal, *i.e.*, most desirable. The techniques applied here are standard techniques, which have been described in greater detail by Myers (1971). The Box-Behnken design of three variables and three levels including 17 trials formed by 5 central points was used (Box and Behnken, 1960). This design was selected as it fulfils most of the requirements needed for optimization of the osmotic dehydration process prior to convective drying. The osmotic dehydration was assumed to be affected by three independent variables (regressor or factors),  $r_i$ , *viz.*, sugar syrup temperature (T), sugar syrup concentration (C) and duration of osmosis ( $\theta$ ). The experimental design of independent parameters and layout are shown in Tables 3.2 and 3.3 for these three levels and three variables under the Response Surface Methodology (Mullen and Ennis, 1979; Floros and Chinnan, 1987; Mudhar *et al.*, 1989; Pokharkar 1994; Saba 1994; Jain 2007; Ranmode 2009). All these variables were closely controlled and accurately measured during experimentation. The dependent variables, also referred as responses,  $Y_k$  (*i.e.*, the percentage of water loss and sugar gain) were measured experimentally.

The independent variables ( $r_i$ ), the coded variables ( $x_i$ ), uncoded variables and their coded and uncoded levels are shown in Table 3.2. It is assumed that the mathematical function  $f_k$  ( $k = 1, 2, 3, \dots, n$ ), exists for each response variable,  $Y_k$  in terms of the processing factors,  $r_i$  ( $i = 1, 2, 3, \dots, m$ ), such as

$$Y_k = f_k(r_1, r_2, r_3, \dots, r_m) \quad \dots 3.7$$

#### Fixed parameters

- Aloe vera gel size : 10 mm cube
- Sample size, g : 250
- Sample to sugar syrup ratio : 1:5
- Equipment : Temperature controller water bath

**Table 3.2 Levels of independent variables for osmotic dehydration of aloe vera samples**

Independent variables	Symbols		Levels	
	Coded	Un-coded	Coded	Un-coded
Sugar syrup temperature (°C)	x <sub>1</sub>	T	1	50
			0	40
			-1	30
Sugar syrup concentration (°Brix)	x <sub>2</sub>	C	1	40
			0	25
			-1	10
Duration of osmosis (min)	x <sub>3</sub>	θ	1	240
			0	150
			-1	60

**Dependent variables**

- Water loss, %
- Sugar gain, %

**Design : Box Behnken**

**Analysis : Response Surface Methodology**

The exact mathematical representation of the function (*f*) is either unknown or extremely complex. However, second order polynomial equation of the following form was assumed to relate the response, *Y<sub>k</sub>* and the factors, *r<sub>i</sub>* as

$$Y_k = \beta_{ko} + \sum_{i=1}^{i=3} \beta_{ki} x_i + \sum_{i=1}^{i=3} \beta_{kii} x_i^2 + \sum_{i=1}^{i=2} \sum_{j=i+1}^{j=3} \beta_{kij} x_i x_j \quad \dots 3.8$$

Where, *Y<sub>k</sub>* is response (i. e. water loss or sugar gain)  $\beta_{ko}$ ,  $\beta_{ki}$ ,  $\beta_{kii}$  and  $\beta_{kij}$  are constant coefficients and *x<sub>i</sub>* and *x<sub>j</sub>* are the coded independent variables that are linearly related to T, C and θ. In practice, the levels of the independent variables change from one application to another. Therefore, the general designs are given in terms of standardized coded variables (*x<sub>i</sub>*), which in any particular application are linearly related to *r<sub>i</sub>* by the following equation:

$$x_i = \frac{(r_i - \bar{r}_i)}{d_i} \quad \dots 3.9$$

Where, *r<sub>i</sub>* = actual value in original units,

$\bar{r}_i$  = mean of high and low levels of *r<sub>i</sub>*

*d<sub>i</sub>* = spacing between the low and high levels of *r<sub>i</sub>*.

In present study, *n* = 2 and *m* = 3 and hence Eqn. (3.7) can be written as

$$Y_k = f_k(T, C, \theta) \quad \dots 3.10$$

Where, T = sugar syrup temperature, °C, C = sugar syrup concentration, °Brix,  $\theta$  = osmotic dehydration duration,  $Y_k$  = water loss or sugar gain, percent, in osmotic dehydration. Hence, Eqn. (3.8) takes the following form as

$$Y_k = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad \dots 3.11$$

Response Surface Methodology (RSM) was applied to the experimental data using the package, Design- Expert version 8.0.4.1 (Statease Inc, Minneapolis, USA, Trial version, 2002).

### 3.5.5.2 Experimental procedure

Seventeen trials were performed as enumerated in Table 3.3 for obtaining the water loss and sugar gain as responses for each treatment. The procedure adopted for this was the same as described earlier in this chapter. The peeled and pre-processed aloe vera samples of 1cm size were fully immersed in the sugar syrup kept in a beaker having sugar syrup to samples ratio of 5:1 (w/w). The beakers were placed inside constant temperature water bath. The samples were removed from the bath at the designated time, and blotted. The samples were weighed and moisture content was determined. The water loss and sugar gain were also calculated. To avoid bias, 17 runs were performed in a random order for estimation of the constants of Eqn. (3.11). The decision for the range and centre points of the variables was taken through preliminary trials.

### 3.5.5.3 Numerical optimization

Numerical optimization technique of the Design-Expert software was used for simultaneous optimization of the multiple responses. The desired goals for each factor and response were chosen. The goals may apply to either factors or responses. The possible goals are: maximize, minimize, target, within range, none (for responses only). All the independent factors (T, C and  $\theta$ ) were kept minimized from economical point of view while the responses viz. water loss was kept maximized and sugar gain was kept targeted.

Table 3.3 Experimental layout for 3 variables and 3 levels response surface analysis

Treatment no.	Syrup temperature, °C	Syrup concentration, °Brix	Duration of osmosis, min
	$x_1$	$x_2$	$x_3$
1	1	1	0
2	1	-1	0
3	-1	1	0
4	-1	-1	0
5	1	0	1
6	1	0	-1
7	-1	0	1
8	-1	0	-1
9	0	1	1
10	0	1	-1
11	0	-1	1
12	0	-1	-1
13	0	0	0
14	0	0	0
15	0	0	0
16	0	0	0
17	0	0	0

#### 3.5.5.4 Graphical optimization

Graphical optimization was also carried out for the process parameters for osmotic dehydration of aloe vera gel for obtaining the best product. For graphical optimization, super imposition of contour plots for all responses was done with respect to process variables using Design-Expert software. The superimposed contours of all responses for syrup temperature, syrup concentration and duration of osmosis and their intersection zone for maximum water loss and targeted sugar gain indicated the ranges of variables which could be considered as the optimum range for best product quality in terms of responses. The optimum combinations of product and process variables for osmotic dehydration of aloe vera gel were derived by averaging those ranges of variables.

#### 3.6 Verification of Optimum Responses

The optimum responses were verified by conducting the osmotic dehydration experiment under optimum conditions. The responses such as water loss and sugar gain at optimum processing conditions were compared to the values predicted by the regression model.

### **3.7 Estimation of Water and Sugar Diffusivities During Osmotic Dehydration of Aloe Vera**

In order to study the diffusivity of water and sugar, a sample of 50g was immersed in sugar syrup of 10 °Brix in 1:5 ratio in nine 500 ml beakers and held at constant temperature 30 °C in a water bath. Sample beakers were removed from water bath after 10 min and thereafter every 30 min interval up to 240 min. Samples were taken out and placed on absorbent paper for 5 min to eliminate excess syrup from the surface before weighing. The same process was repeated for 25 and 40 °Brix sugar syrup and 40 and 50 °C temperature. All samples were analyzed for moisture content. Water and sugar diffusivities were calculated by using experimental data as discussed in Section 4.3. The observations and other details are furnished in Data Sheet 1-9 of Appendix-E. The data were also utilized for determining activation energy.

### **3.8 Convective Drying of Osmotically Dehydrated Aloe Vera Samples**

Osmotically dehydrated product, generally, may not have moisture content low enough to be considered as shelf stable. It is therefore, needed it to be further air dried to obtain a shelf stable product *i.e.* stable with respect to prevention of microbial growth and enzymatic colour changes (Islam and Flink, 1982; Kim and Toledo, 1987).

Hence, the product obtained from the optimized levels of the osmotic dehydration was then air-dried in conventional tray drier as detailed below.

#### **3.8.1 Experimental Set-up**

A laboratory model convective tray dryer (Plate 3.3) was used in the dehydration experiment in this study. The technical specifications of the dryer are presented in Appendix A. The dryer comprised of a drying chamber, heating unit, and a fan. The details of the dryer are described hereunder:

**a) Drying chamber:** It comprised of an insulated box with a single door opening at front. An adjustable speed axial flow fan for regulation of air velocity is provided on left side of drying chamber. A heating unit for increasing the temperature of air is also provided. The air enters the dryer due to suction and transfers heat to the wet product for drying and simultaneously, absorbs moisture from it. The moisture laden air leaves the dryer by another opening of drying chamber. The sample trays having size of 340 x 270 mm made of stainless steel were used in the dryer. The bottom of these trays comprises of SS wire mesh which permitted good flow of drying air through the product. The trays were stacked one over the other in a single column. The size of sample tray permitted enough space to spread 100 g of aloe vera sample uniformly in a single layer. The aloe vera samples were uniformly spread over the tray, thus



**Plate 3.3 Experimental convective tray dryer**

resulted into loading density of  $1.089 \text{ kg/m}^2$ . The dryer is fitted a continuous weighing scale of 0.01 g least count. The sample trays were hanged with the weighing scale to continuously weigh the sample at a selected time interval without interrupting the system.

**b) Heating unit:** It consisted of an electric heater of 2.5 kW placed on inner right wall of drying chamber. A thermostatic controller was used on the heating unit for controlling the temperature of the drying air inside the dryer. The maximum temperature of the drying air which could be attained at highest air velocity of 1.5 m/s was  $92 \text{ }^\circ\text{C}$  (at no load).

**d) Fan:** An axial flow fan provided at inner left side of drying chamber sucks the filtered atmospheric air inside and then through heating unit; forcing it to pass through the wet product placed in trays. This caused quick and effective drying of product. The fan was operated by a single phase, 50 Hz, 0.375 kW AC motor. The drying air velocity was changed with a fan speed regulator provided at the control panel.

The temperature of the drying chamber was pre-set through a temperature indicator cum controller provided on control panel of the dryer. It controlled and maintained the temperature of air by controlling the supply to the heating unit.

### **3.8.2 Convective drying experiment**

The air drying process is influenced by drying air temperature, air velocity, relative humidity, time of drying, loading density, *etc.* Among these parameters listed above, the drying air temperature (T) and air velocity (V) were used in this study. The drying

characteristics of the osmotically dehydrated aloe vera samples were studied. The osmotically dehydrated samples were dried in the above stated laboratory tray dryer at 50, 60, 70 and 80°C drying air temperatures and at three air velocities (1, 2 and 3 m/s). Air velocity was measured with the help of a digital anemometer, while the correct drying air temperature was adjusted by temperature indicator cum controlling unit and conditions were allowed to stabilize at desired temperature. The temperature and relative humidity of the ambient air were measured by a hygrometer.

The osmotically dehydrated aloe vera samples were loaded on the drying trays and inserted into the dryer. Drying data were recorded at 5 minute interval for first half an hour, at 10 minute intervals for next half hour and at 30 minute intervals until completion of experiment. These data are given in Appendix F. The data were used to analyze the moisture movement characteristics viz moisture ratio, drying rate, moisture diffusivity etc.

The moisture ratio (MR) at each moisture content level was determined by the following equation:

$$MR = \frac{M - M_{\infty}}{M_o - M_{\infty}} \quad \dots 3.12$$

Where,

- MR - Moisture ratio
- M - Moisture content at any time (d.b.)
- M<sub>o</sub> - Initial moisture content (d.b.)
- M<sub>∞</sub> - Equilibrium moisture content (d.b.)

Data on equilibrium moisture content (EMC) of air dried aloe vera gel samples with osmotic pretreatment in an air temperature range of 50 to 80 °C and air velocity range of 1 to 3 m/s are not available in the literature. Therefore a separate set of experiments on convective drying was conducted to determine the EMC values by a dynamic method (Kashaninejad *et al.*, 2007). The EMC values were determined by drying osmotically dehydrated aloe vera gel samples in the convective dryer at all temperature and velocity levels as mentioned above until the mass loss of the sample ceased. The final moisture content values were calculated from this constant weight and used as EMC values for calculation of the moisture ratio. The drying data were also used to determine moisture diffusivity as described in section 4.4.

The drying data given in Appendix F were also used to determine activation energy. The activation energy in osmo-convective drying of aloe vera sample under various drying conditions has been estimated from the Arrhenius type of relationship.

$$D_{eff} = D_o \exp\left(\frac{-E_a}{RT_{abs}}\right) \quad \dots 3.14$$

- where,  $D_{\text{eff}}$  = Effective diffusion coefficient,  $\text{m}^2/\text{s}$   
 $D_0$  = Constant equivalent to effective diffusion coefficient at infinite temperature,  $\text{m}^2/\text{s}$   
 $E_a$  = Activation energy,  $\text{kJ/mol}$   
 $R$  = Universal gas constant,  $8.314 \text{ kJ/mol K}$   
 $T_{\text{abs}}$  = Absolute temperature,  $\text{K}$

### 3.8.3 Modeling of convective drying of aloe vera

To determine the most suitable drying equations, the experimental drying data were fitted in the various drying models (Table 3.4). Coefficient of determination ( $R^2$ ) was the main criteria for selecting the best equation. In addition to the coefficient of determination, the goodness of fit was determined by various statistical parameters such as reduced mean square of the deviation  $\chi^2$  and root mean square error  $E_{RMS}$ . For quality fit,  $R^2$  value should be higher close to one and  $\chi^2$  and  $E_{RMS}$  values should be lower (Pangavhane *et al.* 1999; Togrul and Pehlivan, 2002; Erketin *et al.* 2004; Demir *et al.* 2004). The above parameters were calculated as follows:

$$\chi^2 = \frac{\sum_{i=1}^N (M_{R,\text{exp},i} - M_{R,\text{pre},i})^2}{N - z} \quad \dots 3.15$$

$$E_{RMS} = \left[ \frac{1}{N} \sum_{i=1}^N (M_{R,\text{pre},i} - M_{R,\text{exp},i})^2 \right]^{1/2} \quad \dots 3.16$$

Where,  $M_{R,\text{exp},i}$  and  $M_{R,\text{pre},i}$  are the experimental and predicted dimensionless moisture ratios, respectively;  $N$  is the number of observations and  $z$  is the number of drying constants.

**Table 3.4 Mathematical models used under drying study**

Model equation	Name	Reference
$MR = \exp(-kt)$	Exponential	Liu and Bakker-Arkema (1997)
$MR = \exp(-kt^n)$	Page	Zhang and Litchfield (1991)
$MR = \exp(-kt)^n$	Modified page	Overhult <i>et al.</i> , (1973)
$MR = a \exp(-kt)$	Henderson and Pabis	Henderson and Pabis (1961)
$MR = a + b \ln(t)$	Logarithmic	Chandra and Singh (1995)
$MR = At^B$	Power law	Chandra and Singh (1995)

### 3.9 Quality Analysis

As quality is important in food processing, control should be exercised at every stage from pre-processing to packing, storing, *etc.* Quality of osmo-convectively dried aloe vera samples was evaluated on the basis of colour, hardness and ascorbic acid as well as water

activity. All measurements were replicated thrice and the average readings are reported. These three quality parameters viz. colour, ascorbic acid and hardness as well as water activity and drying time were further considered for the optimization of input parameters of convective drying.

### **3.9.1 Colour measurement**

Colour is one of the most important qualities of acceptance for products, reflects sensation to the human eye. Colour is important to consumer as a means of identification, as a method of judging quality and for its basic aesthetic value. Dried products are usually darker in colour, but darker colour does not mean better quality. Too dark may imply that the product is over dried. The advantage is that this parameter can be visually determined for assessing dryness quality. The colorimeter used in the present investigation is shown in Plate 3.4. The technical specifications are presented in Appendix B. Colour of the dried aloe vera was measured using a Hunter Lab Colorimeter (Model CFLX/DIFF, CFLX-45). A cylindrical glass sample cup (63.5 mm in diameter x 40 mm height) was placed at the light port (31.75 mm diameter). The instrument was initially calibrated with a black as well as with standard white plate supplied with the equipment. The 3-dimensional scale  $L^*$ ,  $a^*$  and  $b^*$  were used in a Hunter Lab Colorimeter. The  $L^*$  is the lightness coefficient, ranging from 0 (black) to 100 (white) on a vertical axis. The  $a^*$  is purple-red (positive  $a^*$  value) and blue-green (negative  $a^*$  value) on a horizontal axis. A second horizontal axis is  $b^*$ , that represent yellow (positive  $b^*$  value) or blue (negative  $b^*$  value) colour (Plate 3.5). Hunter L-value, which denotes the degree of whiteness, was chosen to represent the colour value of sample (Anantheswaran *et al.*, 1986).

### **3.9.2 Estimation of ascorbic acid**

In the present study, the fresh aloe vera gel and osmo-convectively dried aloe vera samples were chemically analyzed by volumetric method to determine and compare the retention of ascorbic acid (vitamin C). The volumetric method for determining the quantity of ascorbic acid in a sample is an easy and fast method. It is determined by the method as explained by Ranganna (2000).

Ascorbic acid reduces 2, 6-dichlorophenol indophenol dye to a colourless dye and itself gets oxidised to dehydro-ascorbic acid. The blue coloured compound dye changes to pink colour, in acid medium, at the end point. Oxalic acid is used as the medium for titration.

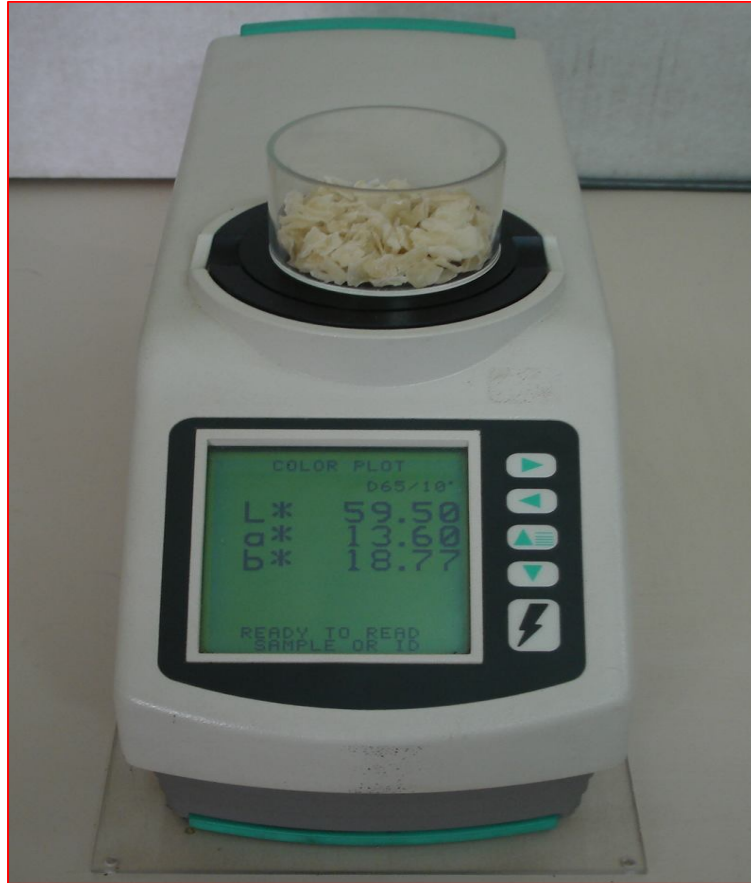


Plate 3.4 Hunter lab colorimeter

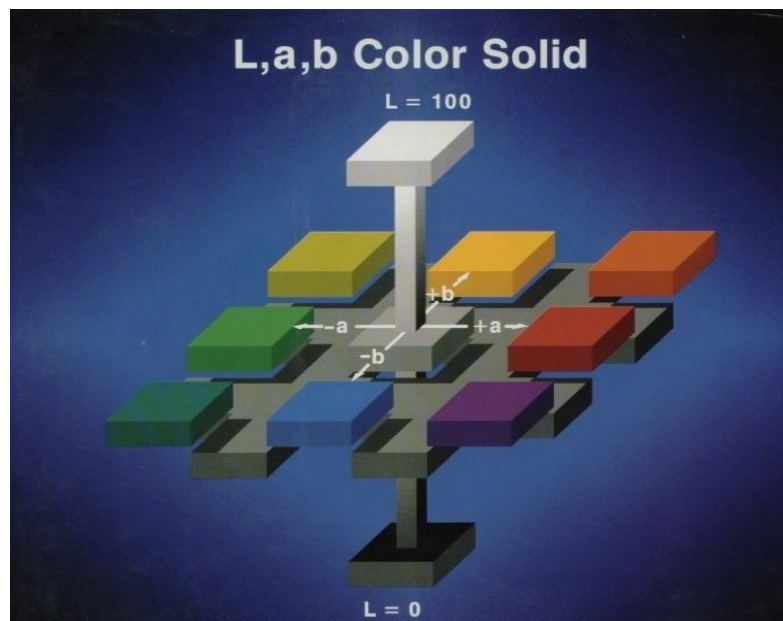


Plate 3.5 Colour Scale representing relationship of colour index ( $L^*$ ,  $a^*$ ,  $b^*$ )

5 ml of working standard solution was pipetted into a 100 ml conical flask. 10 ml of 4% oxalic acid was added to it and the resultant solution was titrated against the dye till persistent pink colour appeared. The amount of dye consumed ( $V_1$  ml) during titration was equivalent to the amount of ascorbic acid. One g (W) of aloe vera sample extracted in 100 ml ( $V_3$ ) of 4 per cent oxalic acid was kept for an hour and centrifuged. Discarding the residue, 5 ml of supernatant was pipetted into a conical flask and 10 ml ( $V_4$ ) of 4 per cent oxalic acid was added to it. The resultant solution was titrated against the dye till persistent pink colour appeared. Again, the volume of dye ( $V_2$  ml) consumed in titration was recorded. Three replications were conducted to determine the exact end point.

$$\text{Ascorbic acid mg/100 g dm} = E V_2 \frac{V_3}{V_4} \times \frac{100 \times 100}{W \times \text{dm}}$$

$$\text{Where, } E = \frac{1}{\text{Titre}} = \frac{1}{V_1} = \text{Ascorbic acid equivalent of the dye in mg/ml}$$

dm = dry matter

### 3.9.3 Textural measurement (Hardness)

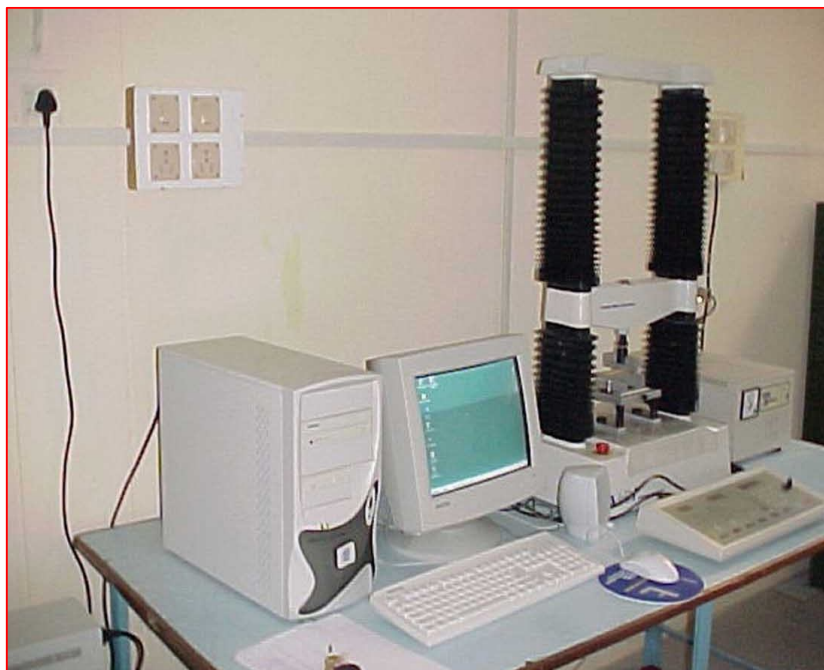
The texture characteristics of osmo-convectively dried aloe-vera sample in terms of hardness were measured using TA.HD Plus Textural Analyzer (Stable Micro System) shown in Plate 3.6. The analyzer is linked to a computer that recorded the data via a software program XT.RA Dimension, version 3.7H (Texture Technologies Corp., Scardale, Ny.) fitted with a 5 mm cylindrical probe. The studies were conducted at a pre test speed of 1.0 mm/s, test speed of 0.5 mm/s, distance of 0.5 mm and load cell of 30 kg. Hardness value was considered as mean peak compression force and expressed in grams (Cruzycelis, *et al.*, 1996: Anonymous, 1998) with the following settings of Texture Analyzer.

#### Settings

Pre- Test speed	: 1.0 mm/s
Test Speed	: 0.5 mm/s
Post-Test speed	: 1.0 mm/s
Distance	: 0.5 mm
Trigger Type Auto	: 3 g

#### Data Acquisition

Rate	: 200 pps
Probe Type	: 5 mm Cylinder (P/5)
Load cell	: 30 kg



**Plate.3.6 Texture analyzer**

### 3.9.4 Water activity ( $a_w$ )

Water plays an important role in the stability of fresh, frozen and dried foods. It acts as a solvent for chemical, microbiological and enzymatic reactions. The water activity,  $a_w$ , is a measure of the availability of water to participate in such reactions. The water in a food exerts a vapour pressure. The extent of this pressure will depend on the amount of water present, the temperature and the composition of the food. Different food components will lower the water vapour pressure to different extents, with salts and sugars being more effective than starches or proteins. Thus two different foods with similar moisture contents may not necessarily have the same  $a_w$ . Water activity can be defined as the ratio of the vapour pressure exerted by the food to the saturated vapour pressure of water at the same temperature.

$$a_w = \frac{\text{Vapour pressure of water exerted by food}}{\text{Saturated vapour pressure of water at the same temperature}} \quad \dots 3.17$$

Water activity is a function of moisture content in the food and the temperature (Ratti and Mujumdar, 1996). Bound molecule of water in food can be defined by water activity:

- Tightly bound water  $a_w < 0.3$
- Moderately bound water  $0.3 < a_w < 0.7$
- Loosely bound water  $a_w > 0.7$
- Free water  $a_w \sim 1.0$ .

Most bacteria do not grow at water activities below 0.91, and most moulds cease to grow at water activities below 0.80 (Leung, 1986). By measuring water activity, it is possible to predict which microorganisms will or will not be potential sources of spoilage. Lower water activity of a dried product implies better potential for storage.

Water activity was determined as a measure of storage stability using a Hygrolab-3 water activity meter. A 2 g sample was used to cover the filling indicator of the sample cup. The filled sample cup was kept in contact with sensor probe of water activity meter and values of water activity were recorded. A digital water activity analyzer used in measuring water activity of the dehydrated aloe vera samples is shown in Plate-3.7.



**Plate 3.7 Water activity meter**

### **3.10 Optimization of Convective Drying Process**

Major problems associated with air drying are considerable shrinkage, poor rehydration and changes in colour, texture, flavour and nutritive value (Mazza, 1983). These quality defects depend upon the drying conditions such as drying air temperature, drying air velocity, etc. The three quality parameters viz. colour (L value), hardness and ascorbic acid as well as water activity and drying time were considered for the optimization of input parameters of convective drying. Efforts were made to optimize the process parameters (drying air temperature and drying air velocity) for convective drying of osmotically dehydrated aloe vera samples for minimum water activity, maximum hardness, maximum L value of colour, maximum vitamin C, and minimum drying time with the following variables.

#### **Fixed parameters**

- Sample size, g : 100
- Equipment : Tray dryer

### **Independent variables**

- Temperature, °C (4 levels) : 50, 60, 70 & 80
- Air velocity, m/s (3 levels) : 1, 2 & 3

### **Dependent variables**

- Ascorbic acid, mg/ 100 g dm
  - Colour (L-value)
  - Hardness, g
  - Water activity
  - Drying time, min
- 
- **Analysis** : ANOVA
  - **Design** : FCRD
  - **Technique** : Numerical optimization technique
  - **Software** : Design Expert (version 8.0.4.1)

The optimization of process parameters (drying temperature and air velocity) for convective drying of osmotically dehydrated aloe vera sample was carried out using numerical optimization technique with the help of Design Expert 8.0.4.1 statistical software (trial version) by adopting full factorial design.

The factorial completely randomized design was adopted for analysis. The influence of various factors was analyzed using analysis of variance (ANOVA) technique and the results were discussed. Full second order model was fitted in various responses and independent variables using multiple regression analysis. Multiple regression analysis was done in MS EXCEL software.

### **3.11 Other Quality Analysis of Aloe Vera Product Prepared by Optimized Drying Conditions**

After optimization, the other quality attributes such as sensory evaluation, rehydration ratio and mineral content were also determined for the aloe vera product prepared by optimized drying conditions by adopting the standard procedure. All measurements were replicated thrice and the average values have been reported.

#### **3.11.1 Sensory evaluation of the product obtained from optimized drying parameters**

Sensory evaluation is important to assess the consumer's requirements. It is difficult to quantify the sensory characteristics of product 100 per cent by machine because it is a

subjective factor. To test the organoleptic characteristics, sensory evaluation was done based on numerical sensory card (BIS – 1971) as given in Appendix G.

Osmo-convectively dried product (Optimized conditions) was evaluated for its sensory characteristics such as colour, taste and over all acceptability and compared with sensory characteristics of convectively dried product (without osmo) (Drying air temperature- 70 °C, Velocity-1m/s) . Both the products were served for the evaluation to ten panelists at a time. The score sheet was provided with product and panelists were requested to mark the product according to their liking. The average scores of all the panelists were computed. The independent sample t test was applied to compare between convectively dried and osmo-convectively dried products for various organoleptic characteristics.

### 3.11.2 Rehydration characteristics

The rehydration characteristics viz. rehydration ratio and coefficient of rehydration of the osmo-convectively dried aloe vera product were determined by the method as explained by Pokharkar (1994).

Eight 250 ml glass beakers were used for rehydration of osmo-convectively dried aloe vera samples in diluted sugar solutions (0, 5, 10, 15 and 20 °Brix). The 5 g osmo-convectively dried sample was taken in each beaker, mixed with 50 ml of 5 °Brix sugar solution and kept in a hot water bath to maintain a sugar solution temperature of 30 °C (Pokharkar, 1994; Jain, 2007). Samples were withdrawn after 5, 15, 30 min and thereafter every 60 min up to 5 hours, their surface was dried with filter paper and weighed to determine the moisture content. The same procedure was repeated for sugar solutions of different concentrations. Various rehydration characteristics were evaluated using following relations (Pokharkar, 1994):

$$(a) \quad \text{Rehydration ratio } (RR) = \frac{C}{D} \quad \dots 3.18$$

$$(b) \quad \text{Coefficient of rehydration } (CR) = \frac{C \times (100 - A)}{\left(D - \frac{BD}{100}\right) \times 100} \quad \dots 3.19$$

Where, A = moisture content of samples before dehydration, IMC (% w.b.)

B = moisture content of dehydrated sample (% w.b.)

C = drained weight of rehydrated sample (g)

D = test weight of dehydrated samples (g)

The osmo-convectively dried products obtained after 5 h of rehydration in distilled water, 5, 10, 15 and 20 °Brix sugar solution were evaluated for organoleptic quality (*viz.* firmness, taste, flavour, and swelling). For each of the product characteristics, numerical

scoring was given by the consumer panel from zero to hundred, representing five quality grades, such as very poor, poor, fair, good and excellent (BIS:6273: Appendix H). Then average scores of the entire panelist were computed.

### **3.11.3 Minerals**

For determination of mineral the dry ashing was carried out and volume was made to 100 ml. This solution was used for determination of minerals. All the determinations were done in triplicate. Standard procedures were used for determination of minerals as given by Ranganna (2000).

#### **3.11.3.1 Calcium**

Twenty ml aliquot of ash solution obtained by dry ashing was added to 250 ml beaker. Ten ml of saturated ammonium oxalate solution & 2 drops methyl red indicator was added to it. Solution was made slightly alkaline & slightly acidic (pH 5) by the addition of dilute ammonia and a few drops of acetic acid respectively. Then the content of flask was heated to boiling point and allowed to stand overnight. The content of flask was filtered through whatman (No. 42) filter paper. The precipitate was washed through hot dilute H<sub>2</sub>SO<sub>4</sub> followed by hot water into the beaker. Then the content of the beaker was titrated with 0.01 N KMnO<sub>4</sub> till faint pink colour is obtained. The precipitate was added finally to solution and the titration was completed till permanent pink colour is obtained. The burette reading (titre) was recorded and used for calcium determination. Calcium was determined by using following formula:

$$\text{Calcium (mg / 100 g dm)} = \frac{\text{Titre} \times 0.2 \times \text{total volume taken for ash} \times 100 \times 100}{\text{Volume taken for estimation} \times \text{weight for sample} \times \text{weight of}}$$

#### **3.11.3.2 Magnesium**

Magnesium was determined by adding 10 ml ash solution into a 15 ml graduated centrifuge tube. One drop of methyl red indicator was added to it and neutralized with 10% ammonium hydroxide. One ml of ammonium oxalate was added to it and volume was made up to 13 ml with distilled water. The content was mixed & allowed to stand overnight. The content was centrifuged for 10 minutes. One ml supernatant was added into a 15 ml centrifuge tube along with 3 ml water, 1 ml of 2 % ammonium phosphate & 2 ml of 10 % ammonium hydroxide solution. The content was mixed and allowed to stand overnight. The content was centrifuged for 7 minutes. The supernatant was discarded. The content was mixed with 5 ml ammonium hydroxide solution (10 %), again centrifuged for 7 minutes and supernatant liquid was discarded. The precipitate was dried by placing it on container of hot water.

One ml of dilute HCl and 5 ml of water was added to dissolve the precipitate. One ml of molybdic acid solution, 0.5 ml of hydroquinone and 0.5 ml sodium sulphite solution was added to it. The content was mixed and allowed to stand for 30 minutes. The content was transferred to colorimeter tube and absorbance reading was noted using red filter.

To prepare standard curve, 0, 0.1, 0.2, 0.3, 0.4 and 0.5 ml of standard potassium dihydrogen phosphate (1 ml  $\text{KH}_2\text{PO}_4$  = 0.1 mg of P = 0.0784 mg of Mg) which correspond to 0, 10, 20, 30, 40 and 50 ppm P was pipetted and the procedure in the previous paragraph was followed. The standard curve was prepared by plotting absorbance against concentration and magnesium content was determined with the help of this curve.

### **3.11.3.3 Phosphorus**

Five ml of molybdate reagent was added to 5 ml of ash solution, obtained by dry ashing, into a 50 ml volumetric flask. Two ml of aminonaphthol sulphonic acid was added to it, mixed and the volume was made up to 50 ml. Similarly a blank was prepared using water in place of the sample. The content was allowed to stand for 10 minutes and the colour was measured at 650 nm, setting the blank at 100 % transmittance.

To prepare standard curve, 0, 0.1, 0.2, 0.3, 0.4 and 0.5 ml of standard potassium dihydrogen phosphate which correspond to 0, 10, 20, 30, 40 and 50 ppm P was pipetted and the procedure given for magnesium was followed. The standard curve was prepared by plotting concentration against absorbance and phosphorus content was determined with the help of this curve.

### **3.11.3.4 Potassium**

To determine potassium, 5 ml of aliquot of ash solution was added into a conical flask. 0.5 % HCl was added to make up the volume 150 ml. The diluted extract was atomized in a calibrated flame photometer with the wavelength dial set at 768 nm and the transmittance set at 100 % for the top standard solution. Standard curve was drawn and concentration (ppm potassium) was recorded from standard curve.

### **3.11.3.5 Sodium**

To determine sodium, 5 ml of aliquot of ash solution was added into a conical flask and 0.5 % HCl was added to make up the volume 150 ml. The diluted extract was atomized in a calibrated flame photometer with the wavelength dial set at 589 nm and the transmittance set at 100 % for the top standard solution. Standard curve was drawn and concentration (ppm sodium) was recorded from standard curve.

## CHAPTER IV

### DEVELOPMENT OF MATHEMATICAL MODEL

This chapter deals with the theories developed for analyzing the data. The existing theories and related mathematical equations (models) of osmotic dehydration and air-drying are cited here to determine water and sugar diffusion coefficients during osmotic and convective drying of aloe vera samples. A mathematical model based on Fick's law is also proposed.

#### 4.1 Modelling of Osmotic Dehydration Process

The process of osmotic dehydration involves soaking of water rich solid food product in concentrated solutions of sugar or salt. During this process two major simultaneous cross flows occur as shown in Fig. 2.1 (Raoult *et al.*, 1994b; Lewicki and Lenart, 1995; Fito *et al.*, 2001). Water flow from product to hypertonic solution is referred to as dewatering. Solute transfer from solution to product is referred to as impregnation. Apart from this a third mass transfer phenomena that could be observed is leaching of product's own solutes such as sugar, organic acids, minerals, vitamins, *etc.* The quantities of these constituents that are lost from these foods are quite negligible in comparison to two major mass transfers. Because of claims of osmotic dehydration process of achieving both dewatering and direct formulation of product (impregnation), this process has been recently named as Dewatering and Impregnation Soaking Process (DISP). An understanding of the above complex mechanism is very essential for the application of osmotic dehydration at industrial scale. Modeling has proved to be a useful tool in getting a better insight about the osmotic dehydration mechanism.

A number of studies on modeling of mass transfer kinetics have been carried out and models have been developed. Various approaches on the basis of which these models have been established are discussed below.

##### 4.1.1 Cellular structure approach

The diffusion of solutes and solvents has attributed to the trans-membrane transport and this process has been modelled by the principles of irreversible thermodynamics (Toupin *et al.*, 1989; Panagiotou *et al.*, 1998). This model takes account of the cell membrane characteristics such as cell volume change, tissue shrinkage and internal volumetric rearrangement. The model developed represented the behaviour of tissues undergoing osmotic

dehydration. This model has been christened as Toupin's model. This model however could not explain the cell membrane as a resistance to mass transfer which is vital during this process and hence its reliability remained doubtful. Later this model was upgraded by several researchers (Marcotte *et al.*, 1991). They have used the concept of irreversible process thermodynamics for trans-membrane transport but for transport in intracellular space (that serves as a resistance), Fick's second law has been applied. The result of this model has been quite successful on macroscopic scale.

#### **4.1.2 Finite element approach**

Recently, finite element technique, as a numerical solution provider for osmotic dehydration process, is being adapted. The weighed residual method was used to generate finite element formulation (equation) which was solved simplistically by use of FEMID software (Reddy, 1984). The predictive model developed by this approach provides a description of changes of rates of osmotic dehydration with time, position and concentrations in various sections of cellular tissue. It was later verified by experimental data and found to be true representation of the actual mass transfer in cellular tissues of food.

#### **4.1.3 Rate approach**

This approach involves use of rate equations (first order Arrhenious kinetics) to model the loss of water from the sample during process of osmotic dehydration. It involves study of rate of loss of water/constituents with time, temperature, concentration and/or other process variables. Once the model is established with respect to a particular food and specific process condition, it could be scaled up for industrial applications. This approach has been well tested for a number of foods like fresh beans (Biswal and Maguer, 1989), apples (Magee *et al.*, 1983) and mushrooms (Yang and Maguer, 1992).

#### **4.1.4 Compartmental approach**

The process of osmotic dehydration involves diffusion of water and solute. Initially surface of the food material is free from any solutes, but with time as solutes form a layer, greater concentration gradient is established than in the earlier stages. Thus with time the rates of diffusion do not remain same (Raoult *et al.*, 1991a, b, c).

#### **4.1.5 Fick's approach**

This approach has been named after Fick's second law that serves as a basis for explaining the mass transfer kinetics during osmotic dehydration. This approach explains the diffusion of soluble components from foods and vice versa. The estimation of diffusion coefficient in this method serves as suitable index for estimating water loss/solid gain in foods during osmotic dehydration.

The modelling of DIS process using various approaches gives a clear picture of the different mechanisms for getting a better understanding and the effects of the process variables. The approach of modelling involved use of a number of assumptions like:

1. Aloe vera samples were considered as hypothetical semi-permeable membrane, which allows only sugar and water to permeate. So, there was no exchange between the samples and sugar solution other than sugar and water.
2. The syrup to sample ratio is sufficiently high so that the syrup concentration can be considered constant throughout the osmotic dehydration process.
3. Apparent diffusion coefficient is constant, not being a function of concentration.
4. There is constant and uniform water/sugar concentration present in samples.
5. The osmotic dehydration process is isothermal.
6. The water and solute diffusion process are independent of one another.

#### 4.2 Fick's Law of Diffusion

When solid material is placed in a concentrated aqueous solution, the solute from solid will be transported through diffusion. The diffusive transfer of solutes from a solid to a surrounding solvent or vice versa is widely used in food industries, *i.e.* desalting of pickles, extraction of oil and sugar, *etc.* In such a process the rate of mass transfer can be approximately predicted by appropriate solutions of simplified unsteady state Fickian equation provided that the apparent or effective diffusivity is known. For one dimension diffusion of solute from sample to the osmotic solution, the differential equation is

$$\frac{\partial C}{\partial \theta} = D \frac{\partial^2 C}{\partial R^2} \quad \dots 4.1$$

Where,

C = concentration of diffusing component

$\theta$  = time, s

D = diffusivity, m<sup>2</sup>/s

R = distance in the direction of diffusion, m

Analytical solution of Eqn (4.1) is available for simple geometries, *i.e.* sphere, infinite cylinder, infinite and semi infinite slabs under constant concentration or fixed volume of the external solution. The mass transfer resistance at the surface of the solid is assumed to be negligible compared to the internal diffusion resistance in the solid. Fick's law of diffusion was applied to osmotic dehydration and air drying process in the present study to find out the sugar and moisture diffusivity.

### 4.3 Mathematical Model for Osmotic Dehydration

Osmotic dehydration is actually a simultaneous water and sugar diffusion process, when cellular solid (aloe vera sample) containing water is immersed in concentrated sugar syrup. The mass of water loss,  $M_{w\theta}$  from a sample after time  $\theta$  when it has been immersed into sugar syrup is given by

$$M_{w\theta} = W_{si} X_{swi} - W_{s\theta} X_{sw\theta} \quad \dots 4.2$$

Similarly the mass of sugar gain,  $M_{s\theta}$  from sugar syrup in time  $\theta$  is given by

$$M_{s\theta} = W_{s\theta} (1 - X_{sw\theta}) - W_{si} (1 - X_{swi}) \quad \dots 4.3$$

Where,

$M_{w\theta}$  = amount of moisture removed from sample in  $\theta$  min, kg

$M_{s\theta}$  = amount of sugar gained from the sugar syrup into sample in time  $\theta$  min, kg

$W_{si}$  = initial mass of sample, kg

$X_{swi}$  = moisture content as a fraction of the initial mass of the sample

$W_{s\theta}$  = mass of the sample after  $\theta$  min, kg

$X_{sw\theta}$  = moisture content as a fraction of the mass of the sample at time  $\theta$

The volumetric equilibrium moisture content, ( $X_{w\infty}$ ) and the volumetric equilibrium sugar content ( $X_{s\infty}$ ) are determined from the following equations

$$X_{w\infty} = \frac{W_{si} X_{swi} + W_{ui} X_{uwi}}{V_{si} + V_{ui}} \quad \dots 4.4$$

and

$$X_{s\infty} = \frac{W_{si} X_{usi} + W_{ui} X_{ssi}}{V_{si} + V_{ui}} \quad \dots 4.5$$

Where,

$X_{w\infty}$  = volumetric equilibrium moisture content in the sample with syrup,  $\text{kg/m}^3$

$X_{s\infty}$  = volumetric equilibrium sugar content in the sample with syrup,  $\text{kg/m}^3$

$W_{ui}$  = initial mass of sugar syrup for given sample, kg

$X_{uwi}$  = moisture content as the fraction of the mass of the syrup

$X_{usi}$  = sugar content as a fraction of the initial mass of the sample

$X_{ssi}$  = sugar content as a fraction of the initial mass of the syrup

$V_{si}$  = initial volume of the sample,  $\text{m}^3$

$V_{ui}$  = initial volume of sugar syrup for given sample,  $\text{m}^3$

If  $M_{w\infty}$  is the maximum possible amount of water which can be removed from the sample in infinite time ( $t = \infty$ ), then  $M_{w\infty}$  can be determined as follows

$$M_{w\infty} = W_{si} X_{swi} - \left( V_{si} - \frac{M_{w\infty}}{\rho_w} + \frac{M_{s\infty}}{\rho_s} \right) X_{w\infty} \quad \dots 4.6$$

During osmotic dehydration some sugar molecules diffuse into the pore structure of sample, resulting in less degree of volume change (shrinkage) in osmosed product (Shahabuddin *et al.*, 1990). It is therefore, assumed that the change in volume of the sample due to the sugar gain is negligible and the movement of water and sugar does not hinder to each other. Hence, the Eqn (4.6) becomes

$$M_{w\infty} = W_{si} X_{swi} - \left( V_{si} - \frac{M_{w\infty}}{\rho_w} \right) X_{w\infty} \quad \dots 4.7$$

or

$$M_{w\infty} = \frac{W_{si} X_{swi} - V_{si} X_{w\infty}}{1 - \frac{X_{w\infty}}{\rho_w}} \quad \dots 4.8$$

Similarly, if  $M_{s\infty}$  is the maximum possible amount of sugar which can be gained in the sample from the syrup in infinite time ( $t = \infty$ ), then the  $M_{s\infty}$  can be calculated as follows

$$M_{s\infty} = W_{si} X_{ssi} + \left( V_{si} - \frac{M_{w\infty}}{\rho_w} + \frac{M_{s\infty}}{\rho_s} \right) X_{s\infty} \quad \dots 4.9$$

If the volume change of sample due to sugar gain is negligible, the Eqn (4.9) becomes

$$M_{s\infty} = W_{si} X_{ssi} + \left( V_{si} - \frac{M_{w\infty}}{\rho_w} \right) X_{s\infty} \quad \dots 4.10$$

By putting the values of  $M_{w\infty}$  in to Eqn (4.10), we get

$$M_{s\infty} = W_{si} X_{ssi} + \left( V_{si} - \frac{W_{si} X_{swi} - V_{si} X_{w\infty}}{1 - \frac{X_{w\infty}}{\rho_w}} \right) \times X_{s\infty} \quad \dots 4.11$$

Where,

$$\left( V_{si} - \frac{M_{w\infty}}{\rho_w} \right) = \text{volume of sample at infinite time}$$

$\rho_w$  = density of water, kg/m<sup>3</sup>

$\rho_s$  = density of sugar, kg/m<sup>3</sup>

$M_{w\infty}$  = maximum possible amount of water removed from samples in infinite time.

$M_{s\infty}$  = maximum possible amount of sugar gained in samples from syrup in infinite time

The water loss ratio can be found out from Eqns (4.2) and (4.8) as

$$\frac{M_{w\theta}}{M_{w\infty}} = \frac{(W_{si} X_{swi} - W_{s\theta} X_{sw\theta}) \left( 1 - \frac{X_{w\infty}}{\rho_w} \right)}{W_{si} X_{swi} - V_{si} X_{w\infty}} \quad \dots 4.12$$

Similarly, sugar gain ratio can be found out from the Eqns (4.3) and (4.11) as

$$\frac{M_{s\theta}}{M_{s\infty}} = \frac{W_{s\theta}(1 - X_{sw\theta}) - W_{si}(1 - X_{swi})}{W_{si} X_{ssi} + \left( V_{si} - \frac{M_{w\infty}}{\rho_w} \right) X_{s\infty}} \quad \dots 4.13$$

From the diffusion of solute from sample immersed into large volume of stirred solution, Crank (1975) gave the equation for diffusion coefficient. Similar expressions were used in osmotic dehydration of aloe vera for water loss ratio and sugar gain ratio as

1. Water loss ratio:

$$\frac{M_{w\theta}}{M_{w\infty}} = 1 - \sum_{n=1}^{\infty} \frac{2\alpha_w(1 + \alpha_w)}{1 + \alpha_w + \alpha_w^2 q_{wn}^2} e^{(-) \frac{D_w q_{wn}^2 \theta}{\ell^2}} \quad \dots 4.14$$

2. Sugar gain ratio:

$$\frac{M_{s\theta}}{M_{s\infty}} = 1 - \sum_{n=1}^{\infty} \frac{2\alpha_s(1 + \alpha_s)}{1 + \alpha_s + \alpha_s^2 q_{sn}^2} e^{(-) \frac{D_s q_{sn}^2 \theta}{\ell^2}} \quad \dots 4.15$$

Where,

$D_w$  = moisture diffusivity, m<sup>2</sup>/s

$D_s$  = sugar diffusivity, m<sup>2</sup>/s

$\ell$  = half thickness of sample, m

$q_{wn}$  and  $q_{sn}$ ,  $n = 1, 2, 3, \dots$  are the roots of the transcendental Eqns (4.16) and (4.17)

$\alpha_w$  = ratio of mass of water in syrup and that in sample

$\alpha_s$  = ratio of mass of sugar in syrup and that in sample

$$\tan q_w = (-)\alpha_w q_w \quad \dots 4.16$$

and

$$\tan q_s = (-)\alpha_s q_s \quad \dots 4.17$$

Keeping the volume of the syrup large enough in comparison to the volume of the sample, the diffusion of water from sample to the syrup will not appreciably increase the values of  $\alpha_w$  and  $\alpha_s$ . The values of  $\alpha_w$  and  $\alpha_s$  may then be estimated from initial values of the syrup and the sample as

$$\alpha_w = \frac{W_{ui} X_{uwi}}{W_{si} X_{swi}} \quad \dots 4.18$$

and

$$\alpha_s = \frac{W_{ui} X_{usi}}{W_{si} X_{ssi}} \quad \dots 4.19$$

As the osmotic concentration of the sample has been carried out in batches and the process lasts for several minutes the values of  $D_w\theta/\ell^2$  and  $D_s\theta/\ell^2$  are large enough to neglect the values of  $n > 1$ . In that case the Eqns (4.14) and (4.15) will be simplified to

For water loss ratio

$$\frac{M_{w\theta}}{M_{w\infty}} = 1 - \frac{2\alpha_w(1+\alpha_w)}{1+\alpha_w+\alpha_w^2q_{w1}^2} e^{(-)\frac{D_wq_{w1}^2\theta}{\ell^2}} \quad \dots 4.20$$

and for sugar gain ratio

$$\frac{M_{s\theta}}{M_{s\infty}} = 1 - \frac{2\alpha_s(1+\alpha_s)}{1+\alpha_s+\alpha_s^2q_{s1}^2} e^{(-)\frac{D_sq_{s1}^2\theta}{\ell^2}} \quad \dots 4.21$$

Considering diffusion in three directions and rearranging Eqns (4.20) and (4.21) we get following equations in more simplified form as

$$\ln\left(1 - \frac{M_{w\theta}}{M_{w\infty}}\right) = \ln k_1 - \frac{3D_wq_{w1}^2\theta}{\ell^2} \quad \dots 4.22$$

and

$$\ln\left(1 - \frac{M_{s\theta}}{M_{s\infty}}\right) = \ln k_2 - \frac{3D_sq_{s1}^2\theta}{\ell^2} \quad \dots 4.23$$

$$\text{Where, } k_1 = \frac{2\alpha_w(1+\alpha_w)}{1+\alpha_w+\alpha_w^2q_{w1}^2}$$

$$\text{and } k_2 = \frac{2\alpha_s(1 + \alpha_s)}{1 + \alpha_s + \alpha_s^2 q_{s1}^2}$$

From the available relation between  $\alpha$  and  $q$  (Crank, 1975) the required values of  $q_{w1}$  and  $q_{s1}$  for a given  $\alpha_w$  and  $\alpha_s$  were obtained by interpolation. The values of  $(M_{w\theta}/M_{w\infty})$  and  $\alpha_w$  were obtained from the Eqns (4.12) and (4.18) respectively. The plot between  $\ln(1 - M_{w\theta}/M_{w\infty})$  and time yielded a straight line. From slope of the line,  $(-D_w q_{w1}^2 / \ell^2)$  the value of moisture diffusivity  $D_w$  was calculated.

Similarly, the value of sugar diffusivity ( $D_s$ ) was obtained from the slope,  $(-D_s q_{s1}^2 / \ell^2)$  of the straight line plot between  $\ln(1 - M_{s\theta}/M_{s\infty})$  and time.

From the known values of water and sugar diffusivities, the water loss ratio  $M_{w\theta}/M_{w\infty}$  and sugar gain ratio  $M_{s\theta}/M_{s\infty}$  in sample after time  $\theta$  were estimated from Eqns (4.20) and (4.21). The required values of  $M_{w\infty}$  and  $M_{s\infty}$  are obtained from Eqns (4.8) and (4.11). The mass reduction of sample  $W_{s\theta}$  after time  $\theta$  was estimated as

$$W_{s\theta} = W_{si} - (M_{w\theta} - M_{s\theta}) \quad \dots 4.24$$

The moisture content  $X_{sw\theta}$  after time  $\theta$  can be predicted as

$$X_{sw\theta} = \frac{W_{si} X_{swi} - M_{w\theta}}{W_{s\theta}} \quad \dots 4.25$$

The water loss from the sample can be given by substituting the value of mass reduction ( $W_{s\theta}$ ) from eqn (4.24) into Eq (4.2), as

$$M_{w\theta} = W_{si} X_{swi} - X_{sw\theta} \times (W_{si} - M_{w\theta} + M_{s\theta})$$

Rearranging the terms as

$$M_{w\theta} \times (1 - X_{sw\theta}) = W_{si} X_{swi} - X_{sw\theta} W_{si} - X_{sw\theta} M_{s\theta}$$

and dividing by  $(1 - X_{sw\theta})$  we get the water loss as

$$M_{w\theta} = \frac{W_{si} \times (X_{swi} - X_{sw\theta}) - X_{sw\theta} M_{s\theta}}{(1 - X_{sw\theta})} \quad \dots 4.26$$

The Eqn (4.26) indicated that the water loss ( $M_{w\theta}$ ) is a function of sugar gain ( $M_{s\theta}$ ), moisture content (MC) and time ( $\theta$ ). The value of  $M_{w\theta}$  for a given moisture content and sugar gain after time  $\theta$  could be estimated from the equation 4.26.

## 4.4 Air Drying Theory

### 4.4.1 Moisture diffusivity during drying

Drying of moist food generally takes place in the falling rate-drying period, during which water migrates from the interior to the surface of the material by diffusion. Moisture diffusivity is, therefore, a transport property (an internal phenomenon) related to the food dehydration and essential for the calculation and modelling of various unit operations in food processing (Saravacos, 1986). Fick's second law has been adopted for evaluation of moisture transport mechanism of the falling rate regions and is mathematically expressed by classical mass balance equation (Crank, 1975) as

$$\frac{\partial M}{\partial \theta} = \frac{\partial}{\partial R} \left( D_d \frac{\partial M}{\partial R} \right) \quad \dots 4.27$$

Where, M = moisture content, kg water per kg dry solids

$\theta$  = time, s

R = diffusion path or length, m

$D_d$  = moisture dependent diffusivity, m<sup>2</sup>/s

The diffusivity ( $D_d$ ) varies considerably with moisture and can be estimated by an analysis of drying data applying the method of slopes. Several researchers have implemented this technique for estimating the effective moisture diffusivity (McMinn and Magee, 1997; Pokharkar and Prasad, 2000; Khodke, 2002; Jain, 2007).

### 4.4.2 Method of slope

The method of slopes is based on the solution of the Fick's law of unsteady state diffusion Eqn (4.27). It involves the comparison between slopes of experimental and theoretical diffusion curves.

The aloe vera gel cube is assumed to be infinite slab (Khodke, 2002) being dried from both the sides with the assumptions that (i) initially moisture is uniformly distributed throughout the mass of sample; (ii) resistance to mass transfer at the surface is negligible compared to the internal resistance of sample and (iii) mass transfer is by diffusion only. Following initial and boundary conditions have, therefore, been fixed for a solution to Eqn. (4.27) as

$$M = M_0 \text{ at } \theta = 0 \text{ for all } R$$

$$M = M_s \text{ at } \theta > 0, \text{ for } R = (+)\ell \text{ at the surface, and}$$

$$M = M_\infty \text{ at } \theta > 0, \text{ for } R = (-)\ell \text{ at the surface}$$

Where,

- $M_s$  = moisture content at the surface
- $M_\infty$  = equilibrium moisture content
- $\ell$  = half the thickness of the sample, m

The solution of Eqn (4.27) for constant diffusivity D in terms of infinite series, the moisture content as a function of time in a slab can be given as:

$$\frac{M - M_\infty}{M_0 - M_\infty} = \frac{8}{\pi^2} \left( e^{(-)\frac{D\pi^2\theta}{4L^2}} + \frac{1}{9} e^{(-9)\frac{D\pi^2\theta}{4L^2}} + \frac{1}{25} e^{(-25)\frac{D\pi^2\theta}{4L^2}} + \dots \right) \quad \dots 4.28$$

When the time becomes large, limiting form of the Eqn (4.28) is as follows:

$$\begin{aligned} MR &= \frac{M - M_\infty}{M_0 - M_\infty} = \frac{8}{\pi^2} \left( e^{(-)\frac{D\pi^2\theta}{4L^2}} \right) \quad \dots 4.29 \\ &= \frac{8}{\pi^2} \left( e^{(-)\frac{\pi^2}{4} F_o} \right) \end{aligned}$$

Where,

- MR = moisture ratio
- $M_0$  = initial moisture content, kg water per kg dry matter
- M = moisture content at time  $\theta$ , kg water per kg dry matter
- $M_\infty$  = equilibrium moisture content, kg water per kg dry matter
- D = moisture diffusivity,  $m^2/s$
- L = thickness of the sample for drying from one side, m
- $F_o$  = Fourier number ( $D\theta/L^2$ )

Eqn (4.29) is evaluated numerically for Fourier number by rearranging and taking logarithm as

$$\begin{aligned} F_o &= (-)\frac{4}{\pi^2} \ln \left( \frac{MR\pi^2}{8} \right) \\ F_o &= (-)\frac{4}{\pi^2} \left( \ln MR + \ln \frac{\pi^2}{8} \right) \\ F_o &= -0.4052847 \ln MR - 0.0851171 \quad \dots 4.30 \end{aligned}$$

The Fourier number from Eqn (4.30) is used in evaluation of “effective moisture diffusivity”, ( $D_{eff}$ ). This established diffusion parameter ( $D_{eff}$ ) is interpreted as an overall mass transfer property, wherein all possible contributory moisture transfer mechanism such as liquid diffusion, vapour diffusion, surface diffusion, capillary flow, hydrodynamic flow and the heterogeneity of food materials are recognized. The measurement of the effective

diffusivity thus allowed a quantitative study of the drying characteristics in relation to controlled experimental variables.

The effective moisture diffusivity ( $D_{eff}$ ) varied considerably with the moisture content (M) of aloe vera sample during drying process, which resulted in nonlinear drying curves. For nonlinear drying curves the method of slope was adopted to estimate  $D_{eff}$  at various moisture contents (Chirife, 1971; Perry and Chilton, 1984). The theoretical moisture ratio MR, was evaluated numerically first for a range of Fourier number. The same ratio MR was evaluated using experimental drying data. Both curves of experimental and theoretical MR were plotted against time (t) and  $F_o$ , respectively, on a semi-log graph. By comparing the slopes of two curves Eqn (4.32), which could be determined by numerical differentiation at a specific MR, moisture diffusivity  $D_{eff}$  was evaluated from the following equation.

$$D_{eff} = \frac{\left( \frac{dMR}{dt} \right)_{exp}}{\left( \frac{dMR}{dF_o} \right)_{the}} \times L^2 \quad \dots 4.31$$

$$D_{eff} = \frac{(F_o)_{th}}{\left( \frac{t}{L^2} \right)} \quad \dots 4.32$$

Since moisture content (M) corresponded to the specific moisture ratio MR,  $D_{eff}$  could be found as a function of moisture content by repeatedly applying Eqn. (4.33).

#### 4.5 Verification of Mathematical Model

The developed mathematical models for osmotic concentration and air drying of aloe vera sample were statistically verified for process parameters under study. The procedure adopted was same as described in Chapter 3 using aloe vera samples of 1cm size (Jain *et al.*, 2011). The results are presented and discussed in Chapter V.

## CHAPTER V

### RESULTS AND DISCUSSION

This chapter deals with the results of the investigations carried out on osmotic dehydration, convective drying of osmotically dehydrated aloe vera gel samples and final product quality analysis. Statistical analysis of experimental data are also presented and discussed here.

#### PART I: OSMOTIC DEHYDRATION OF ALOE VERA GEL

Effects of process parameters such as duration of osmosis, concentration and temperature of syrup on mass reduction, water loss and sugar gain were studied and presented in this section. The water and sugar diffusivity during osmotic dehydration were estimated and activation energy was also determined and presented here. Mathematical model based on experimental results was also developed for prediction of osmotic dehydration kinetics and verified experimentally. The process variables were optimized for maximum water loss and optimum (targeted) sugar gain.

#### 5.1 Some Physical Characteristics of Aloe Vera Leaf

##### 5.1.1 Size and shape

The size of aloe vera leaf was determined from the measurement of its length, width and thickness. The length, width and thickness of aloe vera leaf have been presented in Table 5.1 and found to vary in the ranges from 390 to 630, 81 to 120 and 15 to 23 mm respectively (Appendix C). The mean values of the size of leaf in terms of length, width and thickness were found to be 493.18, 92.73 and 19.77 mm respectively (Table 5.1). Wang and Strong, (1993); Chandegara and Varshney (2007) have given similar results for length, width and thickness of aloe vera leaf. It can be noted from the table that the length is about 25 times to the thickness. The shape of aloe vera leaf may be classified as conic- tapered towards apex as per classification given by Mohsenin (1980).

##### 5.1.2 Apparent volume

The average value of length, width and thickness also decides size which is taken as the apparent volume of aloe vera leaf. The volume of the leaf was determined using mathematical expression as described in section 3.2.2 and presented in Table 5.1. The apparent volume was found to vary from 173.49 to 338.49 cc (Table 5.1). The average apparent volume of aloe vera leaf was found to be 236.77 cc.

##### 5.1.3 Leaf weight

The weight of corresponding 22 leaves was recorded with the help of top pan balance (least count 0.01 g) and reported in Appendix C. The maximum weight of the aloe vera leaf was recorded to be 575 g, whereas, minimum was 317 g with their standard deviation of 65.24. The average weight of the leaf was 428.91 g (Table 5.2).

#### 5.1.4 Gel weight

The weight of gel was measured after removing the skin; with the help of top pan balance (least count 0.01 g) and reported in Appendix C. The gel weight per leaf was found to vary in 137.8 g to 322.2 g range with average value of 213.6 and standard deviation of 43.85 (Table 5.2). It was also observed during the experiment that the aloe vera leaves having higher weight, had recorded better gel recovery.

#### 5.1.5 Gel recovery

The corresponding values of gel recovery of aloe vera leaves are presented in Appendix C. The maximum gel recovery of the aloe vera leaf was recorded to be 56.04 percent, whereas, minimum was 43.47 percent with the standard deviation of 2.59 (Table 5.2). The average gel recovery from the leaf was 49.44 percent. It was observed during the experiment that the aloe vera leaves having higher leaf weight recorded higher gel weight, and better gel recovery.

**Table 5.1 Variation in length, width, thickness and apparent volume of aloe vera leaf**

Particulars	Length, mm	Width, mm	Thickness, mm	Apparent volume, cc
Average	493.18	92.73	19.77	236.77
Range	390 to 630	81 to 120	15 to 23	173.49 to 338.49
SD	54.28	8.87	1.77	41.80

**Table 5.2 Variation in apparent volume, leaf weight, gel weight and gel recovery of aloe vera leaf**

Particulars	Apparent volume, cc	Leaf weight, g	Gel weight, g	Gel recovery, %
Average	236.77	428.91	213.6	49.44
Range	173.49 to 338.49	317 to 575	137.8 to 322.2	43.47 to 56.04
SD	41.80	65.24	43.85	2.59

During experimentation it was observed that, leaf weight and its apparent volume had some relationship. Hence, apparent volume could be taken as a parameter for the estimation of leaf fresh weight. The relationship between apparent volume and fresh weight of leaves is

shown in Fig. 5.1. Taking apparent volume as a function of aloe vera leaf weight, the following 3<sup>rd</sup> degree polynomial equation was developed.

$$y = a + bx + cx^2 + dx^3 \quad \dots(5.1)$$

$$R^2 = 0.65$$

Where,

y = apparent volume, cc; x = leaf weight, g

and the constants of the equation are

a = 552.62, b = -2.014, c = 0.0031 and d = -4 x 10<sup>-7</sup>

As aloe vera gel recovery is also a function of aloe vera leaf fresh weight, leaf weight can be taken as a parameter for the estimation of gel recovery. The relationship between leaf weight and gel recovery is shown in Fig. 5.2. Taking gel recovery as a function of the leaf fresh weight, following 3<sup>rd</sup> degree polynomial equation was developed.

$$y = a + bx + cx^2 + dx^3 \quad \dots (5.2)$$

$$R^2 = 0.98$$

Where,

y = gel recovery, %; x = leaf fresh weight, g

and the constants of the equation are

a = -54.252, b = 0.6547, c = -0.0014 and d = 1 x 10<sup>-6</sup>

Aloe vera gel recovery can also be related with apparent volume. Hence, apparent volume can be taken as a parameter for the estimation of leaf gel recovery. The relationship between apparent volume and gel recovery is shown in Fig. 5.3. Taking aloe vera leaf gel recovery as a function of the apparent volume, following 3<sup>rd</sup> degree polynomial equation was developed.

$$y = a + bx + cx^2 + dx^3 \quad \dots(5.3)$$

$$R^2 = 0.60$$

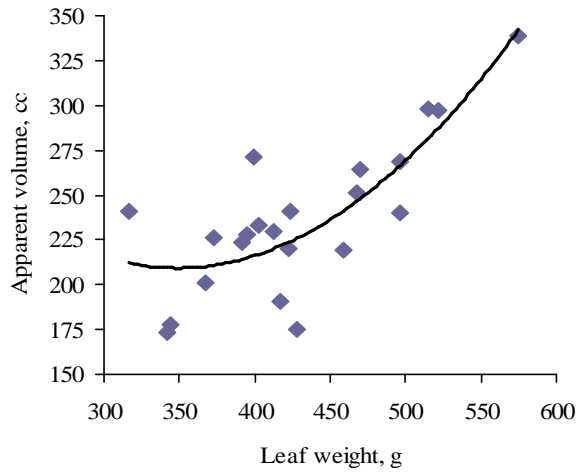
Where,

y = gel recovery, %; x = leaf apparent volume, cc

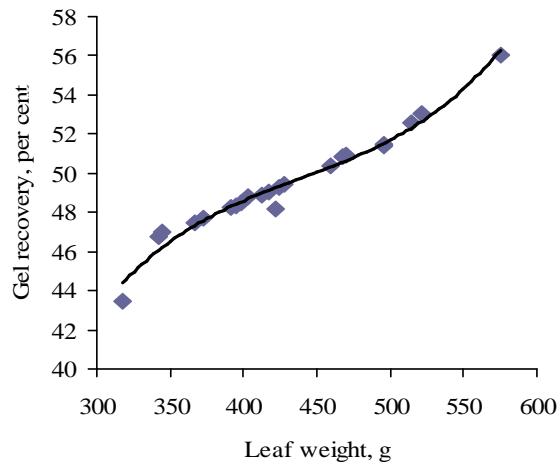
and the constants of the equation are

a = 61.788, b = -0.1534, c = 0.0004 and d = 1 x 10<sup>-7</sup>

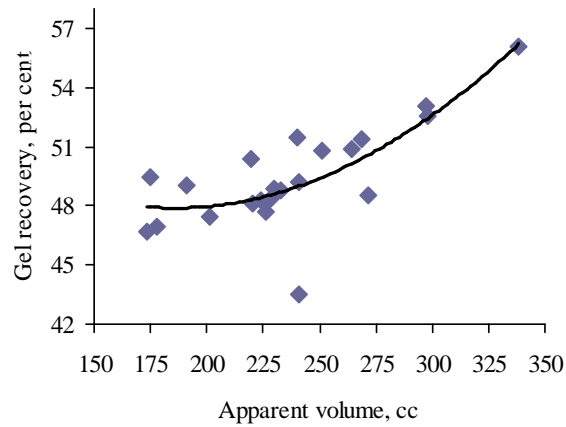
From above equation (5.1, 5.2 and 5.3) it is observed that, aloe vera leaf weight can be taken as a criteria for assessing gel recovery than apparent volume.



**Fig. 5.1 Relationship between apparent volume and fresh weight of aloe vera leaves**



**Fig. 5.2 Relationship between weights of aloe vera leaves and gel recovery**



**Fig. 5.3 Relationship between apparent leaf volume and gel recovery of aloe vera leaves**

## 5.2 Sample preparation by aloe vera peeler

The eleven aloe vera leaves weighing a total of 4731 g were used for the study. The base of leaves weighing 407 g (8.60 %) was removed and not used in sample preparation (Table 5.3). This process of base removed manually required 1.5 min. Similarly, removal of tips (less than 14 mm thickness i.e. not useful for sample preparation) weighing 380 g (8.03 %) required 1.5 min. Similarly, other portions not suitable for sample preparation were cut and time required for it when operation done manually with knife and weight of cut portion are given in Table 5.3. Thus, the total time required for preparation of sample was 140 min when all the operations are done manually with knife. Out of all the operations, the maximum time (60 min) was required to cut epidermis from 10 mm gel slab. By using developed aloe vera peeler this operation needed only 20 min to accomplish it. Thus, about 40 min (66.67 %) time could be saved by using aloe vera peeler as compared to the manual operation and total time required for preparation of sample was reduced to 100 min instead of 140 min.

**Table 5.3 Time required during various operations for sample preparation**

S. No.	Type of operation	Time required, manual with knife	Weight of cut portions of leaf/gel (other than sample)
1.	To cut base	1 min 30 sec	407 g (8.60%)
2.	To cut tips	1 min 30 sec	380 g (8.03%)
3.	To cut margins	7 min	730 g (15.43%)
4.	To cut 10 mm slabs (with epidermis )	40 min	305 g (6.45%)
5.	To cut epidermis for 10 mm gel slab	60 min	1296 g (27.39%)
6.	To cut 10 mm gel cubes	30 min	165 g (3.49%)
<b>Total</b>		<b>140 min</b>	<b>3283 g (69.39%)</b>

Since all other operations (except epidermis cutting from gel slab) were done easily by hand with knife, with accuracy, without fear of drudgery and fast too, efforts were not taken to mechanize the same. The total weight of cut portions of leaf/gel not used for sample was 3283 g (69.39%). Thus, 1448 g (30.61%) gel could be used in the form of 10 mm cubes. This amount of gel will vary as per the size and weight of leaf.

### 5.3 Selection of Process Variables

The initial moisture content of aloe vera gel samples was determined as described in section 3.2.4 and was found in the range 98.13 - 99.16% (wb) (Appendix E). The effect of process variables like duration of osmosis ( $\theta$ ), syrup concentration (C) and its temperature (T) during osmotic dehydration of aloe vera gel samples on mass reduction, water loss and sugar gain was determined as outlined in section 3.5.3. The selection of process variables was done on the basis of these analyses. These trials were replicated thrice and their average values have been reported (Table 5.4 to 5.6). The effects of these process parameters are discussed as follows:

#### 5.3.1 Duration of Osmosis

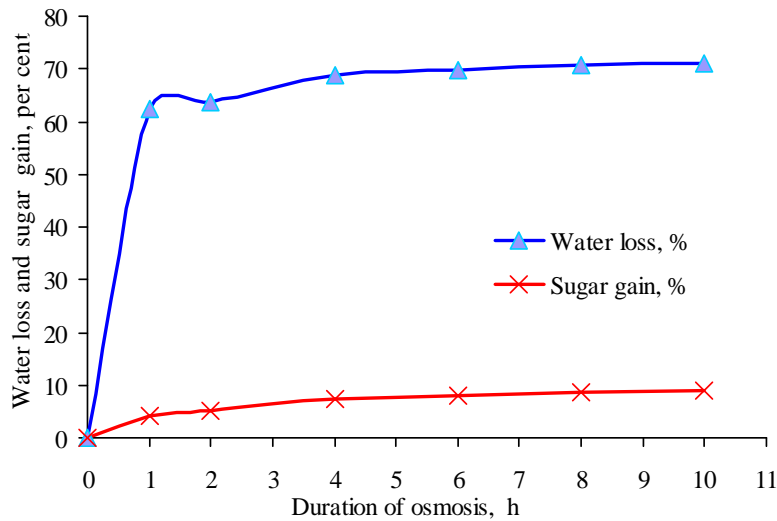
The mass transport data of water and sugar from aloe vera gel samples and syrup with duration of osmosis for 70 °Brix sugar solution at 50°C temperature having syrup to sample ratio (R') as 5 : 1 are presented in Table 5.4 and Fig. 5.4. It is inferred from the table that mass reduction, water loss and sugar gain of sample increased with increase in duration of osmosis. The mass reduction and water loss increased from 0 to 62.34 and 71.15 per cent respectively as the duration of osmosis increased from 0 to 10 h. The sugar gain was also found to increase during this period from 0 to 8.81 per cent, while concentration of syrup after osmotic dehydration was reduced from 70 to 51.3 °Brix.

**Table 5.4 Effect of duration of osmosis on mass transport data for osmotic dehydration of aloe vera gel samples (T = 50°C; C = 70 °Brix; R' = 5:1)**

Duration of osmotic dehydration, h	Initial mass, g	Final mass, g	FMC, %(wb)	Mass reduction, %	Water loss, %	Sugar gain, %	Concentration of syrup after osmotic dehydration, °Brix
0	50.84	50.84	98.93	0.00	0.00	0.00	70.0
1	50.20	20.98	87.43	58.21	62.40	4.18	64.5
2	50.18	20.77	84.83	58.60	63.81	5.21	61.2
4	50.45	19.47	78.42	61.41	68.67	7.26	57.4
6	50.05	19.17	76.31	61.70	69.71	8.01	54.3
8	50.10	18.96	74.67	62.16	70.68	8.52	52.2
10	50.01	18.83	73.78	62.34	71.15	8.81	51.3

It is clear from the Fig. 5.4 that the water loss and sugar gain though increased with duration of osmosis, the rate decreased at later stage and equilibrium could not be achieved

even after 10 h of osmotic dehydration. Rapid water loss (62.40%) was observed in the beginning (first hour of osmosis). The rate of water loss was observed to be 4.86, 1.04, 0.97, and 0.47 per cent in 2 to 4 h, 4 to 6 h, 6 to 8 h and 8 to 10 h respectively. As there was no appreciable increase in water loss beyond 4 hours as compared to time required, the osmosis was limited to 4 hours only.



**Fig. 5.4 Effect of duration of osmosis on water loss and sugar gain (Temp. = 50°C, Conc. = 70 °Brix and R' = 5:1)**

Similarly, the sugar gain increased steadily up to 4h and was found negligible with further advance of time. In the first two hour of osmosis, 5.21 per cent sugar gain was observed while the rate of sugar gain was 2.05, 0.75, 0.51 and 0.29 per cent point as the duration of osmosis increased from 2 to 4 h, 4 to 6 h, 6 to 8 h and then 8 to 10 h, respectively (Table 5.4). Therefore, the osmosis duration of 4h was found to be sufficient for the process of osmotic dehydration of aloe vera gel. The water loss (WL) and sugar gain (SG) in aloe vera gel samples were co-related with duration of osmosis ( $\theta$ ) by fitting third order polynomial model as below.

$$WL = 0.4249 \theta^3 - 7.7251 \theta^2 + 41.153 \theta + 10.379 \quad \dots 5.4$$

$$R^2 = 0.85$$

$$SG = 0.0241 \theta^3 - 0.4934 \theta^2 + 3.3692 \theta + 0.4446 \quad \dots 5.5$$

$$R^2 = 0.98$$

### 5.3.2 Sugar syrup concentration

The effect of syrup concentration (C) on water loss and sugar gain for 6 h of osmosis was studied by constant syrup temperature as 50°C, syrup to fruit ratio as five and initial

moisture content as 98.95 percent. The results of the study are presented in Table 5.5. It can be observed that by increasing the syrup concentration from 10 to 80 °Brix, water loss and sugar gain both increased from 49.33 to 72.94 per cent and 1.41 to 7.56 per cent, respectively, while the mass reduction was in the range of 47.92 to 65.38 per cent. After osmotic dehydration, the concentration of syrup decreased and was observed in the range of 8.5 to 65.4 °Brix.

**Table 5.5 Influence of syrup concentration on mass transport data for aloe vera gel samples (R' = 5:1, T = 50°C and Duration = 6h)**

Syrup concentration, °Brix	Initial mass, g	Final mass, g	FMC, %(wb)	Mass reduction, %	Water loss, %	Sugar gain, %	Concentration of syrup after osmotic dehydration, °Brix
10	50.14	26.11	95.28	47.92	49.33	1.41	8.5
20	50.16	21.40	90.68	57.34	60.27	2.93	17.6
30	50.35	18.33	84.51	63.60	68.19	4.59	24.6
40	50.71	17.87	80.57	64.77	70.56	5.80	34.3
50	50.22	17.57	78.40	65.02	71.52	6.50	44.0
60	50.14	17.53	76.87	65.04	72.08	7.04	53.1
70	50.07	17.43	75.53	65.20	72.66	7.47	59.7
80	50.05	17.33	75.13	65.38	72.94	7.56	65.4

The effect of sugar concentration on water loss and sugar gain has been shown in Fig. 5.5. It can be seen that initially water loss (WL) increased rapidly which gradually reduced. Beyond 40 °Brix sugar syrup concentration, water loss did not increase appreciably. The sugar gain increased with increase in syrup concentration. The sugar gain was relatively higher (5.80%) as the concentration of sugar syrup increased up to 40 °Brix and thereafter the rate of increase was only 0.70, 0.54, 0.43, and 0.09 per cent at 50, 60, 70 and 80 °Brix respectively. Thus, sugar gain of the product was enhanced at higher concentration but beyond 40°Brix of sugar syrup without much advantage of water loss. Therefore range of concentration was limited upto 40°Brix for further study.

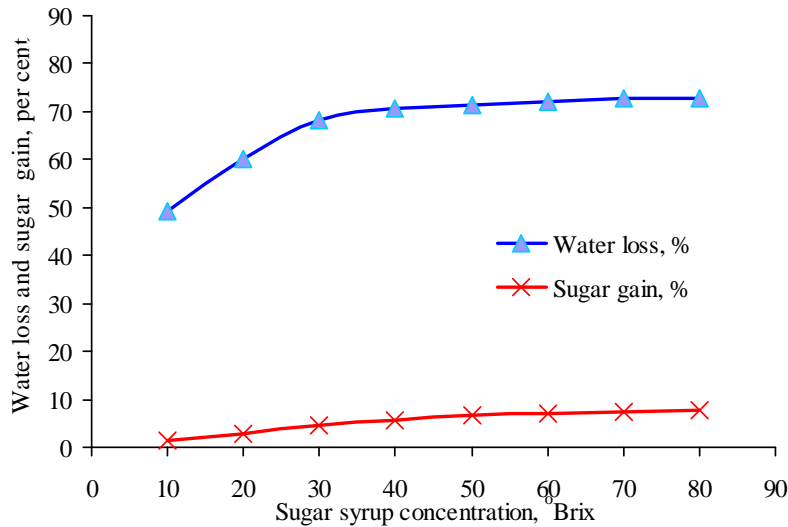
The mass transport data for water loss (WL) and sugar gain (SG) were statistically analyzed and fitted in second order form of polynomial equations as

$$WL = -0.0085 C^2 + 1.0488 C + 41.621 \quad \dots 5.6$$

$$R^2 = 0.955$$

$$SG = -0.0014 C^2 + 0.2123 C - 0.618 \quad \dots 5.7$$

$$R^2 = 0.998$$



**Fig. 5.5 Effect of syrup concentration on water loss and sugar gain (Temp. = 50°C, R' = 5:1 and Duration = 6h)**

### 5.3.3 Sugar syrup temperature

The effect of syrup temperature on water loss and sugar gain for 6 h of osmotic dehydration was studied with syrup concentration at 70 °Brix and syrup to fruit ratio as five. The results of the study are presented in Table 5.6. It is seen that as the temperature of sugar syrup increased from 30 to 50 °C, the water loss increased from 69.59 to 74.64 per cent and thereafter it decreased to 70.23 and 66.63 per cent on increasing the temperature to 60 and 70°C respectively. Similarly sugar gain also increased from 6.16 to 8.68 per cent as the temperature increased from 30 to 50 °C and reduction in sugar gain observed on further increasing temperature which may be due to collapse of cell structure responsible for osmosis due to increase in temperature beyond 50 °C. The mass reduction also increased from 63.43 to 65.96 per cent as the temperature increased from 30 to 50 °C and its reduction was observed on further increasing temperature. Therefore the temperature range of 30 to 50 °C was taken for all the subsequent investigations.

The water loss (WL) and sugar gain (SG) in aloe vera gel samples were co-related with syrup temperature (T). Third order polynomial model was fitted for both water loss and sugar gain as below.

$$WL = 7E-05 T^3 - 0.024 T^2 + 1.7839T + 35.618 \quad \dots 5.8$$

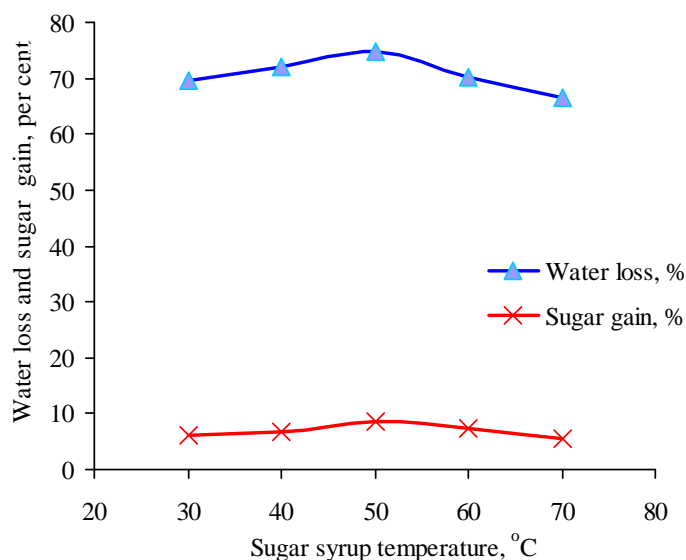
$$R^2 = 0.914$$

$$SG = -0.0001T^3 + 0.0127T^2 - 0.3149T + 7.4011 \quad \dots 5.9$$

$$R^2 = 0.867$$

**Table 5.6 Influence of syrup temperature on mass transport data for aloe vera samples (C= 70 °Brix, R' = 5:1, and Duration = 6 h)**

Temperature, °C	Initial mass, g	Final mass, g	FMC, % (wb)	Mass reduction, %	Water loss, %	Sugar gain, %	Conc. of syrup after osmotic dehydration, °Brix
30	50.06	18.31	80.28	63.43	69.59	6.16	40.0
40	50.09	17.40	77.24	65.26	72.12	6.86	45.2
50	50.30	17.12	71.42	65.96	74.64	8.68	53.2
60	50.10	18.59	77.39	62.89	70.23	7.34	60.2
70	50.05	19.54	82.79	60.97	66.63	5.67	67.3



**Fig. 5.6 Effect of syrup temperature on water loss and sugar gain (Conc. = 70 °Brix, R' = 5:1 and Duration = 6 h)**

### 5.3.4 Levels of input parameters

Based on the results of preliminary investigations on water loss and sugar gain (Table 5.4 to 5.6), the ranges of input parameters were fixed for further experimentation of optimization as shown in Table 5.7. The syrup to sample ratio was taken as constant at 5:1 level which was also suggested by various researchers for various fruits/vegetables (Lenart and flink, 1984a; Simal *et al.*, 1997; Kar and Gupta 2001; Pokharkar and Prasad 2002; and Pisalkar, 2008).

**Table 5.7 Process parameters selected for investigations**

Process parameters	Levels
Sugar syrup temperature	30, 40 and 50°C
Sugar syrup concentration	10, 25 and 40 °Brix
Duration of osmosis	60, 150 and 240 min

### 5.3.5 Product quality

In an osmotic dehydration process the higher the water loss the better is the dehydration process. However, high solid gain affects the fruit quality and sensory characteristics. When high levels of solids are incorporated into the fruit during the osmotic dehydration significant sensory alterations can occur and the final product may present a taste that is very different from the overall acceptability. Thus considering the importance of the sugar gain in product quality and acceptability of osmotic dehydration, this factor was mainly used for optimization of the input parameters of the process. For this purpose, it was necessary to fix the level of sugar gain in the final product, so that it is acceptable by the consumers. Ten judges were given the osmo-convectively dried aloe vera samples having the various levels of sugar gain as shown in Table 5.8. The judges were asked to taste the samples and give the marks according to their liking. The details of quality grade descriptions with corresponding scores are shown in Appendix D. The details of the mean sensory score for the sweetness characteristics of the product as well as the result of these tests are presented in Table 5.8.

**Table 5.8 ANOVA of sensory evaluation done for products having various level of sugar gain**

Product code	Sugar gain, %	Mean score
01	1.41	6.10 <sup>d</sup>
02	2.93	7.10 <sup>c</sup>
03	4.59	8.20 <sup>a</sup>
04	5.90	7.50 <sup>b</sup>
05	6.50	7.30 <sup>b</sup>
06	7.04	7.20 <sup>b,c</sup>
07	7.47	7.10 <sup>c</sup>
08	7.56	7.10 <sup>c</sup>

*\*The values superscripted by similar letters are non-significantly different from each other*  
 F-cal = 27.2 (Sig.), CD (5%) = 0.313, CV = 4.87%

It can be observed from Table 5.8 that, as the sugar gain increased from 1.41 to 7.56 per cent, the mean sensory scores increased up to 4.59 per cent of sugar gain and then decreased.

From the analysis of variance, it could be seen that the coefficient of variance among the different judges was 4.87 per cent, which is less than 10 percent indicating coherence amongst the score attributed by the judges. The F value was significant and the CD (5 %) indicated that the product with 4.59 per cent sugar gain was most liked by the judges. The highest sensory score attributed to the product indicated that osmo-convectively dried aloe vera product having 4.59 per cent sugar gain may acquire higher liking by the consumer. Therefore the input parameters namely, syrup temperature; syrup concentration and duration of osmosis (Table 5.7) were optimized (as discussed in section 5.5) on the basis of maximum water loss and targeted sugar gain (4.59 %).

#### **5.4 Osmotic Dehydration of Aloe Vera Samples**

The experiments on the osmotic dehydration were conducted as mentioned in section 3.7 in order to study its kinetics. The raw data with calculated values of mass reduction, water loss and sugar gain are presented in Data Sheet 1 to 9 of Appendix E. The mass reduction, water loss and sugar gain were observed in the range of 35.17 to 47.32, 36.82 to 53.98 and 1.65 to 6.66 per cent respectively for 4 h duration of osmosis for various concentration and temperature ranges of the syrup. The effect of these process parameters are presented in the following sections.

##### **5.4.1 Water loss**

The initial moisture content of aloe vera gel samples varied from 98.13 to 99.16 per cent (wb). The kinetics of osmotic dehydration are shown in Figs. 5.7 and 5.8 giving the variation in water loss at various concentrations and temperatures of syrup. The water loss from test samples under selected process parameters are shown in different curves, indicating relatively smooth progression of drying despite the fact that almost all data points came from independent test runs. The water loss was different for different conditions of syrup temperatures and concentrations.

The minimum and maximum water loss after osmotic dehydration was 36.82 and 53.98 per cent respectively, corresponding to low levels (10°Brix, 30°C after 4 h) and high levels (40 °Brix, 50 °C after 4 h) of syrup concentration and temperature. The water loss was increased from 0 to 36.82, 38.12 and 45.58 % when duration of osmotic dehydration increased from 0 to 4 h for 10°Brix concentration at 30, 40 and 50°C syrup temperatures respectively. Similarly for 25 and 40 °Brix concentrations, it varied from 0 to 41.98, 45.57, 49.71 and 47.47, 50.98, 53.98 per cent for 30, 40 and 50°C syrup temperatures, respectively (Fig. 5.7) (Appendix E).

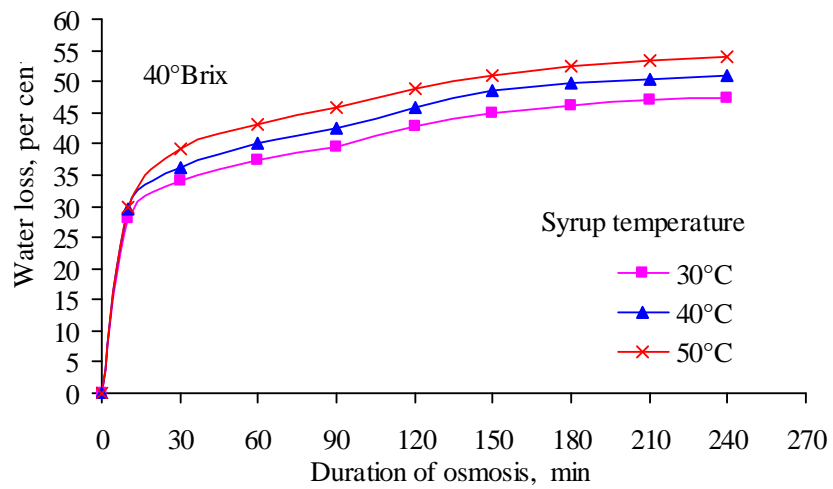
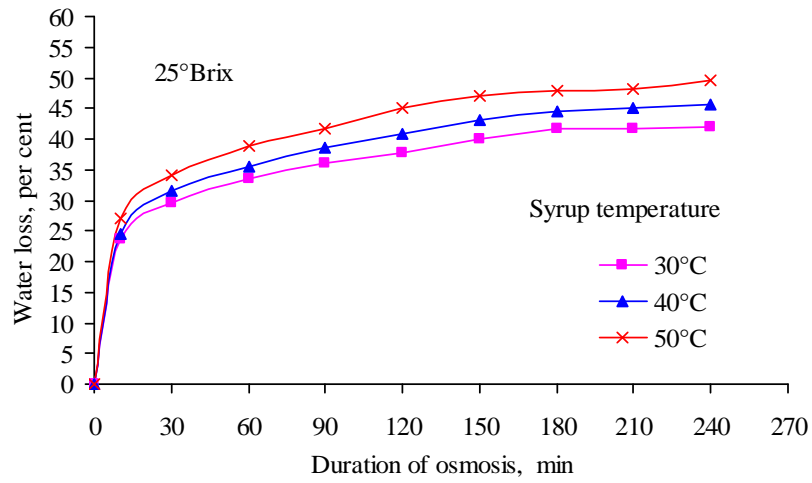
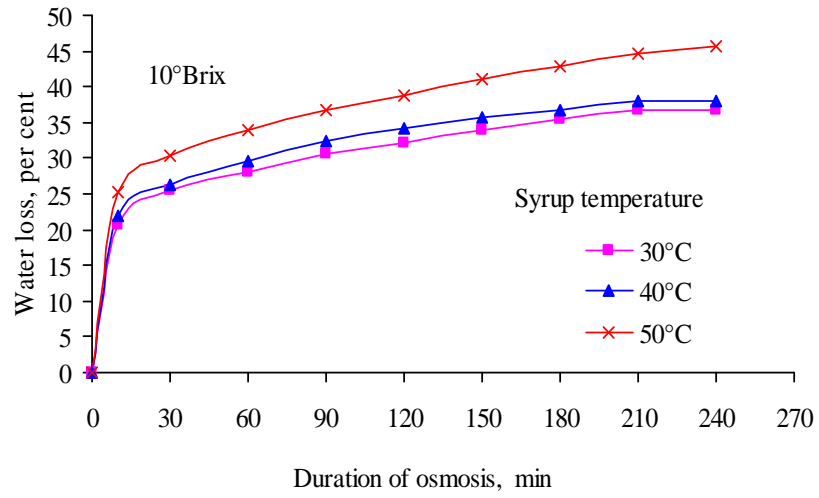
It can be observed that when syrup temperature was increased from 30 to 40°C for 40°Brix syrup concentration, water loss increased from 47.47 to 50.98 per cent after 4 h of

osmotic dehydration causing approximately 3.51 per cent point increase and on further increase in syrup temperature to 50°C, the water loss was observed to be 53.98 per cent (3.0 per cent point increment). For 25°Brix syrup concentration, the water loss increased from 41.98 to 45.57 per cent (3.59 per cent point increment) when syrup temperature increased from 30 to 40°C, and on further increase in syrup temperature from 40 to 50°C, the water loss was increased to 49.71 per cent (4.14 per cent point increment). The similar results were obtained for 10°Brix syrup concentration also with the corresponding increase of 1.31 per cent point when syrup temperature was increased from 30 to 40°C and 7.46 per cent point when temperature was increased from 40 to 50°C (Fig. 5.7).

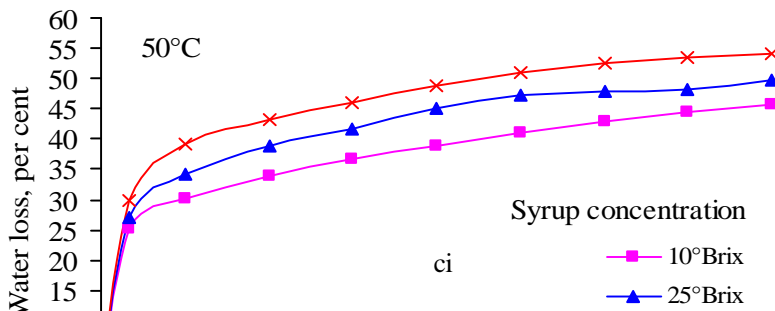
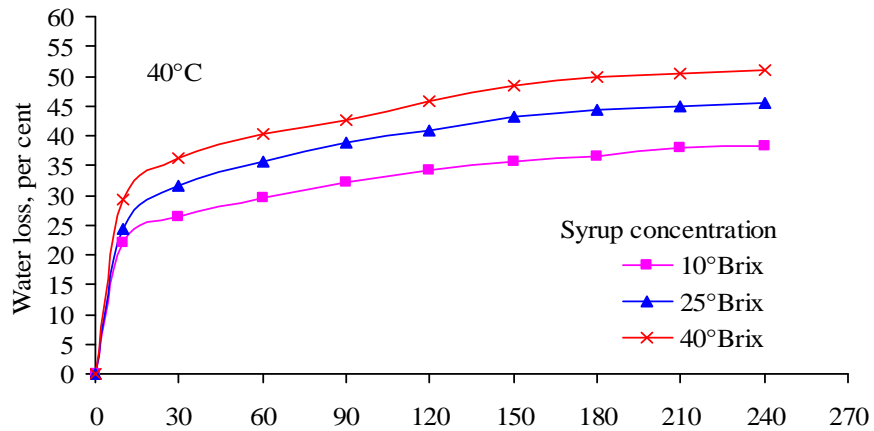
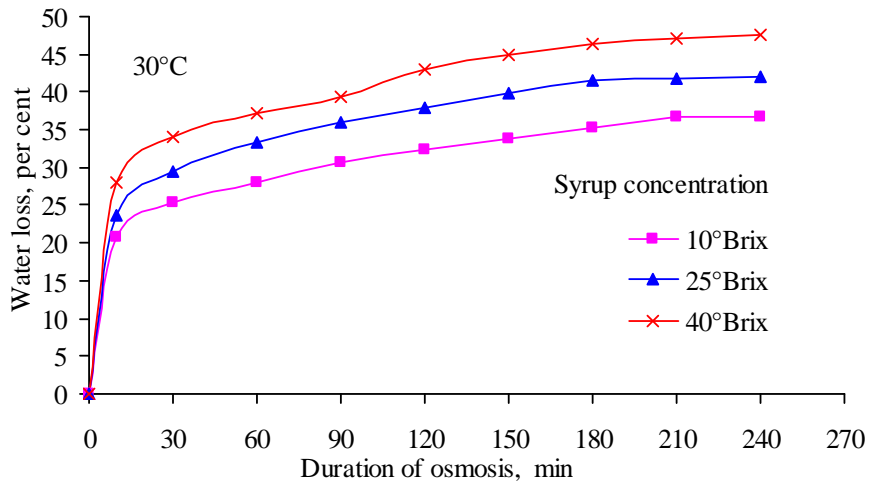
Inference can now be drawn that a low temperature-low concentration condition (30°C-10°Brix) gives a low water loss (36.82 % after 4 h of osmosis) and a high temperature-high concentration conditions (50°C-40°Brix) gives a higher water loss (53.98 % after 4 h of osmosis). The low temperature-high concentration condition 30°C-40°Brix gives a slightly higher water loss of 47.47 per cent after 4 h of osmosis than high temperature-low concentration condition 50°C-10°Brix (45.49 % after 4 h of osmosis) indicating a pronounced effect of concentration on water loss (Fig. 5.7). This indicates that water loss can be increased by increasing either the syrup concentration or temperature of solution. However, an increase in concentration of sugar solution has more influence on water loss (Fig. 5.8) than an increase in temperature (Fig. 5.7).

The water loss at any concentration was affected by the temperature of the syrup and increased with increase in syrup temperature. This may be due to changes in semi-permeability of the cell membrane of the fruit, allowing more water to diffuse out in a shorter period. These findings were in confirmation with the results obtained by Ponting *et al.*, (1966) Farkas and Lazar (1969), Jain (2007) and Pisalkar (2008). Besides, at higher temperatures, the viscosity of the syrup decreased, causing setting of convection currents in the syrup, which in turn eliminated local dilution and favoured osmosis.

Increase in water loss with increase in syrup concentration may be due to increased osmotic pressure in the syrup at higher concentrations, which increased the driving force available for water transport. This is in agreement with Pokharkar *et al.*, (1998a and b), Nieuwenhuijzen *et al.*, (2001), Jain (2007) and Pisalkar (2008). Water loss from the aloe vera samples was very rapid for the first hour of osmosis to the tune of 28.01 percent to 43.06 per cent corresponding to low levels (10°Brix, 30°C) and high levels (40°Brix, 50°C) of syrup concentration and temperature respectively and decreased gradually with further increase of duration of osmosis, but did not approach the equilibrium. The similar results were quoted in case of the osmotic dehydration of green beans (Biswal and Bozorgmehr, 1991), banana slices (Pokharkar *et al.*, 1997), papaya (Jain, 2007) and aloe vera (Pisalkar, 2008).



**Fig.5.7 Variation in water loss with syrup temperature at 10, 25 and 40°Brix concentration**



**Fig. 5.8 Variation in water loss with concentration at 30, 40 and 50°C temperature**  
**5.4.2 Sugar gain**

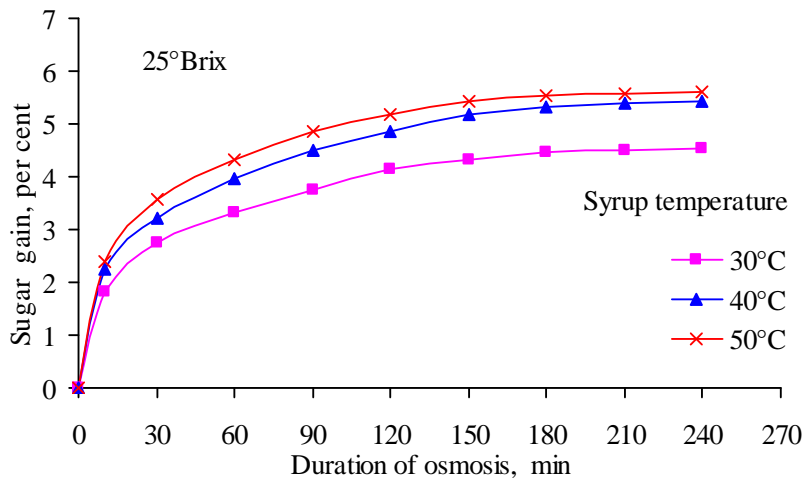
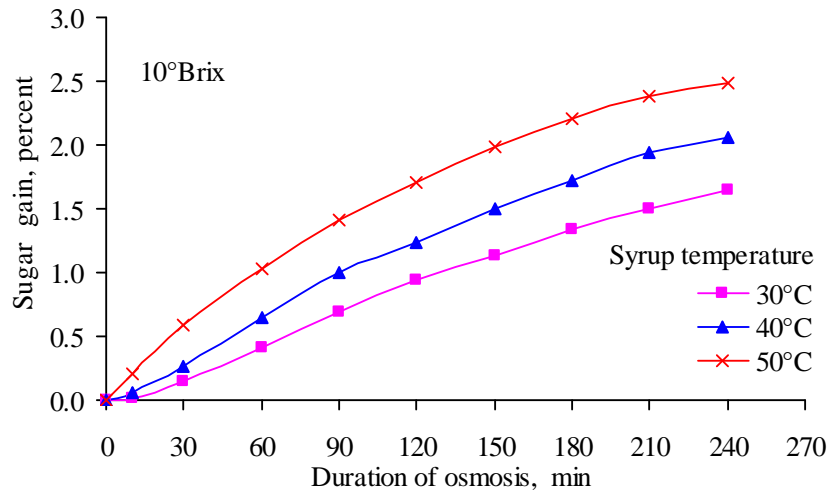
The variations in sugar gain at various concentrations and temperatures of syrup are shown in Fig. 5.9 and 5.10. Similar to water loss, the sugar gain also indicates relatively smooth progression of various curves despite the fact that almost all data points came from independent test runs. It can be seen that sugar gain increased with duration of osmosis and did not approach the equilibrium even after 4 h of osmotic dehydration.

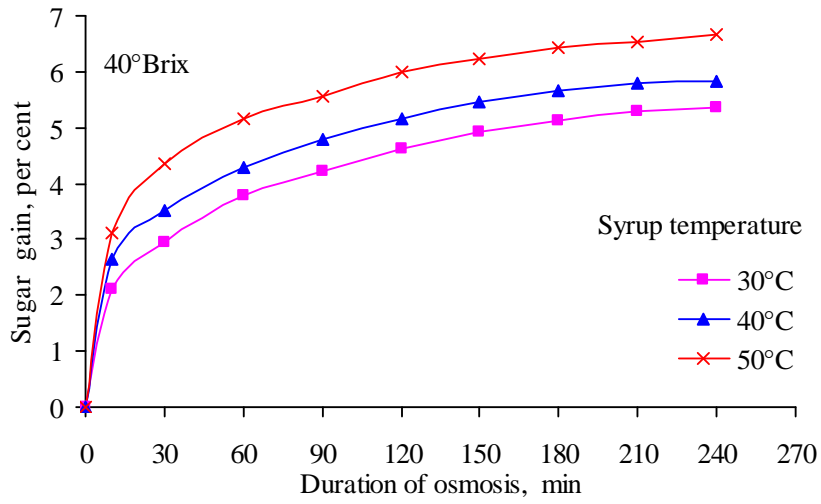
The sugar gain was increased from 0 to 1.65, 2.05 and 2.49 per cent when duration of osmotic dehydration increased from 0 to 4 h for 10°Brix concentration at 30, 40 and 50°C syrup temperatures, respectively. Similarly for 25 and 40°Brix concentrations, the sugar gain was found to vary from 0 to 4.52, 5.41, 5.6 and 5.34, 5.84 and 6.66 per cent for 30, 40 and 50°C syrup temperatures, respectively (Fig. 5.9).

It can be observed from the data that when syrup temperature is increased from 30 to 40°C for 40°Brix concentration, sugar gain increased from 5.34 to 5.84 per cent causing 0.50 per cent point increase after 4 h of osmosis, however for further increase in syrup temperature to 50°C, the sugar gain was 6.66 per cent (0.82 per cent point increase) in the same period of osmosis. Similarly for 25°Brix syrup concentration, the sugar gain increased from 4.52 to 5.41 per cent ( 0.89 per cent point increment) when syrup temperature was increased from 30 to 40°C, while it increased to 5.6 per cent (only 0.19 per cent point increase) with further increase in syrup temperature from 40 to 50°C. However for 10°Brix syrup concentration the increase in sugar gain was 0.4 per cent point and 0.44 per cent point, when the syrup temperature was increased from 30 to 40°C and 40 to 50°C respectively (Fig. 5.9).

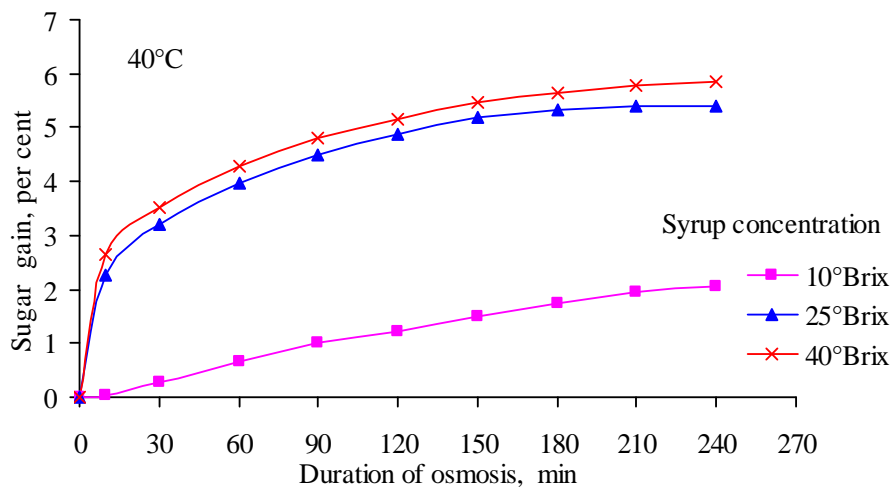
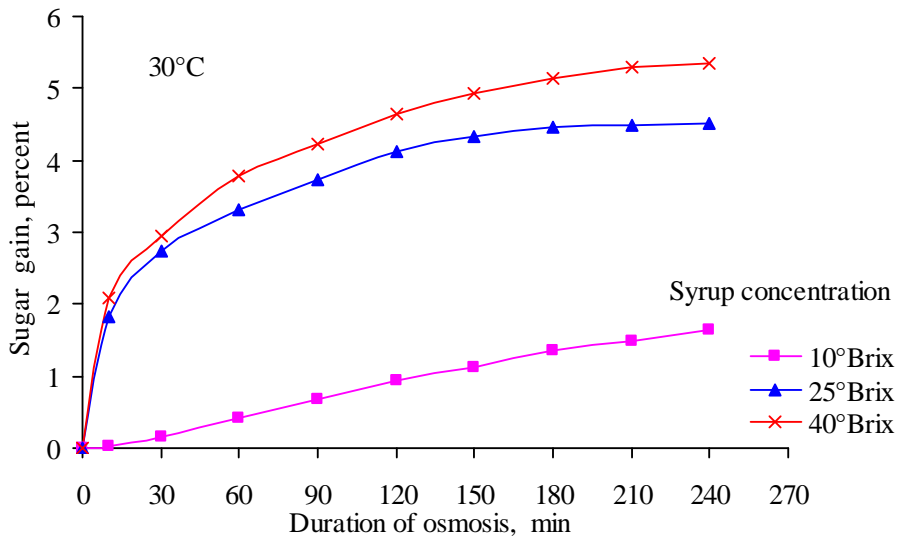
The increase in sugar gain with the concentration of the syrup may be because of the increased concentration difference between fruits and syrup (Fig. 5.10). The increased sugar gain with increase in syrup temperature may be due to the collapse of the cell membrane at higher temperatures. Similar results have also been reported by Ertekin and Cakaloz (1996a and b), Nsonzi and Ramaswamy (1998a), Jain (2007) and Pisalkar (2008).

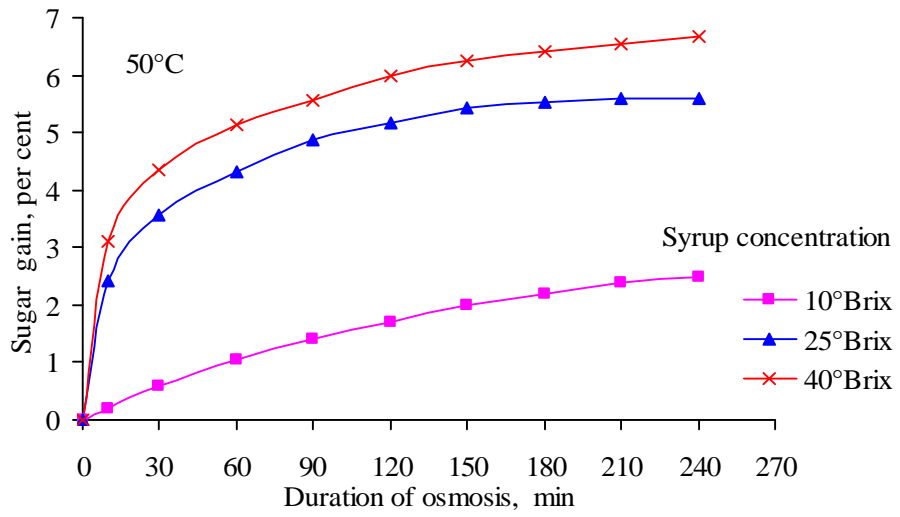
Fig. 5.10 also shows that a low temperature-low concentration condition (30°C-10°Brix) gives a low sugar gain (1.65% after 4 h of osmosis) and a high temperature-high concentration conditions (50°C-40°Brix) gives a higher sugar gain (6.66% after 4 h of osmosis). The low temperature-high concentration condition 30°C-40°Brix gives a higher sugar gain of 5.34 per cent after 4 h of osmosis than high temperature low concentration i.e. 50°C-10°Brix (2.49 % after 4 h of osmosis) indicating a pronounced effect of concentration on sugar gain. This indicates that sugar gain can be increased by increasing either the syrup concentration or temperature of solution. However, an increase in concentration of sugar solution has more influence on sugar gain (Fig. 5.10) than an increase in temperature in the study range (Fig. 5.9).





**Fig. 5.9 Variation in sugar gain with temperature at 10, 25, 40 °Brix concentration**





**Fig. 5.10** Variation in sugar gain with concentration at 30, 40 and 50°C temperatures

### 5.4.3 Osmotic dehydration kinetics

Mass transfer during osmotic dehydration process occurs through the semi-permeable cell membranes that offer the dominant resistance to mass transfer in biological materials. The changes in the state of the cell membrane may cause significant variations in the diffusion coefficients of water through the biological materials, as the cell membrane properties change partially or totally, leading to significant changes in tissue architecture (Rastogi *et al.*, 2002).

#### 5.4.3.1 Water diffusion coefficients during osmotic dehydration

Osmotic dehydration is actually a combination of simultaneous water and sugar diffusion process, when cellular solid (aloe vera gel sample) containing water is immersed in concentrated sugar syrup. The amount of water loss ( $M_{w\theta}$ ) from aloe vera gel sample after time  $\theta$  when it has been immersed into syrup was determined periodically as explained in section 4.3. The data for water loss at various syrup concentrations and temperatures for different duration of osmotic dehydration have been presented in Appendix E.

In all the experiments the amount of water loss from the aloe vera gel samples was found to increase with concentration and temperature of syrup. The quantities of water loss ( $M_{w\theta}$ ) was calculated using Eqn. (4.2) with respect to duration of osmosis. The maximum possible amount of water loss ( $M_{w\infty}$ ), which can be removed from the sample was obtained using Eqn. (4.8). As the osmotic dehydration of the aloe vera samples has been carried out in batches and the process last for several minutes, the values of  $D_w\theta/l^2$  is large enough to neglect the values of  $n > 1$  in Eqn. (4.22), which may be simplified as

$$\ln\left(1 - \frac{M_{w\theta}}{M_{w\infty}}\right) = \ln\frac{2\alpha_w(1+\alpha_w)}{1+\alpha_w+\alpha_w^2q_w^2} - \frac{3D_wq_w^2\theta}{\ell^2} \quad \dots 5.10$$

The values of  $M_{w\theta}$ ,  $M_{w\infty}$  and  $\alpha_w$  are presented in Appendix E. Eqn. (5.10) is rearranged in the form of a straight line equation as

$$\ln\left(1 - \frac{m_{w\theta}}{m_{w\infty}}\right) = a\theta + b \quad \dots 5.11$$

Where,  $\theta$  is time in min and a and b are constants. The constants a and b can be determined by comparing the coefficients of Eqns. (5.10) and (5.11) as

$$a = -\frac{3D_wq_w^2}{\ell^2} \quad \dots 5.12$$

$$b = \ln\frac{2\alpha_w(1+\alpha_w)}{1+\alpha_w+\alpha_w^2q_w^2} \quad \dots 5.13$$

The curves were plotted between  $\ln(1 - M_{w\theta}/M_{w\infty})$  and duration of osmotic dehydration ( $\theta$ ) to determine the slope of Eqn (5.11) (*i.e.* a) and to find the water diffusion coefficient ( $D_w$ ) from Eqn. (5.12). The values of  $\alpha_w$  were determined experimentally from Eqn. (4.18). The values of  $q_w$  as taken from Crank (1975) by interpolation for various concentrations and temperatures of syrup are also presented in the Appendix E.

The plot between  $\ln(1 - M_{w\theta}/M_{w\infty})$  and time yielded straight line as shown in Fig. 5.11. The slopes of these straight lines were used to determine water diffusion coefficient for various concentrations and temperatures of syrup by using the Eqn. (5.12). The variation in water diffusion coefficient with syrup concentration and temperature in the experimental range of study are presented in Table 5.9. It can be seen from the table that the diffusion coefficient for water varied from  $4.2559 \times 10^{-9}$  to  $7.1247 \times 10^{-9} \text{ m}^2/\text{s}$ , at various syrup concentration and temperatures.

**Table 5.9 Variation in water diffusion coefficient with syrup concentration and temperature**

Syrup concentration, °Brix	Syrup temperature, °C	Diffusion coefficient for water, $D_w \times 10^9 \text{ m}^2/\text{s}$
10	30	4.8448
10	40	5.9848
10	50	7.1247
25	30	4.4550
25	40	5.5687
25	50	6.1256
40	30	4.2559
40	40	5.0539
40	50	5.5858

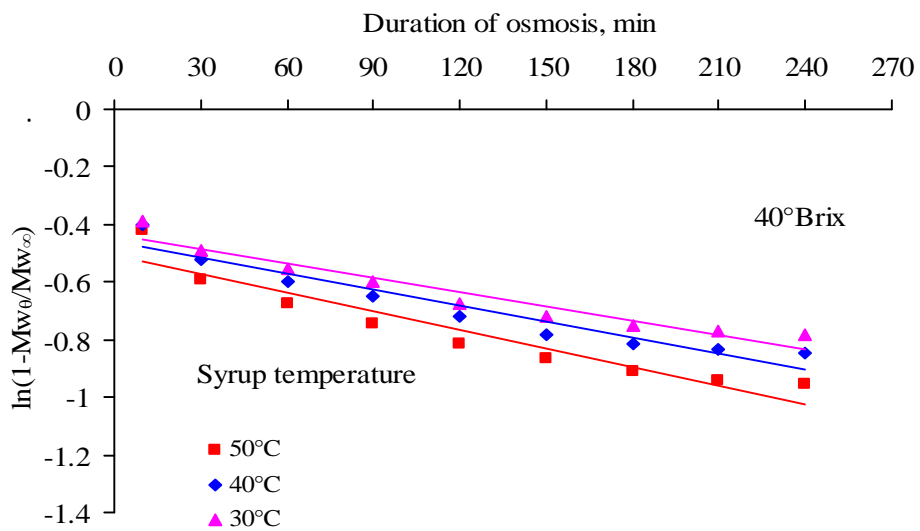
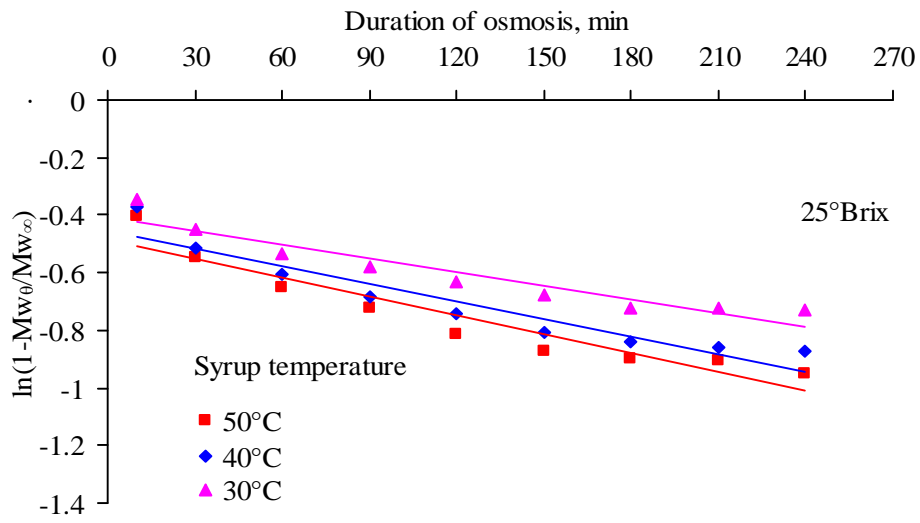
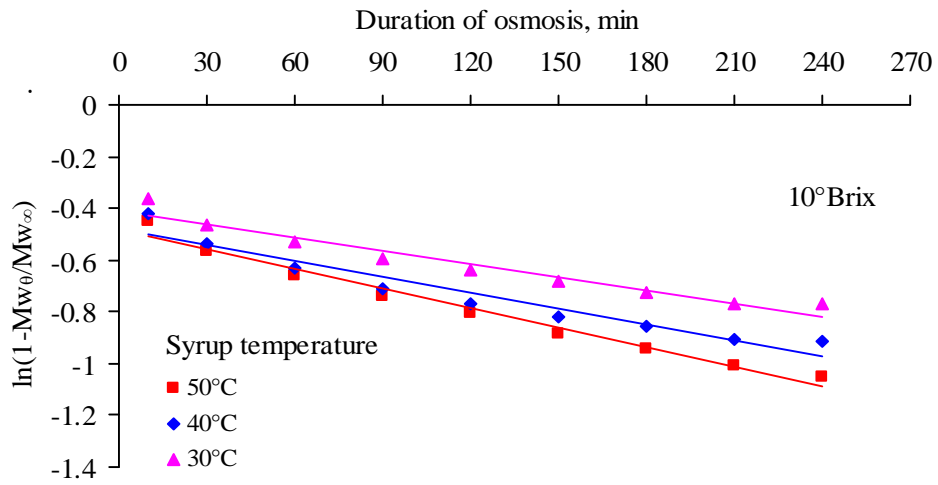
The influence of syrup concentration and syrup temperature on water diffusivity was correlated as follows:

$$D_w = 7.616 \times 10^{-10} \times C^{-0.128} T^{0.64} \quad \dots 5.14$$

$$R^2 = 0.976$$

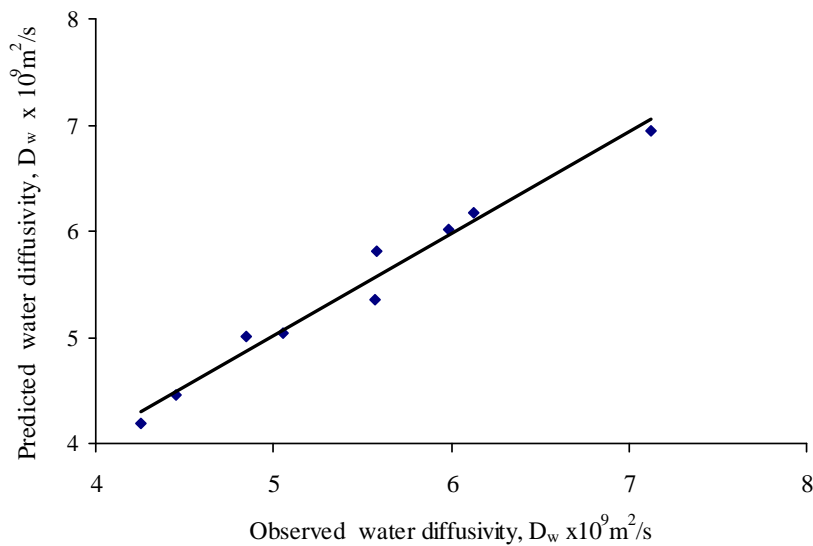
Where,  $D_w$  is water diffusivity in  $\text{m}^2/\text{s}$ , C is syrup concentration in °Brix and T is the syrup temperature in °C.

Eqn. (5.14) suggested that the water diffusivity increased with decreasing syrup concentration and increasing syrup temperatures. The effect of temperature was found more at low syrup concentration. The observed values of water diffusivity ( $D_w$ ) of aloe vera gel sample were comparable with those given by Gracia Segovia *et al.*, (2010) *i.e.* 1.9 to  $19.8 \times 10^{-9} \text{ m}^2/\text{s}$  for peeled and unpeeled aloe vera and Falade *et al.*, (2007) *i.e.* 3.5 to  $10.3 \times 10^{-9} \text{ m}^2/\text{s}$  for water melon.



**Fig. 5.11 Variation in  $\ln(1 - M_{w\theta}/M_{w\infty})$  and time**

The water diffusivity during the osmotic dehydration of aloe vera samples at various syrup concentrations and temperatures as determined experimentally (Table 5.9) and predicted by Eqn. 5.14 are shown in Fig. 5.12. It can be seen from the figure that there is a good co-relation between the observed and the predicted values of water diffusivities with  $R^2$ , 0.974.



**Fig. 5.12 Experimental and predicted values of water diffusivity**

#### **5.4.3.2 Sugar diffusion coefficients during osmotic dehydration**

The amount of sugar gain ( $M_{s\theta}$ ) by aloe vera gel samples when immersed into syrup after time  $\theta$  was determined periodically as explained in section 4.3. The values of sugar gain determined for various syrup concentrations and temperatures for different duration of osmotic dehydration are presented in Appendix E.

In all the experiments the amount of sugar gain from syrup to aloe vera gel sample was found to increase with syrup concentrations and temperatures. The amount of sugar gain ( $M_{s\theta}$ ) was calculated using the Eqn. (4.3) with respect to duration of osmosis. The maximum possible sugar gain ( $M_{s\infty}$ ) was determined using the Eqn. (4.11). As the osmotic dehydration

of the aloe vera gel samples has been carried out in batches and the process lasts for several minutes the values of  $D_s \theta/\ell^2$  are large enough to neglect the values of  $n > 1$  in Eqn. (4.23), which may be simplified as

$$\ln\left(1 - \frac{M_{s\theta}}{M_{s\infty}}\right) = \ln \frac{2\alpha_s(1 + \alpha_s)}{1 + \alpha_s + \alpha_s^2 q_s^2} - \frac{3D_s q_s^2 \theta}{\ell^2} \quad \dots 5.15$$

The above equation is rearranged in the form of a straight line equation as

$$\ln\left(1 - \frac{m_{s\theta}}{m_{s\infty}}\right) = a\theta + b \quad \dots 5.16$$

Where  $\theta$  is time in min and  $a$  and  $b$  are constants. The values of  $a$  and  $b$  can be determined by comparing coefficients of Eqn. (5.15) and (5.16) as

$$a = (-) \frac{3D_s q_s^2}{\ell^2} \quad \dots 5.17$$

$$b = \ln \frac{2\alpha_s(1 + \alpha_s)}{1 + \alpha_s + \alpha_s^2 q_s^2} \quad \dots 5.18$$

The curves were plotted between  $\ln(1 - M_{s\theta}/M_{s\infty})$  and duration of osmotic dehydration to determine the slope of Eqn. (5.16) (*i.e.*  $a$ ) and to find the sugar diffusion coefficients ( $D_s$ ) with the help of Eqn. (5.17). The values of  $\alpha_s$  are determined experimentally using Eqn. (4.19), whereas the values of  $q_s$  are taken from Crank (1975) by interpolation and both are given in Appendix E.

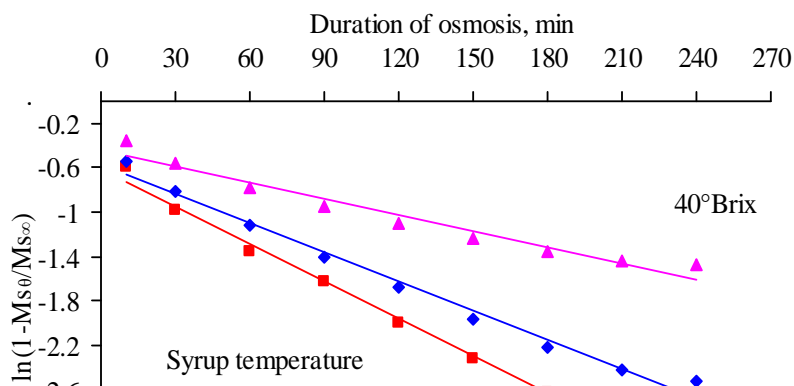
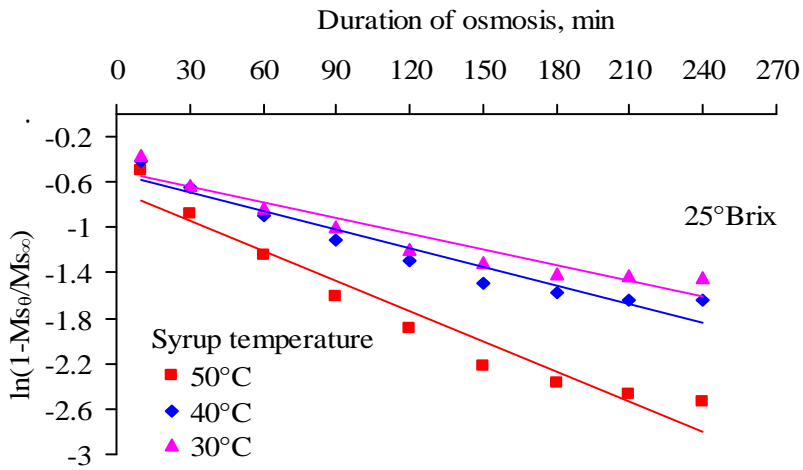
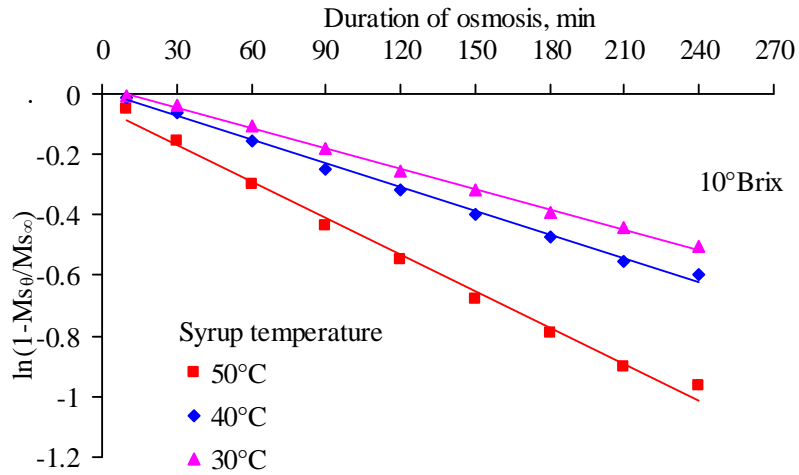
The plot between  $\ln(1 - M_{s\theta}/M_{s\infty})$  and time yielded straight line as shown in Fig. 5.13. The slopes of these equations were used to determine the sugar diffusion coefficients for various concentrations and temperatures of sugar syrup using the Eqn. (5.17).

The sugar diffusion coefficients at various concentrations and temperatures are presented in Table 5.10. It can be seen from the table that the diffusion coefficient for sugar varied from  $0.7430 \times 10^{-8}$  to  $3.7826 \times 10^{-8} \text{ m}^2/\text{s}$ , respectively at various syrup concentrations and temperatures. These observed values of sugar diffusion coefficients were comparable with those given by Falade *et al.*, (2007).

**Table 5.10 Variation in sugar diffusion coefficients with syrup concentration and temperature**

Syrup concentration, °Brix	Syrup temperature, °C	Sugar diffusion coefficient, $D_s \times 10^8 \text{ m}^2/\text{s}$
10	30	0.7430
10	40	0.8781
10	50	1.3509

25	30	1.5536
25	40	1.8576
25	50	3.0059
40	30	1.6211
40	40	2.9721
40	50	3.7826



**Fig. 5.13 Variation in  $\ln(1 - M_{s\theta}/M_{s\infty})$  and time**

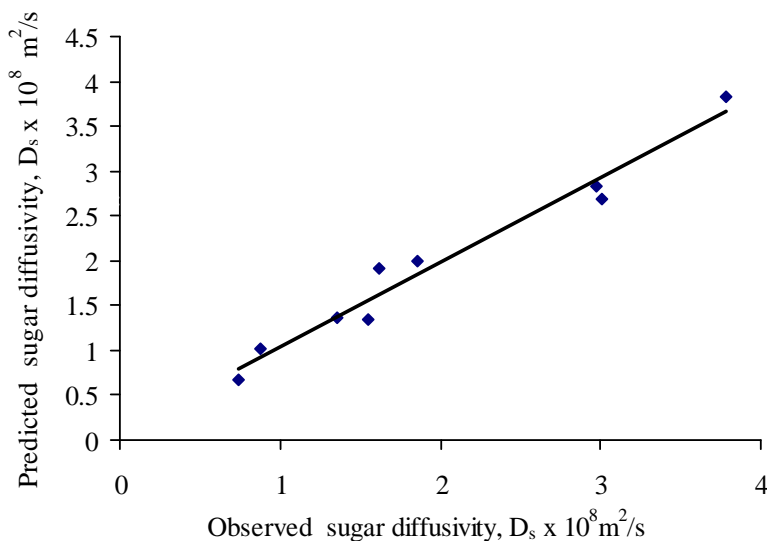
The influence of syrup concentration and temperature on sugar diffusivity during osmotic dehydration was also established as

$$D_s = 1.2 \times 10^{-11} \times C^{0.743} T^{1.361} \quad \dots 5.19$$

$$R^2 = 0.962$$

Where,  $D_s$  is sugar diffusivity in  $m^2/s$ ,  $C$  is syrup concentration in  $^\circ\text{Brix}$  and  $T$  is the syrup temperature in  $^\circ\text{C}$ . Eqn. (5.19) suggested that the sugar diffusivity increased with increase in syrup concentration and temperature.

The sugar diffusivity during the osmotic dehydration of aloe vera gel samples at various syrup concentrations and temperatures as determined experimentally (Table 5.10) and predicted by Eqn. (5.19) are shown in Fig. 5.14. It can be seen from the figure that there is a good co-relation ( $R^2 = 0.968$ ) between the observed and the predicted values of sugar diffusivities.



**Fig. 5.14 Experimental and predicted values of sugar diffusivities**

**5.4.3.3 Activation energy in osmotic dehydration**

The variation in water and sugar diffusion coefficients with syrup concentration and temperature in the experimental range of study are presented in the Table 5.9 and 5.10. It can be seen from the tables that the diffusion coefficient for water varied from  $4.2559 \times 10^{-9}$  to  $7.1247 \times 10^{-9} \text{ m}^2/\text{s}$  and for sugar,  $0.7430 \times 10^{-8}$  to  $3.7826 \times 10^{-8} \text{ m}^2/\text{s}$  at various syrup concentrations and temperatures.

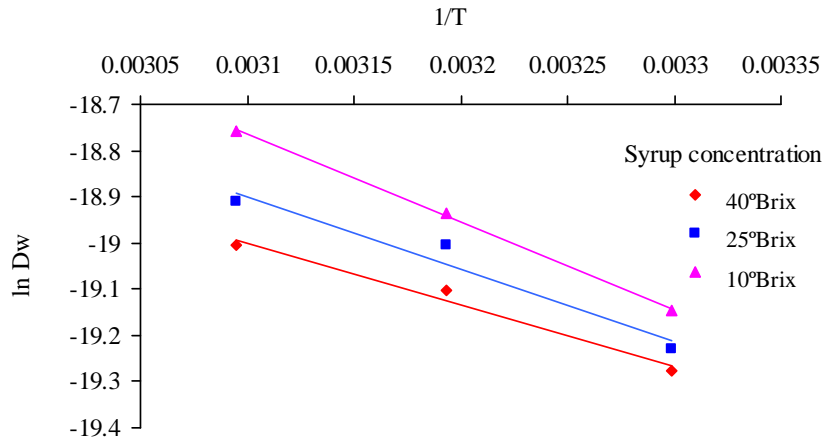
If the values of water and sugar diffusion coefficients are plotted as a function of reciprocal of absolute temperature, (1/T), exponential curves are obtained. Each curve represents the form of Arrhenius equation as

$$D_w = D_{wo} e^{-\frac{E_a}{RT}} \quad \text{and} \quad \dots 5.20$$

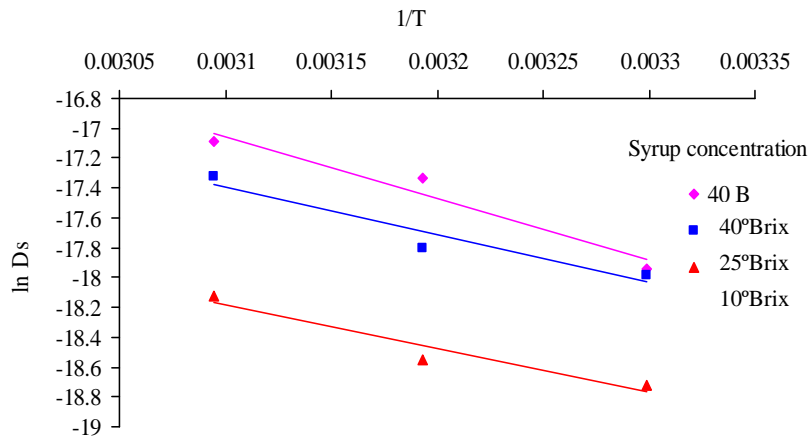
$$D_s = D_{so} e^{-\frac{E_a}{RT}} \quad \dots 5.21$$

Where,  $D_{wo}$  and  $D_{so}$  are water and sugar diffusion coefficient at initial temperature,  $\text{m}^2/\text{s}$ ,  $E_a$  is energy of activation in  $\text{kJ}/\text{mol}$  and  $R$  is gas constant in  $\text{kJ}/\text{mol K}$ .

If the natural logarithm of values of water diffusion coefficient ( $\ln D_w$ ) and sugar diffusion coefficients ( $\ln D_s$ ) are plotted against inverse absolute temperature (1/T), the curves obtained were found to be essentially a straight line in whole range of considered temperatures (Fig 5.15 and 5.16). Activation energy was obtained from the slope of curves and shown in Table 5.11 and Table 5.12 for water and sugar diffusion respectively. It could be seen from the table that activation energy for water diffusion decreased, but increased for sugar diffusion, with increase in syrup concentration. This clearly explained that more amount of energy was required for sugar diffusion in higher concentration of syrup, but a reverse trend was observed in water diffusion process (Pokharkar, 1994 and Jain, 2007).



**Fig.5.15 Variation in water diffusion coefficient with syrup temperature**



**Fig. 5.16 Variation in sugar diffusion coefficient with syrup temperature**

**Table 5.11 Activation energies for water diffusion**

Sugar syrup concentration, °Brix	Slope of Arrhenius plot	R, kJ / mol K	Activation energy, kJ/mol
10	-1890.3	8.314	15.72
25	-1566	8.314	13.02
40	-1335.3	8.314	11.10

**Table 5.12 Activation energies for sugar diffusion**

Sugar syrup concentration, °Brix	Slope of Arrhenius plot	R, kJ / mol K	Activation energy, kJ/mol
10	-2913.6	8.314	24.22
25	-3215.9	8.314	26.74
40	-4167.8	8.314	34.65

### 5.5 Optimization of Osmotic Dehydration Process

The optimization of osmotic dehydration process parameters such as syrup concentration, syrup temperature and duration of osmotic dehydration is necessary so that maximum water loss could be achieved with optimum sugar gain. During the preliminary experiments it was found that water loss and sugar gain during osmotic dehydration process depends on temperature and concentration of syrup and duration of osmosis. However, equilibrium could not be achieved even after 10 h of osmotic dehydration. It is desired that water loss should be maximum with optimum sugar gain of 4.59 per cent as explained in

Section 5.3.5. Therefore, keeping these factors in mind osmotic dehydration process was optimized.

As per 3 variable 3 level Box Behnken model, seventeen trials were performed as enumerated in Table 5.13 for obtaining the water loss and sugar gain as responses for each treatment. All these trials were replicated thrice and the average of the experimental data for mass reduction, water loss and sugar gains are reported. The aloe vera gel samples of 1cm size were fully immersed in the sugar syrup kept in a beaker having syrup to aloe vera gel sample ratio of 5:1 (w/w).The samples were removed from the bath at the designated time and blotted with blotting paper. These samples were then weighed for moisture content determination. The water loss and sugar gain were also calculated. To avoid bias, the 17 runs were performed in a random order for estimation of the constants of Eqn (3.7). The decision for the range and centre points of the variables was taken through preliminary trials. The independent variables, *i.e.* syrup temperature, concentration and duration of osmosis, the coded variables ( $x_i$ ), decoded variables and their levels are presented in Table 5.13, as described by Pokharkar, (1994); Chaudhary *et al.*, (2000); Ravindra and Chattopadhyay, (2000); Jain, (2007); Singh *et al.*, (2008) and Ranmode, (2009).

Table 5.13 Experimental layout for three-variables and three levels response surface analysis

Treatment No.	Syrup temp., °C	Syrup conc. °Brix	Duration of osmosis, h	Syrup temp. °C	Syrup conc. °Brix	Duration of osmosis, min
	$x_1$	$x_2$	$x_3$	T	C	$\theta$
1	1	1	0	50	40	150
2	1	-1	0	50	10	150
3	-1	1	0	30	40	150
4	-1	-1	0	30	10	150
5	1	0	1	50	25	240
6	1	0	-1	50	25	60
7	-1	0	1	30	25	240
8	-1	0	-1	30	25	60
9	0	1	1	40	40	240
10	0	1	-1	40	40	60
11	0	-1	1	40	10	240
12	0	-1	-1	40	10	60
13	0	0	0	40	25	150
14	0	0	0	40	25	150

15	0	0	0	40	25	150
16	0	0	0	40	25	150
17	0	0	0	40	25	150

The initial and final mass of aloe vera gel samples, initial and final moisture content of aloe vera gel samples during osmotic dehydration experiments and mass reduction in per cent have been presented in Table 5.14. It can be seen from the table that the initial mass of the aloe vera samples taken in the study were in the range of 50.10-50.77 g. The final mass after osmotic dehydration was in the range of 27.81-35.58 g showing mass reduction in the range of 28.98 to 45.14 per cent in the experimental range of study. Similarly, initial moisture content of aloe vera gel samples was in the range of 98.13-99.16 % (wb) which was reduced to 86.24-96.57 % after osmotic dehydration in various experiments.

**Table 5.14 Experimental data for mass reduction under different treatment conditions**

S. No.	Syrup temperature, °C	Syrup concentration, °Brix	Duration of osmosis, min	Initial mass, g	Final mass, g	IMC (wb), %	FMC (wb), %	Mass reduction, %
1	50	40	150	50.29	27.86	98.62	86.24	44.60
2	50	10	150	50.33	30.61	99.01	95.11	39.19
3	30	40	150	50.24	30.14	98.61	89.47	40.00
4	30	10	150	50.37	33.88	98.80	96.54	32.74
5	50	25	240	50.28	28.10	98.91	88.03	44.11
6	50	25	60	50.19	32.92	98.91	91.73	34.41
7	30	25	240	50.38	31.51	98.13	89.78	37.46
8	30	25	60	50.12	35.03	98.13	92.56	30.11
9	40	40	240	50.69	27.81	99.16	87.82	45.14
10	40	40	60	50.77	32.54	99.16	92.00	35.90
11	40	10	240	50.46	32.26	98.22	94.00	36.07
12	40	10	60	50.10	35.58	98.22	96.57	28.98
13	40	25	150	50.72	31.39	98.27	89.21	38.11
14	40	25	150	50.40	31.05	98.27	88.91	38.39
15	40	25	150	50.41	31.14	98.27	88.95	38.23
16	40	25	150	50.39	31.11	98.27	88.48	38.26
17	40	25	150	50.35	31.09	98.27	88.39	38.25

### 5.5.1 Effect of variables on water loss

The variation in water loss by changing syrup temperature, concentration and osmosis duration has been presented in Table 5.15. A second order polynomial equation [Eqn. (3.8)] was fitted with the experimental data presented in Table 5.15. Eqn. (5.22) gives the predicted water loss, per cent as a function of syrup temperature ( $x_1$ ), concentration ( $x_2$ ) and duration of osmosis ( $x_3$ ) expressed in coded form. This equation was obtained using step-down regression method where factors with F-values less than one were rejected as described by Snedecor and Cochran (1967). The data for water loss were analysed for stepwise regression analyses as shown in Table 5.16. The quadratic model was fitted to the experimental data and statistical significance for linear, quadratic and interaction terms was calculated for water loss as shown in Table 5.16. The  $R^2$  value was calculated by least square technique and found to be 0.9966 showing good fit of model to the data. The model F value of 377.23 implies that the model is significant ( $P < 0.01$ ). The linear terms (T, C &  $\theta$ ) are significant ( $P < 0.01$ ). The lack of fit F value was non significant which indicates that the developed model was adequate for predicting the response. Moreover the predicted  $R^2$  of 0.9831 was in reasonable agreement with adjusted  $R^2$  of 0.9940. This revealed that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space.

**Table 5.15 Observed water loss under varying processing parameters**

S. No.	Syrup temperature, °C	Syrup concentration, °Brix	Duration of osmosis, min	Water loss, %
1	50	40	150	50.85
2	50	10	150	41.17
3	30	40	150	44.93
4	30	10	150	33.87
5	50	25	240	49.71
6	50	25	60	38.74
7	30	25	240	41.98
8	30	25	60	33.43
9	40	40	240	50.97
10	40	40	60	40.19
11	40	10	240	38.13
12	40	10	60	29.63
13	40	25	150	43.06
14	40	25	150	43.50
15	40	25	150	43.32

16	40	25	150	43.64
17	40	25	150	43.69

High value of coefficient of determination ( $R^2 = 0.9966$ ) obtained for response variable indicated that the developed model for water loss accounted for and adequately explained 99.66 per cent of the total variation. The result of analysis of variance of Eqn. (5.22) indicated that the linear terms of syrup temperature, syrup concentration and duration of osmosis were highly significant at 1 % level (Table 5.16). The presence of quadratic terms of concentration of syrup and duration of osmosis indicated curvilinear nature of response surface. The quadratic terms of concentration of syrup and duration of osmosis were also highly significant at 1 per cent level while interaction terms of temperature of syrup and duration of osmosis as well as concentration of syrup and duration of osmosis were significant at 5 per cent level.

The comparative effect of each factor on water loss was observed by the F values in the ANOVA (Tables 5.16) and also by the magnitudes of coefficients of the coded variables. The F values indicated that concentration of syrup was the most influencing factor followed by duration of osmosis and temperature of syrup was least effective over water loss.

**Table 5.16 Analysis of variance for water loss during osmotic dehydration of aloe vera gel**

Source	Sum of squares	df	Mean sum of squares	F value
Model	557.27	7	79.61	377.23**
T	86.12	1	86.12	408.09**
C	243.81	1	243.81	1155.30**
$\theta$	188.13	1	188.13	891.45**
T $\theta$	1.47	1	1.47	6.94*
C $\theta$	1.32	1	1.32	6.24*
$C^2$	3.99	1	3.99	18.92**
$\theta^2$	31.08	1	31.08	147.26**
Residual	1.90	9	0.21	
Lack of Fit	1.63	5	0.33	4.90 <sup>NS</sup>
Pure Error	0.27	4	0.067	
Cor Total	559.17	16		
$R^2$	0.9966			
Adj. $R^2$	0.9940			
Pred. $R^2$	0.9831			

\*\* Significant at 1 % Level

\* Significant at 5 % Level

NS - Non significant

The regression equation describing the effects of process variables on water loss in terms of coded values of variable is given as

$$WL = 43.55 + 3.28x_1 + 5.52x_2 + 4.85x_3 + 0.61x_1x_3 + 0.57x_2x_3 - 0.97x_2^2 - 2.71x_3^2 \dots 5.22$$

$$R^2 = 0.9966$$

Replacing  $x_1$ ,  $x_2$  and  $x_3$  with  $(T-40)/10$ ,  $(C-25)/15$  and  $(\theta -150)/90$  respectively [Eqn. (3.5)] in Eqn. (5.22), the water loss in real terms of syrup temperature, concentration and duration of osmosis can be given by

$$WL = 8.53 + 0.23T + 0.52C + 0.12\theta + 6.73 \times 10^{-4}T\theta + 4.25 \times 10^{-4}C\theta - 4.32 \times 10^{-3}C^2 - 3.35 \times 10^{-4}\theta^2 \dots 5.23$$

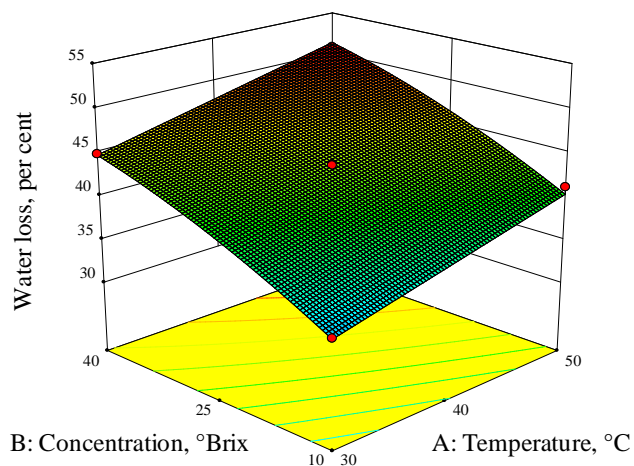
The linear positive terms [Eqn. (5.22)] indicated that water loss increased with increase in syrup temperature, syrup concentration and duration of osmosis. The presence of positive interaction terms between syrup temperature and duration of osmosis as well as syrup concentration and duration of osmosis indicated that increase in their levels increased water loss. The negative values of quadratic terms of syrup concentration and duration of osmosis indicated that higher values of these variables further reduced water loss.

To visualize the combined effect of two variables on the water loss, the response surface and contour plots (Fig 5.17 A, B and C) were generated for the fitted model as a function of two variables while keeping third variable at its central value. The water loss increased rapidly in the early stages of osmosis, after which the rate of water loss from aloe vera gel sample into sugar syrup gradually slowed down with time. Rapid removal of water in early stages of osmosis has been reported for apple (Conway *et al.*, 1983), carrots (Uddin *et al.*, 2004), etc.

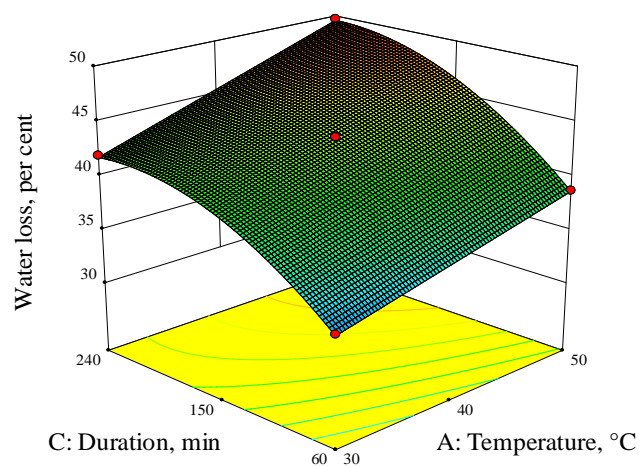
Higher temperatures seem to promote faster water loss (Fig. 5.17 A and B) through swelling and plasticizing of cell membranes as well as the better water transfer characteristics on the product surface due to lower viscosity of the osmotic medium (Conteras, 1981; Uddin *et al.*, 2004).

Water loss increased with concentration of syrup (Fig 5.17 A and C) as well as with duration of osmosis (Fig 5.17 B and C) over the entire osmotic dehydration process. In the

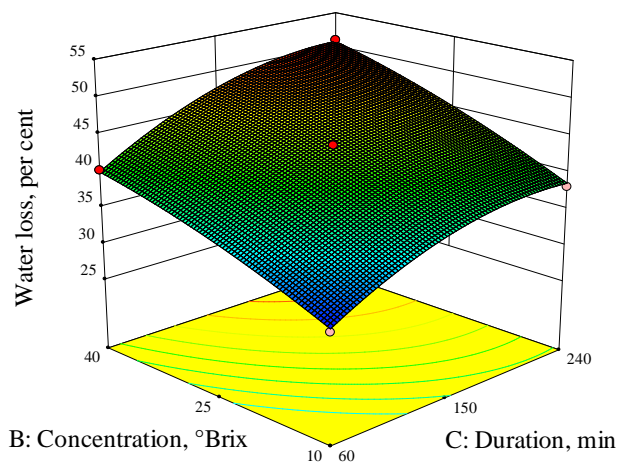
osmosis of other fruits and vegetables, also such effect has been observed (Ponting *et al.*, 1966; Hawkes and Flink 1978; Beristain *et al.*, 1990; Biswal and Bozorgmehr 1991; Ertekin and Cakaloz, 1996 and Pokharkar and Prasad, 1998 a and b).



**(A) At 150 min duration of osmosis**



**(B) At 25°Brix syrup concentration**



**(C) At 40 °C syrup temperature**

**Fig 5.17 The contour and response surface showing the effect of temperature, concentration and duration on water loss during osmotic dehydration**

### 5.5.2 Effect of variables on sugar gain

The sugar gain during the osmotic dehydration was found to be dependent on the syrup temperature, concentration and duration of osmosis (Table 5.17).

A second order polynomial equation [Eqn. (3.7)] was fitted with the experimental data presented in Table 5. 17. Eqn. (5.24) gives the predicted sugar gain, per cent as a function of syrup temperature ( $x_1$ ), concentration ( $x_2$ ) and duration of osmosis ( $x_3$ ) expressed in coded form. This equation was obtained using step-down regression method where factors with F-values less than one were rejected as described by Snedecor and Cochran (1967). The data for sugar gain were analyzed for stepwise regression analysis as shown in Table 5.18. The quadratic model was fitted to the experimental data and statistical significance for linear and quadratic terms was calculated for sugar gain as shown in Table 5.18. The  $R^2$  value was calculated by least square technique and found to be 0.9932 showing good fit of model to the data. The model F value of 243.48 implies that the model is significant ( $P < 0.01$ ). The linear terms (T, C and  $\theta$ ) are significant ( $P < 0.01$ ). The lack of fit F value was non significant, which indicates that the developed model was adequate for predicting the response. Moreover the predicted  $R^2$  of 0.9819 was in reasonable agreement with adjusted  $R^2$  of 0.9891. This revealed that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space.

High value of coefficient of determination ( $R^2=0.9932$ ) obtained for response variable indicated that the developed model for sugar gain accounted for and adequately explained 99.32 % of the total variation.

**Table 5.17 Observed sugar gain under varying processing parameters**

S. No.	Syrup temperature, °C	Syrup concentration, °Brix	Duration of osmosis, min	Sugar gain, %
1	50	40	150	6.25
2	50	10	150	1.98
3	30	40	150	4.93
4	30	10	150	1.13
5	50	25	240	5.60
6	50	25	60	4.33
7	30	25	240	4.52
8	30	25	60	3.33
9	40	40	240	5.84
10	40	40	60	4.29
11	40	10	240	2.05
12	40	10	60	0.65
13	40	25	150	4.95
14	40	25	150	5.10
15	40	25	150	5.10
16	40	25	150	5.38
17	40	25	150	5.44

The result of analysis of variance indicated that the linear terms of syrup temperature, syrup concentration and duration of osmosis were highly significant at 1 per cent level (Table 5.18). The presence of quadratic terms of concentration of syrup and duration of osmosis indicated curvilinear nature of response surface. The quadratic terms of concentration of syrup and duration of osmosis were also highly significant at 1 per cent level while quadratic term of temperature was non significant.

The comparative effect of each factor on sugar gain could be observed by F values in the ANOVA (Table 5.18) and also by the magnitudes of the coded variables. The F values indicated that concentration of syrup was the most influencing factor followed by duration of osmosis and temperature of syrup was least effective over sugar gain.

**Table 5.18 Analysis of variance for sugar gain during osmotic dehydration of aloe vera gel**

Source	Sum of squares	df	Mean sum of squares	F value
Model	46.56	6	7.76	243.48**
T	2.25	1	2.25	70.75**
C	29.97	1	29.97	940.43**
$\theta$	3.66	1	3.66	114.86**
$T^2$	0.16	1	0.16	4.93 <sup>NS</sup>
$C^2$	8.59	1	8.59	269.55**
$\theta^2$	1.31	1	1.31	41.08**
Residual	0.32	10	0.032	
Lack of Fit	0.14	6	0.024	0.55 <sup>NS</sup>
Pure Error	0.17	4	0.044	
Cor Total	46.88	16		
$R^2$	0.9932			
Adj. $R^2$	0.9891			
Pred. $R^2$	0.9819			
C.V. %	4.28			

\*\* Significant at 1% level

NS - Non significant

The regression equation describing the effects of process variables on sugar gain in terms of coded values of variables is given as

$$SG = 5.19 + 0.53x_1 + 1.94x_2 + 0.68x_3 - 0.19x_1^2 - 1.43x_2^2 - 0.56x_3^2 \quad \dots 5.24$$

$$R^2 = 0.9932$$

Replacing  $x_1$ ,  $x_2$  and  $x_3$  with  $(T-40)/10$ ,  $(C-25)/15$  and  $(\theta - 150)/90$  respectively [Eqn. (3.5)] in Eqn. (5.24), the sugar gain in real terms of syrup temperature, concentration and duration of osmosis is given by

$$SG = -9.89 + 0.21T + 0.45C + 0.03\theta - 1.93 \times 10^{-3} T^2 - 6.35 \times 10^{-3} C^2 - 6.89 \times 10^{-5} \theta^2$$

...5.25

The linear positive terms [Eqn. (5.24)] indicated that sugar gain increased with increase in syrup temperature, syrup concentration and duration of osmosis. The negative values of quadratic terms of temperature of concentration of syrup and duration of osmosis indicated that higher values of these variables further reduced sugar gain.

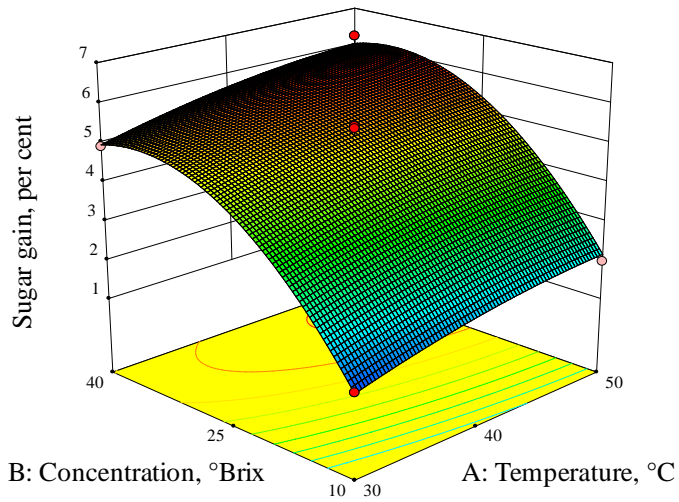
To visualize the combined effect of two variables on the sugar gain, the response surface and contour plots (Fig 5.18 A, B and C) were generated for the fitted model as a function of two variables while keeping third variable at its central value.

The sugar gain increased rapidly in the early stages of osmosis after which the rate of sugar gain from sugar syrup to aloe vera gel sample slowed down with time.

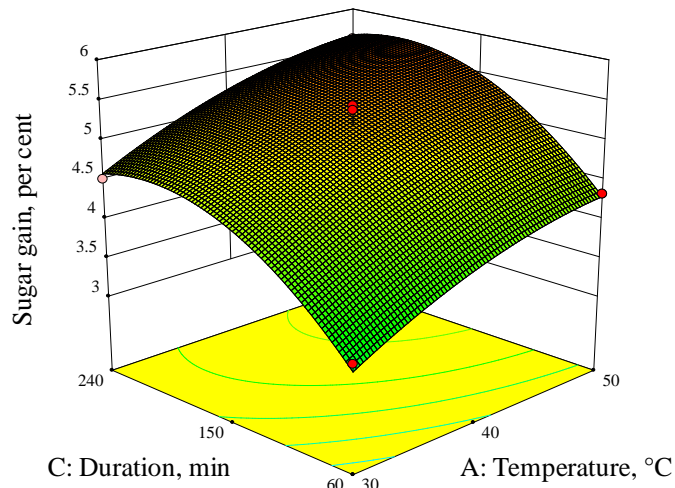
The sugar gain was found to increase with temperature (Fig 5.18 A and B). As it was explained for water loss, temperature has an effect on the cell membrane permeability that could allow solute to enter by loosing its selectivity. Decrease of solution viscosity at higher temperature may influence sugar gain due to fact that lower viscosity decreases the resistance to diffusion of solutes into the sample (food product) tissue.

Increased concentration of the sugar syrup also led to increase in sugar gain (Fig 5.18 A and C) probably due to an increase of osmotic pressure gradient and consequent loss of functionality of cell plasmatic membrane that allows solute to enter.

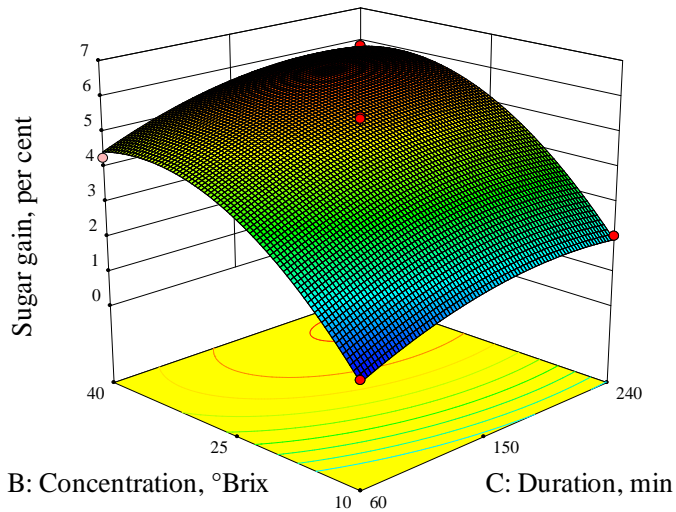
It was observed from Fig 5.17 and 5.18 that the moisture loss as well as the solid gain increased non linearly with time at all concentrations. Both moisture loss and solid gain were faster in the initial period of osmosis and then the rate decreased. This was because osmotic driving potential for moisture as well solid transfer will keep on decreasing with time as the moisture keeps moving from sample to solution and the solids from solution to sample. Further progressive solid uptake would result in the formation of high solid sub surface layer, which would interface with the concentration gradients across the sample solution interface and would set as barrier against removal of water and uptake of solid (Hawkes and Flink, 1978). Besides, rapid loss of water and uptake of solids near the surface in the beginning may result in structural changes leading to compaction of this surface layers and increased mass transfer resistance for water and solids (Lenart and Flink 1984). Similar trends have been reported for other fruits and vegetables during osmosis (Conway, *et al.*, 1983; Lazarides *et al.*, 1995; Ertekin and Cakaloz, 1996b; Ghosh *et al.*, 2006).



**(A) At 150 min duration of osmosis**



**(B) At 25 °Brix syrup concentration**



**(C) At 40 °C syrup temperature**

**Fig 5.18 The contour and response surface showing the effect of temperature, concentration and duration on sugar gain during osmotic dehydration**

### 5.5.3 Optimization of osmotic dehydration of aloe vera gel

Numerical multiresponse optimization technique was carried out for the process parameters of the osmotic dehydration of aloe vera gel. To perform this operation, Design expert version 8.0.4.1 of the STAT-EASE software (Statease Inc, Minneapolis, USA, Trial version), as discussed in section 3.5.5 was used for simultaneous optimization of the multiple responses.

The constraints were set such that the selected variables (T, C and  $\theta$ ) would be minimum from economical point of view for the most important product attribute and close to the optimum for the others (Jain *et al.*, 2011). The main criteria for constraints optimization were maximum possible water loss and targeted sugar gain of 4.59 percent as most important quality (sweetness) attribute (Themelin *et al.*, 1997; Ade-Omowaye *et al.*, 2002). The desired goals for each factor and response are shown in Table 5.19. In order to optimize the process parameters for osmotic dehydration process by numerical optimization which finds a point that maximizes the desirability function; equal importance of '3' was given to all the 3 process parameters and 2 responses. The goal setting begins at a random starting point and proceeds up the steepest slope on the response surface for a maximum value of water loss and targeted value of sugar gain.

**Table 5.19 Optimization criteria for different process variables and responses for osmotic dehydration of aloe vera gel**

Parameter	Goal	Lower limit	Upper limit	Importance
Temperature, °C	minimize	30	50	3
Concentration, °Brix	minimize	10	40	3
Duration, min	minimize	60	240	3
Water loss, percent	maximize	29.63	50.98	3
Sugar gain, percent	target = 4.59	0.65	6.25	3

Table 5.20 shows the software generated optimum conditions of independent variables with the predicted values of responses.

**Table 5.20 Solution generated by the software for osmotic dehydration of aloe vera gel**

No.	Temperature, °C	Concentration, °Brix	Duration, min	Water loss, %	Sugar gain, %
1	31.44	25.35	128.73	39.69	4.45*

\*Selected

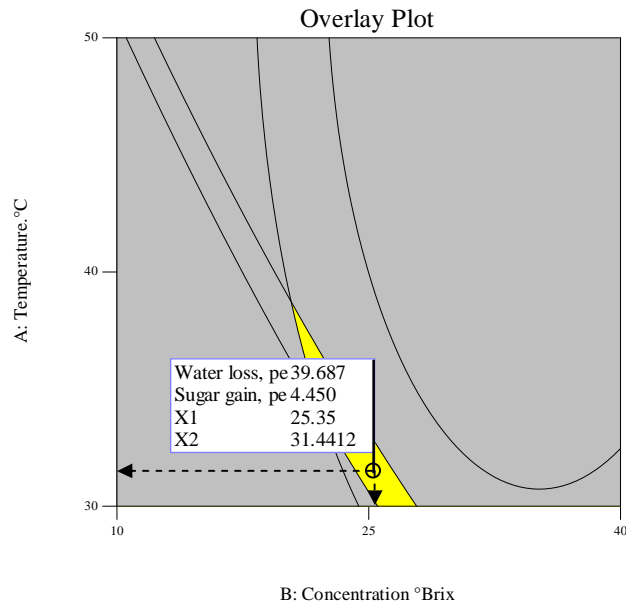
The optimum values of process variables obtained by numerical optimization:

Temperature of syrup, °C : 31.44 ≈ 31  
Concentration of syrup, °Brix : 25.35 ≈ 25  
Duration of osmosis, min : 128.73 ≈ 129

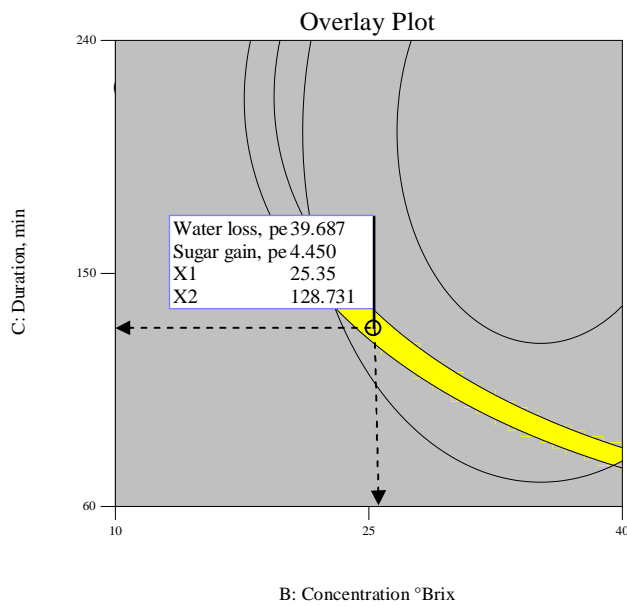
A graphical multiresponse optimization technique was adapted to determine the workable optimum conditions for the osmotic dehydration of aloe vera gel. The contour plots for all responses were superimposed and regions that best satisfy all the constraints were selected as optimum conditions. The criteria for constraint optimization are already given in Table 5.19. These constraints resulted in feasible zone of the optimum solutions (yellow coloured area in the superimposed contour plots). Superimposed contour plots having common superimposed area for all responses for osmotic dehydration of aloe vera gel are shown in Fig. 5.19.

The superimposed contours of all responses for C and T (Fig 5.19 A) and C and  $\theta$  (Fig 5.19 B) and their intersection zone for maximum water loss and targeted sugar gain (4.59%) indicated the range of optimum values of process variables as follows,

Temperature of syrup (T) : 30-38 °C  
Concentration of syrup (C) : 18-29 °Brix  
Duration of osmosis ( $\theta$ ) : 77-150 min



**(A) At duration of osmosis = 128.73 min**



**(B) At concentration of syrup = 25.35 °Brix**

**Fig 5.19 Superimposed contours for water loss (%) and sugar gain (%) for osmotic dehydration of aloe vera gel at varying (A) concentration of syrup and temperature of syrup and (B) concentration of syrup and duration of osmosis**

#### **5.5.4 Verification of the model for osmotic dehydration of aloe vera gel**

Osmotic dehydration experiments were conducted at the optimum process conditions ( $x_1 = 31$  °C,  $x_2 = 25$  °Brix and  $x_3 = 129$  min) for testing the adequacy of model equations for predicting the response values.

**Table 5.21 Predicted and experimental values of response at optimum process conditions for osmotic dehydration of aloe vera gel**

<b>Response</b>	<b>Predicted value</b>	<b>*Experimental value (<math>\pm</math>SD)</b>	<b>C.V., %</b>
Water loss, %	39.69	40.72 $\pm$ 1.53	3.76
Sugar gain, %	4.45	4.68 $\pm$ 0.368	7.85

\* Average of three replications

The observed experimental values (mean of 3 experiments) and values predicted by the equations of the model are presented in Table 5.21. The experimental values were found to be very close to the predicted values for water loss and sugar gain, with the value of C.V. as 3.76 percent and 7.85 percent respectively. Therefore, it could be concluded from above discussion that model Eqns. (5.23) and (5.25) are quite adequate to assess the behaviour of the osmotic dehydration.

## **PART II: CONVECTIVE DRYING OF OSMOTICALLY DEHYDRATED ALOE VERA SAMPLES**

This section deals with results of various investigations pertaining to convective drying behaviour of osmotically dehydrated aloe vera samples, mathematical modelling of drying curves and optimization of convective drying parameters

### **5.6 Convective Drying Behaviour of Osmotically Dehydrated Aloe vera Samples**

The investigations were carried out on (i) drying characteristics and (ii) estimation of effective moisture diffusivity and activation energy during the convective drying of osmotically dehydrated aloe vera samples.

#### **5.6.1 Drying characteristics**

The convective drying behaviour was investigated for osmo-dehydrated aloe vera samples dried at air temperature of 50, 60, 70 and 80°C with air velocities of 1.0, 2.0 and 3.0 m/s. The experimental data are presented in Appendix F. The variation in moisture content of osmotically dehydrated aloe vera samples with drying time, drying rate and effective moisture diffusivities as well as effect of air velocities were calculated and presented in following sections.

##### **5.6.1.1 Variation in moisture content with time**

The typical curves showing variation in moisture content with drying time for osmotically dehydrated aloe vera samples dried with air temperature of 50, 60, 70 and 80°C are shown in Fig. 5.20 for air velocities of 1.0, 2.0 and 3.0 m/s respectively.

The initial moisture content of the osmotically dehydrated aloe vera samples was ranging 976.45 to 982.31 per cent (db) for all the samples investigated and after drying upto (nearly) constant weight, the moisture content was reduced in the range of 8.83 to 19.29 per cent (db) (Fig 5.20) (Appendix F). It can also be observed from these curves that moisture content of aloe vera samples decreased exponentially with drying time under all drying conditions. Similar types of results have been reported by various researcher viz. Pokharkar (1994), Maskan (2002), Jain (2007), Pisalkar (2008), etc. for air drying of osmotically dehydrated pineapple, papaya, aloe vera and banana slices respectively.

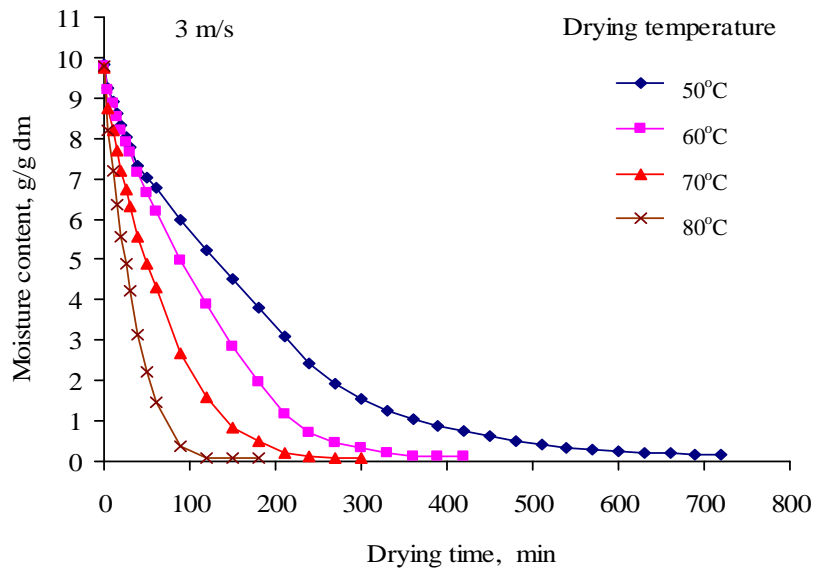
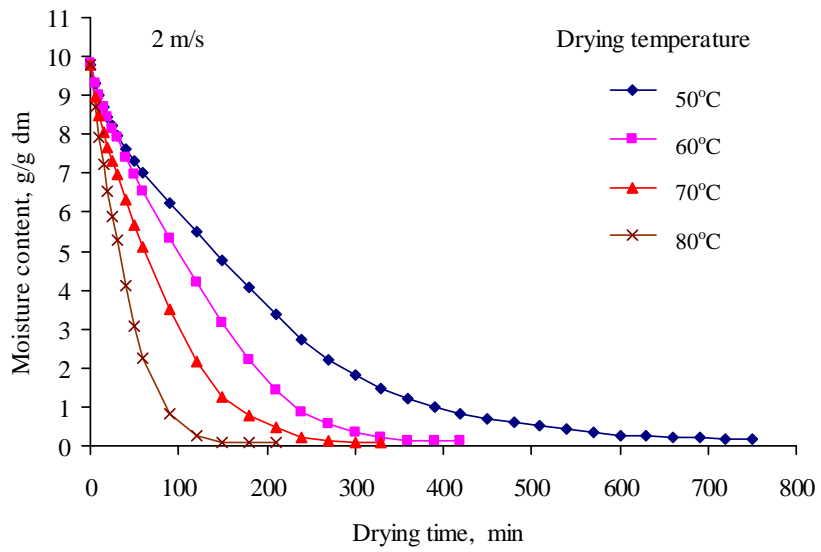
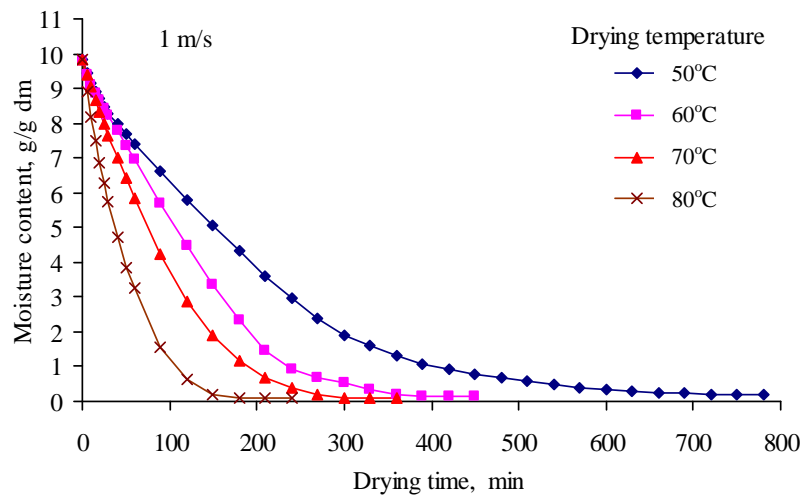
The drying data were statistically analysed and regression equations of exponential form were predicted as

$$MC = Ae^{(-kt)} \quad \dots 5.26$$

Where, MC is moisture content of the aloe vera samples and t is time in min and A and k are constants. The average values of regression coefficients A and k are given in Table 5.22 with R<sup>2</sup> value for air velocity 1, 2 and 3 m/s for the temperature range of 50 to 80°C. It can be seen from the table that for all experiments the coefficients of determination is more than 0.95 showing good correlation between the data collected. It can also be seen that values of regression coefficients (A and k) of the Eqn. (5.26) varies with variation in drying air temperature.

**Table 5.22 Overall regression coefficients of Eqn. (5.26) with R<sup>2</sup>**

S.N.	Drying air velocity, m/s	Regression coefficient		Coefficient of determination, R <sup>2</sup>
		A	k	
1.	1.0	11.049	0.0131	0.9859
2.	2.0	10.730	0.0145	0.9836
3.	3.0	10.192	0.0162	0.9818



**Fig 5.20 Variation in moisture content with drying time at different air velocities**

The total drying time for convective drying of osmotically dehydrated aloe vera gel is shown in Table 5.23. It can be seen that there was a wide variation in drying time from 180 to 780 min for the range of drying air temperatures and air velocities taken for study. It can also be seen that minimum time in drying was observed for higher air temperature (80°C) and maximum time was recorded for low air temperature (50°C) for all air velocities. Drying time in general decreased with increase in drying air temperature as well as increase in drying air velocity but the effect of air temperature was much more as compared to air velocity.

The ANOVA was carried out to study the effect of process variables *i.e.* air temperature and velocity on drying time for air drying of osmotically dehydrated aloe vera samples and the same is presented in Table 5.24. It can be inferred from the table that drying air temperature and velocity both have significant effect at 1 per cent level on drying time during convective drying process, but influence of velocity was not as high as drying air temperature.

**Table 5.23 Effect of temperature (T), velocity (V) and their interaction on total drying time**

Temp.	Velocity			Mean
	V <sub>1</sub>	V <sub>2</sub>	V <sub>3</sub>	
T <sub>1</sub>	780	750	720	750
T <sub>2</sub>	450	420	420	430
T <sub>3</sub>	360	330	300	330
T <sub>4</sub>	240	210	180	210
Mean	457.5	427.5	405	

Where, T<sub>1</sub> = 50°C, T<sub>2</sub> = 60°C, T<sub>3</sub> = 70°C and T<sub>4</sub> = 80°C  
and V<sub>1</sub> = 1m/s, V<sub>2</sub> = 2m/s and V<sub>3</sub> = 3m/s

**Table 5.24 Analysis of variance for the effect of process variables on the drying time**

Source of variation	SS	df	MS	F <sub>cal</sub>
T	1447000	3	482400	630.59**
V	16650	2	8325	10.88**
T xV	1350	6	225	0.25 <sup>NS</sup>
Error	21600	24	900	
Total	1487000	35		

\*\*Significant at 1% level NS – Non significant

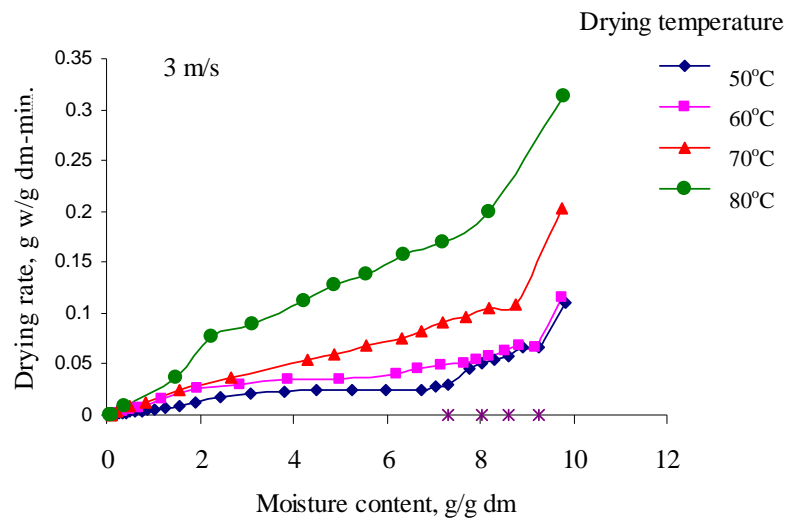
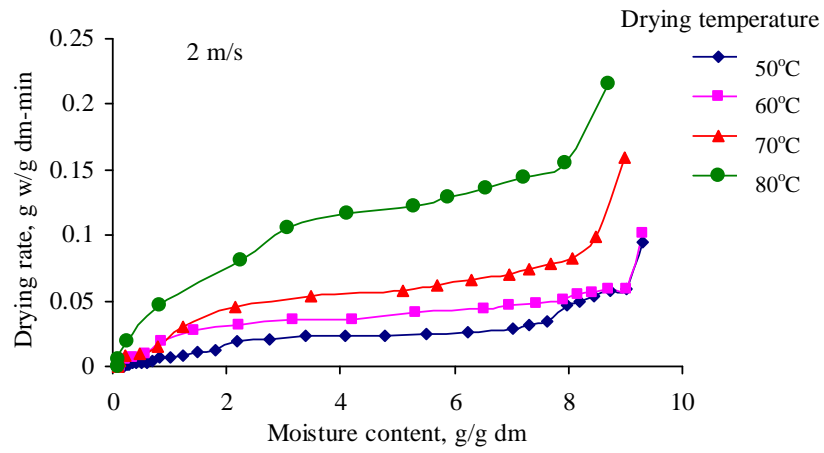
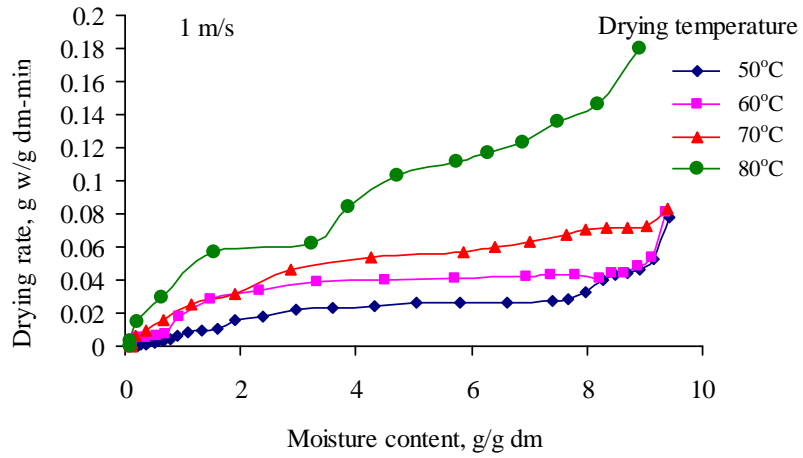
The typical curves showing variation in drying rate with moisture content of osmotically dehydrated aloe vera samples dried with air temperature of 50, 60, 70 and 80°C

for air velocities of 1.0, 2.0 and 3.0 m/s, respectively are shown in Fig. 5.21. The drying rates were determined from the mass balance calculations at various moisture contents.

The convective drying of osmotically dehydrated aloe vera samples followed a typical trend. As the drying air temperature increased, the drying curves exhibited steeper slope indicating that the drying rate increased with increase in drying air temperature. This resulted into substantial decrease in drying time. This is according to kinetic theory, due to the increased energy of water molecules as temperature is increased. Hence escaping of molecules becomes easier from the medium and faster. It has been reported by Prabhanjan *et al.*, (1995) that the higher temperature provide a larger water vapour pressure deficit (the difference between the saturated water vapour pressure and partial pressure of water vapour in air at a given temperature), which is one of the driving forces for the outward moisture diffusion process (drying).

Similar behaviours were observed by Vergara, *et al.*, (1997) for osmotically dehydrated apple, Maskan and Gogus (1998) for mulberry, Pokharkar (1994) for pine apple, Jain (2007) for papaya and Pisalkar (2008) for aloe vera. It was also found that the drying rate increased with increase in drying air velocity that might be due to modification in cellular structure, which minimized the effect of superficial hardening.

It can be observed from the Fig. 5.21 that as the drying proceeds, the moisture content of the sample decreased and the rate of drying also decreased. It can also be seen from the figure that the rate of drying was higher for high temperature of drying air. This is expected also because high temperature of drying will remove moisture quickly from the sample which resulted in high drying rate. Further, it can be seen from the figures that no constant rate period was found during convective drying of osmotically dehydrated aloe vera samples and entire drying has taken place in falling rate period.



**Fig 5.21 Variation of drying rate with moisture content**

The drying rate curves presented in Fig. 5.21 were statistically analyzed and regression equation of third order with overall regression coefficients and coefficient of determination can be given as :

$$y = 0.00049 x^3 + 0.00669x^2 + 0.03293x + 0.00403$$

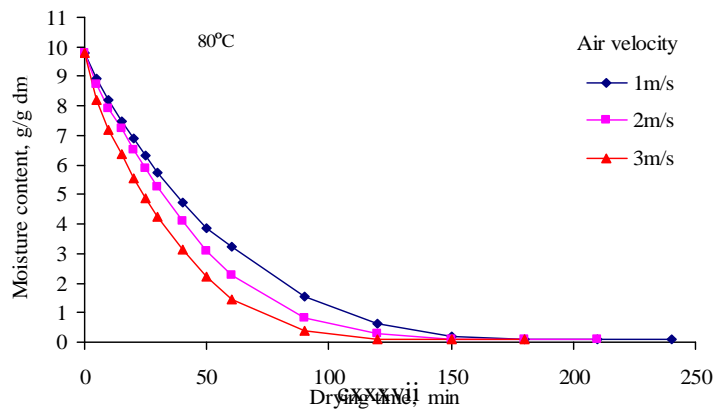
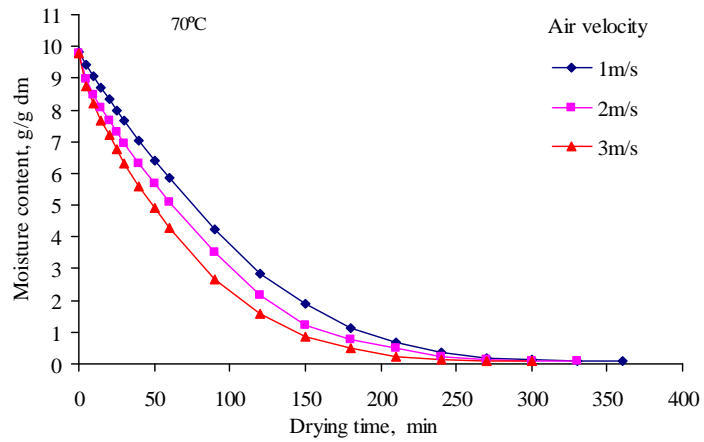
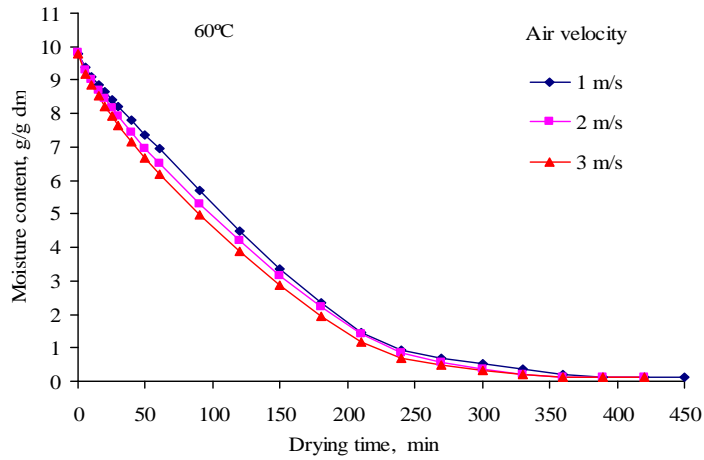
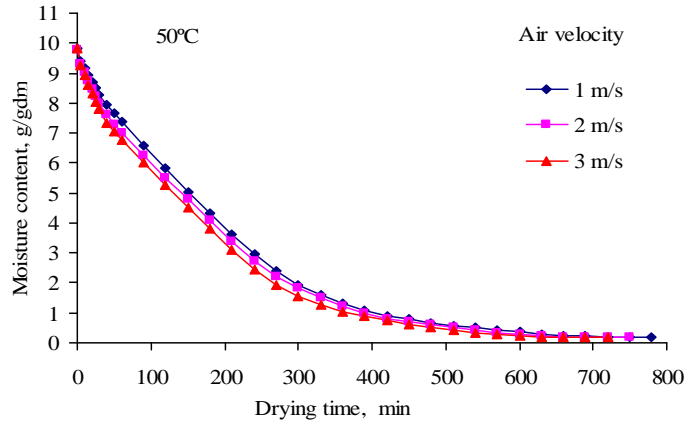
...5.27

$$R^2 = 0.97$$

Where y is the drying rate (g water/g dry matter-min), a, b, c, and d are regression coefficients and x is the moisture content (g water/g dry matter). The details are given in Appendix J. It can also be seen that the values of coefficient of determination were more than 0.94 (Appendix J) for all the experiments conducted which shows the good correlation among the predicted and observed data. Similar trend was also reported by various research workers for different food products such as for papaya by Ghatge (2003), for mushroom by Murumkar (2006), for onion by Revaskar (2005) and for aloe vera by Pisalkar (2008).

#### 5.6.1.2 Effect of air velocity

The effect of air velocities on moisture content of osmotically dehydrated aloe vera samples during air drying is shown in Fig. 5.22 for air temperature of 50, 60, 70 and 80°C. The figure revealed that under the prevailing conditions, the three drying curves at different air velocities are not very different from each other and the slopes of the lines are almost identical, especially at higher moisture levels, but effect becomes more pronounced in the later stage of drying where higher flow rate helps in attaining the final moisture content quickly in comparison to the lower air velocity. These findings are in conformity of the results reported for air drying of osmotically dehydrated banana, pineapple and papaya slices by Bongirwar and Sreenivasan (1977), Pokharkar (1994), Jain (2007) and Kaya *et al.*, (2009) respectively. The effect of velocity was more pronounced at higher temperatures.



**Fig. 5.22 Effect of air velocity on drying of osmo-dehydrated aloe vera samples for 50, 60, 70 & 80 °C air temperature**

**5.6.2 Effective moisture diffusivity ( $D_{eff}$ )**

It has been discussed earlier that entire drying process in the present study occurred during falling rate period (section 5.6.1.1). Further analysis of the falling drying rate period was carried out to estimate effective moisture diffusivity ( $D_{eff}$ ), to study the influence of moisture content on effective moisture diffusivity and to estimate the activation energy for different drying air temperatures.

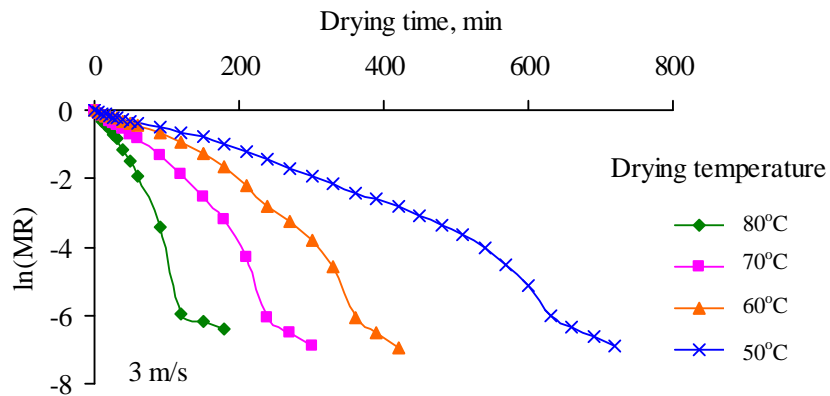
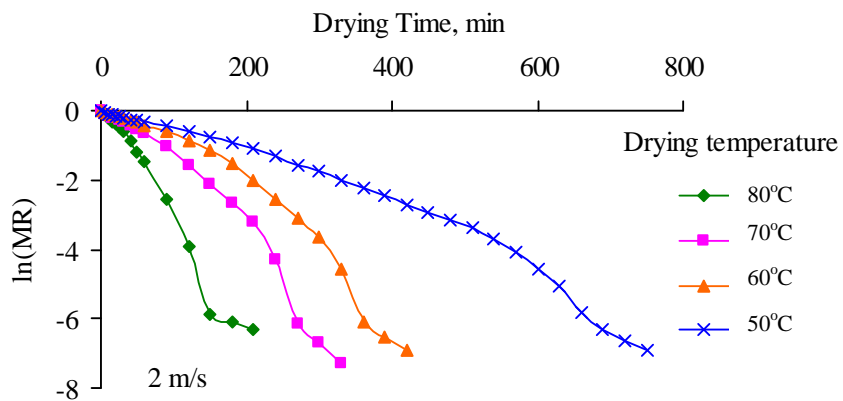
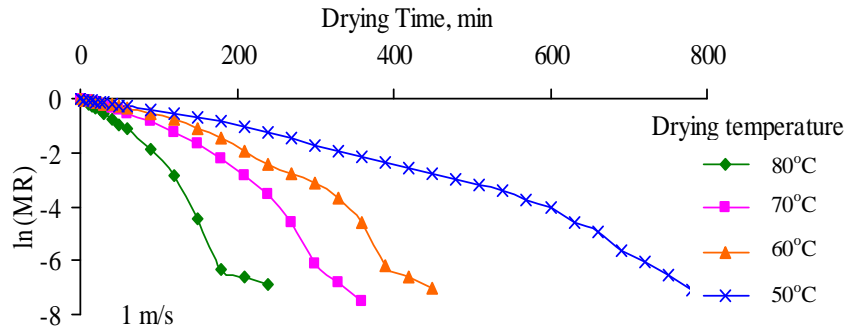
The equilibrium moisture content of osmotically dehydrated aloe vera gel sample at different temperatures and air velocity levels used in the drying experiments were obtained using dynamic method as described in section 3.8.2. Equilibrium moisture content for each experimental run is given in Table 5.25 varying between 7.22 and 18.47% (db).

**Table 5.25 Equilibrium moisture content at different experimental conditions**

Air velocity, m/s	Temperature, °C	EMC, % (db)
1	50	18.47
1	60	11.39
1	70	9.60
1	80	8.30
2	50	17.85
2	60	11.12
2	70	9.22
2	80	7.43
3	50	17.48
3	60	10.93
3	70	9.00
3	80	7.22

The variation of moisture content with drying time under different drying air temperatures is presented in Appendix-F. The drying data were used to compute moisture ratio (MR) using Eqn. (3.13). The natural logarithms of moisture ratio ( $\ln MR$ ) were plotted against average drying time ( $t$ ) for different drying air temperatures, and are shown in Fig. 5.23. It was observed from the figure that the relationship was non-linear in nature for all drying conditions. This non-linearity in the relationship might be due to reasons like

shrinkage in the product, variation in moisture diffusivity with moisture content and change in product temperature during drying (Hawlder *et al.*, 1991; Kannan and Bandopadhyay, 1995). The non-linearity of the curves, an indicative of the variation in moisture diffusivity with moisture content, was used to estimate effective moisture diffusivity of osmo-convectively dried aloe vera samples at corresponding moisture content, under different drying conditions. Fourier number ( $F_o$ ) and effective moisture diffusivity ( $D_{eff}$ ) were estimated for corresponding moisture content using Eqns. (4.31) and (4.33) and are presented in Appendix-F.



**Fig. 5.23 Variation in  $\ln(MR)$  versus time for convective drying of osmotically dehydrated aloe vera samples**

The experimental  $D_{\text{eff}}$  values during convective drying of osmotically dehydrated aloe vera samples were obtained by the modified method of slopes described in section 4.4.2. The average effective moisture diffusivity ( $D_{\text{eff}})_{\text{avg}}$  values of osmo-convectively dried aloe vera samples varied considerably with moisture content and air drying temperature from  $0.959 \times 10^{-9}$  to  $6.103 \times 10^{-9}$ ,  $1.170 \times 10^{-9}$  to  $7.351 \times 10^{-9}$  and  $1.39 \times 10^{-9}$  to  $10.405 \times 10^{-9}$  m<sup>2</sup>/s for air velocity of 1.0, 2.0 and 3.0 m/s, respectively (Table 5.26). These values are within the general range of  $10^{-8}$  to  $10^{-12}$  m<sup>2</sup>/s for drying of food materials (McMinn and Magee, 1999). These values are in fact consistent with those existing in literature e.g.  $56$  to  $137 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of pear (Park *et al.*, 2001);  $4.28 \times 10^{-10}$  to  $6.8 \times 10^{-9}$  for hot air drying of okra (Gogus and Maskan, 1999);  $2.32 \times 10^{-10}$  to  $2.76 \times 10^{-9}$  for hot air drying mulberry (Maskan and Gogus, 1998);  $4 \times 10^{-10}$  to  $21 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of apple (Karathanos *et al.*, 1995);  $1 \times 10^{-10}$  to  $2 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of blueberries (Ramaswami and Nsonzi, 1998);  $3 \times 10^{-10}$  to  $17.21 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of kiwifruit (Simal *et al.*, 2005a);  $5.30 \times 10^{-10}$  to  $17.73 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of fresh aloe vera (Vega-Galvez *et al.*, 2007);  $4.2 \times 10^{-10}$  to  $11.5 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of kiwifruit (Simal *et al.*, 2005b);  $0.589 \times 10^{-10}$  to  $6.574 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of kiwifruit (Kaya *et al.*, 2009);  $5.64 \times 10^{-10}$  to  $18.1 \times 10^{-10}$  m<sup>2</sup>/s for hot air drying of fresh aloe vera (Simal *et al.*, 2000);  $2.93 \times 10^{-9}$  to  $7.99 \times 10^{-9}$  m<sup>2</sup>/s and  $2.69 \times 10^{-9}$  to  $4.59 \times 10^{-9}$  m<sup>2</sup>/s for hot air drying of osmotically dehydrated and fresh aloe vera respectively (Pisalkar, 2008), etc.

It can be observed from the table that moisture diffusivity values increased with both parameters *i.e.* drying air temperature as expected and drying air velocity. The increase of moisture diffusivity with increase of air velocity for the osmo-convectively dehydrated aloe vera can be explained by the reduction in the effect of superficial hardening due to osmotic dehydration treatment. The increase in air velocity, decreasing the external resistance, increases the effective diffusivity.

The values of  $D_{\text{eff}}$  corresponding to positive  $F_0$  values were plotted against moisture content under all drying air temperature and are shown in Fig. 5.24. It is evident from these figures that  $D_{\text{eff}}$  increased with decrease in moisture content for all drying temperatures. The  $D_{\text{eff}}$  started decreasing as the moisture content decreased further below at the end of drying. This might be due to the fact that at the end of drying when most of the moisture has been removed, sample cells might have collapsed and overlapped each other reducing porosity and resulted in reduction in diffusion. Similar results were found by Khodke (2002) for drying of potato cubes. McMinn and Magee (1997) also reported that the variation of effective moisture diffusivity with moisture content is a complex function, dependant on variation of physical parameters, *i.e.* porosity and density during the process and interaction of the food component *e.g.* starch, cellulose or protein with water.

**Table 5.26 Moisture diffusivity in air drying of osmotically dehydrated aloe vera samples**

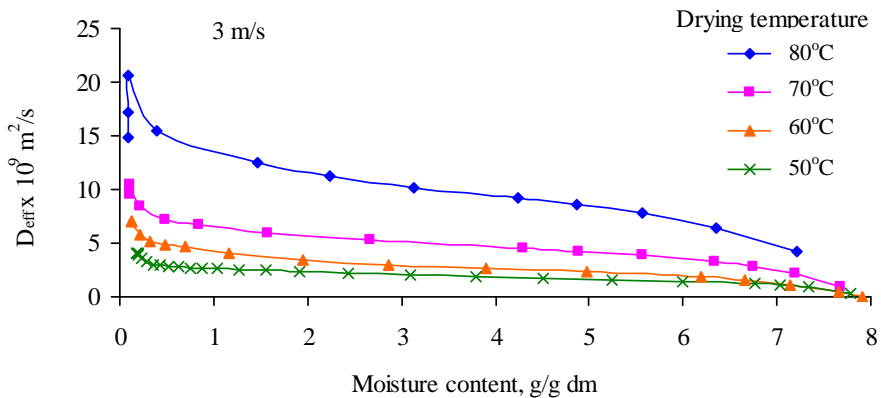
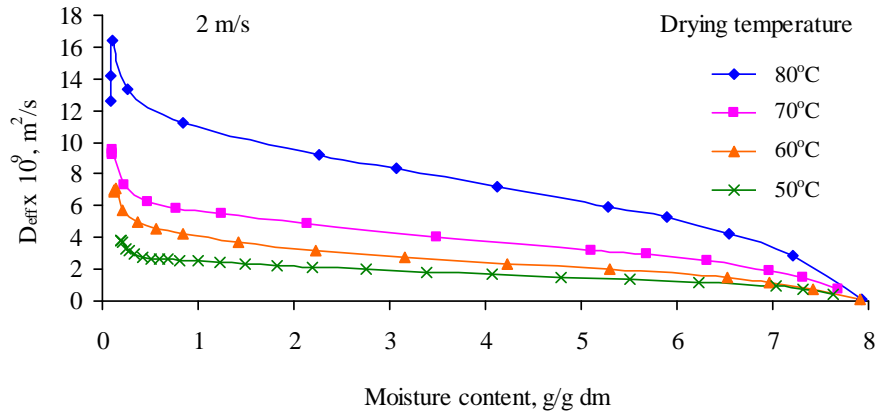
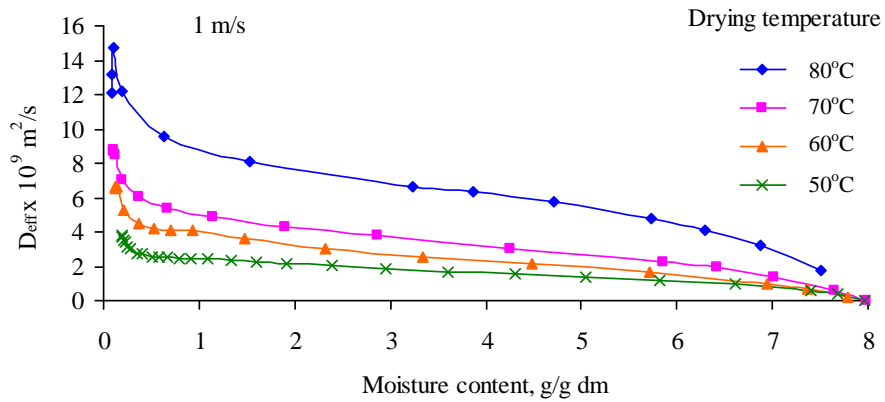
S. No.	Temperature of drying, °C	Air velocity, m/s	Diffusivity coefficient, $(D_{\text{eff}})_{\text{avg}} \times 10^9, \text{m}^2/\text{s}$	Predicted diffusivity coefficient $\times 10^9, \text{m}^2/\text{s}$
1	50	1.0	0.959	0.748
2	50	2.0	1.170	1.02
3	50	3.0	1.39	1.223
4	60	1.0	1.362	1.547
5	60	2.0	1.664	2.11
6	60	3.0	1.984	2.53
7	70	1.0	2.275	2.861
8	70	2.0	3.366	3.901
9	70	3.0	4.445	4.677
10	80	1.0	6.103	4.872
11	80	2.0	7.351	6.644
12	80	3.0	10.405	7.966

The ANOVA was carried out to study the effect of the process variables on  $(D_{\text{eff}})_{\text{avg}}$  and the same is presented in Table 5.27. It was observed from F-values that drying air temperature, velocity and their interactions has significant effect on  $(D_{\text{eff}})_{\text{avg}}$  at 5% level.

**Table 5.27 ANOVA showing effect of process variables on average effective moisture diffusivity**

Source	DF	SS	MSS	F <sub>cal</sub>
<b>T</b>	3	$8.19 \times 10^{-17}$	$2.73 \times 10^{-17}$	413.70*
<b>V</b>	2	$4.07 \times 10^{-18}$	$2.04 \times 10^{-18}$	30.85*
<b>T x V</b>	6	$2.11 \times 10^{-16}$	$3.52 \times 10^{-17}$	533.59*
<b>Error</b>	24	$1.58 \times 10^{-18}$	$6.6 \times 10^{-20}$	
<b>Total</b>	35	$2.99 \times 10^{-16}$		

\*Significant at 5% level.

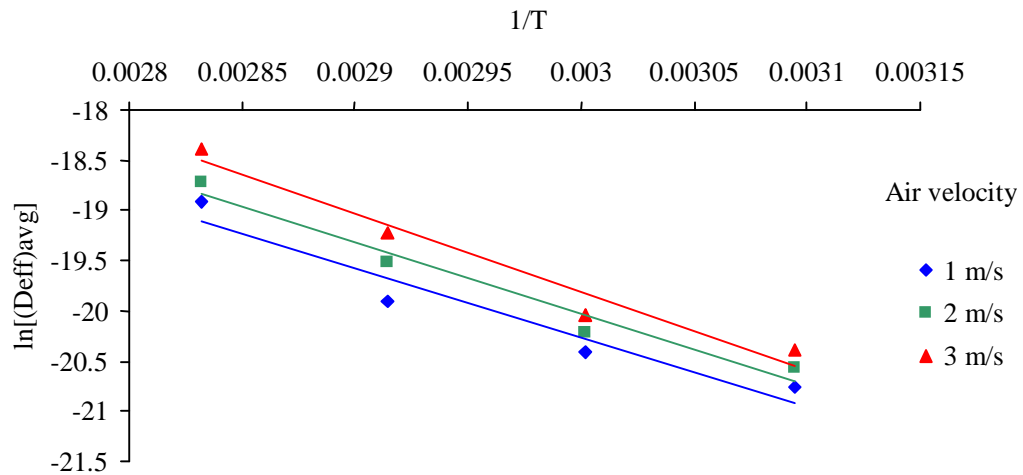


**Fig. 5.24 Variation in effective moisture diffusivity with moisture content of osmotically dehydrated aloe vera samples at various drying temperatures**

### 5.6.2.1 Activation energy

It is universally accepted that temperature renders a dominant influence on moisture diffusivity. In general, this is manifested by a progressive increase in the effective moisture diffusivity with increasing temperature (Zogzas *et al.*, 1994).

The temperature dependence of average effective moisture diffusivity ( $D_{\text{eff}})_{\text{avg}}$  during convective drying can be expressed as Arrhenius type relationship. The activation energy for moisture diffusion were obtained from the slope of plot of  $\ln [(D_{\text{eff}})_{\text{avg}}]$  versus reciprocal of temperature in  $\text{K}^{-1}$  (Fig. 5.25) and found to be 57.164, 58.835 and 64.636 kJ/mol (Table 5.29) for 1.0, 2.0 and 3.0 m/s air velocities, respectively. It is implied from the figure that moisture diffusivity of aloe vera sample decreased linearly with increase in  $1/T_{\text{abs}}$ . These values are closed to the  $E_a$  values reported by various researchers e.g.15-40 kJ/mol (Rizvi,1986) for various foods, 49-54 kJ/mol (Azzouz *et al.*,2002) for grapes, 39.47 kJ/mol (Senadeera *et al.*,2003) for potatoes, 30.37 kJ/mol (Vega-Galvez *et al.*, 2007) for fresh aloe vera and 36.11 kJ/mol (Mwithiga and Olwal, 2004) for kale, 51.26 kJ/mol (Doymaz, 2005) for okra and 82.93 kJ/mol (Park *et al.*, 2002) for mint.



**Fig. 5.25 Relationship between moisture diffusivity with reciprocal of absolute Temperature**

**Table 5.28 Activation energies for convective drying of osmo-dehydrated aloe vera**

Drying air velocity, m/s	Slope of Arrhenius plot	R, kJ /kg mol K	Activation energy, kJ/mol
1	- 6805.6	8.314	57.164
2	- 7046.7	8.314	58.835
3	- 7774.3	8.314	64.636

Based on the diffusivity values presented in Table 5.27, the data were statistically analysed to develop a semi empirical equation demonstrating the relationship between moisture diffusivity and other related process parameters using the regression technique. The proposed relationship is as follows:

$$(D_{eff})_{avg} = 1.1863 \times 10^{-16} \times e^{\frac{-0.058}{T}} + 0.4341V \quad \dots 5.29$$

$$R^2 = 0.94$$

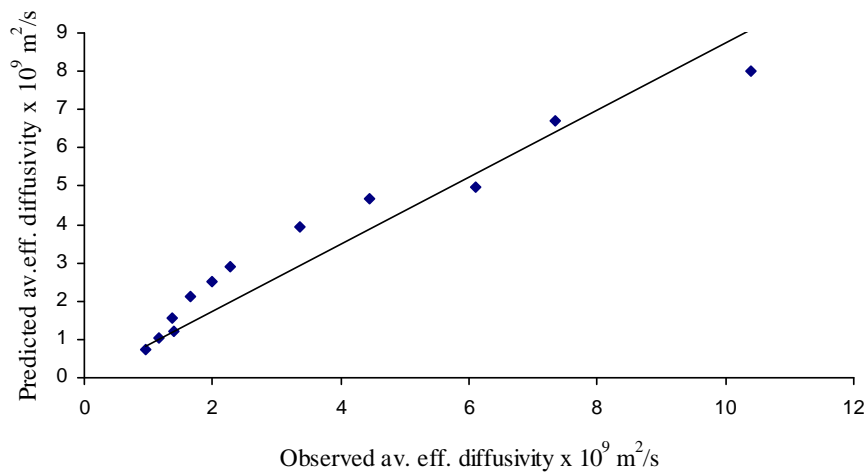
Where,

$(D_{eff})_{avg}$  = average effective moisture diffusivity,  $m^2/s$

T = temperature, K

V = drying air velocity, m/s

The diffusivity values calculated by the Eqn. (5.29) are also shown in Table 5.26 as predicted values. The above expression shows that the drying air temperature has a pronounced influence on the moisture diffusivity, whereas, the effect of air velocity was very limited as indicated by its predicted values Table (5.26). The average effective diffusivity during the convective drying of osmotically dehydrated aloe vera samples at various air temperatures and velocities as determined experimentally (Table 5.27) and predicted by Eqn. (5.29) are shown in Fig. 5.26. It can be seen from the figure that there is a good co-relation between the observed and the predicted values of water diffusivities with  $R^2 = 0.95$ .



**Fig. 5.26 Experimental and predicted values of moisture diffusivities**

## 5.7 Modelling of Drying Curve

To determine the most suitable drying equation the moisture ratio data of osmosed aloe vera sample dried at various air temperatures and velocity of air were fitted into the six thin-layer drying models (Exponential, Henderson and Pabis, Page, Modified page, Logarithmic and Power law) in their linearized form using regression technique. Among all these models, the best model suitable to fit the data were selected on basis of highest values of  $R^2$  and the lowest value of reduced mean square of the deviation ( $\chi^2$ ) and root mean square error ( $E_{RMS}$ ), which were calculated using Eqn. (3.14) through Eqn.(3.15).

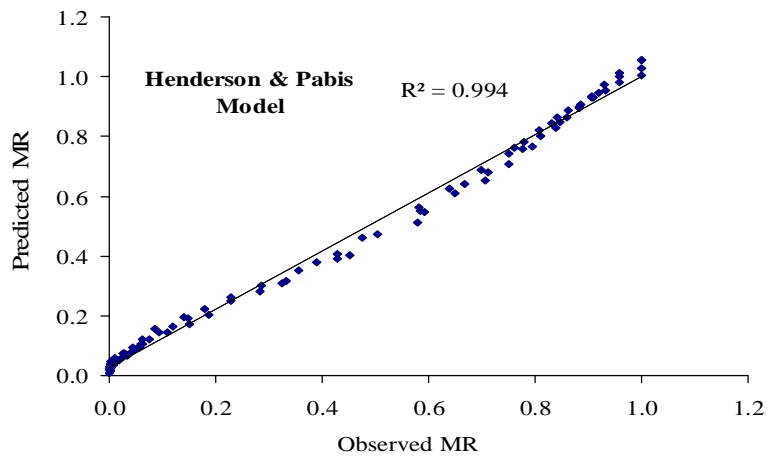
The overall statistical parameters for different models used for osmo-convective dried aloe vera samples have been presented in Table 5.29. The results show that the highest values of coefficient of determination ( $R^2$ ) and the lowest values of root mean square error ( $E_{RMS}$ ), reduced mean square of the deviation ( $\chi^2$ ) was obtained for Henderson and Pabis drying model. The details are presented in Appendix K. Hence, Henderson and Pabis model was found to be the most satisfactory among the models to represent the thin-layer drying of aloe vera samples.

This was another confirmation of the suitability of Henderson and Pabis model to thin layer drying, which has been reported by Jain (2007) for air drying of papaya and Koua *et al.*, (2009) for thin layer solar drying of mango, banana and cassava.

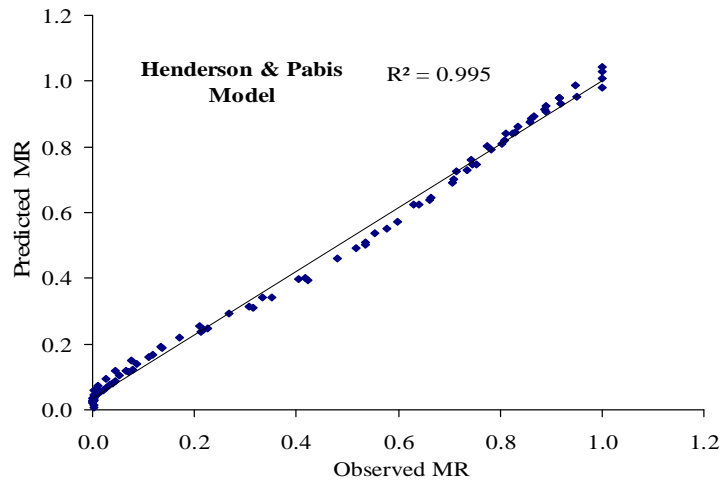
**The selected Henderson and Pabis model for osmo-convective drying studies was validated by comparing the predicted and observed values of moisture ratio in all drying experiment. The predicted and observed values of moisture ratio were plotted as shown in Fig 5.27, 5.28 and 5.29 (Pangavhane *et al.*, 1999; Sarsavadia, *et al.*, 1999; Togrul and Pehlivan, 2002; Erketin, *et al.*, 2004; Demir, *et al.*, 2004) for all drying air velocities.**

Table 5.29 Overall values for model constants and statistical parameters used in convective drying of osmotically dehydrated aloe vera sample

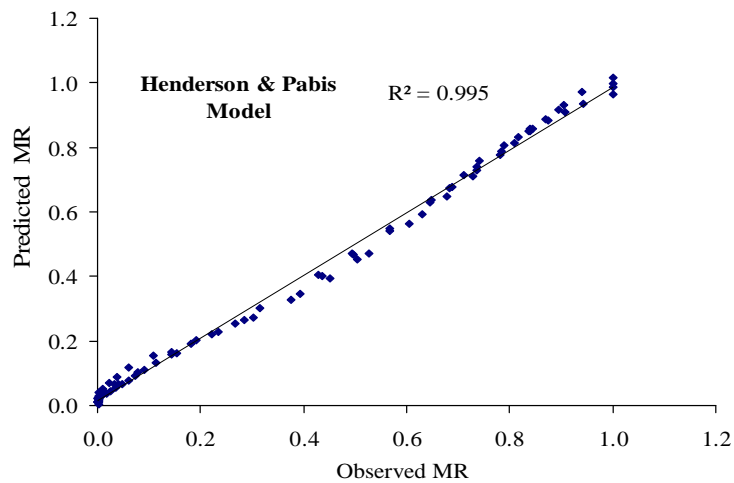
Name of Model	Drying constant				Statistical parameters		
	k	n	a	b	$R^2$	$\chi^2$	$E_{RMS}$
<b>Exponential</b>	0.013	-	-	-	0.992	0.0011	0.030
Henderson and Pabis	0.013	-	0.014	-	0.993	0.0010	0.029
Page	0.009	1.100	-	-	0.973	0.0252	0.100
Modified page	0.014	1.100	-	-	0.969	0.0129	0.095
Logarithmic	-	-	1.542	-0.265	0.952	0.1505	0.293
Power law	-	-	2.506	-0.457	0.783	0.1270	0.270



**Fig. 5.27** Experimental and predicted values of moisture ratio by Henderson and Pabis model for 1m/s air velocity and at various temperatures.



**Fig. 5.28** Experimental and predicted values of moisture ratio by Henderson and Pabis model for 2m/s air velocity and at various temperatures.



**Fig. 5.29** Experimental and predicted values of moisture ratio by Henderson and Pabis model for 3m/s air velocity and at various temperatures.

## 5.8 Optimization of Convective Drying Parameters

The factorial CRD was adopted for analysis. Influence of various factors was analysed using ANOVA technique. The experimental values of independent variables were fitted in second order model for various responses using multiple regression analysis. The optimization of input parameters (drying air temperature and air velocity) was carried out using numerical optimization technique with the help of Design Expert 8.0.4.1 statistical software (trial version) by adopting full factorial design.

### 5.8.1 Ascorbic acid

Ascorbic acid is precursor of Vitamin C and is lost during the drying process because it is heat liable nutrient. When a food is sliced and its cells are cut, the surfaces that are exposed to air loose some vitamin C content. The retention of vitamin C depends upon the water content, sugar content, the size of the sample of food material, amount of air circulation when food is dried, the level of humidity in the air entering the dehydrator and the air temperature and time of exposure inside the dehydrator.

In the present study the ascorbic acid content of fresh aloe vera gel sample was 149.08 mg/100 g dm, whereas ascorbic acid content of osmo-convectively dried aloe vera sample was ranging between 22.03 and 28.74 mg/100 g dm (Table 5.30). It revealed that in all the dried samples, the loss of ascorbic acid was greater than 80 per cent compared to fresh gel. Individual effect of temperature on ascorbic acid revealed that the retention of ascorbic acid increased with decrease in drying temperature from 80 to 60°C and it decreased with further decrease in drying air temperature to 50°C. The loss of ascorbic acid at higher temperature (80°C) is maximum because it is thermo sensitive compound (Hawlder *et al.*, 2006) but comparative higher loss at lower temperature (50°C) might be due to long period of exposure (e.g. 720 to 780 min for 3 m/s to 1 m/s air velocity respectively) required to dry the sample at 50°C. Similar results are quoted by Miranda *et al.*, (2009a). The drying air temperature of 60°C was found better and recorded significantly highest ascorbic acid (27.54 mg/100 g dm). Similarly, with respect to individual effect of velocity, the sample dried with lower velocity (1 m/s) recorded highest ascorbic acid (26.69 mg/100 g dm). As the velocity increased the ascorbic acid decreased slightly.

The ANOVA with respect to ascorbic acid is shown in Table 5.31. The table shows that the effects of temperature, air velocity, and their interaction on ascorbic acid are significant at 5 per cent level of significance. From Table 5.30, it revealed that the sample with the highest retention of ascorbic acid (28.74 mg/100 g dm) was the one dried at 60 °C temperature and 1 m/s drying air velocity and was significantly superior over all other samples.

**Table 5.30 Effect of temperature (T), velocity (V) and their interaction on ascorbic acid**

Temp.	Velocity			Mean
	V <sub>1</sub>	V <sub>2</sub>	V <sub>3</sub>	
T <sub>1</sub>	28.08	26.83	25.56	26.82
T <sub>2</sub>	28.74	27.05	26.82	27.54
T <sub>3</sub>	27.34	23.86	23.58	24.93
T <sub>4</sub>	22.59	22.31	22.03	22.31
Mean	26.69	25.01	24.50	

Where, T<sub>1</sub> = 50°C, T<sub>2</sub> = 60°C, T<sub>3</sub> = 70°C and T<sub>4</sub> = 80°C  
and V<sub>1</sub> = 1m/s, V<sub>2</sub> = 2m/s and V<sub>3</sub> = 3m/s

**Table 5.31 Analysis of variance for two factor CRD for ascorbic acid**

Source	DF	SS	MSS	F <sub>cal</sub>	SE (m) ±	CD at 5%	C.V.%
T	3	147.28	49.09	3487.75*	0.0396	0.0115	0.468
V	2	31.48	15.74	1118.15*	0.0343	0.1002	
T x V	6	11.40	1.90	134.98*	0.687	0.2005	
Error	24	0.34	0.014				
Total	35	190.50					

\*Significant at 5% level

Second order regression model fitted well to the experimental data (Table 5.30). This model [Eqn.(5.30)] shows the effect of drying temperature (T) and air velocity (V) on ascorbic acid.

$$\text{Ascorbic acid} = 8.54 + 0.8795T - 4.7232 V - 0.0083 T^2 - 0.5786 V^2 + 0.2020 TV$$

....(5.30)

$$R^2 = 0.92$$

### 5.8.2 Colour (L-value)

Colour is often used as an indication of quality and freshness for food products. Hence, it has become important for food processors to evaluate and grade their products based on colour.

Colour values measured using a Colourflex Hunterlab Colourimeter, were relative to the absolute values of perfect reflecting diffuser as measured under the same geometric conditions (ASTM method). Observations were taken at room temperature 30.5°C and 25 percent relative humidity.

The colour of dried aloe vera product was measured in terms of L-value (brightness/darkness) and shown in Table 5.32. The L-value of fresh aloe vera gel was about

34.68. The L-values of osmo-convectively dried aloe vera sample at various experimental conditions were ranged between 38.93 and 78.13 (Table 5.32). As the temperature increased L-value of colour increased that means sample became lighter in colour from 50°C to 70°C and thereafter decreased at 80°C which may be due to discolouring the sample slightly because of elevated temperature. The drying air temperature 70°C was found better and recorded significantly highest colour (L-value = 71.45). Increase in velocity also resulted in increased colour (L-value) slightly. The sample dried with 3 m/s drying air velocity was found significantly superior in recording better colour (L-value = 62.82).

From Table 5.33 it can be seen that temperature, air velocity and their interactions are significant at 5 per cent level of significance. The sample with highest L-value (78.13) was the one dried at 70°C temperature and 3 m/s air velocity and was significantly superior over rest of the samples dried with all other combinations of temperature and velocity (Table 5.32).

**Table 5.32 Effect of temperature, velocity and their interaction on colour (L-value)**

<b>Temp. \ Velocity</b>	<b>V<sub>1</sub></b>	<b>V<sub>2</sub></b>	<b>V<sub>3</sub></b>	<b>Mean</b>
<b>T<sub>1</sub></b>	38.93	42.80	47.29	43.01
<b>T<sub>2</sub></b>	52.04	55.84	60.84	56.24
<b>T<sub>3</sub></b>	66.35	69.86	78.13	71.45
<b>T<sub>4</sub></b>	58.71	63.56	65.02	62.43
<b>Mean</b>	54.01	58.02	62.82	

**Table 5.33 Analysis of variance for two factor CRD for colour (L-value)**

<b>Source</b>	<b>DF</b>	<b>SS</b>	<b>MSS</b>	<b>F<sub>cal</sub></b>	<b>SE (m) ±</b>	<b>CD at 5%</b>	<b>C.V.%</b>
T	3	3852.01	1284	2050.43*	0.263	0.77	1.35
V	2	467.33	233.66	373.14*	0.228	0.666	
T x V	6	39.55	6.56	10.53*	0.456	1.33	
Error	24	15.03	0.63				
Total	35	4373.92					

\*Significant at 5% level

Second order regression model for colour (L-value) was fitted to the experimental data (Table 5.32. This model shows the effect of drying temperature (T) and air velocity (V) on colour (L-value).

$$L = -206.24 + 7.2638T + 3.4851 V - 0.0505T^2 + 0.3634 V^2 - 0.0144TV \quad \dots 5.31$$

$$R^2 = 0.92$$

### 5.8.3 Hardness

Textural characteristics of osmo-convectively dried aloe vera samples were studied in terms of hardness. Hardness of dried aloe vera product was measured using Texture analyzer as discussed in section 3.10.5. Each trial was repeated three times and the average hardness data are presented in the Table 5.34. The average hardness values varied between 582.8 and 1214.31 g for the range of drying air temperature and air velocity. As regards the individual effect of temperature, it was observed that as the temperature increased, hardness increased significantly for each velocity and was the highest at 80°C temperature. Hardness also increased with increase in air velocity slightly and was the highest (913.19 m/s) at 3 m/s.

From Table 5.35 it can be seen that for temperature, air velocity, and for their interaction calculated F value ( $F_c$ ) is more than tabulated F-value ( $F_t$ ) at 5 per cent level of significance. So the effect of drying temperature, air velocity, and their interactions were significant on hardness. From Table 5.34 the osmo-convectively dried sample with interaction of 80°C drying air temperature and 3 m/s air velocity was having better hardness (1214.31 g) and was significantly superior over all other combination of temperature and velocity.

**Table 5.34 Effect of temperature, velocity and their interaction on hardness**

Temp. \ Velocity	V <sub>1</sub>	V <sub>2</sub>	V <sub>3</sub>	Mean
T <sub>1</sub>	582.8	610.77	648.07	613.88
T <sub>2</sub>	718.01	773.96	801.93	764.63
T <sub>3</sub>	848.56	918.49	988.43	918.49
T <sub>4</sub>	1011.74	1077.04	1214.31	1101.03
<b>Mean</b>	790.28	845.07	913.19	

**Table 5.35 Analysis of variance for two factor CRD for hardness**

Source	DF	SS	MSS	F <sub>cal</sub>	SE (m) ±	CD at 5%	C.V.%
<b>T</b>	3	1.77 x 10 <sup>6</sup>	3.92 x 10 <sup>5</sup>	1430.42*	5.52	16.11	1.94
<b>V</b>	2	90998.04	45499.02	165.93*	4.77	13.95	
<b>T x V</b>	6	19883.49	3313.91	12.09*	9.56	27.9	
<b>Error</b>	24	6581.09	274.21				
<b>Total</b>	35	1.29 x 10 <sup>6</sup>					

\*Significant at 5% level

The best fitted model for the experimental data was second order regression model [Eqn. (5.32)]. This model shows the effect of drying temperature (T) and air velocity (V) on hardness.

$$\text{Hardness} = - 214.937 + 16.27 T - 5.6 V - 0.023 T^2 + 0.839 TV \quad \dots 5.32$$

$$R^2 = 0.99$$

#### 5.8.4 Water activity

The water activity of fresh aloe vera gel sample was 0.982 whereas water activities of osmo-convectively dried samples with all combinations of temperatures and velocities were ranging between 0.282 and 0.422. As regards to individual effect of temperature, it revealed that as the temperature increased water activity decreased significantly. The sample dried at 80°C drying air temperature was having significantly lowest (0.286) water activity. Similarly as air velocity increased water activity decreased significantly but the rate was comparatively less. The sample dried with 3 m/s drying air velocity showed lowest (0.347) water activity (Table 5.36).

The ANOVA for water activity is presented in Table 5.37. From this table, it can be seen for temperature, air velocity and for interaction that the effect of drying temperature, air velocity, and their interactions were significant on water activity at 5 per cent level of significance. From Table 5.36 it revealed that the sample with lowest water activity (0.282) was one dried at 80°C drying air temperature and 3 m/s drying air velocity and was significantly superior over the sample dried by all other combinations of temperature and velocity.

**Table 5.36 Effect of temperature, velocity and their interaction on water activity**

<b>Temp. \ Velocity</b>	<b>V<sub>1</sub></b>	<b>V<sub>2</sub></b>	<b>V<sub>3</sub></b>	<b>Mean</b>
<b>T<sub>1</sub></b>	0.422	0.415	0.412	0.416
<b>T<sub>2</sub></b>	0.392	0.385	0.382	0.386
<b>T<sub>3</sub></b>	0.332	0.325	0.313	0.323
<b>T<sub>4</sub></b>	0.292	0.285	0.282	0.286
<b>Mean</b>	0.360	0.353	0.347	

**Table 5.37 Analysis of variance for two factor CRD for water activity**

<b>Source</b>	<b>DF</b>	<b>SS</b>	<b>MSS</b>	<b>F<sub>cal</sub></b>	<b>SE (m) ±</b>	<b>CD at 5%</b>	<b>C.V.%</b>
<b>T</b>	3	0.094	0.031	19478.76*	0.0004	0.0012	0.359
<b>V</b>	2	9.44 x 10 <sup>-4</sup>	4.72 x 10 <sup>-4</sup>	293.03*	0.0004	0.0011	
<b>T x V</b>	6	1.17 x 10 <sup>-4</sup>	1.96 x 10 <sup>-5</sup>	12.14*	0.0007	0.0021	
<b>Error</b>	24	3.86x10 <sup>-5</sup>	1.61x10 <sup>-6</sup>				
<b>Total</b>	35	0.095					

\*Significant at 5% level

The model that fits well to the experimental data was second order regression model [Eqn. (5.33)]. This model shows the effect of drying temperature and air velocity on water activity ( $a_w$ ).

$$a_w = 0.5858 - 0.00219T - 0.0064V - 0.000017T^2 + 0.00092V^2 - 0.00005VT \quad \dots 5.33$$

$$R^2 = 0.98$$

### 5.8.5 Optimization of independent variables for production of osmo- convectively dried aloe vera sample

Optimization is a process of making compromises between responses, to achieve a common target. Numerical optimization was carried out using Design-Expert 8.0.4.1 statistical software (trial version). The goal was fixed to be in the range for hardness, ascorbic acid, colour (L-value), water activity and drying time. The goal seeking begins at a random starting point and proceeds up and down the steepest slope on the response for a maximum or minimum value of the response respectively. All the responses and independent variables were given similar (+++) importance. The goal setup for optimization is given in the Table 5.38.

Based on above mentioned criteria the optimization was carried out. During optimization 12 solutions were obtained, out of which the one that suited the criteria most was selected. The most suitable optimum point is given in the Table 5.39. Thus convective drying of osmotically dehydrated aloe vera sample with 70°C drying air temperature and 1 m/s air velocity was found to be the best for optimum response among the range of variables.

**Table 5.38 Constraints for optimization for convective drying of osmotically dehydrated aloe vera sample**

S.No.	Parameter	Goal	Lower limit	Upper limit
1.	Temperature, °C	Is in range	50	80
2.	Air velocity, m/s	Is in range	1	3
3.	Ascorbic Acid, mg / 100g dm	Maximum	21.97	29
4.	Colour (L-value)	Maximum	38.48	78.33
5.	Hardness, g	Maximum	573.48	1128.98
6.	Water activity	Minimum	0.281	0.423
7.	Drying time, min.	Minimum	150	810

**Table 5.39 Optimum levels of variables for convective drying of osmotically dehydrated aloe vera sample**

<b>S. No.</b>	<b>Variable</b>	<b>Optimum level</b>
1.	Drying air temperature, °C	70
2.	Air velocity, m/s	1

The osmo-convectively dried aloe vera sample with optimized input parameters is shown in Plate 5.1.



**Plate 5.1 Osmo-convectively dried aloe vera sample**

### PART III: QUALITY EVALUATION OF DRIED ALOE VERA SAMPLES

The osmo-convectively dried aloe vera samples with the optimized input parameters were further studied for their quality aspects, such as sensory evaluation, re-hydration ratio and mineral contents.

#### 5.9 Quality Evaluation

##### 5.9.1 Sensory evaluation

Convectively dried product (Drying air temperature-70 °C, Velocity-1m/s) and osmo-convectively dried product (Optimized conditions) were served for the evaluation to a ten panelists at a time. The score sheet (Appendix G) was provided with product and panelists were requested to mark the product according to their liking. The sensory evaluation was carried out for colour, taste and overall acceptability. The independent sample t test was applied to compare between convectively dried and osmo-convectively dried product for various organoleptic characteristics.

**Table 5.40 Mean sensory score data for individual characters**

Character	Mean		Sd		SEd	t
	Convectively dried	Osmo-convectively dried	Convectively dried	Osmo-convectively dried		
Colour	7.3	7.5	0.483	0.527	0.226	0.885 <sup>NS</sup>
Taste	6.1	7.8	0.316	0.422	0.167	10.200**
Overall acceptability	6.3	7.6	0.483	0.516	0.224	5.814**

\*\* Significant at 1% level

NS - Non-significant

The difference between convectively dried and osmo-convectively dried product was significant for taste and overall acceptability at 1 per cent level of significance (Table 5.40) whereas colour was non-significant. Hence the osmo-convectively dried product was found superior with respect to taste and overall acceptability as compared to convectively dried product (without osmo). The osmo-convectively dried product was appreciated by the panelist because of its sweet and sour taste, but convectively dried product (without osmo) showed little bit bitterness. It is in agreement with an earlier study (Raoult-Wack *et al.*, 1991; Allaeddini and Emam-Djomeh, 2004) where osmotic pre-treatment was able to improve quality of dried product.

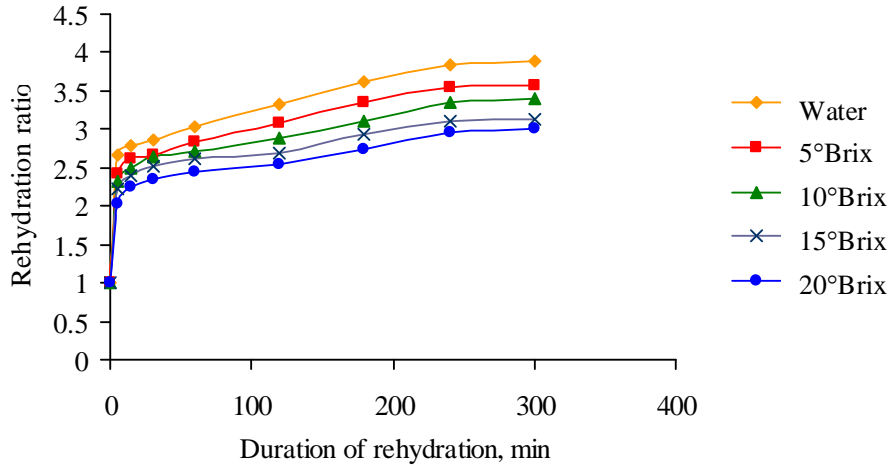
### 5.9.2 Rehydration characteristics of osmo-convectively dried aloe vera samples

The reconstitution qualities of osmo-convectively dried aloe vera samples were determined by conducting re-hydration tests as described in 3.9.5. The osmo-convectively dried samples were immersed in water, 5, 10, 15 and 20 °Brix sugar solution and the mass of the products after every 5, 15, 30 and thereafter every 60 minutes upto 5 hours were measured. The data pertaining to moisture content of the sample during the rehydration tests are reported in Table 5.41. The dehydrated sample absorbed water during rehydration and became soft. Three replications of each sample were rehydrated to avoid any experimental error and average values are reported in Table 5.41.

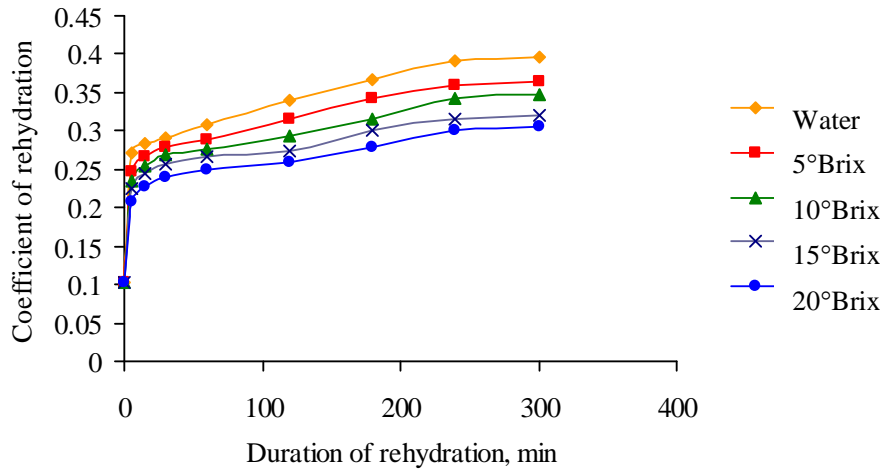
The initial moisture content of the samples was 10.14% (db). The moisture content of the samples increased to 328.35 % during rehydration test when immersed in water for 5 h. Similarly this increment was 293.82, 275.04, 245.85 and 231.15 % for 5, 10, 15 and 20 °Brix sugar solution for the same duration of rehydration (Table 5.41).

**Table 5.41 Moisture content (% db) data during rehydration behaviour of osmo convectively dried aloe vera samples**

S. No.	Time, min	Concentration of sugar solutions , ° Brix				
		0	5	10	15	20
1	0	10.14	10.14	10.14	10.14	10.14
2	5	193.75	165.72	155.04	143.86	123.98
3	15	207.63	187.81	175.42	165.06	146.61
4	30	215.07	201.58	190.18	177.89	159.28
5	60	233.13	213.25	198.55	187.20	170.35
6	120	266.34	240.46	216.89	196.29	178.94
7	180	297.35	269.75	241.34	224.15	202.29
8	240	322.02	289.64	269.59	240.79	224.82
9	300	328.35	293.82	275.04	245.85	231.15



**Fig. 5.30 Rehydration ratio of osmo-convectively dried aloe vera samples**



**Fig. 5.31 Rehydration ratio and coefficient of rehydration of osmo-convectively dried aloe vera samples**

The rehydration ratio and coefficient of rehydration of osmo-convectively dried aloe vera samples were determined using Eqns. (3.19) and (3.20) and are presented in Fig. 5.30 and 5.31, respectively. The rehydration ratio of the samples increased to 3.89 with duration of rehydration when immersed in water for 5 h. Similarly this increment was 3.58, 3.41, 3.14 and 3.01 for 5, 10, 15 and 20 °Brix sugar solution for the same duration of rehydration. Similarly for coefficient of rehydration, the corresponding values were 0.40, 0.36, 0.35, 0.32 and 0.31 for 0, 5, 10, 15 and 20 °Brix sugar solutions for the same duration of rehydration. It can be seen from the figures that maximum rehydration ratio and coefficient of rehydration

were present for the samples dipped in water and minimum for the samples soaked in 20 °Brix sugar solution.

It can be observed from the Figs. 5.30 and 5.31 that moisture absorption during rehydration decreased with increase in the sugar solution concentration from 0 to 20 °Brix for a period of 1 to 5 h. This may be due to the increase in sugar content of the rehydration solution. This is a good feature that the sugar treated materials do not absorb water i.e. their rehydration is less that gives a crisper texture and can be stored for a longer period without impairing quality (Mazza, 1983). These results are also in confirmation with the results obtained by Pokharkar and Prasad (1998a) for osmo-convective drying of pineapple slices and Jain (2007) for osmo-convective drying of papaya.

The osmo-convectively dried products obtained after 5 h of rehydration in distilled water, 5, 10, 15 and 20 °Brix sugar solution were also evaluated for organoleptic quality (*viz.* firmness, taste, flavour, and swelling). For each of the product characteristics, numerical scoring was given by the consumer panel from zero to hundred, representing five quality grades, such as very poor, poor, fair, good and excellent (BIS:6273) (Appendix H). The details of the mean scores given by the consumer panel for individual character are presented in Table 5. 42.

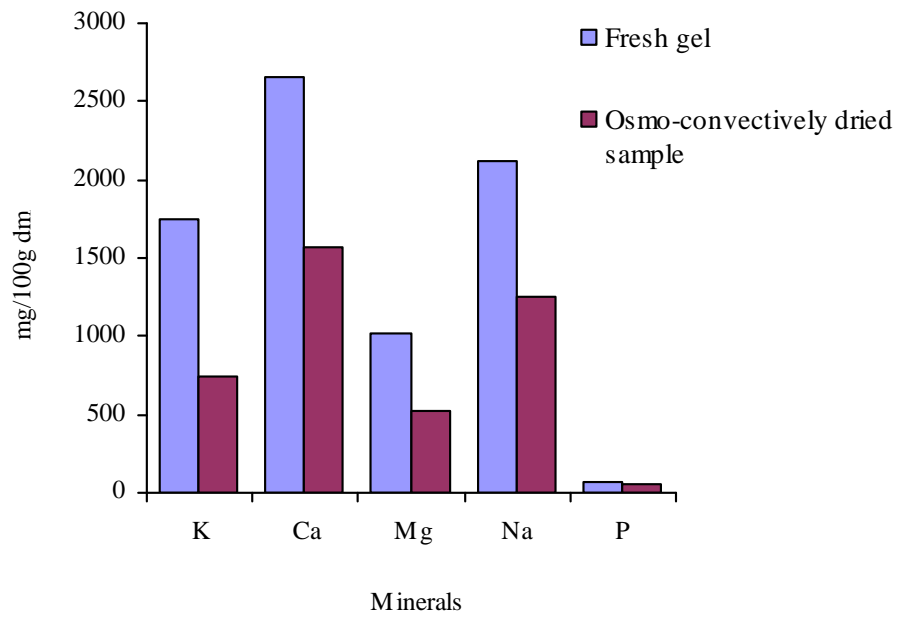
**Table 5.42 Relative rating for quality evaluation of rehydrated aloe vera samples**

Quality characteristics	Rehydrated in				
	Water	Sugar solution, °Brix			
		5	10	15	20
Firmness	44	53	71	74	84
Swelling	87	75	70	56	45
Taste	42	65	75	76	78
Flavour	77	76	74	69	63
Overall acceptability	62.5	67.25	72.5	68.75	67.5

It can be observed from the Table 5.42 that the texture was firmer for the product which was rehydrated in higher concentrated sugar solution. The osmo-convectively dried product rehydrated in water showed higher swelling than product rehydrated in higher concentration of sugar solution because aloe vera samples absorbed more sugar molecules from the solution. It can also be seen that rehydrated product obtained from 10 °Brix sugar solution had a higher overall acceptability rating.

### 5.9.3 Minerals

The osmo-convectively dried product were evaluated for their mineral content and reported in Fig. 5.32. Ca and Na were found the predominant mineral elements in the fresh and osmo-convectively dried aloe vera sample (Fig. 5.32). The peak value in the fresh gel were  $2658.09 \pm 92$  mg/100 g dm and  $2118 \pm 43.31$  mg/100 g dm for Ca and Na respectively, followed by K ( $1741.02 \pm 37.94$  mg/100 g dm), Mg ( $1013.33 \pm 36.02$  mg/100 g dm) and P ( $67.67 \pm 14.01$  mg/100 g dm). The Ca and Na contents in the osmo-convectively dried sample were 59.04 and 59.43 per cent whereas K, Mg and P contents were 42.30, 51.75 and 75.85 per cent respectively of the fresh gel. Similar results were reported for aloe vera by Femenia *et al.*, (1999), Garcia- Hernandez *et al.*, (2006) and Miranda *et al.*, (2009a). However all the osmo-convectively dried aloe vera sample showed considerable decrease in mineral content with respect to the fresh samples may be due to leaching of inorganic compounds into osmotic solution during osmosis (Sablani, 2006). Nevertheless, the mineral content may vary widely among vegetables, depending on several factors such as ripeness, variety, soil type, the use of fertilizers, intensity and exposure time to sunlight, temperature, rain and cultivation area (Garcia – Hernandez *et al.*, 2006). Among the minerals studied, the level of Ca was high ( $1569.33 \pm 44.11$  mg/100 g dm) followed by Na ( $1258.67 \pm 38.07$  mg/100 g dm) in osmo-convectively dried samples. Ca is important for human given its role in bone and tooth development (Ozcan and Hacisferogullari, 2007). Furthermore, Ca may be used for the prevention and treatment of hypertension, since it stabilizes vascular membranes, blocks its own entry into cells and reduces vasoconstriction. In combination of other similar ions such as  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$  provides an ionic balance for the vascular membrane, promoting vasorelaxation and a reduction in blood pressure (Houston, 2005). Similarly, the presence of Ca is of special technological interest, since  $\text{Ca}^{2+}$  ions have the capacity to form bridges between galacturonic acid and adjacent units of pectic chains, and therefore can significantly influence the texture and porosity of tissues post-processing (Alonso *et al.*, 1995). The Mg and K content in osmo-convectively dried sample was observed to be  $524.41 \pm 22.48$  mg/100 g dm and  $736.47 \pm 37.94$  mg/100 g dm. Mg and K are important in that many enzymes use them as cofactors (Ozcan and Hacisferogullari, 2007). Femenia *et al.*, (1999) suggested that the level of K in aloe vera could affect its healing properties. Garcia-Hernandez *et al.*, (2006) reported similar results for the mineral content of aloe vera; moreover they suggested that the plant has greater efficiency in terms of these inorganic elements, because plants grown in nutrient rich soils do not tend to accumulate these minerals in their leaves. The P content in osmo-convectively dried sample was found to be  $51.33 \pm 6.11$  mg/100 g dm. Phosphorus is the second most abundant mineral in the human body. Together with Ca, P constitutes part of the inorganic structure of bones. Phosphorus also contributes to the maintenance of acid base equilibrium in the blood (Martinez and Garcia, 2001).



**Fig. 5.32 Mineral content of fresh and osmo-convectively dried aloe vera sample**

## CHAPTER VI

### SUMMARY AND CONCLUSIONS

Aloe vera (*Aloe barbadensis* Miller) is a member of the family Liliaceae, which comprises more than 360 different species found in the arid regions of Africa, Asia, Europe and the America. It is widely used as a natural treatment and alternative therapy for various types of diseases and several studies have suggested the healing, cosmetic and nutritional benefits of aloe vera (Femenia, *et al.*, 1999, 2003; Simal *et al.*., 2000; Chang *et al.*, 2006; Vega-Galvez, *et al.*, 2007). Accordingly to a report on aloe vera cultivation, published by [IASC \(International Aloe Science Council\)](#), there are close to 23,600 hectares of aloe vera being cultivated worldwide. In India total production of aloe vera is estimated to be about 1, 00,000 tonnes (Anonymous, 2006). The annual consumption of aloe vera extract by the Indian pharmaceutical industries is about 200 tonnes.

Aloe vera, an industrial crop, has long been used in food, pharmaceutical, cosmetic and toiletry industry. Unfortunately, because of improper processing procedure, many of these so-called aloe products contain very little or virtually no active ingredients. In view of the known wide spectrum of biological activities possessed by the leaves of the aloe vera plant and its wide spread use, it has become very important to evolve a better method of preservation for increasing the shelf life and maintaining the quality of aloe vera gel. Drying or dehydration have inherent advantages, such as longer periods of storage, minimize packaging requirements and reduce shipping weights (Okos *et al.*,1992). Generally drying has been done with convective dryers, but some physical properties are changed by this techniques e.g. colour, texture, flavour, nutrients and rehydration quality (Krokida *et al.*, 2003). So, in order to improve the final product quality and drying rate of high moisture materials with low energy consumption, pretreatment prior to drying can be considered.

Osmotic dehydration is less energy intensive technique than air or vacuum drying process, since it can be conducted at low or ambient temperature. It is the process of water removal from a product by immersion in concentrated aqueous solution. The driving force for water removal is the concentration gradient between the solution and the intercellular fluid of the product. The osmotic dehydration process may not yield a product of low moisture content enough to make it shelf stable, and therefore further processing, such as air drying, vacuum drying or freeze drying is necessary (Ponting *et al.*, 1966). Osmotic dehydration process besides being energy efficient process, leads to better quality and stability of product

during storage. The limitation of the osmotic dehydration process is the increase in sweetness and decrease in acidity of the product. However, it may be possible to control the rate of solute diffusion and optimize the process (Rahman, 1992).

The advantage of osmo-convective drying process for aloe vera gel necessitates the study of osmotic kinetics and air drying behaviour of osmosed product. As controlling and regulating the osmotic process plays an important role in improving the quality of the dehydrated product and this can be achieved by optimization of osmotic dehydration process. Also, there is a need to investigate the osmo-convective drying process in order to get shelf stable quality dehydrated products. Air drying leads to physical and biochemical change in the food. These changes are frequently disastrous for the product quality (browning reaction and vitamin destruction). So, to improve the product quality it is important to optimize the drying conditions. The quality of the product obtained by optimized treatment needs to be evaluated for the acceptance of the consumer. In addition, the functional characteristics of the final dehydrated product obtained by optimized input parameters such as rehydration and mineral content retention are essential to get better picture about the product.

The present investigation was undertaken to study the process of osmotic dehydration and convective drying of aloe vera samples with the following specific objectives:

6. To study osmotic dehydration behaviour of aloe vera gel.
7. To optimize the process parameters for osmotic dehydration of aloe vera gel.
8. To study convective drying behaviour of osmotically dehydrated aloe vera gel
9. To optimize drying parameters for convective drying of osmotically dehydrated aloe vera gel.
10. To evaluate quality of dried product

Initially, the physical properties of aloe vera leaf namely size, leaf weight and pulp weight were studied. The average values of tri-axial dimensions viz. length, width and thickness of aloe vera leaves were found to be 493.18, 92.73 and 19.77 mm respectively with apparent volume of leaf 236.77 cc. The average leaf weight, gel weight and gel recovery was found to be 65.24 g, 43.85 g and 2.59 per cent respectively.

An 'aloe vera peeler' was developed for removal of epidermis (skin) from aloe vera leaves during sample preparation and found that, use of the peeler could save 66.67 per cent time during peeling of aloe vera leaves as compared to manual operation.

Fresh aloe vera leaves (*Aloe barbadensis* Miller) were obtained from Banki Research Farm, Sisarama, Department of Forest, Udaipur (Rajasthan). The two years old and matured aloe vera leaves were selected for the study. Initial moisture content of aloe vera (gel) samples varied in the range of 98.13 - 99.16 per cent (wb). The osmotic dehydration process depends on many factors, such as type of osmotic agent, concentration of solution, osmotic temperature, agitation of osmotic solution, solution to fruit ratio, the duration of osmosis and size and shape of the product (Ponting *et al.*, 1966). However, in the present preliminary investigation only effect of solution concentration (10 to 80 °Brix), osmotic temperature (30 to 70 °C) and duration of osmosis (1 to 10 h) were studied to fix the range of input parameters for optimization of osmotic dehydration process. The mass transport data, *viz.* water loss and sugar gain were used to study the effect of independent variables. It was observed that higher rate of water loss was achieved up to 4 h, whereas the rate of sugar gain increased steadily upto 4 h and was found negligible with further advancement of time. Similarly, water loss and sugar gain increased appreciably with increase in syrup concentration upto 40 °Brix and to maximum for sugar syrup temperature upto 50 °C. The constant syrup to sample ratio of 5:1 was used in all osmotic dehydration experiment. Based on this study, the input parameters selected for optimization of osmotic dehydration of aloe vera sample were, sugar syrup temperature - 30, 40 and 50 °C ; sugar syrup concentration - 10, 25 and 40 °Brix and duration of osmosis - 60, 150 and 240 min. Considering the limitations of the sugar gain in the product quality, the osmo-convectively dried products having different sugar gain were organoleptically evaluated and product having 4.59 percent sugar gain was found to be acceptable by the consumer panel. This was considered as a targeted response along with maximum water loss during optimization of input parameters for osmotic dehydration of aloe vera sample.

The experiments on the osmotic dehydration of aloe vera sample were conducted in order to study its kinetics on the basis of preliminary experiment. The osmotic dehydration was carried out up to 240 min (10, 30, 60, 90, 120, 150, 180, 210 and 240 min) with 10, 25 and 40 °Brix sugar syrup at 30, 40 and 50 °C syrup temperature and constant syrup to sample ratio (5:1). The syrup in the beakers was manually stirred at regular intervals to maintain uniform temperature. The minimum and maximum mass reduction, water loss and sugar gain after osmotic dehydration were observed in the range of 35.17 to 47.32, 36.82 to 53.98 and 1.65 to 6.66 per cent respectively corresponding to low levels (10°Brix, 30°C after 4 h) and high levels (40 °Brix, 50 °C after 4 h) of syrup concentration and temperature respectively. In all the experiments, the water loss from the aloe vera samples was very rapid in the beginning of process particularly for the first hour of osmosis to the tune of 28.01 percent to 43.06 percent corresponding to low levels (10°Brix, 30°C) and high levels (40 °Brix, 50 °C) of syrup

concentration and temperature respectively and decreased gradually with further increase of duration of osmosis, but did not approach the equilibrium. It indicated that an increase in concentration of sugar solution had more influence on water loss and sugar gain than an increase in temperature in the study range. The water and sugar diffusivity varied in the range from  $4.2559 \times 10^{-9}$  to  $7.1247 \times 10^{-9}$  m<sup>2</sup>/s and  $0.7430 \times 10^{-8}$  to  $3.7826 \times 10^{-8}$  m<sup>2</sup>/s respectively. The mathematical models for osmotic dehydration based on the Fick's law of diffusion were developed and found quite adequate to predict the mass transport data. The activation energy was found to be decreased for water diffusion (15.72 to 11.10 kJ/mol), but increased for sugar diffusion (24.22 to 34.65 kJ/mol), with increase in syrup concentration. This clearly explained that more amount of energy was required for sugar diffusion in higher concentration of syrup, but a reverse trend was observed in water diffusion process.

The optimization of osmotic dehydration process parameters such as syrup concentration, syrup temperature and duration of osmotic dehydration was necessary so that maximum water loss could be achieved with optimum sugar gain (4.59 %). The process parameters for osmotic dehydration of aloe vera gel were optimized using response surface methodology. Numerical and graphical multiresponse optimization technique were adopted for the process parameters for the osmotic dehydration of aloe vera gel. Sugar syrup temperature (30-50 °C), syrup concentration (10-40 °Brix) and duration of osmosis (60-240 min) were the factors investigated with respect to water loss (WL) and sugar gain (SG). The solution to sample ratio of 5/1 (w/w) was used. The Box- Behnken design of three variables and three levels including 17 experiments formed by 5 central points was used for optimizing input parameters. With respect to water loss and solid gain, the linear, quadratic and interaction effects of three variables were analysed. For each response, second order polynomial models were developed using multiple regression analysis. Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted models. The response surfaces and contour maps showing the interaction of process variables were constructed. The result of analysis of variance indicated that the linear terms of syrup temperature, syrup concentration and duration of osmosis were highly significant at 1 per cent level for both the responses viz. water loss and sugar gain. The F values indicated that concentration of syrup was the most influencing factor followed by duration of osmosis and temperature of syrup for both the responses viz. water loss and sugar gain. As per graphical optimization technique, the superimposed contours of all responses for concentration of syrup (C), temperature of syrup (T) and duration of osmosis ( $\theta$ ) and their intersection zone for maximum water loss and targeted sugar gain (4.59%) indicated the range of optimum values of process variables as follows,

Temperature of syrup (T)	:	30-38 °C
Concentration of syrup (C)	:	18-29 °Brix
Duration of osmosis ( $\theta$ )	:	77-150 min

According to numerical optimization technique, the optimum operating conditions were found to be syrup temperature of 31.44 °C, syrup concentration of 25.35°Brix and osmosis time of 128.73 min. At this optimum point, water loss and sugar gain were predicted to be 39.69 % and 4.45 % respectively.

Osmotic dehydration experiments were conducted at the optimum input parameters for testing the adequacy of model equations for predicting both the response values. It could be concluded from the results that model equations developed for water loss and sugar gain are quite adequate to assess the behavior of the osmotic dehydration.

The convective drying behaviour of osmotically dehydrated aloe vera sample (at optimized conditions) was assessed at drying air temperatures of 50, 60, 70 and 80 °C with drying air velocity of 1, 2 and 3 m/s. The initial moisture content of the osmotically dehydrated aloe vera samples was ranging from 976.45 to 982.31 per cent (db) for all the samples investigated and after drying upto (nearly) constant weight, the moisture content was reduced to 8.83 to 19.29 (db). It was observed that the moisture content of the product was reduced exponentially with drying time and no constant rate period was observed. It was also seen that increasing the air velocity from 1 to 3 m/s in the temperature range of 50 to 80°C did not change the drying rate appreciably. This indicated that the total mass transfer resistance was primarily due to the internal moisture diffusion within the sample. It could be seen that there was a wide variation in drying time from 180 to 780 min for the range of drying air temperatures and air velocities taken for study. It could also be seen that minimum time in drying was observed for higher air temperature (80°C) and maximum time was recorded for low air temperature (50°C) for all air velocities. Air temperature was observed to be more influencing factor for reduction of drying time than air velocity. It also observed that, the drying rate increased with increase in drying air temperature. Drying air velocity had little effect on drying rate.

The mass transport data during air drying of osmotically dehydrated aloe vera samples showed that the moisture diffusivity increased from  $0.959 \times 10^{-9}$  to  $10.405 \times 10^{-9} \text{ m}^2/\text{s}$  with increase in temperature from 50 to 80°C. The activation energy for moisture diffusion process was 57.164, 58.835 and 64.636 kJ/mol for 1, 2 and 3 m/s air velocity respectively. The drying data were statistically analyzed to develop a semi empirical equation demonstrating the relationship between moisture diffusivity and other related process

parameters (air temperature and air velocity) using the regression technique. The mathematical expression showed that the drying air temperature had a pronounced influence on the moisture diffusivity, whereas, the effect of air velocity was very limited as indicated by its predicted values.

To determine the most suitable drying equation the moisture ratio data of osmosed aloe vera sample dried at various air temperatures and velocity of air were fitted into the six thin-layer drying models (Exponential, Henderson and Pabis, Page, Modified page, Logarithmic and Power law) in their linearized form using regression technique. Henderson and Pabis model was found to be the most satisfactory than the other models to represent the thin-layer drying of aloe vera samples on the basis of highest values of  $R^2$  and the lowest value of reduced mean square of the deviation ( $\chi^2$ ) and root mean square error ( $E_{RMS}$ ).

The optimization of input parameters (drying air temperature and air velocity) of convective drying was carried out using numerical optimization technique with the help of Design Expert 8.0.4.1 statistical software (trial version) by adopting full factorial design on the basis of various quality parameters viz. ascorbic acid, hardness, colour (L value) as well as water activity. Study revealed that, convective drying of osmotically dehydrated aloe vera sample with 70°C drying air temperature and 1 m/s air velocity was best for optimum responses among the range of variables taken for the study.

With the optimized input parameters the osmo-convectively dried aloe vera product having 10.14 % (db) moisture content, 848.56 g hardness and 0.332 water activity could be obtained within 360 min. The osmo-convectively dried aloe vera samples with optimized input parameters were further studied for their quality aspects, such as sensory evaluation, re-hydration ratio and mineral contents. Sensory evaluation revealed that, the osmo-convectively dried product was appreciated by the consumers than convectively dried product (without osmo). The rehydration ratio of the osmo-convectively dried aloe vera samples was in the range of 3.14 to 3.89. The moisture absorption during rehydration was observed to decrease with increase in the concentration of sugar solution from 0 to 20°Brix for the period of 1 to 5 h. The osmo-dehydrated product absorbed less water in sugar solution, resulting in a product of crispier texture. The rehydrated product obtained from 10°Brix sugar solution had a highest overall acceptability rating. The osmodehydrated aloe vera sample convectively dried with optimized input parameters could retain 28.74 mg/100 g dm ascorbic acid. Among the minerals studied, the level of Ca was high.(1569.33 ± 44.11 mg/100 g dm) followed by Na (1258.67 ± 38.07 mg/100 g dm) in osmo-convectively dried samples. The Mg, K and P content in osmo-convectively dried sample were observed to be 524.41 ± 22.48 mg/100 g dm, 736.47 ± 37.94 and 51.33 ± 6.11 mg per 100 g dry matter respectively.

Based on the results of the investigations, the following conclusions were drawn:

1. The average values of length, width, thickness and apparent volume of aloe vera leaves studied were observed to be 493.18 mm, 92.73 mm, 19.77 mm and 236.77 cc respectively. So also, the average values of corresponding leaf weight, gel weight and gel recovery were found to be 428.91 g, 213.6 g and 49.44 per cent respectively. Third order polynomial equations were developed to establish relationship between leaf weight and apparent volume, leaf weight and gel recovery as well as apparent volume and gel recovery.
2. Use of developed aloe vera peeler could save 66.67 per cent time during operation of epidermis removal from aloe vera leaves as compared to manual operation.
3. Osmotic dehydration of aloe vera samples was influenced by syrup concentration, temperature and duration of osmosis.
4. The osmo-dehydrated aloe vera samples with 4.59 per cent sugar gain gave excellent acceptability and the same level was considered for optimization of osmotic dehydration.
5. Water loss and sugar gain both increased with increase in syrup concentration and temperature.
6. After 4 h of osmotic dehydration, the minimum and maximum mass reduction, water loss and sugar gain were in the range of 35.17 to 47.32, 36.82 to 53.98 and 1.65 to 6.66 per cent corresponding to low levels (10°Brix, 30°C) and high levels (40 °Brix, 50 °C) of syrup concentration and temperature respectively.
7. Water loss from the aloe vera samples was very rapid for the first hour of osmosis to the tune of 28.01 percent to 43.06 per cent corresponding to low levels (10°Brix, 30°C) and high levels (40 °Brix, 50 °C) of syrup concentration and temperature respectively and decreased gradually with further increase of duration of osmosis, but did not approach the equilibrium.
8. The water and sugar diffusivity varied in the range from  $4.2559 \times 10^{-9}$  to  $7.1247 \times 10^{-9}$  m<sup>2</sup>/s and  $0.7430 \times 10^{-8}$  to  $3.7826 \times 10^{-8}$  m<sup>2</sup>/s respectively.
9. The mathematical model for osmotic dehydration and air drying based on the Fick's law of diffusion were found quite adequate to predict the mass transport data during osmotic dehydration and air drying process respectively.

10. The activation energy was found to be decreased for water diffusion (15.72 to 11.10 kJ/mol), but increased for sugar diffusion (24.22 to 34.65 kJ/mol), with increase in syrup concentration.
11. Model equations developed for water loss and sugar gain were found quite adequate to assess the behavior of the osmotic dehydration.
12. Concentration of syrup was found to be the most influencing factor followed by duration of osmosis and temperature of syrup was least effective for both the responses viz. water loss and sugar gain.
13. For maximum water loss and optimum sugar gain, the optimum input parameters were found to be, syrup concentration - 25.35 °Brix, syrup temperature - 31.44 °C and duration of osmosis -128.73 min for osmotic dehydration of aloe vera samples. The predicted water loss and sugar gain was 39.69 per cent and 4.45 per cent respectively at optimized input parameters.
14. Air drying of osmotically dehydrated aloe vera samples exhibited falling rate period of drying. Drying air velocity had little effect on the drying rate.
15. There was a wide variation in drying time from 180 to 780 min for the range of drying air temperatures (50 – 80 °C) and air velocities (1-3 m/s) taken for study. Minimum drying time was observed for high air temperature (80°C) and maximum time was recorded for low air temperature (50°C) for all air velocities.
16. The moisture diffusivity varied in the range of  $0.959 \times 10^{-9}$  to  $10.405 \times 10^{-9}$  m<sup>2</sup>/s during air drying depending on the drying air temperature and air velocity.
17. The activation energy for moisture diffusion process was 57.164, 58.835 and 64.636 kJ/mol for 1, 2 and 3 m/s air velocity respectively.
18. Henderson and Pabis model was found to be the most satisfactory to represent the thin-layer drying of osmo-dehydrated aloe vera.
19. The input parameters were optimized to 70°C drying air temperature and 1 m/s air velocity for better response during convective drying of osmotically dehydrated aloe vera sample. With these input parameters the osmo-convectively dried aloe vera product having 10.14 % (db) moisture content, 848.56 g hardness and 0.332 water activity could be obtained within 360 min.
20. The osmodehydrated aloe vera sample convectively dried with optimized input parameters could retain 27.34 mg/100 g dm ascorbic acid, 1569.33 mg/100 g dm Ca, 1258.67 mg/100 g dm Na, 736.47 mg/ 100g dm K, 524.41 mg/100g dm Mg and 51.33 mg/100g dm P.

21. The rehydration ratio of the osmo-convectively dried aloe vera samples was in the range of 3.01 to 3.89.
22. The moisture absorption during rehydration decreased with increase in the concentration of sugar solution from 0 to 20°Brix for the period of 1 to 5 h. The rehydrated product obtained from 10°Brix sugar solution had a highest overall acceptability rating.
23. Osmo-convectively dried aloe vera product at optimized input parameters was highly appreciated by the consumers than convectively dried product (without osmo).

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