COLD GRINDING TECHNOLOGY OF FEW SPICES

THESIS

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1998

CERTIFICATE - I

This is to certify that the thesis entitled "COLD GRINDING TECHNOLOGY OF FEW SPICES" submitted in partial fulfilment of the requirements for the degree of MASTER OF SCIENCE IN AGRICULTURE of the Jawaharlal Nehru Krishi Vishwa Vidyalaya, Jabalpur, is a record of the bonafied research work carried out by SHRI D.C. SHRIVASTAVA under my guidance and supervision. The subject of the thesis has been approved by the student's advisory committee and Director of Instructions.

No part of the thesis has been submitted for any other degree or diploma (certificate awarded etc.) or has been published/published part has been fully acknowledged. All the assistance and help received during the course of the investigation has been duly acknowledge by him.

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CERTIFICATE - II

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ABBREVIATIONS

ac Critical relative humidity

Cp Specific heat at constance pressure.

°C Degree centrigate

CG-S Cold ground samples

CON-S Conventional ground sample

d.b. Dry basis

D- Diameter

Df Temperature differences

Eq Equation

Fig Figures

g Gram

hc Surface heat transfer (JM-2S-1°C-1)

k Thermal conductivity (JM⁻¹S⁻¹°C⁻¹)

Kg Kilogram

M Moisture content at any time "t"

Mc Critical moisture content

Mo Initial moisture content %

Me Equlibrium moisture content %

NRe Reynolde number

Nu Nusselt number

Pr. Prandtl number

Q Rate of heat loss, J/S

RH Relative Humidity

```
t Storage time in days
T' Temperature in °k
V Velocity(ms-1)
```

X Mean

P Density Kg/m³

 μ Viscosity(MSm⁻²)

CHAPIER - I INTRODUCTION

INTRODUCTION

occupies the place of pride since time immemorial in the production and export of spices. Spice production, processing and trade now have a prominent role in national economy. They provide employment to millions rural people and earn substantial foreign exchange for the late, there is a substantial increase Of in the It is estimated that the production of spices. present production of spices is around 25 lakh tonnes per Spices export have also shown a remarkable improvement recent years. During 1995-96, the country earned Rs. 785.89 foreign exchange through the export of 202.197 crores as tonnes of spices Whereas during April-Dec. '1997, it earned about Rs. 955.00 crores (Table -1). Considering the demand in the domestic consumption and export, a target of per cent annual growth rate, in production and 15-30 per cent in the export is being envisaged during the Ninth Plan period. therefore, imperative to increase the production, productivity and diversify the spice processed value-added products of high quality.

The major drive in the area of food processing and preservation is towards reducing the loss of organoleptic properties of food during processing and preservation. Spices are valued for their flavour which consists of both aromatic

Table 1 : SPICE EXPORTS FROM INDIA

Item	Qty.	APR.DEC 1997 Value
Pepper	26,000	344.94
Cardamom	200.00	0.67
Chilli	35,950	110.99
Ginger	15,050	42.34
Turmeric	19,900	55.76
Coriander	14,950	41.08
Cumin	7,400	36.77
Celery	2,500	5.85
Fennel	10,250	30.41
Fenugreek	3,200	4.97
Garlic	2,520	5.40
Curry pow⊖der	3,825	16.05
Spice Oils Oleoresins	1,505	45.56
Total :	1,60960	955.00

Value in Rs. Grores Quantity in tonnes

Source : Process food industry, 1998

volatiles and non-volatile constituents which are equally important to impart the desired flavour. Thus, the processing steps should ensure proper conservation of the above qualities and keeping the loss at minimum, so as to fetch premium price in International trade.

Food industry employs conventional grinders used chemical industry for size reduction, where raw materials non-biological in nature. These grinders usually have inherent disadvantages of high heat generation during grinding and low efficiency. This method of grinding, is not suitable for heat sensitive, high fat content and fibrous materials of including spices. Not origins, much research is development of specific grinders directed towards for food applications.

grinding of spices, the mill In conventional and product high temperature rises 95°C as as and at high temperatures, there will be considerable loss in volatile oil in the volatile constituents. and changes An alternative method is freeze-grind the material under cryogenic condition create a cold environment around the mill that grinding operation takes place in a chilled condition. Murthy et al. (1995) have compared the retention of black pepper volatiles obtained from Chilled, ambient and cryogrinding and reported that there were not much difference in

the volatile oil content of chilled and cryogenic-ground black pepper. Cryogenic technology has some limitations, viz;(i) liquid nitrogen is needed for this purpose, (ii) trained operators are required to handle as this causes sever cold burns (iii) high investment on imported equipments. Therefore, cold or chilled grinding technique offers a less sophisticated less capital investment and easier operating system.

There is a need to optimize the process parameters viz; the ground material temperature, types of grinder, rpm of the grinder, powder particle size, feed rate of the spice and coolant, type of cooling etc; which give the best quality product at the minimum cost. These conditions are product specific and depends on the physical characteristics of the spices.

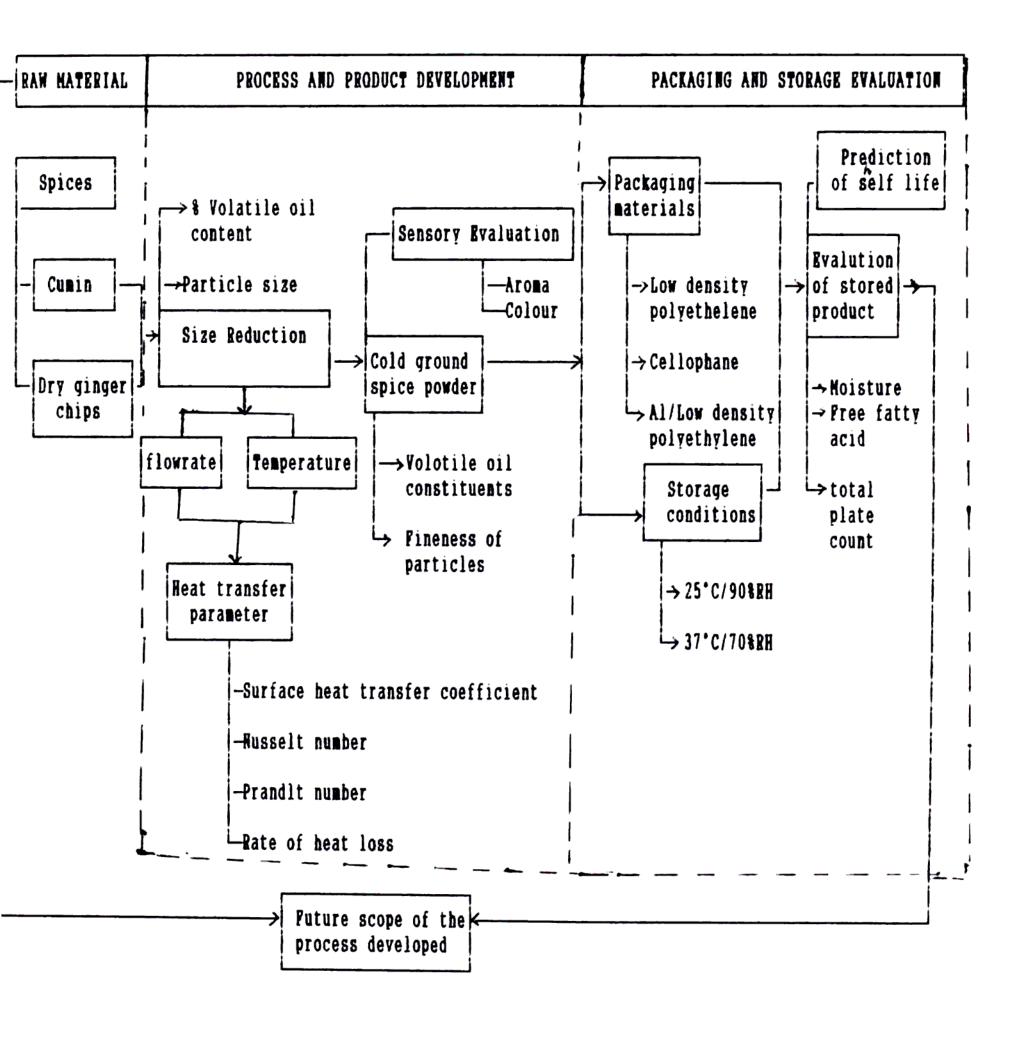
The present series of investigations were undertaken to make systematic study on cold grinding process, rate of heat loss during grinding, packaging and storage study of cold ground cumin and dry ginger powder with the following objectives:

 To study effect of machine variables and grinding temperature on the fineness and volatile oil content of ground spices.

- To determine the various heat transfer parameters during cold grinding of spices.
- 3. To study the biochemical and microbiological changes during storage of both conventional(ambient) and cold ground spices.

The details of the investigations are presented in project design (Figure.I) formulated for the purpose.

FIGURE I : PROJECT DESIGN



CHAPIER - II REVIEW OF LITERATURE

REVIEW OF LITERATURE

This chapter deals with the review of pertinent literatures on chemical composition, microbiological status, size reduction process technologies, isotherm of food, packaging and storage conditions for spices.

2.1 Chemical composition of spices oils:

Spices like cardamom, ginger, Cumin and coriander etc. have volatile oils which give them attractive spicy odours. These oils can be obtained from the ground spices by steam distillation. Although some of these have a more attractive odour when they are fresh.

Dried ginger (Zingiber officinale Rose) contains about 1.5 to 3.5 percent, Volatile oil depending upon varieties. The pungency of ginger is due to compound gingerol (Pruthi, 1980).

Cumin seeds (Cuminum cyminum L.) contains about 2-4% volatile oil and its active principle is an aldehyde "cumirol", yield of volatile oils from few spices are given The in the 2.1. Lewis and Krishnamurthy (1974) reported that table the spice oils are also made up of terpene, sesquinterpenes and other corresponding oxygenated derivatives (Table 2.2). The amount of hydrocarbons in some oil is small (5-10%), in others

Tabel 2.1: Essential oil contents of important spices.

Spice	% Oil	
Cardamom(seeds)	5.0- 7.0	
Ginger	1.5- 3.5	
Pepper	1.5- 3.5	
Turmeric	1.5- 4.0	
Cloves	16.0- 18	
Nutmeg and Mace	5.0- 6.0	
Cinnamon and Cassia	2.0- 3.5	
Coriander	0.5- 2.0	
Cumin	2.0- 4.0	
Celery	2.0- 2.5	

Tabel 2.2: Approximate composition of spice oils.

Spice	Monoterpens %	Sesquiterpenes	Oxygenated compounds
Pepper	70	25	05
ginger	05	65	30
Cardamom	08	· ——	92
Turmeric	10	25	65
Cloves	02	08	90
Nutmeg	90	_	10
Cinnamon	10	04	86
Coriander	20	-	80
Cumin	50	_	50
Celery	60	15	25

they form 95% of the oil. The oxygenated derivatives are recognised as the most important odoriferous compounds in the oil.

2.2 Microbiological profile of spices

Spices are known to carry heavy microbiological contamination. Pruthi (1964) and Krishnaswamy et al. (1974) reported that spices are infested with bacteria, yeast and molds. Krishnaswamy et al. (1974) have studied the type of coliforms, aerobic spore formers, yeasts and molds in some of spices. Cloves, currypowder turmeric, onion, garlic, mustard, black pepper and chilli powder were found to carry heavy loads, of mold (Table 2.3).

Singh et al. (1988) reported 21 x 10⁴ to 97 x 10⁵ total bacterial count ,25 x 10¹ to 11 x 10⁴ coliforms, 15 x 10^1 to 66×10^3 spore formers, 27 x 10^1 yeast and 11 x 10^1 to 89 x 10^2 molds per gram of sample, respectively in black pepper, turmeric and chilli powder.

Schinder and Lisenberg (1968), Pal and Kundu (1972) reported that different strains of Aspergillus and Penicillium produced aflatoxin on different Indian spices. Shrivastava and Jain (1992) reported fungal contaminants namely, Aspergillus flavous, A. niger, A. ochracens, Penicillium spp. Rhizopus arrhizus. R. stolonifer and Syneephalastrum racemosum

Table 2.3: Mcrobiological profile of spices.

Spice	Standard plate count	Coliforms	Yeast and molds	Aerobic thermophiles	Aerobic spore formers	Mesophilic putrefactives	Salm- onella
Black pepper	10x10 ³ to9x10 ⁷	0 to 24x10 ⁷	40 to 93x10 ³	2x10 ² to 8x10 ⁴	4x10 ² to 10x10 ⁴	0-102	-ve
Dry ginger	40x104 to 8x106	0 to > 2400	8x10 ² to 44x10 ³	16x104 to 83x104	45x10 ² to 10x16 ⁶	10x10 ³ to 10x10 ⁷	-ve
cardamom	4x10 ³ to 14x10 ⁵	0 to 60	0 to 8x10 ³	0 to 23x10 ³	0 to 22x10 ³	0-90	-ve
Dehydrated Onion	7x10 ³ to 10x10 ⁴	NOTE SEAS (\$20)	0 to 53x10 ²	0 to 53x10 ²	3x10 ² to 13x10 ³		-ve

in seeds of bistop weed, black pepper, coriander and cumin.

Singh (1996) reported, the microbiological profile of thermally processed, and unprocessed red chilli powder were consisted of <u>Aspergillus</u> and <u>Rhizopus</u> spp. of fungus.

2.3 Size reduction processes of spices :

Grinding of spices is an important step in processing, as it involves the loss of volatile oil and aroma present in them. Generally, spices are ground either for direct use or for making value-added products like oleoresins or oils from them. Usually, grinding facilitates the release of aroma/flavour principles and better uniform mixing with food materials.

2.3.1 Mechanism and energy requirement in size reduction :

reduction can Size achieved be by different mechanisms may be compression, cutting, mechanisms. These impact, shear and rubbing etc. Sometimes two more or mechanisms may play a role during size reduction or grinding. Table (2.4) shows grinders of different mechanisms with their peripheral speeds, with product types (Loncin and Merson. 1979).

Grinding is an energy intensive operation wherein almost 99% of the energy appears as heat resulting in high

Table 2.4; Type of grinders and applications.

Name of the equipments	Methods of size reduction	Peripheral speed(m/s)	Product example
Pinned Disk Mill	Impect	80-160	Papper, Nut, Clove, Must- ard, Roasted nut, Sugar, Cocoa.
Hammer Mill	Impact	40-50	Sugar agglomerate, Cocoa Press cake, Tapioca, Dry- vagetables, Extracted - Bones
Blast Mill	Impact	40-110	Papper, Pactin, Cocoa, dry ugs, Tobaco leaver and stems.
Toothed Disk Mill	Abrasion	5-16	Wheat, Pepper, Linseed, Bitter orange, Rough- grinding of rye, corn, junifer berries
Cutting Granulator	Section	5-18	Fish meal, Dried fruits and vegetable, frozen coffee extract, Cocoa press cake,

product and equipment temperatures (Loncin and Merson, 1979).

Remaining only 1% of energy is useful in creating new surfaces. There is no general method for predicting the energy needed for size reduction. However, the following laws can be used for theoretical estimation of energy for grinding of solids (Loncin and Merson, 1979):

Kick's law: $E = K_1 \log (D_1/D_2)$

Rittinger's law : $E_2 = K_2 (1/D_2 - 1/D_1)$

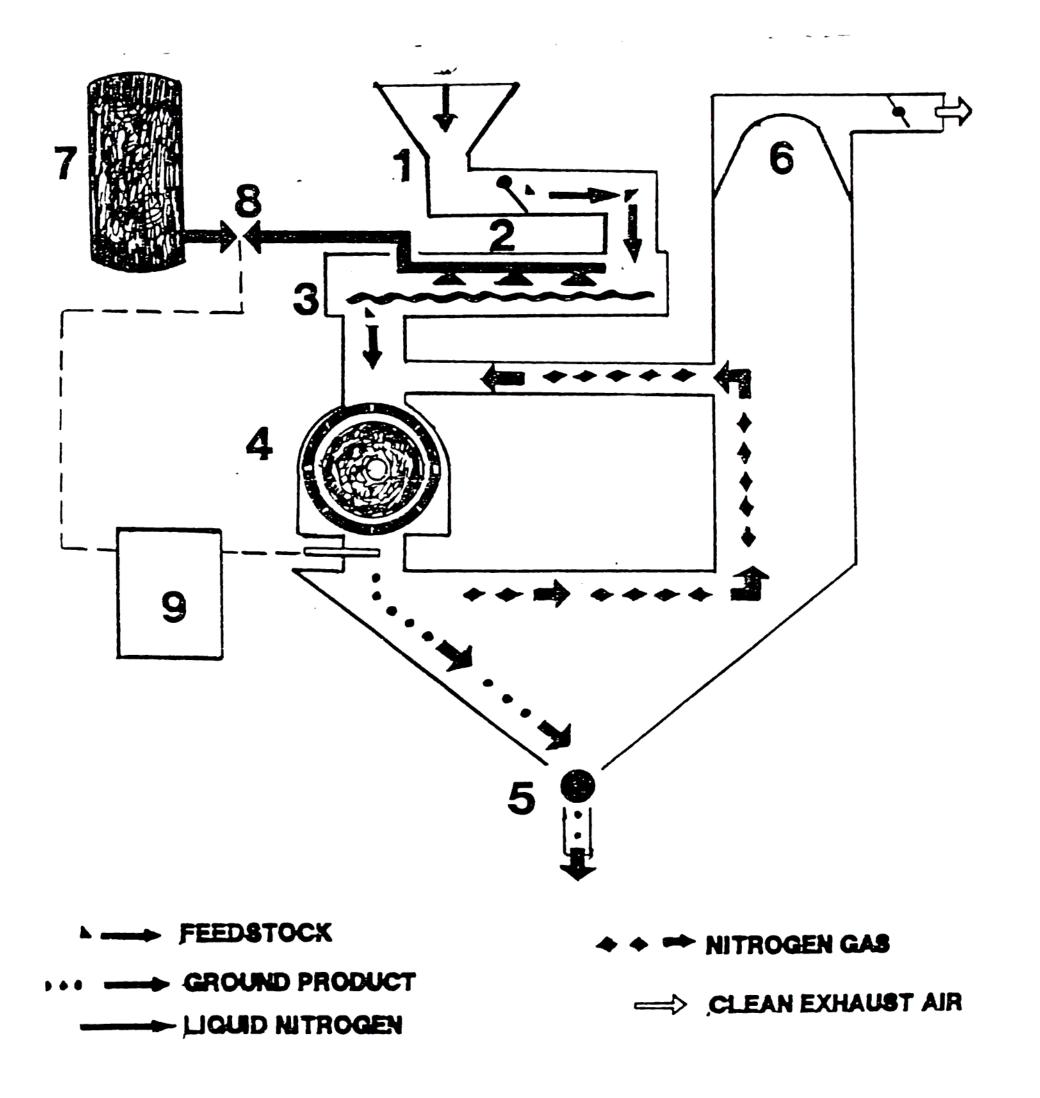
Bond's law: $E/Wi = (100 - D_2)^{1/2} - (100/D_1)^{1/2}$

Where, D_1 D_2 are initial and final characteristic size of particles, K_1 , K_2 are Constants, W_1 , is the work index and E, is the required energy.

conventional grinding of spices, the mill and In product temperature rises as high as 95°C and as a result of there is considerable loss of volatile oil (Wistreich Schafer 1962). The loss of volatile oil during grinding and could be overcome partly by lowering or maintaining the mill temperature as low (less possible then the boiling as temperature of the volatile constituents of the spice volatile This could be achieved by grinding the spices oil). condition and to a some extent by circulating chilled water around the grinding zone (Murthy et al. 1996).

2.3.1 Cryogenic grinding process:

The term "cryogenics" refers ultra low temperature. Bureau of standards, UK has defined cryogenic The national temperature as -150°C and below. One of the popular cryogene is LN2 (liquid nitrogen B.P-150°C). During cryogenic grinding spices, there is evaporation of LN2 which quickly chills of spice and the grinder. Fibrous materials become the both brittle and crisp making size reduction easy. The use of LN₂ in a cubic particle with good product flow usually results Wistreich et al. (1962), Anon (1962) and characteristics. Pruthi (1991) have reported many advantages of cryogrinding of some of them are; i) reduced oxidation of spice spices; as LN2 evaporates in the grinding zone, it tends to expel any air in the mill, and increased stability as cryo-treatment has a pasteurizing effect on spices ii) extremely fine grinding is obtained as spice become very brittle and spice oil solidify, resulting in more uniform dispersal of flavour, reduced visual reduced settling rate in liquid preparation, speckling and reduced loss of volatiles, increases flavour iii) strength. On an average, 30% increase in quality; iv) lowered microbial load inert as grinding is done at low temperature and in atmosphere, v) visually lighter colour due to the absence browning. A typical cryogenic grinding system is shown in Figure (2.1). The difference between essential oil retention and the head space analysis of different spice volatiles



- 1. VIBRATION FEEDER 4. UNIVERSAL MILL
- 2. LIQUID NITROGEN 5. ROTARY VALVE INLET
- 3. SCREW CONVEYOR 6. FILTER BAG

- 7. LIQUID NITROGEN TANK
- 8. TEMPERATURE
 CONTROLLED VALVE
- 9. CONTROL PANEL

extracted from ambient, and cryogenically ground spices respectively given in the Table (2.5 and 2.6). Which revealed that cryogenically ground spices retain more volatile component than the ambient ground samples (Pruthi, 1991).

2.3.2 Chilled grinding:

Spices are ground in the process by circulating chilled water around the surface of the mill or grinder to extract much of heat from the body of the mill during grinding. Murthy et al. (1996) modified the stainless steel mixer/grinder by providing an outer jacket for circulating chilled water during grinding of black pepper and reported that ground pepper obtained from chilled water and LN2 grinding (cryogenic) system retained almost same quantity of volatile oils (3.56 ml and 3.60 ml/g respectively). Similarly, there were no variations in the volatile oil contents of nutmeg obtained from chilled, ambient and LN2 grinding system (McKee et al. 1993).

2.4 General isotherms of food:

The relationship between equilibrium moisture content and the corresponding water activity (aw) of a food over a range of values at a constant temperature yield a moisture sorption isotherm (MSI) when graphically expressed.

Table 2.5: Gas chromatographic headspace analysis of diffrent spice volatiles.

Area (in integration units $\times 10^5$

Spice	Component	Grin	ding techniques	0
		Ambient	Cryogenic	<pre>% increase due to cry- ogrinding</pre>
White	a-Pinene	4.68	9.50	95.47
papper	b-Pinene	6.95	10.63	53.00
	Unidentified	9.64	13.36	38.59
	Limonene	5.59	6.76	21.93
Nutmeg	a-Pinene	16.75	19.38	15.70
	b-Pinene	8.84	9.38	5.67
Cinnamon	a-Pinene	1.15	8.32	623.48
	Camphene	0.40	2.53	532.50
	b-Pinene	0.45	2.42	437.78
	Cineole	0.11	0.47	327.27
	Limonene	0.24	0.54	125.00
Cumin	a-Pinene	0.24	0.42	75.00
	b-Pinene	3.23	4.49	39.00
	y-Pinene	1.24	1.39	12.10
Oregano	Unidentified a-Pinene Camphene b-Pinene Myrcene p-Cymene y-Terpinene	0.62 1.49 0.82 1.40 0.00 8.32 4.92	2.86 5.71 2.44 4.10 3.98 17.31 12.87	361.30 283.22 197.56 192.86 Very high 108.05 16.59

Table 2.6: A comprison of the spice volatile oils retention by ambient and cryogenic grinding.

Spice	Ambient Grinding (ml\100g)	Cryo-grinding (ml\100g)	<pre>% increase due to cryo-grinding</pre>
White paper	1.95	3.19	64
Black paper	2.21	3.09	40
Pimento	2.71	3.08	14
Mace	9.10	14.50	59
Cloves	11.50	16.50	43

Source : M/s. Alpine, Germany

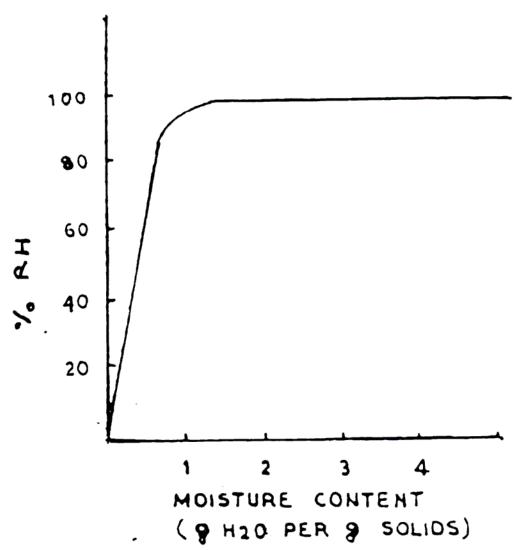


FIG. 2-9 GENERAL ISOTHERMS OF FOOD MOISTURE VS ERH

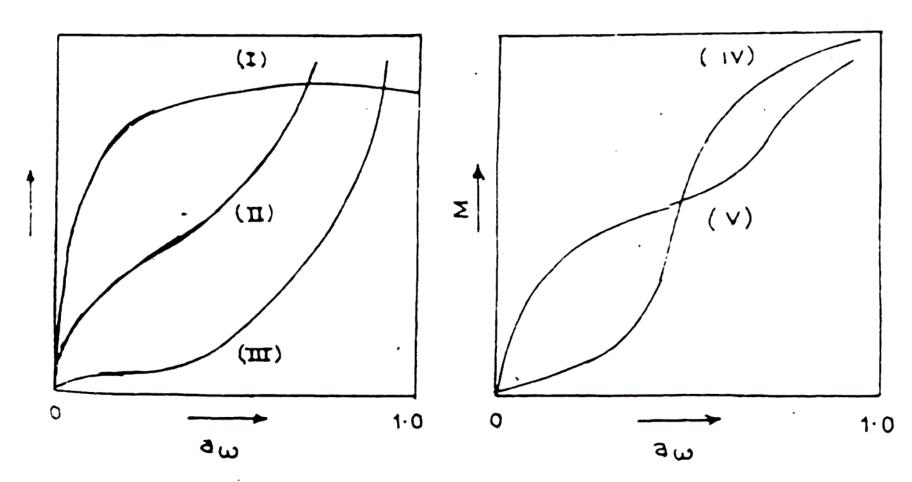


FIG. 2.3 THE FIVE TYPES OF VANDER-WAALS ADSORPTION ISOTHERMS

The equilibrium moisture content (EMC) of a food material corresponding to a given water activity and temperature is determined experimentally and is presented as water or moisture sorption isotherm of the material. Gal (1967) Bera et al(1990) and Selot et al. (1989) improved upon the existing methods to determine the sorption isotherms of food materials, the principle being the determination of weight changes of the samples in equilibrium with different water vapour pressure.

(1974) reviewed the food isotherms. The Labuza isotherms for a general type of food is given moisture Figure 2.2. It has three parts. The upper part of the curve shows aw close to one over a wide range of moisture contents. corresponds to fresh tissue such as meat, fish, This area vegetables, fruits etc. The lowest part of the curve aw < 0.5, corresponds to most dehydrated foods such as milk powder, cereals, instant coffee etc. The effect of water on stability structure is vastly different between these two areas and isotherms. The middle portion of the curve is called the intermediate moisture food range. Foods falling in this range for example; jams, candies and newer semi-moist al. (1940), classified adsorption foods, etc.Brunauer et into five general types (Figure 2.3). Type I isotherms Langmuir and type II is the sigmoid or S-shaped adsorption isotherm. No special names have been attached to the three other types. Types II and III are closely related to types IV

V, except that the maximum adsorption occurs at and pressure lower than the vapour pressure of the gas. MSI's of most foods are non-linear, generally sigmoid in shape and have been classified as type II isotherms. Foods rich in soluble components, such as sugars, have been found to show type III behaviour. Another behaviour commonly observed is, that different paths are followed during adsorption and desorption processes, resulting in a hysterisis. The desorption isotherm lies above the adsorption isotherm (Figure 2.4) and therefore, in the desorption process moisture is retained more compared to adsorption at a given equilibrium Relative humidity.

2.5 Packaging and storage of spices:

Spices and spice products being highly sensitive to and its moisture, absorption may result in caking, discolouration, hydrolytic rancidity and mold growth. Since products contain aromatic essential oils and the flavouring ingredients, loss of these and pick up of foreign odour may pose serious problem especially in ground spices.

Heat and light accelerate deterioration, especially with oxygen sensitive product. Almost all the spices are highly prone to the insect attack.

Loss of volatile oils, caking, microbial spoilage

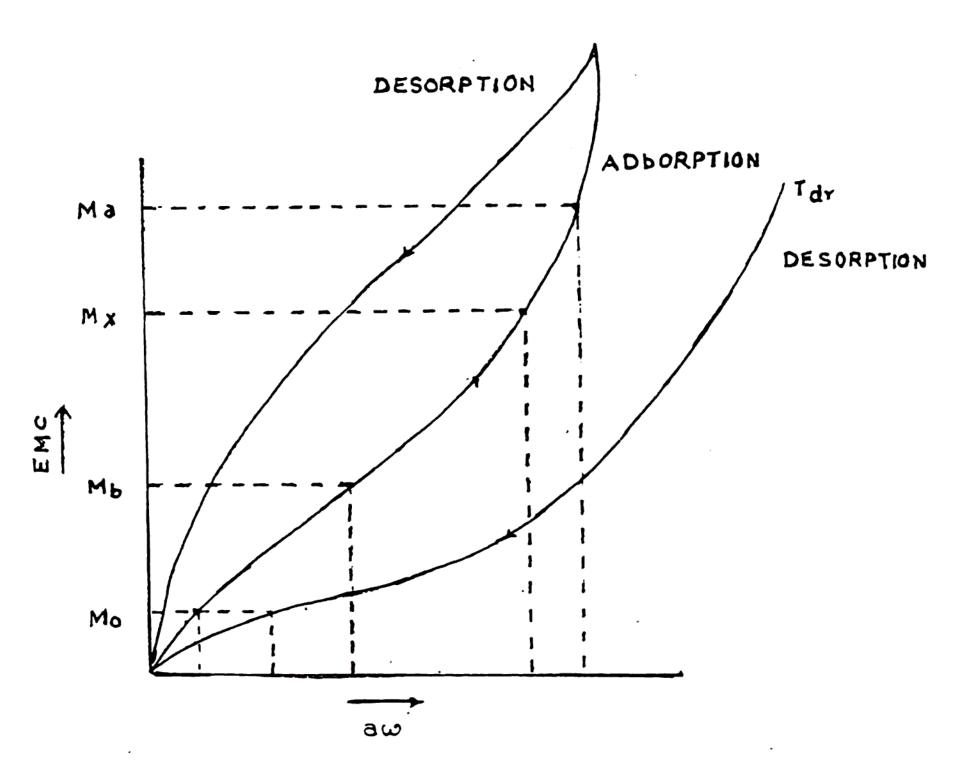


FIG 24 TYPICAL MARKING POINTS ON A SCHEMATIC SORPTION ISOTHERM FOR USE IN DRYING AND STORAGE CALCULATIONS

and insect infestation are main deteriorative changes during storage and distribution. In pepper, mold grow at moisture content, of 13% (ERH 88%) but not at 11% (ERH 67%). Moisture content of less than 10% (6-8% in hygroscopic spices) is best for retention of quality of spices. Green pepper and red chillies discolour considerably when exposed to light and oxygen.

Functional package especially for ground spices should offer protection against microbiological spoilage occurring due to environmental conditions like humidity, temperature, light and oxygen; prevent loss of volatile oils, flavours and pick up of foreign odour; prevent insect infestation and spoil age; assist in selling by suitable attractive outer graphics; should conform to the food laws of the user countries; and be economical and easily available. For evaluating storage-life, overall quality with respect to volatile oil, colour, flavour leading to overall loss of of should be considered (Kumar and Anandaswamy, 1974, freshness Properties of various packaging materials 1988) are shown in the Table (2.7).

Balasubrahmanyam et al; (1978, 1979, 1980, 1982) studied Packaging and storage of ground spices in consumer unit packs using several types of flexible packaging materials and reported that low and high density polyethylene (LDPE, HDPE) and polyprophlene (PP) film pouches retard only the

Table 2.7: Properties of packaging materials.

Packaging materials		WVTR g/m²/24hr mil	GTR ml(NTP)cm ² /sec./mil) cm.Hg at 25-Cx10 ⁴			Grease resestance	Tensile strength	Elongation (%)	
		at 37.8°C/ 90% R.H.	0 ² C0 ²		J ²		kg/cm²		
1	Low density Polye- thylene	18.6	15.00	55.00	5.00	Poor	70.3-162	500	
2	High density Poly- thylene	3-5	3.00	13.00	1.0	Fair	218-246	300	
3	Polypropyleme	10.8	4.00	12.00	0.80	Good	387	800	
į	Polystyreme	120.0	4.00	35.00	1.00	Excellent	105-844	10-66	
5	Umplastized P.V.C.	54.0	0.50	0.40	0.20	Good	492	5-500	
ŧ	Polyvinyledeme chl- oride	3.9	0.01	0.12	0.002	Excellent	844	60	
7	Polyster	28.0	0.10	0.50	0.02	Good	1757	-1	
å	Dylon 6	140.0	0.13	0.50	0.04	Good	844	200	
ş	300 MSAT Cellophame	3-15	ca 0.05	ca 0.12		Good	700	20	

ingress of moisture but not loss of volatiles. Printing polyethylene pouches containing turmeric and pepper the get disfigured and smudged to great extent. PP and HDPE (200 gauge) are better suited than LDPE in offering moisture flavour protection. These two offer about 90 days life at 65% RH and 27°C at the sacrifice of 20-50% volatile loss in turmeric and pepper, and 120-150 days for cumin, oil and chilli powder. Double pouch of cellophane coriander and 250 gauge LDPB outside, laminates of cello/poly, inside glassine/poly and 300 MXXT cellophane retain volatiles provide adequate moisture protection for about 200 days for all spice powders at 65% RH, 27°C. Metallised polyester/poly and paper/foil/poly laminate pouches offer adequate protection for over a year for all spice powders, against volatile loss ingress under normal conditions of storage. and moisture However, in met. PET/poly laminate pouches, turmeric having higher volatile oil content caused slight delamination.

retention of paprikas Colour in clear glass containers was superior to that of opaque polyethylene bags (Stringhetsa and Coelho, 1974) probably due to higher oxygen the latter. Losses in essential oils permeability of were 23% over one year in whole coriander stored in paper upto cotton bags. Hermetically sealed cans ensured minimal quality Laminates of polyamide/poly, and cello /foil deterioration. /poly were satisfactory for storage of spices with respect to

volatile components. For packaging of mustard, foil/paper/PB packs were found unsatisfactory due to its gas permeability, whereas cello/foil/poly was satisfactory. Flavour retention in curry powder packed in polybags was minimum due to loss of volatile oils but the product packed in cans was good for 5 months both at room temperature and 37°C(Mishra, 1981).

Natural polymers like gelatin, gums and starches are wall materials for being used as microencapsulated flavours. Flavour loss in these were very little regardless flexible packaging used. Packaging materials of like paper/foil/ploy or paper/ploy and opaque polyethylene been suggested. The total loss was 10% over 12 weeks, also while ground spice seasoning lost 20% volatiles foil in between 70 and 80% packaging and in cheaper packaging materials.

Green and red pepper for export is packed in A 2} tall or 50z babycans in 2% brine or in glass bottles as 12% brine. For dehydrated green and pickles in red having moisture content of 3-4% (ERH 20-30%), coloured plastic containers or poly bags of various sizes are being term storage. Foil laminates are best for suited short consumer unit packs. Oleoresins and volatile oils extracted from ground dried spices are generally packed in glass, aluminium or suitable lined tin containers. Of late, they are

packed in high density polyethylene containers. Aluminium tube with inner coating is suitable as consumer unit container for oleoresins and oils, as it helps in easy dispensing.

Traditionally, jute bags are used for bulk packaging spices. Due to problems of moisture whole of ingress and sifting, polylined jute bags and high density polyethylene woven sacks are being considered. For ground spices, multi paper, woven plastic and textile sacks, glass, metal or wall aluminium containers are generally employed. Whole pepper packed in double burlap bags with polyliners of 3 mil or more, moisture increase was only 0.4% even under monsoon conditions Package for dry whole chillies, due to 1957). bulk density, becomes voluminous and the transport becomes freight is charged on volume basis. At the expensive as moisture content of 10%, and pressure of 2.5 kg/sq.cm the volume of dry whole chilli is reduced by 78% of its original value offer economic transportation (Viraktamath, 1964).

CHAPTER - III THEOREGICAL CONGIDERATION

THEORETICAL CONSIDERATION

This chapter deals with the theoretical considerations required for the determination of rate of heat removal or heat loss during cold grinding of spices and prediction of shelf life of cumin and dry ginger powder packed in various packaging materials.

3.1 Rate of heat removal during cold grinding process :

During grinding process heat is generated which heats up the surface of the grinder machine. In cold grinding process, conditions are created to drive out heat from the surface of the machine. To accomplish this, the stainless steel jar of an electrical grinder is provided with the plastic jacket which holds chilled water and maintains the product temperature minimum (Fig. 3.1). During grinding process the rate of heat loss could be calculated from the equation (Earle 1956):

$$Q = hc A (To - Tx)$$
(3.1)

hc = Surface heat transfer coefficient, J/m²S°C

Q = rate of heat loss, J/S

Following assumptions are made to calculate the rate of heat loss:

i) Geometry of the jar is assumed to be cylinderical.

- 123-THERMOCOUPLE POINTS
- (Metalic PRODS)
- A -STAINLESS JAR

- B -WATER JACKET C NEEDLE VALVE
 D MULTICHANNEL TEMPERATURE
- E -INDICATER
- F PUMP
- G-WATER TANK

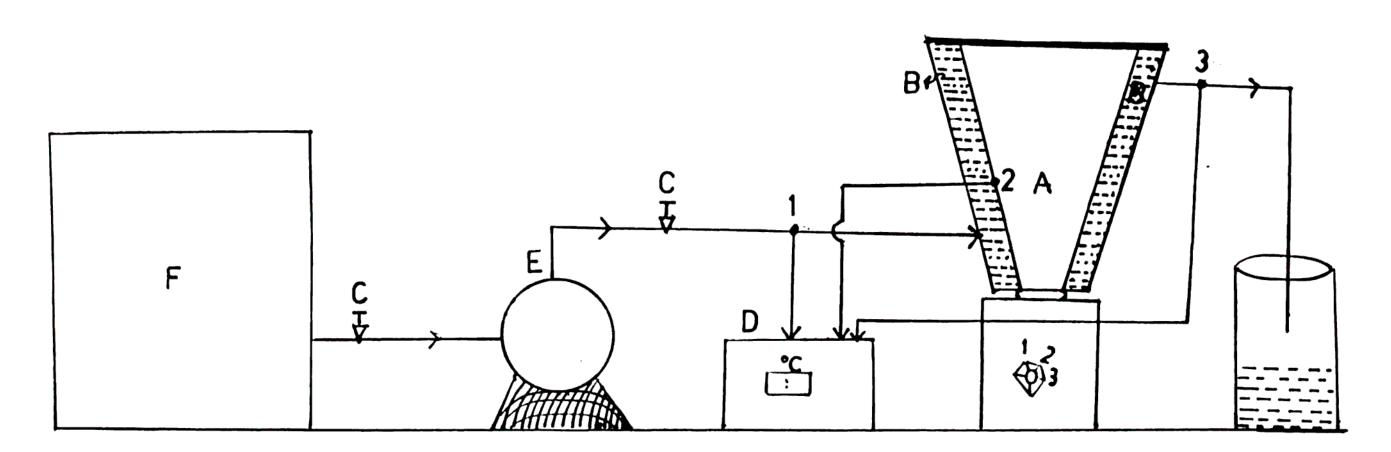


FIG -31 LINE DIAGRAM OF EXPERIMENTAL SET UP

- ii) Chilled water is being circulated across the cylindrical jar to have a forced convective heat transfer condition.
- iii) Steady state condition is obtained instantaneously.
- iv) There is no radiation heat loss during grinding.
- v) High flow rate of chilled water circulation would increase the rate of heat loss.
- vi) Faster the rate of heat removal, faster will be the reduction in the product temperature.
- vii) There is no change in viscosity (μ) and specific heat (C_P) of cooling media during grinding process.

The surface heat transfer coefficient is calculated from the equation (Loncin and Merson, 1979) given below for liquids at high or moderate Reynold numbers.

$$Nu = 0.26 (NR_{\bullet})^{0.6} (Pr)^{0.3} \dots (3.2)$$

Nu = Nusselt number (hcD/k)

 $Pr = Prandtl number (Cp \mu/k)$

 $NR_e = Reynold number (Dv / \mu)$

Fluid (Cooling media) properties (Cp,μ,k,ρ etc) in the equations are obtained at the mean film temperature which is the arithmetic mean temperature between the temperature of the tube walls and the temperature of the bulk fluid.

The surface heat transfer coefficient (hc) could be calculated from the value of Nusselt number (Nu=hcD/k). Having obtained the value of "hc" the rate of heat loss(Q) from the surface of the stainless steel jar during grinding process could be calculated.

3.2 Prediction of Shelf-life:

Shelf-life is the period for which the package will preserve the contents of the product without deterioration.

The Prime factor to be considered in package is their ability to keep the moisture contents of the product at a minimum.

knowledge of the water vapour transmission (WVTR) of the package components will be helpful to some extent for predicting the shelf life but other factors such as sealing effectiveness, storage conditions, time, temperature make water vapour transmission value relatively useless etc. predicting ultimate shelf-life. Although actual field for tests with package of the product are desirable in order to out the shelf-life of a particular product but they find quite expensive and time consuming. Testing the package under accelerated conditions of high temperatures and humidity provides the manufacturer with valuable shelf-life data correlated with previously run field test. In addition the

cost of accelerated testing is relatively low and less time consuming.

3.2.1 Half value period(product equivalent method) :

The half value period (H.V.P.) is the time required for the moisture content of the packed foods to move half way between the initial value and the value that would be obtained when equilibrium has been reached with storage conditions. It may be used either to compare the efficiency of two packs or to estimate the shelf-life

The shelf-life of different packages can be estimated from the following derivation:

The rate of change of moisture content is directly proportional to the driving force. Here the driving force is the difference between equilibrium moisture content and the moisture content at any time "t"

$$\frac{dM}{dt} = K (M_e - M) \dots (3.3)$$

$$\frac{dM}{(M_{\bullet} - M)} = K dt \tag{3.4}$$

Integrating on both sides with suitable limits.

$$\int_{M_0}^{M_0} \frac{dM}{M_0} = K \int_{M_0}^{t} dt$$

$$\ln \frac{(M_{\bullet}-M)}{-1} \quad M_{\bullet} = Kt \quad f$$

$$-\ln(M_{e}-M) - \ln(M_{e}-M_{0}) = Kt$$
(3.5)

For determining the shelf life at critical moisture content level the equation (3.5) can be modified as,

$$\begin{array}{cccc}
 & M_{e} & - M_{c} \\
 & 1n & \hline{ & & & & & & & & & \\
 & M_{e} & - M_{0} & & & & & & & & \\
\end{array} = -Kt &(3.6)$$

3.2.2 Equilibrium moisture content (EMC) and critical moisture content(Mc):

There are two methods of determining equilibrium moisture content viz; static method (Labuza, 1975) and dynamic method (Bandyopadhyay and Das, 1987, Bera et al. 1990). Among them, static method is easier and more suitable for storage studies. In this method changes in the appearance of product such as colour, flavour, texture and microbial growth can be observed after a long period of exposure to a given relative humidity. However, some assumptions are made in determining the EMC by static method.

- i) The salt solution or sulphuric acid solutions are assumed to maintain a constant relative humidity inside the air-tight desiccators.
- ii) The EMC is not varied to a great extent by the temperature fluctuation of \pm 2°C.

From the moisture equilibrium curve Fig. 3.2 three points have been identified.

- a) The initial point(I)- The moisture content and equilibrium relative humidity of the product as prepared.
- b) The critical point(C)- stage at which the product just become lumpy.
- c) The danger point (D)- a point which is of 5% lower relative humidity than the critical point.

The portion of the curve between D and C is generally defined as the safety range (SR). The portion of the curve between the points I and D may be defined as the safety margin(SM) as a helpful guide in the packaging of material. The package adopted should not permit the product to reach the danger point so that there may be a safe margin of error.

An analytical method has been derived for the determination of critical moisture content by using Henderson's equation (Henderson, 1952) and the derivation is given below:

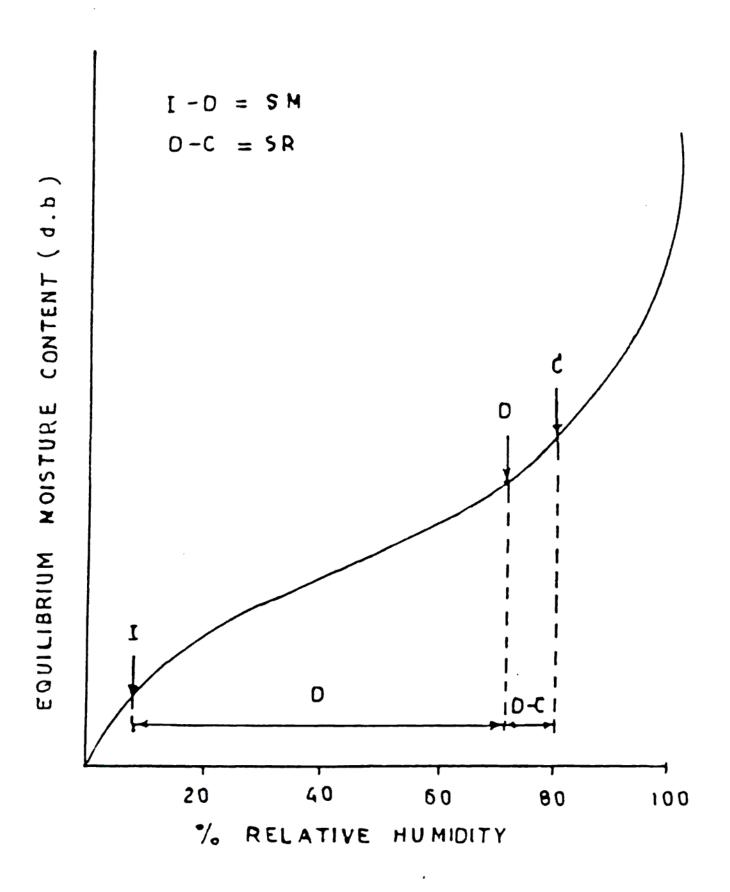


FIG. 3.2: MOISTURE ADSORPTION ISOTHERM

$$\log \log \left[\frac{1}{(1-aw)} \right] = n \log M + \log K + \log T$$

$$M^{n} = - \frac{\log (1-aw)}{KT'}$$

$$M = \begin{bmatrix} -\frac{\log (1-aw)}{KT'} \end{bmatrix}$$

$$M = \begin{bmatrix} \frac{1}{KT'} \end{bmatrix} \begin{bmatrix} \log \frac{1}{1-aw} \end{bmatrix}$$

Now differentiating for M in the equation with respect to aw, we have,

$$\frac{dM}{daw} = \left[\frac{1}{KT'}\right]^{1/n} \frac{1}{n} \left[\log \frac{1}{1-aw}\right]^{1/n-1} \left[-(1-aw)\right]...(3.8)$$

equating (3.8) to zero and solving for aw, we get aw = 0 and aw=1, M is maximum. So at aw = 0 and aw = 1. Now we need to find the turning point of dM/daw vs, aw graph.

So again differentiating dM/daw with respect to aw and equating it to zero and then solving for aw gives the critical relative humidity (ac). Now differentiating eq. (3.8) again we get;

$$\frac{d}{daw} \left[\frac{dM}{daw} \right] = -\frac{1}{(KT')^{1/n}} \frac{1}{n} \left[\ln \frac{1}{1-aw} \right]^{1/n-1}$$
(-1)

$$+ \left[\frac{1}{(KT')} \right]^{1/n} \frac{1}{n} \left[\frac{1}{n} -1 \right] (1-aw) \left[\frac{1}{1-aw} \right]^{1/n-2}$$

(1-aw)(-1)

= 0 at aw = aw critical (a/c)

So,
$$\left[\frac{1}{KT'}\right]^{1/n} \left[\ln \frac{1}{1-aw}\right]^{1/n-1} = -\left[\frac{1}{KT'}\right]^{1/n} \frac{1}{n} \left(\frac{1}{n} - 1\right)$$

$$\ln \frac{1}{(1-ac)^{1/n-2}} \qquad (1-ac)^{2}$$

Cancelling common terms on either side we get;

$$\left[1n \frac{1}{1-ac}\right]^{1/n-1} = \left[1 - \frac{1}{n}\right] \left[1n \frac{1}{1-ac}\right]^{1/n-2} (1-ac)^{2}$$

$$\left[\begin{array}{cc} 1n & \frac{1}{1-ac} \end{array}\right]^{1/n-1-(1/n-2)} = \left[\begin{array}{cc} 1 & -\frac{1}{n} \end{array}\right] (1-ac)^2$$

Hence In
$$\frac{1}{a-ac} = \left[\begin{bmatrix} 1 - \frac{1}{n} \end{bmatrix} \right] (1-ac)^2$$

$$-\ln (1-ac) = \left[\frac{1}{n} -1 \right] (1-ac)^2$$

$$\ln (1-ac) + \left[\frac{1}{n} -1\right] (1-ac)^2 = 0 \dots (3.9)$$

let 1 - ac = x

$$\ln x + \left[\frac{1}{n} - 1 \right] \quad x^2 = 0 \qquad \dots (4.0)$$

The value of n in the above equation(4.0) could be substituted from the Henderson's equation(3.7) fitted to the adsorption isotherm data of cumin and ginger powder obtained at 25°C.

Having substituted the n value in the equation 4.0, the value of 'x' could be calculated by Newton's method of iteration for which a computer program is being developed, given below.

Computer programme for calculation value of x

10 INPUT " VALUE OF N"; N

$$20 x = 10 - 999:B = 1$$

30
$$X = X - \{(LOG(X)+(X 2)\}0 * N) * X/(2* X*N+1):B=B+1$$

40 IF B < 100 GO TO 30

50 PRINT "N = "; N, "
$$X = "$$
; X

60 END

Solving equation (4.0) for x will give the value of (1-ac), hence "ac" (critical relative humidity) can be determined. From this corresponding critical moisture content (Mc) could be obtained from the EMC-relative humidity curve.

CHAPIER - IV MATERIAL AND METHODS

MATERIAL AND METHODS

This chapter deals with materials selected for the study and methods used for grinding of spices, equilibrium moisture content determination, physico-chemical and microbiological analysis, packaging and storage evaluation of cumin and dry ginger powder.

4.1 Cold grinding of spices

Freshly harvested Cumin seed(Cuminum cyminum L.) and ginger (Zingiber officinale Rose) were procurred from the local market. The materials were cleaned. Ginger were had peeled and cut into slices and then dried in the hot aircirculating electrical drier. Dried spices were packed air tight in the aluminum container and used as when required for the experiment.

A kitchen grinder (waring blender-type) was used for grinding of cumin seeds and dried ginger chips. The stainless steel mixer jar was modified by providing an outer jacket (Fig. 3.1). The outer wall of the jacket was insulated. For cold grinding, chilled water was circulated around the mixer jar.

4.1.1 Experimental procedure:

Chilled water was circulated using a centrifugal pump($0.5 h_P$) in the grinder which is fabricated for the experiment. The flow rate of the chilled water was adjusted using a needle valve. Mass-flow rate of the circulating chilled water was determined interms of amount of water collected per minutes. The temperature of the water was adjusted by mixing properly ice-cold water and water at ambient temperature. The temperature of circulating water was adjusted to 10, 15, 25°C. The temperatures of inlet, outlet water, surface of the stainless steel jar were monitored, using metalic probes (1.5 mm dia) connected with multichannel temperature indicator (Model 44/E, Century digital Instruments),

Twenty five grams of the dried sample was taken into the stainless steel jar of the grinder and conditioned with the temperature of the circulating water. Grinding speed (12000 rpm) and 3 minutes time was fixed for each set of experiment. The ground materials were quickly collected in the aluminium boxes and used for further experiments. In another set of experiment(control), samples were ground in the grinder without circulating cold water or the grinding was done at room temperature (35°C).



The moisture contents of the cold ground spice (CG-S) and conventional ground samples or control (CON-S) were determined by following the method described in ASTA(1968).

4.3 Determination of fineness Modulus:

Fineness of the ground product was determined by standard method of sieve analysis using 8, 14, 28, 60 and 100 mesh sieve (Taylor series) with opening of 2 mm, 1 mm, 0.5 mm, 0.25 mm and 0.1 mm. In this 25 grams sample (m.c.- 9.5% d.b.) was taken in the sieve shaker (Raw-tap) and shaken for 5 minutes. The sample collected in each sieve was weighed and % fraction was calculated. The fineness modulus of the sample was also calculated using the equation (Pandey, 1994).

4.4 Volatile oil determinations:

Steam volatile oil was determined separately for ground cumin and dry ginger sample by following the method described in ASTA(1968).

4.5 Microbiological analysis:

The plate count of the samples was done by following the standard method (Ranganna, 1979). Briefly, the procedure given below:

4.5.1 Preparation of Media:

Potato Dextrose Agar :

Potato-Dextrose-agar media was prepared by mixing the potato infusion (200 ml), dextrose (20 g), agar (15 g) and water (800 ml). Potato infusion was prepared previously by boiling peeled and crushed potato (200 g) in 1 lit. of water. The media was distributed in the cleaned and dried conical flask and sterilized in the autoclave at 15 psi for 20 min.

4.5.2 Sterilization of glassware and distilled water:

Petridishes, pipettes and conical flasks were cleaned and oven dried. These glasswares were wrapped with craft papers and kept inside the autoclave. Some conical flasks (25 ml) and testtubes were filled with distilled water, and cotton plugged and sterilized at 25 psi for 20 min. Sterilized glasswares and distilled water were used as and when required.

4.5.3 Plate counting:

The procedure for the quantitative determination of micro- organism in the ground cumin and dry ginger samples is given below:

Apparatus:

- Potato-daxtrose agar, sterilized in plugged conical flask.
- 2. Sterilized dishes of about 10 to 12 cm diameter.
- 3. Sterilized tubes containing 9 ml of distilled water.
- Inoculation needle, inoculation chamber and B.O.D. incubator.

Procedure:

Initially, conical flasks containing potato dextrose agar were placed in the boiling water until the media was completely melted. The molten media was poured into the sterilized petridishes by lifting the lid just enough to permit pouring and allowed to solidify in the form of a thin layer.

One gram ground powder was taken to prepare 1/10, 1/100, 1/1000... 1/100000 dilutions from the same solution. From these serial dilutions, small portion was taken with the help of sterilized inoculation needle and was spread on the surface of media containing petriplates. Inoculation was done in the sterilized inoculation chember. The inoculated plates were kept in inverted position inside the B.O.D. incubator at 25°C. After one week of incubation time plates were taken out for observation. The development of various colonies of micro organism was recorded and confirmed under the micro scope.

4.6 Determination of adsorption isotherms:

Five grams each of the sample (CON-S and CG-S of cumin and dry ginger powder) was taken and placed in lid covered petridishes. These dishes were placed in the desiccators which maintained the desired water activities(0.1 to 0.9) by using various concentrations of sulphuric acid solution (Reugg, 1980) at 25°C and 37°C.

The desiccators were placed in the B.O.D. incubators where predetermined temperature was adjusted. The weight of the samples was noted every 48 hours. Equilibrium moisture content (EMC) was attained at a point when there was no further loss or gain in weight of samples.

The equilibrium moisture content (EMC) data at their corresponding water activity were plotted to obtain moisture adsorption isotherms at 25°C and 37°C. The isothermal data were analysed using Henderson equation (1952), to obtain constants n' and k'. (Eq. 3.7)

4.7 Determination of self-life and storage studies of ground spices:

For the storage studies, low density polyethylene (150 gauge) Cellophane (150 gauge), and dluminium(0.9 μ)/ low density polyethylene (150 gauge) pouches size (5 x 5 cm) were used for packaging 10 grams of CG-S and CON-S samples

Separately of cumin and dry ginger powder, These packed samples were stored separately in the desiccators maintained at 25°C/90% RH and 37°C and 70% RH. The relative humidity in the desiccators were maintained by sulphuric acid solution as mentioned earlier. The desiccators were placed inside the BOD incubators where pre-determined temperature was maintained. The pouches were weighed every week and changes in any physical parameters (colour and caking) moisture content, microbial growth (total plate count) free fatty acid were recorded.

Equilibrium moisture content(EMC) of samples were also determined at 25°C at 90% RH and 37°C and 70% RH by following method mentioned earlier.

4.8 Sensory analysis of spice oil:

Ginger and cumin have volatile oils which give attractive spicy odours.

The volatile oils of dry ginger and cumin powder were obtained from CG-S and CON-S spice samples by steam distillation and were used to prepare soup for sensory evaluation.

4.8.1 Taste Pannels:

Soup Samples were prepared by mixing 0.1 ml volatile

oil of the spice, and 1.0 gm cornflour in 100 ml salt solution (0.5%) at 60 °C using waring Blender. The mixing was done for one minute then serve hot to the taste pannelist.

A reference sample was also prepared in the same manner, except that spice oils were omitted. During each session 10 pannelist were presented first with a reference sample to acquaint them with the mouth feel of the soup and then served with three 10-ml samples (coded) in 3 oz dixie cups. The triangle tests were conducted to quantify the sensory evaluation data. Statistical analysis of the data was done and presented in the form of analysis of varience (ANOVA).

CHAPICR - V RESULTS AND DISCUSSION

RESULTS AND DISCUSSION

This chapter deals with the results of the study on cold grinding of cumin seed and dry ginger chips, heat transfer parameters during grinding, adsorption isotherm, prediction of shelf-life and changes occurring in the physicochemical and microbiological profile of ground spices at various storage conditions and packaging systems.

5.1 Cold grinding processing of cumin and dry ginger:

Cold grinding processing of cumin seed and dry ginger chips was done in an electrical cold grinder fabricated for the purpose (Figure 3.1). The volatile oil contents, and fineness modulus data of the ground cumin and dry ginger powder are given in the Table 5.1, 5.2 and 5.3. The volatile oil contents of the conventional ground samples (CON-S) i,e, ground at 35°C (room temp. and without circulating water) of cumin and ginger were 2.5 and 2.0 percents, respectively(Table 5.1).

The volatile oil contents of the cold ground samples (CG-S) were increased, when the temperatures of the grinding zone of the grinder were lowered by circulating chilled water (10-25°C). The volatile oil contents of cumin powder increased from 2.5% to 3.5% and in ginger powder it increased

Table 5.1: Volatile Oil Content and fineness modulus of Cumin and dry ginger powder ground at 35°C.

Name of spices °C	Protiduct temp.°C after grinding	% Volatile Oil. contents	Fineness module
Cunin	69	2.5	3.51
Ginger	67	2.0	3.48

Table 5.2: Effect of granding temperatures on the % Volatile Oil Contents and fineness modulus of Ground Cumin.

Temp.of water°C	Prouduct temp. °C	% Volatile Oil. contents	Fineness modulus
10	22.80	3.5	3.26
15	24.00	3.5	3.28
25	32.92	3.0	3.30

Table 5.3: Effect of granding temperatures on the % Volatile Oil Contents and fineness modulus of ground Cumin.

Temp.of water c	Prouduct temp.°c	% Volatile Oil. contents	Fineness modulus
10	20.40	3.0	2.91
15	23.10	3.0	3.01
25	30.80	2.5	3.30

from 2.0% to 3.0% (Table 5.2 and 5.3). There was 40% increase in the volatile oil content of both cumin and dry ginger powder in comparison to the spices ground at 35°C. This was expected because grinding at higher temperature results in loss of volatile oil (Pruthi, 1980). Murthy et al. (1996) reported that volatile oil contents of the black pepper increased when grinding operation was done at chilled and cryogenic conditions.

fineness modulus data of cumin and dry ginger The powder ground at 35°C were respectively 3.51 and respectively (Table 5.1). It decreased when the temperature of the grinding zone of the grinder was lowered from 35°C to The fineness modulus of cumin decreased from 3.51 to 3.26 and in ginger powder it decreased from 3.48 to 2.91 (Table 5.2 and 5.3). Lower fineness modulus value of the powder indicates fine particle size. That means by grinding spices at cold temperatures improves the fineness of ground products. The grinding operation conducted at increased the product temperature (67°C) and there frequent stopping of the grinding blades because of "gumming In this temperature the size reduction was shattering technique. Which leads to tearing than In large scale grinders where continuous products. ground grinding is undertaken, gumming of grinder walls and results in frequent stopping of grinder for cleaning work

reduces the granding rate and makes ground product Distre-Amon 1961).

The grinding of cumin seed and dry ginger chips stabilled temperature not only increased the volstile oil contents of the product but it improved the fineness

5.2 Sensory evaluation the cold ground sumin and iry ginger powder :

Spices are valued for their aroma and flavour including appearance or visual quality. On visual examination of the ground products, indicated that the COM-5 products were farker in colour in comparison to the CG-5 products. Figh product temperature during grinding at 35°C might have initiated browning type reaction and hence fark onloured products. Wistreich et al. 1962 Anon 1962 and Pritail 1991 have reported that visually light onloured spice products were obtained at low temperature grinding the to absence of browning reaction.

The cake-formation of ground spice products was also observed in the spices ground at high temperature 35°C out the spice ground at low temperature (10-25°C, gave free flowing spice products.

Triangle text performed on the 2000 prepared cases on the 2000 prepared cases on the 2000 prepared cases

of the CG-S-samples were better in comparison to the aroma CON-S samples. Taste pannelist clearly differentiated the aroma soup prepared from the CON-S and CG-S-samples. of The data related to the sensory evaluation and analysis variance are given in the Table 5.4 and 5.5. Statistical analysis of the aroma of soup prepared from volatile oils of both CON-S and CG-S samples of cumin and ginger powder significantly (F>0.05). This results verify that cold ground samples contain more volatile compounds than that high temperature or conventional ground spice samples. et al. (1996) and Pesek et al. (1985) reported that there were retention of volatile constituents of volatile oil better cold ground and cryo-ground spice samples of pepper, cumin and incomparison to the spices ground at ambient orgegano temperature.

The sensory evaluation data related to the aroma of the soup prepared from the volatile oils of the CON-S and CG-S samples would have been explained authentically if these data were supported by gas chromatographic analysis of the volatile constituents of the volatile oils. Nevertheless, sensory evaluation of the products prepared from the volatile oil of ground spice product revealed that better quality of the ground spice could be prepared from the cold grinding technique.

Table 5.4: Sensory evaluation of the aroma of soup prepared from volatile oil of cumin powder

Treatment	Replication								Average		
	1	2			5			8	_	10	
Cold ground	8	7	8		7		8	7	6		7.2
Conventional ground	7	6	7	6	7	6	7	5	5	7	6.3
Analysis of V	/aria	ance	. (A	AVON)						
Source of variance		đf			SS]	MS		F value	₽ .05 %
Treatment		(2-	-1)=1	L	4.(05	4	4.05		6.23*	4.41

Within treatment (20-2)=18 11.7 0.65

Table 5.5: Sensory evaluation of the aroma of soup prepared from volatile oil of dry ginger powder.

Treatment Replication							Average				
	1	2	3	4	5	6	7	8	9	10	
Cold ground	7	6	7	8	6	7	7	7	8	7	7
Conventional	7	7	6	5	6	7	5	6	6	5	6

Analysis of Variance (ANOVA)

Source of variance	df	SS	MS	r value	.05 %
Treatment	1	5	5	9.09*	4.41
Within treatment	18	10	0.55		

5.3 Evaluation of convective heat transfer parameters during cold grinding :

Heat transfer parameters viz; surface heat transfer coefficient (hc) and rate of heat transfer or heat loss for design considerations. are required In the present investigation chilled water (10-25°C) was circulated grinding in order to have forced circulation the zone heat transfer condition and to maintain convective forced required temperature at the grinding Ιn zone. convection, the fluid is constantly being replaced and the of heat transfer are therefore higher than the natural rate (Earle, 1956). Effect of flow rate of chilled convection circulation at various temperature on heat transfer water parameters during grinding are given in the Table 5.6, 5.7, It has been observed that as the velocity of 5.8 5.9. and chilled water circulation increased from 0.11 m/s to 0.22 the outlet water temperature decreased from 26.4°C to 19.01°C indicated that there was gradual heat loss from the which zone of the grinder (Table 5.7). Since Reynold grinding from dependent on flow rate also, increased (NRe) number 18560.16 to 37120.16.

Heat transfer parameters viz; Nusselt number (Nu=hcD/k), Prandtl number (Pr = $Cp~\mu/k$) and surface heat transfer coefficient (hc) were changed with the changes in the outlet water temperature. Although Prandlt number did not change

Table 5.6: Effect of flow rates of water circulated at 25°C on heat transfer parameters during grinding of cumin.

Velocity of water, m/s	Out let temp. of water °C	NRe	Pr.	Nu.	hc (Jm ⁻² S ⁻¹ C ⁻¹	Q.) (J/S)
0.22	30.2	36948.2	1.46	803.35	1407.10	485.53
0.18	30.6	30230.3		712.18	1243.89	425.11
0.14	32.2	23512.5		612.52	1070.49	350.60
0.11	33.3	18474.1		530.01	925.65	294.10

Table 5.7: Effect of flow rates of water circulated at 15°C on heat transfer parameters during grinding of cumin.

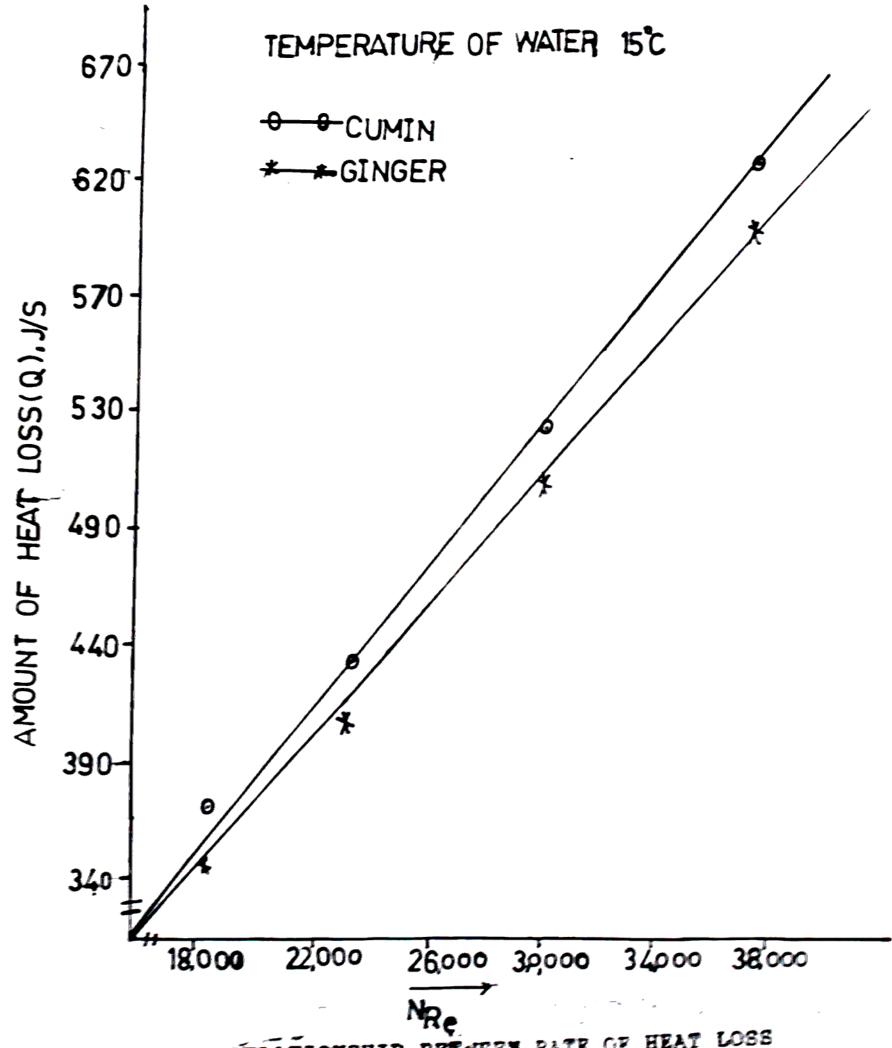
Velocity of water, m/s	Outlet temp. of water °C	NRe	Pr.	Nu.	hc (Jm ⁻² S ⁻¹ C ⁻¹)	Q. (J/S)
0.22	19.9	37120.16	1.47	811.11	1434.62	626.91
0.18	22.2	30371.20		719.10	1271.89	529.76
0.14	24.4	23622.20		618.45	1093.87	434.30
0.11	26.3	18560.16		535.13	946.50	359.69

Table 5.8: Effect of flow rates of water circulated at 25°C on heat transfer parameters during grinding of dry ginger.

Velocity of water, m/s	Outlet temp. of water, °C	NRe	Pr.	Nu.	hc (Jm-2S-1°C-1)	Q. (J/S)
0.22	28.6	36949.80	1.47	803.35	1407.10	480.89
0.18	29.3	30227.50		712.12	1243.87	417.36
0.14	30.6	23526.25		612.52	1070.49	346.79
0.11	32.1	18983.54		530.01	925.65	287.51

Table 5.9: Effect of flow rates of water cirrculated at 15°C on heat transfer parameter during grinding of dry ginger.

Velocity of water, m/s	Outlet temp. of water °C	NRe	Pr.	Nu .	hc (Jm ⁻² S ⁻¹ °C ⁻¹)	Q. (J/S)
0.22	19.01	37120.16	1.47	811.11	1434.62	612.67
0.18	22.40	30371.20		719.10	1271.89	505.80
0.14	24.60	23622.20		618.45	1093.87	413.51
0.11	26.40	18560.16		535.13	946.50	341.25



5.1 RELATIONSHIP BETWEEN RATE OF HEAT LOSS (Q) AND REYNOLD NUMBER (No.)

significantly because it depends upon the physical properties of the cooling water evaluated at the mean film temperature i.e. temperature of the surface of the grinding zone and the temperature of the bulk fluid.

Heat transfer parameter i.e. Nusselt number $(Nu=\frac{hcD}{k})$ increased with NR_e, which also increased the heat transfer coefficient (hc). At NR_e of 37120.16 the hc was 1434.62 J/m²S°C while at NR_e 18560.16 it was 946.50 J/m²S°C. The heat transfer coefficient has a linear relationship with the rate of heat loss(Q). Increased values of hc increased the rate of heat loss. At a value of hc = 1434.62 the rate of heat loss (Q) was 612 J/S, while at hc=946.50 J/m²S°C it was 341.25 J/S.

Similarly, water (15°C) circulated at NR_e 37120.16 the rate of heat loss (Q) was 629.91 J/S while at NR_e 18560.16 it was 359.69 J/S. (Fig. 5.1). Data related to the heat transfer parameter viz; Nu, Pr, hc and flow rate parameter(NR_e) could be used for calculation of required capacity (surface area of the grinding zone) and designing of a continuous type cold grinding equipment.

5.4 Adsorption isotherm of ground cumin and dry ginger powder :

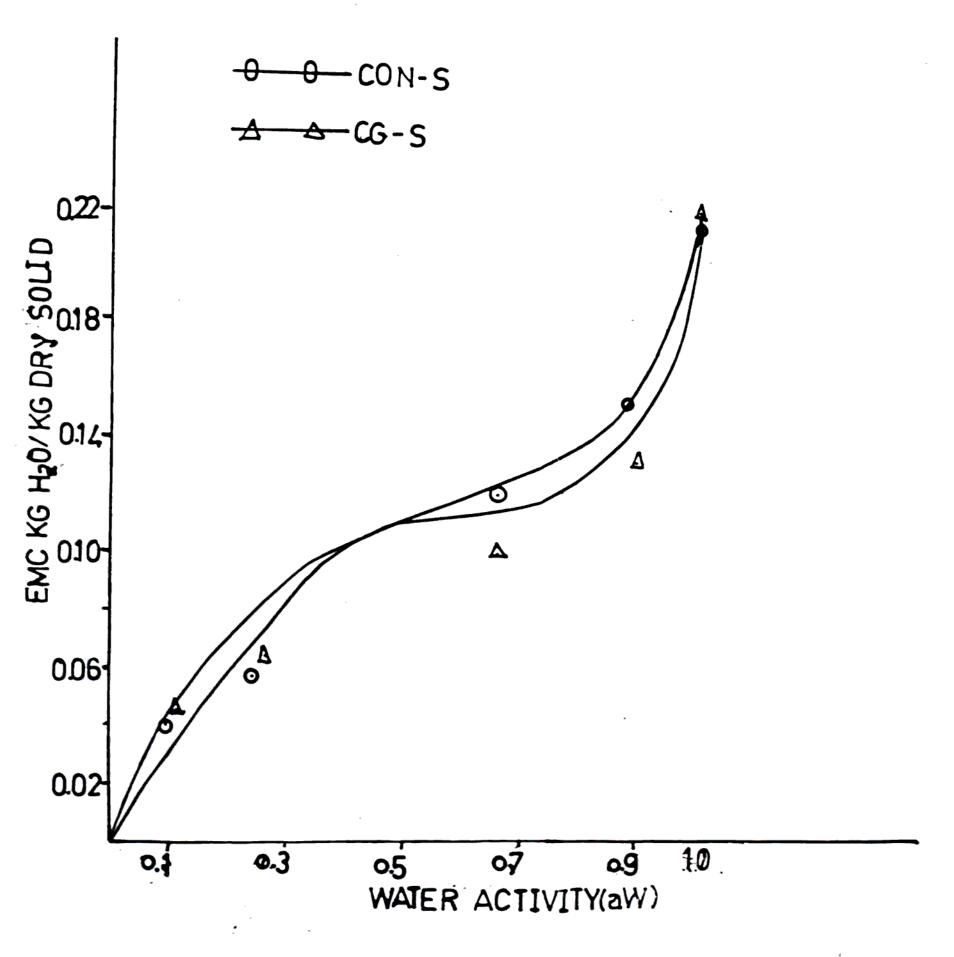
It was observed that isotherms at 25°C and 37°C temperature were sigmoid shape (Type II) (Figure 5.2 and

5.3). The rate of moisture adsorption of the sample at 25 and 37°C were more or less same at all levels of water i.e. , adsorption isotherms obtained at 25 and 37°C were similar. The rate of moisture adsorption of both 50%-2 and 55samples of cumin and dry ginger powder was slow upto the activity of 0.7 but further increase in the levels of water activity changed the pattern of moisture 3030700 characteristics (Figure 5.2 and 5.3). At a water activity of 0.9 the COM-S samples of cumin and ginger sample had 1.15 kg water/kg dry solid and 0.15 kg water/kg dry respectively. Similarly, the CG-8-samples of dumin and quages powder had 0.13 kg water/kg dry solid and 0.14 kg water kg solids, respectively. However, both COM-2 and CS-2-2amp.ex of dry ganger powder had less equalibrium moisture context. "BMC value in comparison to the cumin samples Senderson equation was used to fit the isothern data. The graphics. 3.5 representation of the Benderson's equation for outline and for y princer powder are shown in the Pigure 5 4 and 5 5. The Mailles different constants I' and a lave been taloughter from the slop and intercept of the straight line and the evening form of the equations are shown below

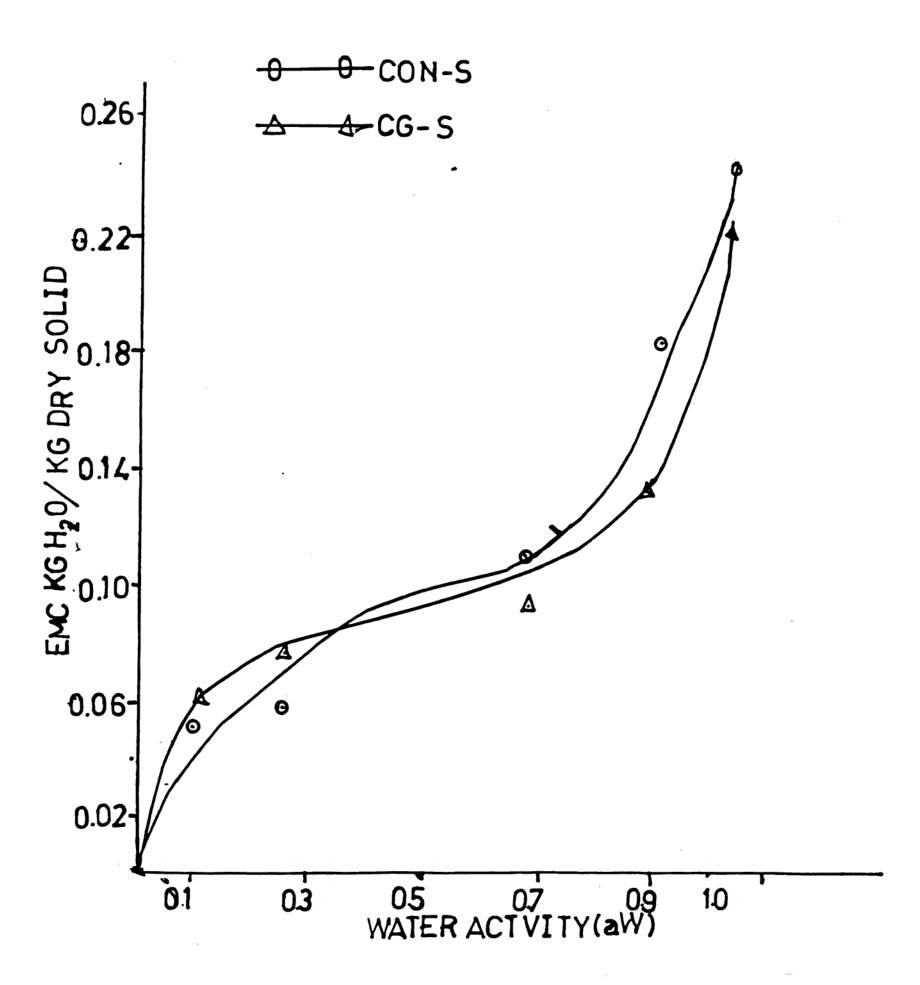
^{1.} Sumin powder

E Doministations of dual sample in the single set of the single

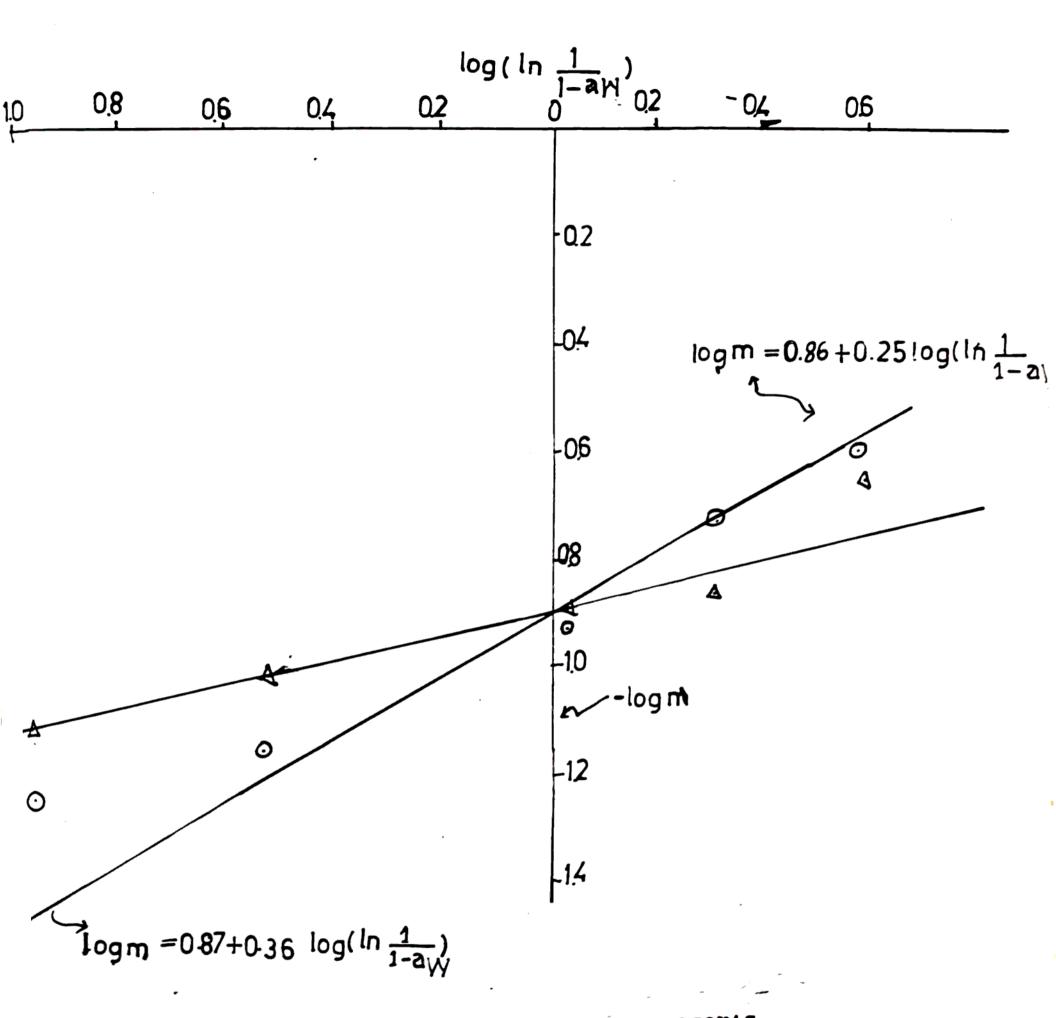
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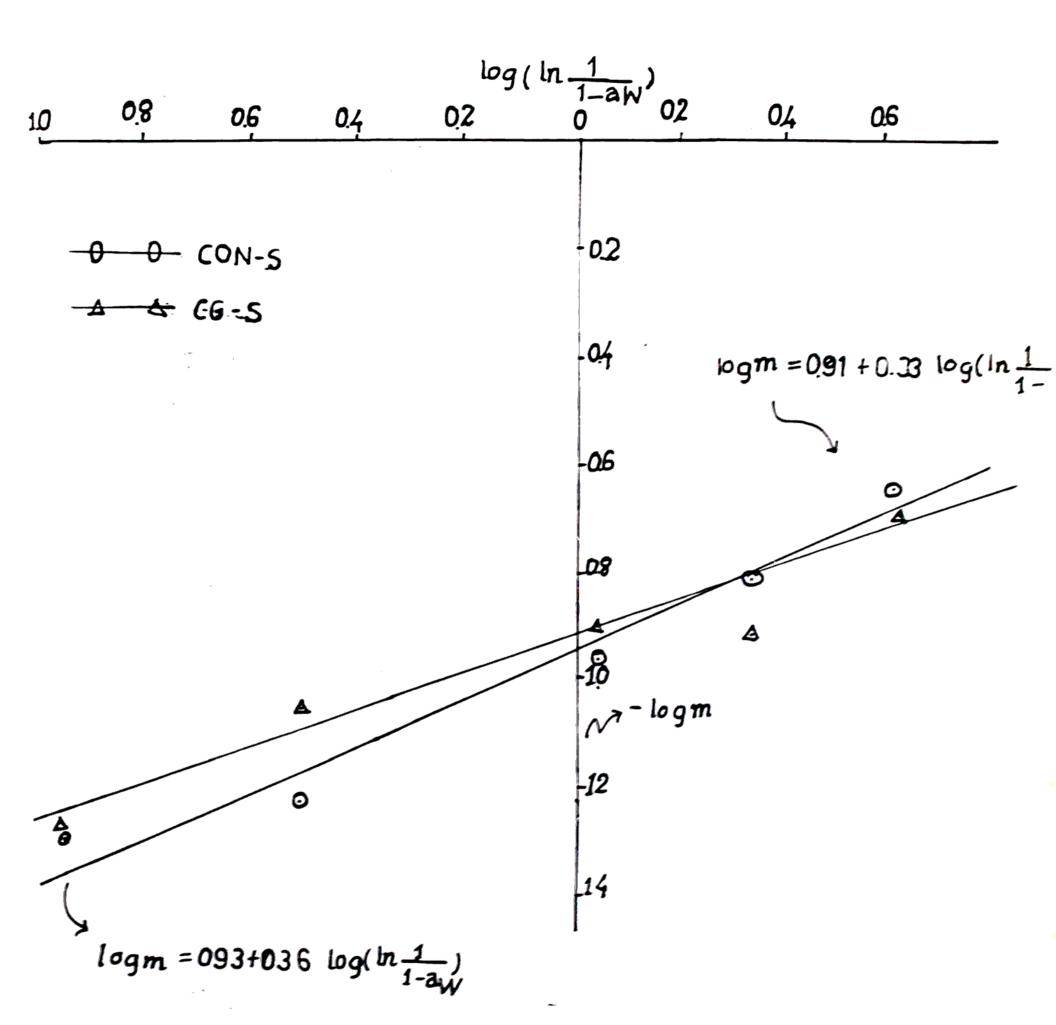
5.2 MOISTURE ADSORPTION ISOTHERM OF CUMIN POWDER.



5.3 MOISTURE ADSORPTION ISOTHERM OF DRY GINER POWDER.



5.4 GRAPHICAL REPRESENTATION OF HENDERSON'S EQUATION FOR CUMIN POWDER AT 25°C.



5.5 GRAPHICAL REPRESENTATION OF HENDERSON'S EQUATION FOR DRY GINGER POWDER AT 25°C.

2. Dry ginger powder

- a) Conventional ground sample: $1-aw = e^{-2.53}M^{0.36}$
- b) Cold ground sample: $1-aw = e^{-2.48}M^{0.33}$

The correlation coefficient (R) value for each straight line equations (Henderson equation) were calculated analytically which gave correlation coefficient (R = 0.8-0.9). The constant of the Henderson equation "n" was later used for the calculation of critical relative humidity (ac) and critical moisture content (Mc) to determine shelf-life of the ground spices product stored at various storage conditions.

5.5 Storage characteristic of cumin and dry ginger powder packed in various packaging materials :

moisture contents, free fatty acid Changes in and total plate count (microbial lead) of CON-S contents CG-S-samples of both cumin and dry ginger powder packed in the density polyethylene (150 gauge), Cellophane (150 gauge) low and Aluminum (0.9 micro)/low density polyethylene (150 laminated pouches and stored at 25°C and 90% relative humidity and 70% relative humidity for four months. Data 37°C and related to these are summarised in this section.

5.5.1 Changes in moisture content :

The changes in moisture contents of the samples

Table 5.10: Effect of storage conditions on % moisture content, % free fatty acid and total plate count of the ground ginger powder packed in different packaging materials.

Cample	Dagkaging	Ctorago	Moisture Content(d.b.)		Plate	Count	free fa	•
Sample	Packaging material	Storage condition	Initial	Final	Intial	Final	Intial	of Oleic acid) Pinal
CON-S	Polyethelen Cellophane Al + Poly.	25°c/90%RH	9.6	12.3 12.0 11.54	76x104	142x10 ⁴ 125x10 ⁴ 110x10 ⁴	0.056	0.068 0.064 0.062
CG-S	Polyethelen Cellphane Al foil + Poly.	25°c/90%RH	9.6	12.10 11.80 11.40	55x104	85x10 ⁴ 78x10 ⁴ 68x10 ⁴	0.042	0.050 0.048 0.046
CON-S	Polyethelen Cellophane Al foil + Poly.	37°c/70%RH	9.6	12.1 11.7 11.35	76x104	110x10 ⁴ 95x10 ⁴ 86x10 ⁴	0.056	0.070 0.065 0.063
CG-S	Polyethelean Cellophane Al foil + Poly.	37°c/70%RH	9.6	11.98 11.58 11.29	55x104	78x104 66x104 59x104	0.042	0.052 0.048 0.047

Table 5.11: Effect of storage conditions on % moisture content, % free fatty acid and total plate count of the ground cumin powder packed in different packaging materials.

Sample	Packaging	Storage	Moisture	Content(d.b.)	Plate	Count	<pre>free fatty acid (In term of Oleic acid)</pre>		
	material	condition	Initial	Final	Intial	Final	Intial	Final	
CON-S	- Polyethelen Cellophane - Al + Poly.	25°c/90%RH	9.5	121.41 12.10 11.16	80x104	151x10 ⁴ 136x10 ⁴ 110x10 ⁴	0.11	0.16 0.15 0.13	-
CG-S	Polyethelen Cellphane Al foil + Poly.	25°c/90%RH	9.5	12.21 11.71 11.52	42x104	75x104 68x104 56x104	0.11	0.14 0.13 0.12	- C
CON-S	Polyethelen Cellophane Al foil + Poly.	37°c/70%RH	9.5	12.1 11.91 11.4	80x104	148x104 131x104 92x104	0.11	0.17 0.15 0.14	
CG-S	Polyethelean Cellophane Al foil + Poly.	37°c/70%RH	9.5	11.89 11.60 11.31	42x104	70x104 62x104 48x104	0.11	0.16 0.14 0.13	

stored at 25°C/90% RH and 37°C/70% RH are shown in the Table 5.10, 5.11. It has been observed that moisture content CON-S and CG-S samples of cumin and dry ginger powder both increased irrespective of the type of packaging materials used conditions. However, increase in the and storage contents of the sample packed in the Al/Polylaminate pouches in comparison to the samples packed in the low were less density polyethylene and cellophane pouches. CG-S and CON-S samples of dry ginger powder packed in Al / Poly Laminate & polyethylene pouches had 18.75% and 20.20 % moisture increase when stored at 25°C/90% RH while it had 12.25% and 18.75% moisture increase at 37°C/70% RH. (Figure 5.6 and 5.7). Similarly, CG-S and CON-S samples of cumin powder packed in Al/poly laminate, polythylene pouches had 21.26% and 22.10% moisture increase at 25°C/90% RH and at 37°C/70% RH it had 17.05% and 20.0% increase in moisture content. The increase in moisture content of the ground spice products influence the storage stability or shelf-life.

5.5.2 Changes in the free-fatty acid content:

The changes in the free-fatty acid contents of both CON-S and CG-S samples of cumin and dry ginger powder stored at 25°C/90% RH and 37°C/70% RH are shown in the Table 5.10 and 5.11. It has been observed that free fatty of CON-S and CG-S samples of both cumin and dry ginger powder packed in low

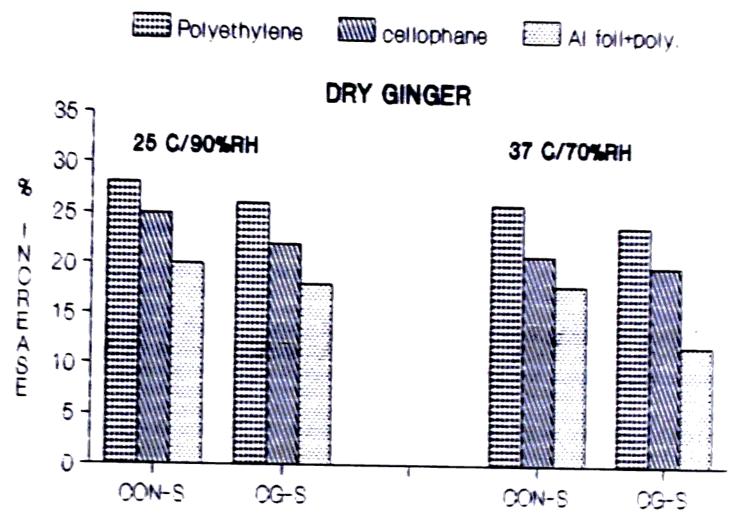


Fig. 5.6: % INOREASE IN MOISTURE CONTE-NT OF DRY GINGER POWDER PACKED IN VARIOUS PACKAGING MATERIALS.

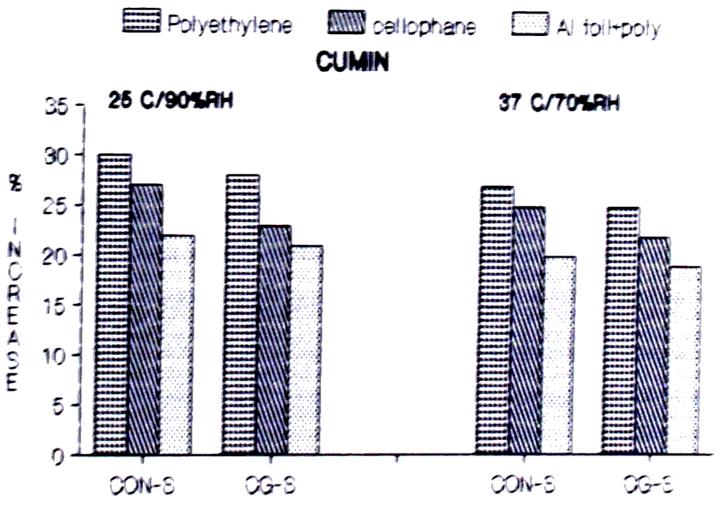
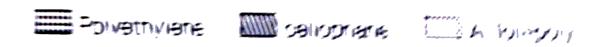


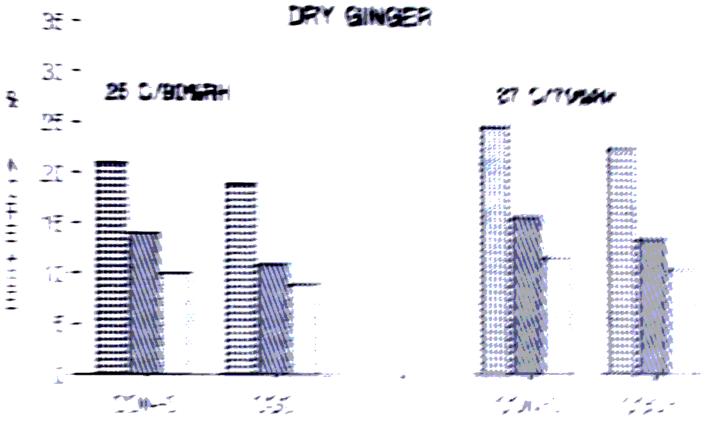
Fig.5.7: % INCREASE IN MOISTURE CONTENT OF CUMIN POWDER PACKED IN VARIOUS PACKAGING MATERIALS.

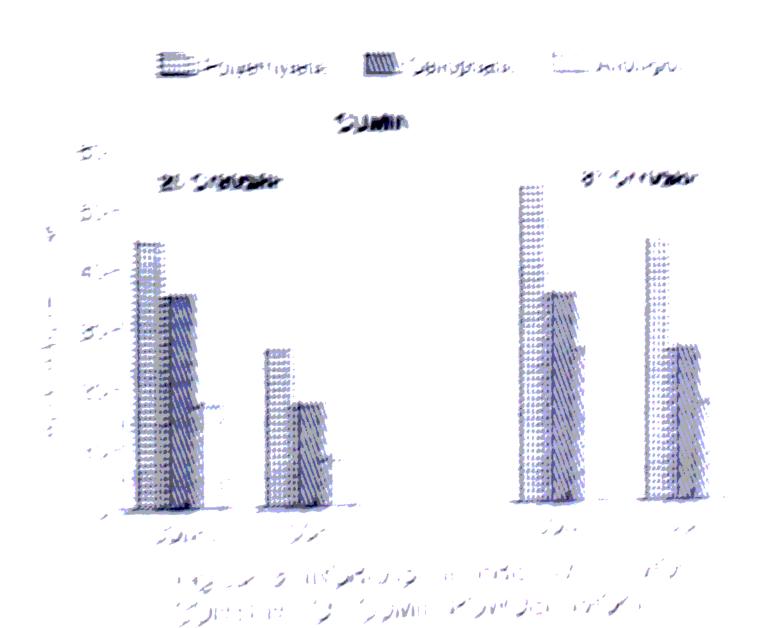
density polyethylene, cellophane and Al/ polyliminate pouches increased when stored at 25°C/90% and 37°C/70% RH The percentage increase in free fatty acid contents of all the spice products was more when packed in the low density polyethylene pouches in comparison to the cellophane and Al/poly liminate pouches irrespective of the conditions (Fig. 5.8 and 5.9). There was 45.45 to 54.54% increase in fatty acid content of CON-S samples of cumin powder packed in the polyethylene pouches when stored 25°C/90% RH and 37°C/70% RH . It was interesting to note that Increase in the free fatty acid contents of both cumin SIL dry ginger powder packed in different packaging materials was more when stored at 37°C/70% RH. The production of free fatty acids during storage of all the oil bearing materials including spice assumes greater significance in terms of their sensory quality.

5.5.3 Microbiological evaluation

A common problem faced by both importing and exporting countries, is that the spices are often heavily contaminated with microorganisms which forms source of contamination in the prepared food products. Changes in the total plate count of microorganism in the ground cumin and first ginger powder packed in different packaging materials and





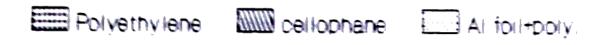


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stored at various storage conditions are given in the table 5.10 and 5.11.

The initial microbial loads of CON-S and CG-S samples of cumin powder were 80 x 104 and 42 x 104 and in dry ginger powder it were 76 x 104 and 55 x 104. The initial microbial load of CG-S samples of both cumin and dry ginger powder was less in comparison to the CON-S samples (Plate-I).

The microbial loads of the cumin and dry ginger powder packed in the various packaging materials increased when stored for 4 months at 25°C/90% RH and 37°C/75% Plate II &III shows the microbial load of the CON-E and RH. CG-S samples packed in various packaging materials and stored 25°C/90% RH. The microbial load of CG-S samples of cumin in the polyethylene also increased from 80 x 10^4 to packed 151x104 when stored at 25°C/90% and it increased marginally 52 104 at 37°C/70% RH. Similarly, CG-8 samples of dry gimper powder stored at 25°C/90% RH had microbial load of 142 z 154 increase) and at 37°C/70% RH it had 110 x 104 44 0% increase). Percentage increase in the microbial load was more in all the samples packed in polyethylene pouches irrespective of the storage conditions(Figure 5.10 and 5.11). However. samples packed in the Al/ polylaminate and stored at 15°5 TV4 RE had lowest percentage increase in the microbial Load



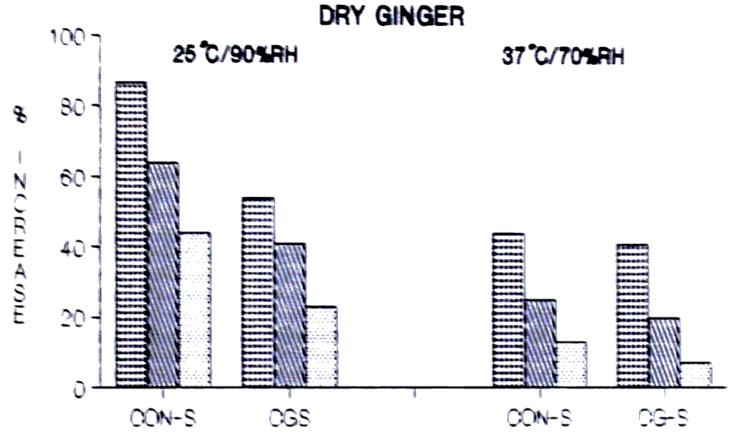


FIG.5.10 % INCREASE IN TOTAL PLATE COUNT OF DRY GINGER POWDER PACKED IN VARIOUS PACKAGING MATERIALS.

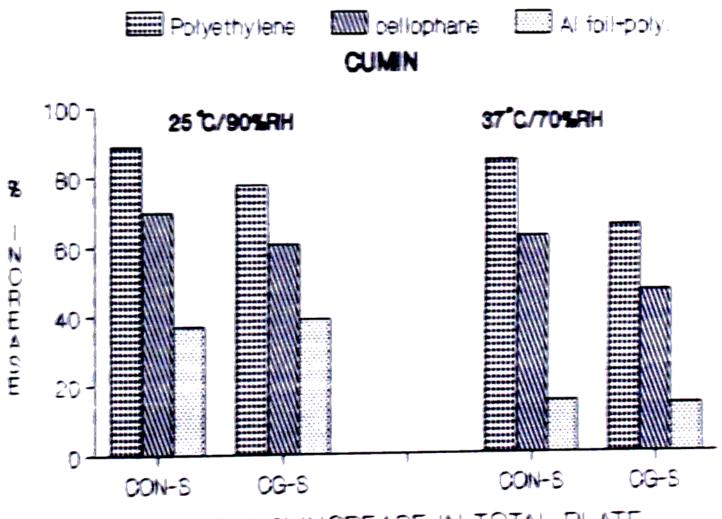


Fig.5.11: % INCREASE IN TOTAL PLATE COUNT OF CUMIN POWDER PACKED IN VARIOUS PACKAGING MATERIALS

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Major portion of the microorganisms consisted of fungi.

Although few colonies of bacteria were also observed.

As stated in the foregoing paragraph that percentage the moisture content of all increase in the ground samples packed in the low density polyethylene and stored at 25°C/90% RH, was more and this could also explain the increase microbial load of the sample packed in low in polyethylene pouches. Al/poly laminate have excellent gases and water vapour including against light properties (Kumar and Anandaswamy, 1974), could be used for packaging of both ground cumin and dry ginger powder.

5.6 Prediction of shelf-life period :

The storage studies at accelerated conditions may not give true picture of shelf-life since the maintenance of accelerated conditions are precarious. Hence, shelf-life of the CON-S and CG-S samples of both cumin and dry ginger powder at normal service conditions were predicted. Here, the shelf-life period of the spice samples were predicted at the level of critical moisture content.

The critical moisture content (M_c) is requisite in the prediction of shelf-life period. Critical relative humidity (a_c) is obtained by solving the equation (4.0). The value of critical relative humidity is used to obtain critical



Plate - No. I

Association of microflora of ground cumin seeds and dry ginger chips before storage.

- 1. Cumin (CON-S)
- 2. Ginger (CON-S)
- 3. Cumin (CG-S)
- 4. Ginger (CG-S)



Plate - No. II

Association of microflora of ground dry ginger packed in various packaging materials, and stored for 4 months at 25°C/90% RH.

- 1. Aluminium foil / polyethylene Laminate (CG-S)
- 2. Cellophane (CG-S)
- 3. Low density polyethylene (CG-S)
- 4. Aluminium foil / polyethylene (CON-S)
- 5. Cellophane (CON-S)
- 6. Low density polyethylene (CON-S)

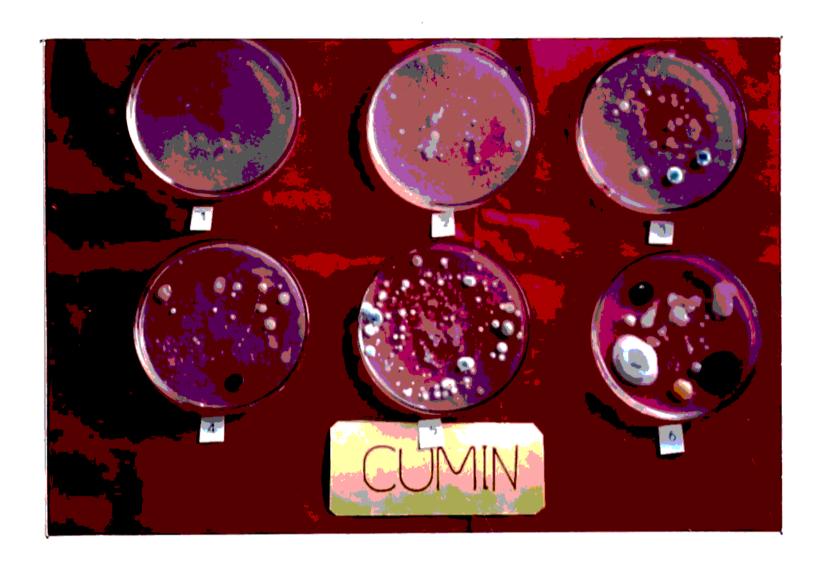


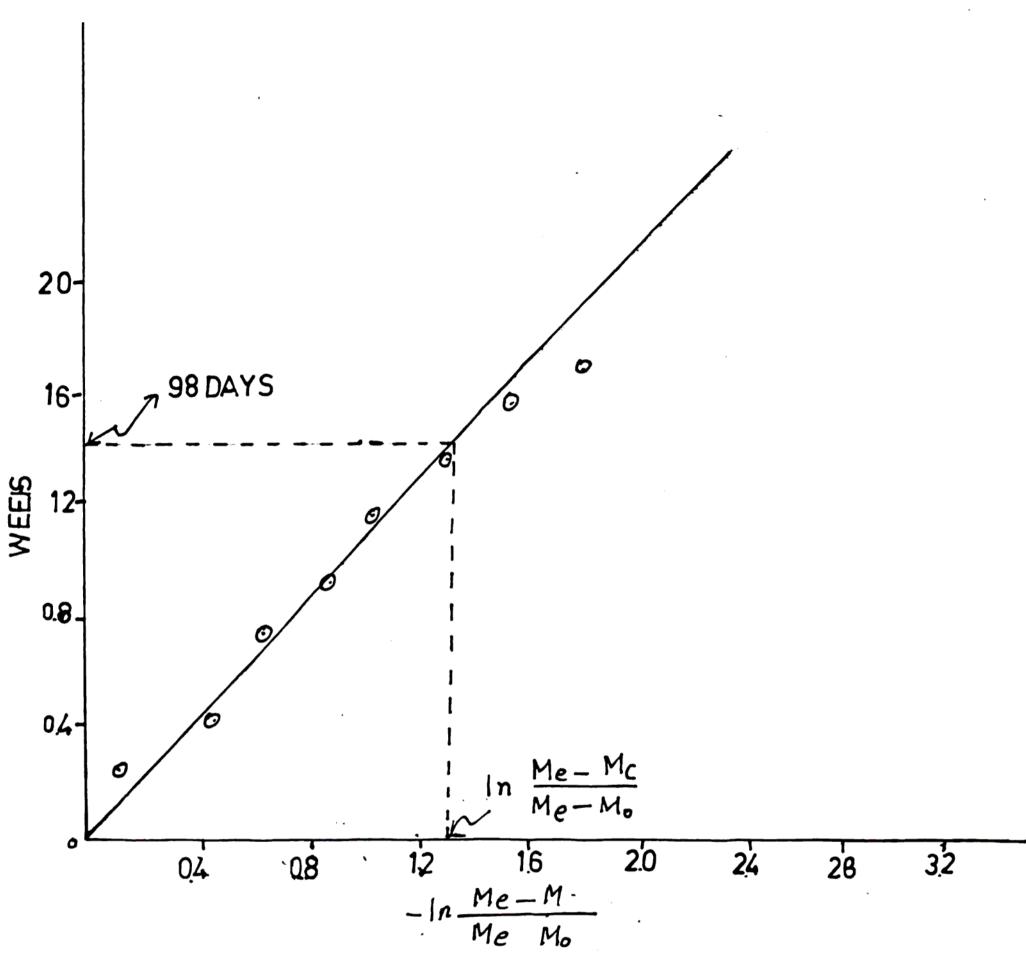
Plate No. III

Association of microflora of ground cumin seeds packed in various packaging materials and stored for 4 months at 25°C/90% RH.

- 1. Aluminium foil / polyethylene Laminate (CG-S)
- 2. Cellophane (CG-S)
- 3. Low density polyethylene (CG-S)
- 4. Aluminium foil / polyethylene (CON-S)
- 5. Cellophane (CON-S)
- 6. Low density polyethylene (CON-S)

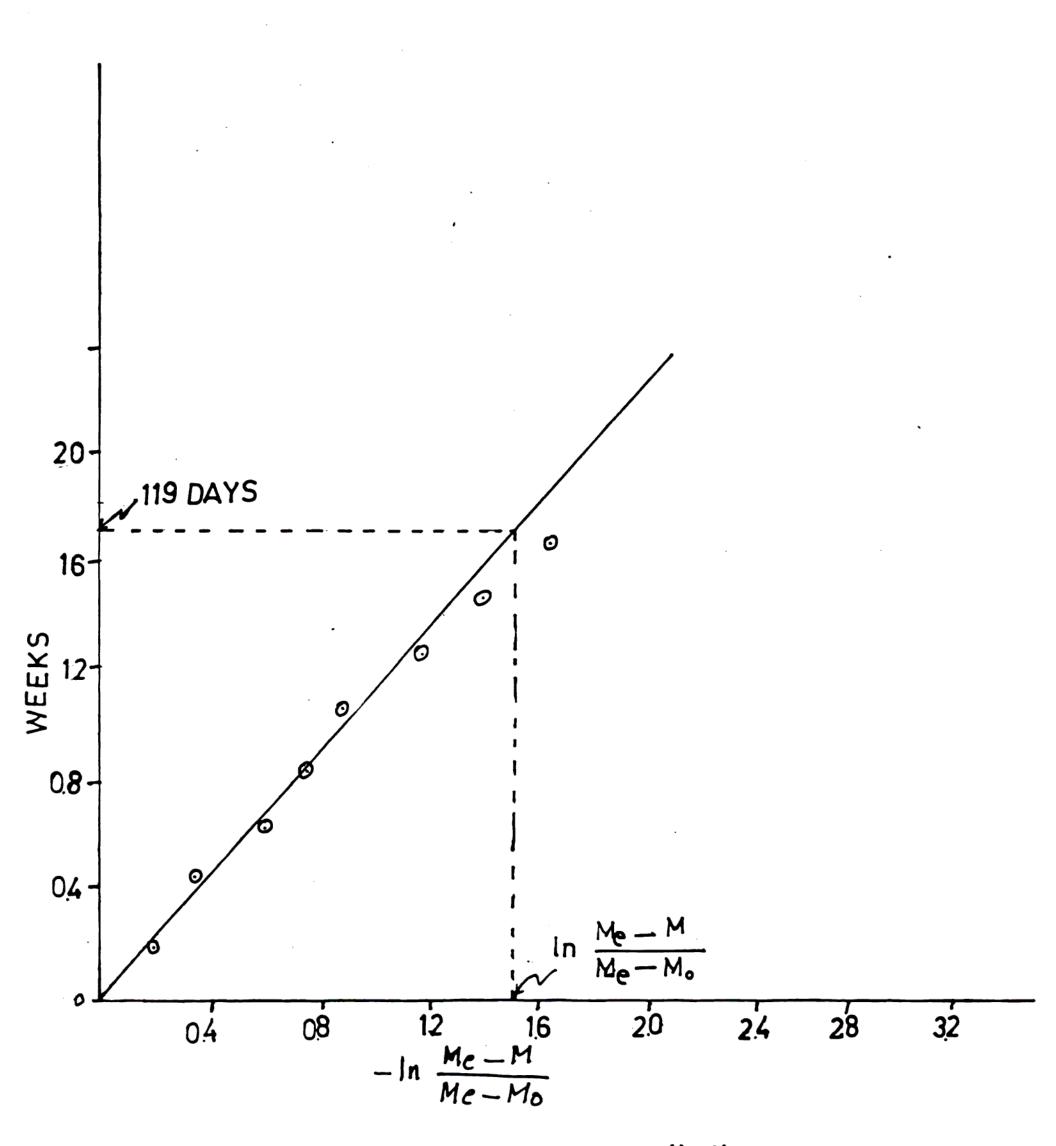
content from the adsorption isotherm graph plotted moisture for CON-S and CG-S samples of both cumin and dry ginger powder 25°C(Figure 5.2 and at 5.3). The values critical relative humidity obtained for CON-S and CG-S samples of both cumin and dryginger powder at 25°C were 43.3, 51.3 and percentage respectively. Referring the isotherms(Figure 5.2 and 5.3) and values οf critical relative humidity, the critical moisture contents of and CG-S samples of cumin and dry ginger CON-S powder found approximately 10.5%. be Since the slopes the isotherms curve did not increase much upto 70% RH (0.7 aw) and therefore, 70% RH level was considered critical relative humidity point and critical moisture contents of ground samples corresponds for this, were used for the determination shelf-life. The values of critical moisture contents of of CG-S of cumin and ginger powder CON-S and at 25°C there considered 11.0% (db). Since was no significant observed in the adsorption isotherm of samples of cumin and dry ginger powder at 37°C hence, CG-S 11.0% (d.b) critical moisture content was also considered for further calculation of shelf-life of spice products stored at 37°C.

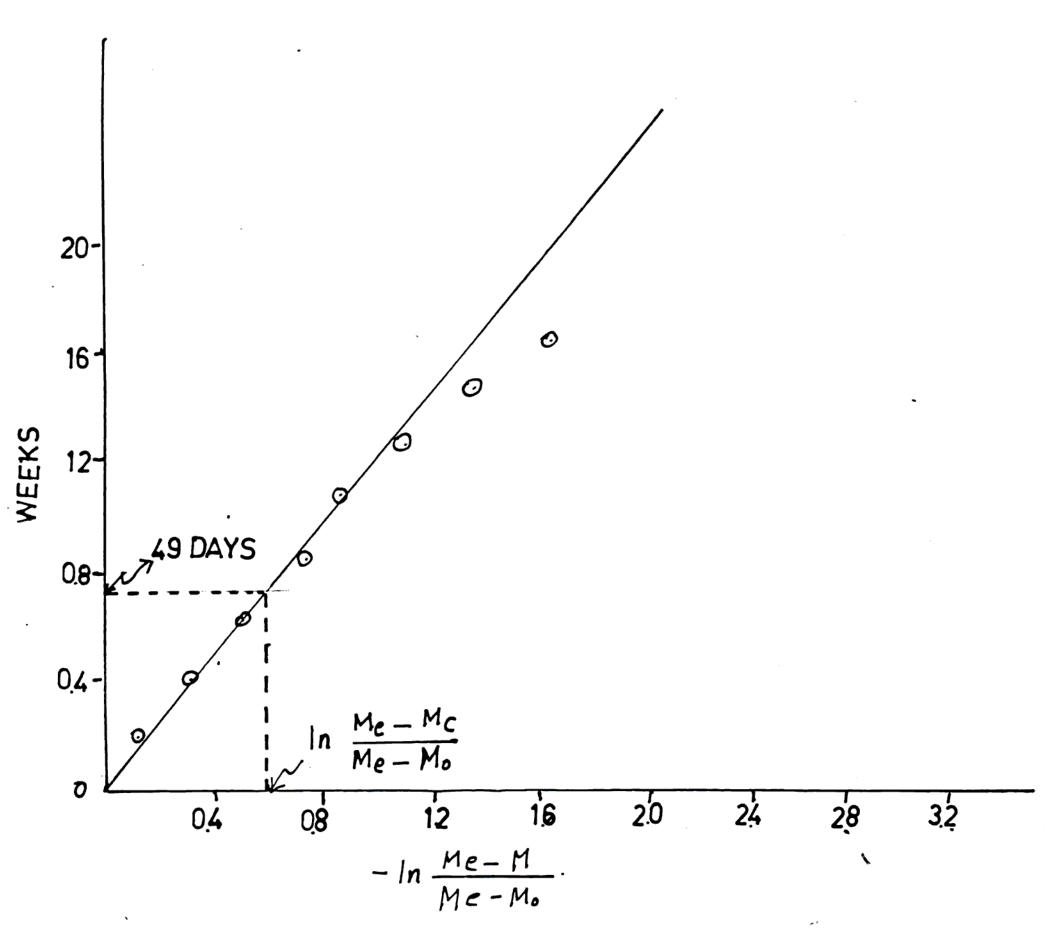
The relationship between the storage period (weeks) and $\ln(M_e-M)/(M_e-M_o)$ are shown in the Figure 5.12, 5.13, 5.14 and 5.15. From the plot of storage period and $\ln(M_e-M)/(M_e-M_o)$



5.12 GRAPHICAL REPRESENTATION OF -In Ma-Mo

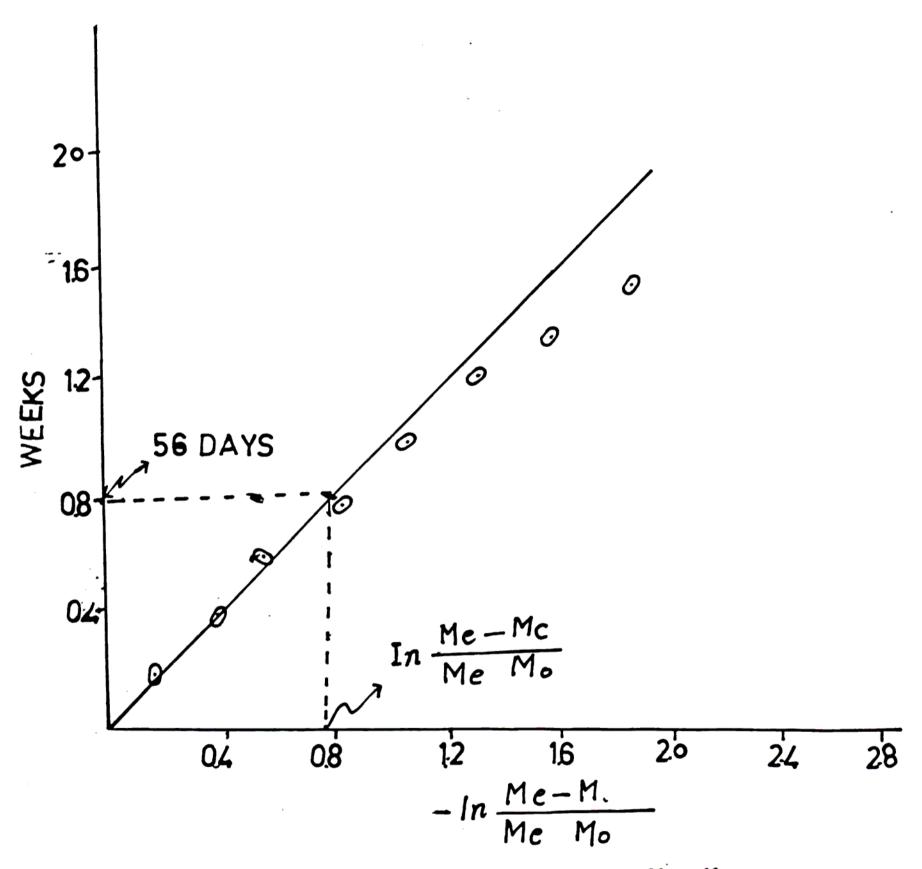
STORAGE PERIOD AT 25°C AND 90% RH OF COLD
GROUND DRY GINGER POWDER PACKED IN AL FOIL/
POLIETHYLENE.





5.14 GRAPHICAL REPRESENTATION OF -1n Ma-Mo

STORAGE PERIOD AT 25°C AND 90% RH OF CONVENTIONAL GROUND CUMIN POWDER PACKED IN POLYETHYLENE POUCH.



5.15 GRAPHICAL REPRESENTATION OF -ln AND

Ma -Mo

STORAGE PERIOD AT 37°C AND 70% RH OF CO'NVENTIONAL GROUND CUMIN POWDER PACKED IN POLYETHYLENE POUCH.

it is evident that CG-S sample of cumin powder packed in low density polyethylene, cellophane and Al/ polylaminate pouches had 56 days 77 days and 98 days shelf-life, respectively at 25°C/90% RH. Similarly, CG-S samples of dry ginger powder had shelf-life, respectively 63 days, 84 days and 119 days.

Predictions of shelf-life of both CON-S and CG-S cumin and dry ginger powder packed samples of in above packaging materials and stored at 37°C/70% RH mentioned were also made and given in the Table 5.12 and 5.13. The shelf-life both cumin and dry given powder packed in Al/polylaminate of found to be more in comparison to the pouches were ground spices packed in other two packaging materials. The shelf-life CON-S and CG-S-samples of cumin packed **A1** / in of both polylaminate pouches increased from 98 to 119 days and 84-91 days, respectively when storage condition changed from 25°C/90% to 37°C/70% RH.

The shelf-life of the ground cumin and dry ginger products packed in different packaging materials was more when stored at high temperature and low relative humidity (37°C/70%) (Table 5.12 and 5.13).

Water plays a very important role in the stability of fresh, frozen and dried foods; it acts as a solvent for chemical, microbiological and enzymatic reaction. Ground

Table 5.12: Shelf life of ground ginger packed in various packaging materials and storage conditions.

S. No.	Types of packaging	Storage conditions							
materials		25°C/ Self li: CON-S	90%RH fe, days CG-S	37°C/70%RH Self life, days CON-S CG-S					
1.	Polyethylene	42	56	56	63				
2.	Cellophane	63	78	77	84				
3.	Al/Polyethylene	77	98	98	119				

Table: 5.13 Skelf life of ground cumin packed in various packaging materials and storage conditions.

S. No.	Types of	Storage conditions						
	packaging materials	25°C/9 Self li CON-S	90%RH fe, days CG-S	Self CON-S	37°C/70%RH life, days CG-S			
1.	Polyethylene	49	56	56	70			
2.	Cellophane	56	77	63	84			
3.	Al/Polyethylene	84	98	91	119			

CON-S = Conventional ground samples at 35°C

CG-S = Cold ground samples at 15°C

cumin and dry ginger products stored at high relative humidity adsorbed more moisture than stored in low relative humidity and would undergo various spoilage reactions.

CHAPIER - VI SUMMARY AND CONCLUSIONS

SUMMARY AND CONCLUSION

The present investigations were taken up with the following objectives:

- i) To study the effect of machine variables and grinding temperature on the fineness and volatile oil content of ground spices.
- ii) To determine the various heat transfer parameters during cold grinding of spices.
- iii) To study the biochemical and microbiological changes during storage of both conventional and cold ground spices.

Cumin (Cuminum cyminum L.) and dry ginger (Zingiber officinale Rose) chips were ground in a water circulating jacketted -type electrical grinder. The temperatures of water were maintained at 10, 15 and 25°C. The effect of various levels of temperatures and flow rates of circulating water on volatile oil content, fineness modulus, sensory quality and heat transfer parameters were studied. Beside these, adsorption isotherm of the ground spice products, prediction of shelf-life of the ground spice products packed in the low density polyethylene, cellophane, Al/ polylaminate pouches and stored at 25°C/90% RH and 37°C/70% RH were also studied.

The results of the different experiments are summarised below:

- The volatile oil content of cumin and dry ginger powder were 2.5% and 2.0% respectively.
- The volatile oil contents of cumin powder increased from 2.5 to 3.5% and in ginger it increased from 2.0 to 3.0%, respectively when grinding temperature was reduced from 35°C to 10°C.
- 3. The fineness modulus data decreased with the lowering of the grinding temperature i.e. fineness of the ground product improved.
- 4. Aroma of the soup prepared from the volatile oils of both cold ground and conventional ground cumin and dry ginger powder differ significantly (F>5.0%).
- 5. Sensory quality of the cold ground sample was better in comparison to the conventional ground sample. Cold ground spice sample had lighter colour, free flowing and better aroma.
- 6. The heat transfer parameters i.e. Nusselt number (Nu=hd/k), the surface heat transfer coefficient (hc)and rate of heat loss (Q) increased with the increase in Reynold number (NR_e).

- 7. Adsorption isotherms of both cumin and dry ginger powder samples at 25°C and 37°C were more or less same.
- 8. Adsorption isotherms were sigmoid in shape(Type-II). The rate of moisture adsorptions were slow or less upto a water activity of 0.7.
- 9. Henderson equation fitted the adsorption isotherm data reasonably well (R = 0.8-0.9).
- 10. Critical relative humidity and critical moisture content of both cumin and dry ginger powder were; ac = 70% RH ≈ Mc = 11.0%(d.b).
- 11. Moisture content, free fatty acid content and total plate count (microbial-load) of both cumin and dry ginger powder packed in the low density polyethylene, cellophane and Al/ polylaminate pouches were increased during storage.
- 12. The percentage increase in the moisture content, free fatty acid and total plate count was less in the ground spice product packed in the Al/ polylaminate pouches in comparison to the cellophane and low density polyethylene pouches.
- 13, The percentage increase in the moisture content, free fatty acid content and total plate count was less in all

the packed samples stored at 37°C/70% RH.

- 14. Major portion of the microorganism was consisted of fungi.
- 15. The predicted shelf-life of the ground cumin and dry ginger products packed in different packaging materials were more when stored at 37°C/70% RH.
- 16. The predicted shelf-life of the ground spices packed in the Al/ polylaminate pouches were more in comparison to the samples packed in the low density polythylene and cellophane pouches.

Conclusion :

the volatile oil content in has great economic significance in the manufacture and sale of ground products, particularly when/where sale or trading spice based on sensory qualities. Lowering the temperature of the grinding zone of the grinder by circulating chilled water (10shown profound influence of the retention of volatile 25°C) oil, fineness of the particle, and sensory quality of the ground products. Data on surface heat transfer coefficient and rate of heat loss during grinding operation could be used cooled-jacketed continuous water in designing a type electrical grinder machine. Aluminium /low density polyethylene laminate pouch a storage conditions of 37°C/70% could be used for packaging and storage of cumin and ginger powder.

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