DEVELOPMENT OF STARCH BASED BIODEGRADABLE PLASTIC

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M. Tech. (Agril. Engg.)

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COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

JUNAGADH AGRICULTURAL UNIVERSITY

JUNAGADH – 362 001

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DEVELOPMENT OF STARCH BASED BIODEGRADABLE PLASTIC

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IN

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CERTIFICATE – I

This is to certify that the thesis entitled **"DEVELOPMENT OF STARCH BASED BIODEGRADABLE PLASTIC"** submitted by **HIRPARA NEHA JAYANTILAL (Reg. No. 1050217002)** in partial fulfilment of the requirements for the award of the degree of **DOCTOR OF PHILOSOPHY (AGRICULTURAL ENGINEERING)** in the subject of **PROCESSING AND FOOD ENGINEERING** to the Junagadh Agricultural University is a record of bonafide research work carried out by her under my guidance and supervision and the thesis has not previously formed the basis for the award of any degree or other similar title. The candidate had fulfilled all prescribe requirements. The assistance and help received during the course of investigation have been fully acknowledged. She has successfully completed the comprehensive/ preliminary examination held on **JUNE 09, 2020** as required under the regulation for Post-graduate studies. She has submitted *Kachcha* bound thesis on**, FEBRUARY 23, 2021.**

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C E R T I F I C A T E – II

Date:01/09/2021

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DEDICATION

To my parents
The reason of what I become today.
Thanks for your great support and continuous care.

To my only best friend
I am really grateful to you.
You have been my inspiration and my soul mates.

To my respected guide
Who is the continuous Source of
Inspiration and constant encouragement for me.

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Neha...

Abstract

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Name of Student **Name of Major Guide**
HIRPARA NEHA JAYANTILAL **Dr. M. N. DABHI**

DEVELOPMENT OF STARCH BASED BIODEGRADABLE PLASTIC

ABSTRACT

Key words: Starch, Synthetic polymer, Biodegradable polymer, Packaging

The present investigation entitled "Development of Starch Based Biodegradable Plastic" in 2019-20 were conducted at Biochemical Laboratory, Department of Processing & Food Engineering, College of Agricultural Engineering & Technology Junagadh Agricultural University, Junagadh. The experiments were aimed to prepare a biodegradable plastic from renewable sources such as starch that would be environment-friendly. Optimization of preparation condition would help us to study the feasibility and potential of this starch with other additives to obtain a biodegradable as well as high tensile strength plastic.

Synthetic polymers are important in many branches of industry, particularly in the packaging industry. However, it has an undesirable influence on the environment and causes problems with deposition of waste and consumption. Therefore, there is a tendency to replace the polymer with biodegradable polymer that undergoes a process.These artificial macromolecular substances are usually generated from petroleum and most of are regarded as non-degradable. However, the resources of petroleum are limited, and the flourishing use of non biodegradable polymers has caused serious environmental tribulations. The Non-biodegradable Plastics are the derivative of renewable resources that cannot be easily broken down in the environment by micro-organisms. Biodegradable plastics are plastics synthesized using renewable resources which facilitate the straight forwardly decomposition in the atmosphere by means of microbes. Among the natural polymers, starch takes prior attention. Starch is a biodegradable polysaccharide, produced in plenty at low expenditure and exhibits thermoplastic in nature. Thus, it has become most promise alternative material to replace conventional plastics in individual market segments.

Corn, potato and rice starch were used for the experimental work. The physical properties of starch (Corn, Potato & Rice) powder *viz.,* WAI and WSI were determined as per the standard analytical methods.

Development of starch film was carried out at different levels of starch concentration (5, 6.5, 8, 9.5 &11) and glycerol concentration (0.5, 0.875, 1.250, 1.625 & 2) whereas distilled water 100 ml and acetic acid 1 ml were kept constant throughout the experiment. The films were prepared by casting technique using a film-forming solution. The results on biodegradable film were analysed using Central Composite Rotatable Design (CCRD), Response Surface Methodology with two factors. The characteristic evaluation on the basis of physical properties, viz., thickness, physico-chemical properties viz., moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology, mechanical

properties viz., tensile strength and puncture strength as well as biodegradation properties viz., reduction in weight of developed biodegradable packaging film were determined using standard analytical methods and instruments.

The physical properties of starch powder *viz.*, water absorption index of potato, corn and rice starch powder with their standard deviation was found as 139 ± 1.53%, 155 ± 2 % and 130 ± 2.51% respectively and water solubility index of potato, corn and rice starch powder with their standard deviation was found as 82 ± 1.52%, 86 ± 2.50 % and 79 ± 2.08 % respectively.

Physico-chemical properties of corn starch biodegradable plastic *viz.*, moisture content, transparency, water absorption capacity and water vapour permeability was found as 22.83%, 76.92%, 180%, 0.0045 g mm/m^2 kPa and mechanical properties *viz.*, tensile strength 14.78 MPa and puncture 12.1 MPa respectively. The response surface quadratic model for corn starch film optimized the treatment condition as 7 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 18.38 %, transparency 68.36, water absorption capacity 145.93%, water vapour permeability 0.002 g mm/m^2 day KPa, tensile strength 10.84 MPa and puncture strength 8.18 MPa. The production cost for development of corn starch biodegradable packaging film of 1m^2 was estimated by 28.5 Rs.

Physico-chemical properties of potato starch biodegradable plastic *viz.*, moisture content, transparency, water absorption capacity and water vapour permeability was found as 23.1 %, 69.54%, 190%, 0.0058 g mm/m^2 kPa and mechanical properties *viz.*, tensile strength 13.62 MPa and puncture strength 10.54 MPa respectively.

The response surface quadratic model for potato starch film optimized the treatment condition as 7.1 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 18.52 %, transparency 65.47%, water absorption capacity 159 %, water vapour permeability 0.004 g mm/m^2 day KPa , tensile strength 8.90 MPa and puncture strength 7.17 MPa. The production cost for development of potato starch biodegradable packaging film of 1m^2 was estimated by 35.9 Rs.

Physico-chemical properties of rice starch biodegradable plastic *viz.*, moisture content, transparency, water absorption capacity and water vapour permeability was found as 24.12%, 53.6%, 195%, 0.0071 g.mm/m^2kPa and mechanical properties *viz.*, tensile strength 10.98 MPa and puncture 8.63 MPa. The response surface quadratic model for rice starch film optimized the treatment condition as 6.6 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 15.39 %, transparency 50.08, water absorption capacity 157.90 %, water vapour permeability 0.005 g mm/m^2 day KPa, tensile strength 6.11 MPa and puncture strength 5.03 MPa respectively. The production cost for development of corn starch biodegradable packaging film of 1m^2 was estimated by 53.7 Rs.

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Now, as I carry this in my hand, I carry with me memories that will enrich my nostalgia.

Place: *Junagadh*
Date:23/ 02/2021 ***(Hirpara Neha J.)***

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LIST OF ABREVIATIONS

@	:	At the rate of	m^2	:	Square meter
C.D.	:	Critical difference	cm^2	:	Square centimeter
cm	:	Centimeter	Max.	:	Maximum
C.V.	:	Coefficient of Variation	Min.	:	Minimum
NS	:	Non-significant	ml	:	Milliliter
e.g.		Exempli gratia	mg	:	Milligram
^0C	:	Degree Celsius	ANOVA	:	Analysis of variance
J		Journal	kPa		Kilopascal
df	:	Degree of freedom	M. Tech.	:	Master of Technology
S.S.	:	Sum of squares	CCRD		Central composite rotatable design
M.S.	:	Mean squares	Ph.D.	:	Doctor of philosophy
E	:	Error	PE	:	Polyethylene
et al.	:	Co-worker (et alli)	RNA	:	Ribonucleic acid
etc	:	Etcetera	DNA	:	Deoxyribonucleic acid
g	:	Gram	TPS	:	Thermoplastic starch
hrs.	:	Hours	FAO	:	Food and Agriculture Organization
µm	:	Micrometre	SEM	:	Scanning electron microscopy
ppm	:	Parts per million	t	:	Tonne
i.e.	:	That is	Temp.	:	Temperature
W/V	:	Weight per volume	*viz*.	:	Videlicet (namely)
W/W	:	Weight per weight	/	:	Per
m	:	Meter	%	:	Per cent
ALD	:	Atomic layer deposition	LDPE	:	Low-density polyethylene
G	:	Glycerol	PLA	:	Polylactic acid
GS	:	Glycerol sorbitol	PCL	:	Polycaprolactone
TS	:	Tensile strength	WVP	:	Water vapor permeability
SiO_2	:	Silicon dioxide	FS	:	Film solubility
PVA	:	Polyvinyl alcohol	XPS	:	X-beam photoelectron spectroscopy

CA	:	Citric acid	XRD	:	X-ray diffraction
CMC	:	Carboxymethyl cellulose	DSC	:	Differential scanning calorimeter
MPa	:	Megapascal	SCBP	:	Starch-based totally biodegradable polymers
Pa	:	Pascal	RPM	:	Revolutions per minute
<	:	Less-than	ZEO	:	Zataria multiflora Boiss
>	:	Greater-than	MEO	:	Menthapulegium
DMSO	:	Dimethyl sulfoxide	PEG	:	Polyethylene glycol
MB	:	Mungbean	N	:	Newspaper pulp fibre
min.	:	Minute	B	:	Bioplastic
JIS	:	Japanesse industrial standard	AOAC	:	Association of Official Analytical Chemists
mm	:	Millimetre	wb	:	Wet basis
RH	:	Relative humidity	ASTM	:	American Society for Testing and Materials
Ma	:	Microampere	ISO	:	International Organization for Standardization
Kv	:	Kilovolt	N	:	Newton
USA	:	United State of America	RSM	:	Response surface methodology
WSI	:	Water solubility index	WAI	:	Water absorption index
S	:	Sorbitol	FTIR	:	Fourier-transform infrared spectroscopy
RVP		Relative vapour pressure	CS	:	Cassava starch

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CHAPTER I

INTRODUCTION

Petrochemical based plastics films such as polyolefin, polyesters and polyamides have been increasingly used as packaging materials because of their availability in large quantities at low cost and functionality characteristics such as good tensile and tear strength, good barrier properties to oxygen and aroma compounds and heat seal ability. However, these plastics are made of petroleum-based materials that are not readily biodegradable and therefore lead to environmental pollution, the most obvious form of pollution associated with plastic packaging is waste plastic dump to the landfills.

Polymer materials are strong, non-metallic mixes of high subatomic loads (Callister, 1999). They contain rehashing macromolecules, and have shifting highlights relying on their creation. A biopolymer is any natural polymer. They are long chain mixes made up of long chain atom subunits. Notable biopolymers incorporate starch, proteins and peptides, DNA, and RNA. These are biodegradable, eco-accommodating and are gotten from normal sources. Biopolymers are regularly biodegradable, and not lethal to deliver. Biopolymers are an alternative to petroleum-based polymers (traditional plastics).

This biopolymer is also called a biodegradable "green plastic", as it is gotten from plants and microorganisms. Biodegradable plastics are those that can be totally degraded in landfills, composters or sewage treatment plants by the activity of naturally occurring micro-organisms. Really biodegradable plastics leave no dangerous, obvious or discernable deposits following degradation (Mooney, 2009).

As packaging industry relies strongly on the petroleum derived plastics that concerns to future economy and environment. Additionally, petroleum products are non-renewable and non-degradable, the shortage of this raw materials, represent a danger to the accessibility and their biodegradability. Biodegradable plastics are produced using plant-based raw materials that permit the common decay process by microscopic organisms and parasites present in the general condition and assists with advancing breakdown the structure of a biodegradable plastic. The final product of which is less harmful to the environment to the as compared to regular plastic bags.

The genuine inclination in packaging research is to create and advance the utilization of "bio-plastics" which are valuable in decreasing waste removal and are acceptable replaces of petroleum, a non-renewable resource with lessening amounts (Ruban, 2009; Souza *et al.,* 2010). Utilization of polymers from renewable sources in food packaging is growing (Mensitieri *et al.,* 2011).The issues in discarding the tremendous amounts of waste produced by non-biodegradable food packaging have prompted the investigation of biopolymers as materials to be utilized as edible coatings in food packaging (Azeredo *et al*., 2012). Improvement of materials from bio polymers for various applications has been an interesting issue for quite a long while because of expanding costs of petrochemicals and expanding natural concerns (Farris *et al*., 2009; Laine *et al*., 2012).

To extend the shelf-life of all types of foods with increasing the preservation and protection from oxidation and microbial spoilage the tendency is to use more natural compounds. The use of synthetic films has led to big ecological problems because this materials are non-biodegradable (Sabiha-Hanim and Siti-Norsafurah, 2012). The common polymers that are utilized in food packaging have the favorable circumstances to be accessible from replenishable resources, biocompatible, biodegradable, and these attributes prompted natural wellbeing (Prashanth and Tharanathan, 2007). Polymers separated or expelled from normal sources can be degraded and changed under various ecological conditions and under the activity of various microorganisms (Mensitieri *et al*., 2011).

Starch, and cellulose polysaccharides are biopolymers, which are found in nature during the growth cycles of all organisms. Protein is also another natural polymer for production biodegradable materials. Whey proteins are capable to form elastic films, and they have been utilized as raw material for biodegradable packaging since they have great oxygen barrier and moderate moisture permeability (Shingala *et al*., 2019). These polymers are regularly artificially changed modification of the degradation rate and improvement of the mechanical properties (Vroman and Tighzert, 2009).

Among the natural polymers, starch is of interest. It is recovered from carbon dioxide and water by photosynthesis in plants. It is one of the most abundant natural polysaccharide raw materials. It is a renewable resource, inexpensive, and widely available (Lourdin *et al*., 2003). Attributable to its total biodegradability, ease and sustainability, starch is considered as a promising possibility for creating maintainable

materials. Numerous efforts have been applied to create starch-based polymers for moderating the petrochemical resources, decreasing natural effect and looking through more applications.

Starch is a notable hydrophilic hydrocolloid biopolymer which is produced by agricultural plants in the form of granules with different sizes inside the endosperm. The most important sources of starch extraction are potatoes, corn, wheat and rice. Starch comprises most of two types of polysaccharides, to be specific amylose and amylopectin. Amylose is a linear molecule with a few branches, whereas amylopectin is a highly branched molecule. In this way, amylose content adds to film quality and extended structure of amylopectin by and large prompts film with low mechanical properties (Mali *et al*., 2002). With 30% amylose (poly-α-1, 4-D-glucopyranoside), a crystalline polymer and 70% amylopectin (poly-α-1, 4-D-glucopyranoside and α-1, 6-D-glucopyranoside) it contents 1% proteins and lipids. Amylose is solvent in water and structures a helical structure. The relative sums and molar masses of amylose and amylopectin shift with the starch source, respecting materials of various mechanical properties and biodegradability (Ratnayake *et al*., 2001; Svihus *et al*., 2005; Castillejo *et al*., 2012).

As the amylose substance of starch builds, the stretching and quality increment as well. The strength of starch under pressure isn't high. The glucoside joins begin to break at 150 °C or more 250 °C the granules breakdown. Starch is normally utilized as a thermoplastic. It is plasticized within the sight of explicit measures of water or plasticizers and warmth. Thermoplastic starch (TPS) has a high affectability to dampness. Thermal properties of TPS have been demonstrated to be more impacted by the substance of water than the starch atomic weight. TPS in this way acquired is practically formless. Starch is extraordinary to uphold the textural properties of numerous nourishments and is generally utilized in food and mechanical applications as a thickener, colloidal stabilizer, gelling agent, bulking agent and water retention agent (Singh *et al*., 2007).

Most regular substances lying on earth are in cooperation as the structure of polysaccharides. An imperative polysaccharide is starch. It is kept within the sight of little granules or cells with distance across stuck between 1-100 μm. Starch is begun in seeds (for instance corn, wheat, rice, grain, or peas) and tubers or roots (for instance potato) of the plants. A ton of the starch conveyed generally speaking starts from corn, additional sorts, for instance, potato, wheat, cassava and yam starch are

likewise made in huge aggregates. Aggregation of starch by potato around 70-72 % of the completely dry mass in the tubers has given in as much as 20 tonne starch for each hectare. While a corn seed involves starch about 60-75 weight %, with a normal give in of 5 tonne for every hectare.

Starch and starch subordinates are seen as promising opportunities for the improvement of biopolymer based packaging materials environment friendly packaging materials mainly due to their renewability, abundance, low cost, film forming properties, bland taste and colour, low solubility, biodegradability etc. (Guohua *et al*., 2006; Tang *et al*., 2008; Yun *et al*., 2008).

Lately, there is a developing enthusiasm for creating starch based biodegradable polymers to supplant manufactured non-degradable materials. Research has been performed concerning the use of these films as a way of improving shelf life of food. Be that as it may, starch has serious restrictions due to its dissolvability and poor water-opposition, making starch items exceptionally delicate to the relative mugginess at which they are put away and utilized. Films developed from starch are described as isotropic, odorless, colorless, non-toxic and biologically degradable (Flores *et al.,* 2007).

Potato (*Solanum tuberosum* L.) is one of the world's major farming yields. India contributes about 11.26% of the absolute potato creation on the planet. Starch is the principle part of solids in potato tubers. In correlation with different sorts of starch, confined potato starch offers a few innovative favorable circumstances for applications in the food and non-food industry. Also plastic substitutes (e.g., shopping bags) have become of strong interest. Starch contains commonplace huge oval round granules extending in size between 5 to 100 μm. Potato starch is an extremely refined starch, containing negligible protein or fat. This gives the powder a reasonable white shading, and the cooked starch common attributes of unbiased taste, great clearness, high restricting quality, long surface and an insignificant inclination to frothing or yellowing of the arrangement. Potato starch has around 20 to 22 % amylase (Kolybaba *et al*., 2003).

Potato starch contains around 800 ppm phosphate bound to the starch; this expands the thickness and gives the arrangement a some what anionic character, a low gelatinisation temperature of roughly 60 °C and high growing force. Numerous kinds of potatoes are developed; for the creation of potato starch, potato assortments with high starch substance and high starch yields are chosen. The development of potatoes

for starch mostly happens in Germany, the Netherlands, China, Japan, France, Denmark, Poland, Canada and India (Toshiko, 1983).

Maize has become a staple food in numerous pieces of the world, with the complete creation of maize outperforming that of wheat or rice. Notwithstanding, little of this maize is expended straightforwardly by people: most is utilized for corn ethanol, animal feed and other maize items, for example, corn starch and corn syrup (Jonathon, 2019).

Corn starch or maize starch is the starch acquired from the corn (maize) grain. The starch is acquired from the endosperm of the part. Corn starch is a typical food ingredient utilized in thickening sauces or soups, and in making corn syrup and different sugars. It is flexible, handily changed, and finds numerous utilizations in industry as cements, in paper items, anti-sticking agent, and textile manufacturing. Corn starch is used for many purposes in several industries, ranging from its use as a chemical additive for certain products, to medical therapy for certain illnesses. Corn starch has 29 % amylose (Kolybaba *et al*., 2003).

Rice is a significant food staple, being one of the most abundant crops produced worldwide (FAO, 2018). Rice starch is a characteristic polymeric sugar and the primary part of rice. In its local structure it is a dissolvable white powder consisting of both amylose and amylopectin. Rice starch has an amylose: amylopectin proportion of about 20:80. In the wake of warming with water it forms a gel with a smooth and rich surface.

The starch content, amylose/amylopectin composition and its characteristics vary with botanical variety and are influenced by climatic and soil conditions during grain development. In view of the natural assortment, be it for instance Japonica or Indica, amylose substance can change from 2-3 % up to 35 %, giving a huge number of conceivable outcomes with regards to making customized gels, simply messing with various local starch varieties (Verma *et al*., 2018).

The earliest utilization of starch in food was as a nutritional material obtained from fruits, vegetables, and roots or seeds, the latter having the advantage of stability for storage. Starch and its subsequent refinements is a nutritive, abundant, and economical food source in an overpopulating world. Food starches perform two basic roles. As a nutritive stabilizer, starches provide the characteristic viscosity, texture, mouth-feel, and consistency of many food products.

The commercial preparation of starch from rice is limited owing to the high cost of brewer's rice relative to other cereals and tubers. Brewer's rice is used as an ingredient in beer making and as starting material for starch manufacture. The unique structure of rice amylopectin gives rice starch exceptional shelf-life stability, and the branched rice amylose and small starch granule size make rice starch an ideal starch when it comes to creating soft and creamy textures.

Rice starch also has a neutral taste and clear white colour, assuring preservation of the authentic taste and colour of your food product. Rice starch with high amylose is an attractive raw material for use as barriers in packaging materials. Differences in eating quality exist among varieties of similar amylose content that are related to other quality factors, such as gel consistency. The widespread use of rice starch is limited by its higher price relative to corn, wheat, and potato starches. He has been used to produce biodegradable films to partially or entirely replace plastic polymers because of the low cost and renewability, as well as possessing good mechanical properties (Juliano, 1984).

Food packaging and edible films are two major applications of the starch-based biodegradable polymers in food industry. The requirements for food packaging include reducing the food losses, keeping food fresh, enhancing organoleptic characteristics of food such as appearance, odour, and flavour, and providing food safety (Zhao *et al*., 2008). Traditional food packaging materials such as LDPE have the problem of environmental pollution and disposal problems (Ozdemir and Floros, 2004). The starch based biodegradable polymers can be a possible alternative for food packaging to overcome these disadvantages and keep the advantages of traditional packaging materials.

However, the components in the conventional starch-based polymer packaging materials are not completely inert. The movement of substances into the food potentially occurs, and the segment that moves into food may cause hurt for the human body. Starch-based edible films are odorless, tasteless, colorless, non-toxic, and biodegradable. They display very low permeability to oxygen at low relative humidity (Debeaufort *et al*., 2009) and are proposed for food product protection to improve quality and shelf life without impairing consumer acceptability (Flores *et al*., 2007).

Also, starch can be changed into a frothed material by utilizing water steam to supplant the polystyrene froth as packaging material. It very well may be squeezed

into plate or expendable dishes, which can break up in water and leave a non-lethal arrangement, at that point can be consumed by microbic environment (Siracusa *et al*., 2018).

Starch-based biodegradable polymers have discovered two significant applications in horticulture: the covering of greenhouse and mulch film (Dilara and Briassoulis, 2000). The utilization of agriculture films is inexhaustible. By and large, the removal techniques for tradition films are landfill, reusing or consuming. But they are time-consuming, not economic and lead to environmental pollution (Bohlmann and Toki, 2004). Subsequent to utilizing, starch-based films can be blasted through soil and discarded legitimately. Besides, no poisonous deposits shaped after the degradation of starch based biodegradable polymers (Scott, 2000; Malinconico *et al*., 2002). In this way, the improvement of starch-based materials for agribusiness applications is being preceded.

Starch-based biodegradable polymers have a few points of interest to be medical polymer materials (Mendes *et al*., 2001)

a) Decent biocompatibility

b) Biodegradable and its corruption items are non-lethal

c) Legitimate mechanical properties

d) Degradation as requirement

A significant number of these applications can be found in the clinical field and can be generally partitioned into three classifications: drug delivery systems, wound closure and healing products, and surgical implant devices (Sinha and Kumria, 2001). Medication conveyance inside the human body can be handily controlled with the utilization of biodegradable capsules (Reis *et al*., 2008). In wound mending, resorbable non-wovens for the substitution of human tissue, just as basic sutures, staples, clasps or networks are accessible (Boesel *et al*., 2004).

Hence, our target is to set up a biodegradable plastic from inexhaustible sources, for example, starch that would be well disposed of. Advancement of planning conditions would assist us with studying the attainability and capability of this starch with different added substances to acquire a biodegradable just as high rigidity plastic.

Practical utility of the research problem

Synthetic polymer materials have been broadly utilized in each field of human movement during the most recent decades. These artificial macromolecular substances are typically starting from petroleum and a large portion of the ordinary

ones are viewed as non-degradable. Nonetheless, the petroleum resources are constrained and the sprouting utilization of non-biodegradable polymers has caused genuine ecological issues. In this manner, the polymer materials which are degradable and additionally biodegradable have been given increasingly more consideration.

Food is the necessity of our day to day life. Now a day's most of the food items are packed. In regular day to day existence, packaging is a significant zone where biodegradable polymers can be utilized. For increasingly characteristic items, bio-based films or biopolymers, improving the nature of numerous items is essential to fulfill the customers' requests of all the more environmentally friendly packaging. This methodology will keep on assuming a significant job in the food industry (Cutter, 2006; Satyanarayana *et al.,* 2009). In order to reduce the volume of waste, biodegradable polymers are often used.

Other than their biodegradability, biopolymers have different attributes as air permeability, low temperature sealability, accessibility and low cost. A few biopolymers, for example, starch, cellulose, chitosan, PLA, PCL, PHB and so forth are utilized for packaging purposes. The present pattern in food packaging is the utilization of mixes of various polymers like starch-PLA mixes, starch-PCL mixes and so on. Conventional food packaging materials, for example, LDPE have the issue of ecological contamination and removal issues. The starch based biodegradable polymers can be a potential option for food packaging to overcome these disadvantages and maintain the benefits of conventional packaging materials. Clearly, the starch-based biodegradable polymers are attractive for food industry and will make great progress in the future.

Lately, there has been a marked increment in the enthusiasm for utilization of biodegradable materials in packaging. The principal function of packaging is protection and preservation of food from external contamination. This function involves retardation of deterioration, extension of shelf life, and maintenance of quality and safety of packaged food. Biodegradable polymers are the one which fulfill all these functions without causing any threat to the environment. The belief is that biodegradable polymer materials was reduce the need for synthetic polymer production (thus reducing pollution) at a low cost, thereby producing a positive effect both environmentally and economically. Therefore, the current study was undertaken with the following objectives.

Objectives

1. To study the physical properties of potato, corn and rice starch powder.
2. To optimize the process parameters for development of starch based biodegradable plastic using response surface methodology.
3. To study the physico-chemical and mechanical properties of developed starch based biodegradable plastic.
4. To evaluate starch based biodegradable plastic film for packaging.
5. To study the biodegradability of developed starch based biodegradable plastic.
6. To carry out the cost analysis of developed starch based biodegradable plastic.

CHAPTER II

REVIEW OF LITERATURE

The related review of literature on research work carried out in India and elsewhere in recent years on amylose and amylopectine content of starch, biopolymers used in food packaging, factor affecting starch-based biodegradable film and development of biodegradable film from starch and their properties are reviewed and presented here as under:

2.1 Amylose and amylopectine content of starch

Kolybaba *et al.* (2003) determined the amylose content of some frequent starches.

Table 2.1: Approximate content of amylose on common starch

Starch Source	Amylose (%)
Oat	27
Corn	29
Manioc	15
Sweet potato	17
Arrowroot	21
Potato	20-22
Rice	19
Tapioca	16
Wheat	25

Garc *et al.* (2015) measured amylose and amylopectine of different starches.

Table 2.2: Amylose and amylopectin concentration of various starch sources.

Type of starch	Amylose (%)	Amylopectin (%)
Amylomaize	48-77	23-52
Banana	17-24	76-83
Corn	17-25	75-83
High amylose corn	55-70	30-45
Potato	17-24	76-83
Rice	15-35	65-85
Cassava	19-22	28-81
Sorghum	25	75
Wheat	20-25	75-80
Yam	9-15	85-91
Waxy	<1	>99

Gadhve *et al.* (2018) measured amylose and amylopectin content of common starches.

Table 2.3 Approximate amylose and amylopectin content of common starches

Starch Type	Amylose Content (%)	Amylopectin Content (%)
Dent corn	25	75
Waxy corn	<1	>99
Tapioca	17	83
Potato	20	80
High amylose corn	55 -70 (or higher)	45 -30 (or lower)
Wheat	25	75
Rice	19	81

2.2 Biopolymers used in food packaging

Catherine (2006) reviewed on opportunities for bio-based packaging technologies to improve the quality and safety of fresh and further processed muscle foods. Their examination on improvements have shown the plausibility, usage, and business use of an assortment of bio-based polymers or bio-polymers produced using an assortment of materials, including inexhaustible/practical horticultural products, and applied to muscle nourishments.

Gabor and Tita (2012) reviewed on biopolymers used in food packaging. The most relevant biopolymers like polysaccharides (alginate, carrageenan, cellulose, chitosan, curdlan, gellan, pectin, pullulan, starch and xanthan gum), proteins (collagen, gelatin, soy protein, whey proteins and zein) aliphatic polyesters (poly lactic acid (PLA), poly hidroxy butyrate (PHB)) which are used and have great potential applications in food packaging. Most works that they have examined ordered the biopolymers positioned in three classes to be specific: biopolymers from inexhaustible sources, artificially orchestrated biopolymers and microbial incorporated biopolymers.

Pawar and Purwar (2013) reported on biodegradable polymers in food packaging. The principal function of packaging is protection and preservation of food from external contamination. This capacity includes impediment of crumbling, expansion of time span of usability, and upkeep of value and wellbeing of packaged food. Biodegradable polymers are the one which satisfy every one of these capacities without making any danger to the earth. The belief is that biodegradable polymer

materials will reduce the need for synthetic polymer production (thus reducing pollution) at a low cost, thereby producing a positive effect both environmentally and economically.

Vartiainen *et al.* (2014) reviewed biopolymer films and coatings in packaging applications. They reviewed with special emphasis on the barrier properties, which are crucial in terms of food packaging. The State-of-the-specialty of a few biopolymers including gelatin, starch, chitosan, xylan, galactoglucomannan, lignin and cellulose nanofibrils was examined. As much of the time the packaging related properties of single layer biopolymer films are lacking, the thin film coatings, for example, sol-gel and ALD (nuclear layer testimony), just as the multilayer coatings.

Galgano *et al*. (2015) focused on biodegradable packaging for fresh-cut fruits and vegetables and their effects on the product quality. They reported that biodegradable packing did not allow full control of the product moisture loss. Better outcomes could be accomplished by the joined utilization of biodegradable packaging with eadible coatings and enrichment with silver nanoparticles.

Gadhave *et al*. (2018) reviewed on starch based bio-plastics for future of sustainable packaging. They reported that due to the growing concern over environmental problems of these materials, interest has shifted towards the development and promoting the use of “bio-plastics”. This bio-plastic is produced from agro/food sources, materials such as starch, cellulose, etc. and which are considered safe to be used in food applications. To enhance the mechanical properties, and water barrier properties, it can be blended easily with other polymer as well as nano fillers.

2.3 Factor affecting starch-based biodegradable film

2.3.1 Amylose and amylopectin content

Westling *et al.* (2002) prepared films from potato starch, amylose, and amylopectin and blends by solution casting. Results showed that amylose films had a relative crystallinity of about 30% whereas amylopectin films were entirely amorphous. The mixing of amylose and amylopectin brought about film with a significantly higher level of crystallinity than could be anticipated. This is clarified by co-crystallization among amylose and amylopectin and perhaps by crystallization of amylopectin. The solidified material offered ascend to an endothermic recognized with differential filtering calorimetry. The enthalpy and pinnacle temperature of the

change additionally expanded as the amylose content diminished. When the amylose extent in the mixes was low, separate periods of amylose and amylopectin were seen by light microscopy. At higher amylose extents, nonetheless, the stage division was obviously forestalled by amylose gelation and the development of constant amylose organize. Addition, the amylose network in the films, observed with transmission electron microscopy, consisted of stiff strands and open pores and became less visible as the amylose proportion decreased.

Alves *et al.* (2007) studied the effect of amylose enrichment on cassava starch films properties. This investigation indicated the mechanical and boundary properties of cassava films were impacted by the amylose substance. The amylose enrichment originated stronger films and this could be explained because during drying of film-forming solutions, water evaporates, allowing the formation of starch network. During this stage the proximity of starch chains induced by higher amylose contents could facilitate the formation of matrix with more polymer content per area.

Ming *et al.* (2011) characterized the biodegradable films from corn starch with different amylose content. They reasoned that amylose content had altogether influenced the mechanical and thermal properties of the biodegradable starch-based films. The high amylose starch films displayed better mechanical properties, for example, higher modulus and tensile strength, and very high impact strength. The reasons for this include not only the easy entanglement of long linear amylose chains, but also the retained granular structure in high amylose films, which may act as self-reinforcement.

Muscat *et al.* (2012) studied the effect of low and high amylose starches on film forming behavior. They found that, films with high amylose content showed higher glass transition temperature, tensile strength and modulus of elasticity values and lower elongation values than low amylose starch films. There was an expansion in thermal and mechanical properties of high amylose starch films. This could be a result of what happens when the drying of film-shaping arrangements, water evaporates, and permitting the development of starch organize happens. During this stage, the proximity of starch chains induced by higher amylose contents could facilitate the formation of a matrix with more polymer content per area.

Colussi *et al.* (2017) evaluated acetylated rice starches films with different levels of amylose: Mechanical, water vapor barrier, thermal, and biodegradability

properties. Biodegradable films from native or acetylated starches with different amylose levels were prepared. Rice grains cultivars of IRGA 417 (high-amylose), IRGA 406 (medium-amylose), and Motti (low-amylose), with amylose contents of 32%, 20%, and 8%, respectively, were used. The films from acetylated high amylose starches had higher moisture content and water solubility than the native high amylose starch film. However, the acetylation did not affect acid solubility of the films, regardless of the amylose content. Films produced using high and medium amylose rice starches were gotten; anyway low amylose rice starches, regardless of whether local or acetylated, didn't frame films with alluring qualities. The acetylation decreased the tensile strength and increased the elongation of the films. The acetylated starch-based films had a lower decomposition temperature and higher thermal stability than native starch films. Acetylated starches films exhibited more rapid degradation as compared with the native starches films.

2.3.2 Type and content of plasticizers

Srinivasa *et al.* (2007) studied the effect of plasticizers and fatty acids (stearic and palmitic acids) on the mechanical and permeability characteristics of chitosan films. No considerable differences in water vapor permeability were observed in fatty acid blend films.

Talja *et al*. (2007) investigated the effect of various polyols and polyol contents on physical and mechanical properties of potato starch-based films. Plasticizers, such as glycerol, sorbitol or xylitol, are typically used for reducing the brittleness. At low glycerol concentrations both strain and strength decreased but above 20% glycerol concentration the elongation reached larger values. Impacts of glycerol, sorbitol or xylitol on physical and mechanical properties of starch films were highest for glycerol and lowest for sorbitol. High substance of xylitol and sorbitol brought about changes in physical and mechanical properties of films most likely because of stage detachment and crystallization. The amount of water absorbed by starch films increases with the glycerol content. However, the increase in moisture is greater as the starch content in the film increases. This behavior could be explained because increasing the starch content promotes the formation of more hydrogen bridges and plastic containing glycerol absorb more moisture which is likely due to the hydrophilic nature of glycerol.

Bourtoom and Chinnan (2008a) determined plasticizer effect on the properties of biodegradable blend film from rice starch-chitosan. The results of these studies demonstrated that sorbitol plasticized films provided the films with highest mechanical resistance, but the poorest film flexibility. In contrast, glycerol and polyethylene glycol plasticized films exhibited flexible structure; however, the mechanical resistance was low, while inversely affecting the water vapor permeability.

Dai *et al*. (2010) reported that type and content of plasticizer affected the properties of corn starch films. Increasing the plasticizer content resulted in increasing water vapor permeability of the resulting film. Increase the starch concentration transparency was decrease due to more molecules of starch give darker colour of film. These outcomes would be identified with basic changes of the starch organize by the plasticizer accompanying with the hydrophilic character of plasticizer, which favored the absorption and desorption of water molecules. Plasticizers decreased intra-and inter-molecular forces in starch. Plasticizers could broaden, weaken and modify the structure adequately; at that point the starch chain versatility would be expanded.

Muhammed *et al.* (2015) studied the effect of plasticizer type and concentration on tensile, thermal and barrier properties of biodegradable films based on sugar palm (*Arengapinnata*) starch. The effect of different plasticizer types (glycerol (G), sorbitol (S) and glycerol sorbitol (GS) combination) with varying concentrations (0, 15, 30 and 45 w/w %) on the tensile, thermal and barrier properties of sugar palm starch (SPS) films was evaluated. Regardless of plasticizer types, the tensile strength of plasticized SPS films decreased, whereas their elongation at break (E %) increased as the plasticizer concentrations were raised. However, the E% for G and GS-plasticized films significantly decreased at a higher plasticizer concentration (45% w/w) due to the anti-plasticization effect of plasticizers. Change in plasticizer concentration showed an insignificant effect on the thermal properties of S-plasticized films. The glass transition temperature of SPS films slightly decreased as the plasticizer concentration increased from 15% to 45%. The plasticized films exhibited increased water vapor permeability values from 4.855×10^{-10} to 8.70×10^{-10} $g \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$, irrespective of plasticizer types. Overall, the current study manifested that plasticized sugar palm starch can be regarded as a promising biopolymer for biodegradable films.

2.3.3 Type and content of lipids

Yang and Paulson (2000) investigated gellan/lipid composite films through emulsification and determining the effect of lipid (beeswax and 1:1 blend of stearic palmitic acids) on the moisture barrier, and mechanical and optical properties of the films. The result showed that the expansion of the lipids to gellan films altogether improved the water vapour penetrability ($p<0.05$), however brought down the mechanical properties and caused the films become obscure. Beeswax was more powerful than stearic-palmitic acids in diminishing the water vapour penetrability and movies with beeswax demonstrated preferable mechanical properties in general over those with stearic-palmitic acids.

Bourtoom and Chinnan (2009) investigated improvement of water barrier property of rice starch chitosan composite film incorporated with lipids. Tensile strength and water vapor permeability of rice starch-chitosan composite film decreased with the addition of lipids, whereas elongation at break increased in these films. Therefore, incorporation of lipids into hydrophilic polysaccharide films in an effort to decrease their water vapor permeability can negatively affect film strength as expressed by tensile strength measurements. In addition, rice starch-chitosan films added with oleic acid provided the films with smoother surface and higher values of tensile strength and elongation at break but lower water vapor permeability than with margarine and palm oil, respectively. The differences in mechanical and barrier properties between these films could be related to their physical state, structure, and chemical nature of the lipids.

2.3.4 Relative Humidity

Masclaux *et al.* (2010) reported that experimental and modelling studies of transport in starch nanocomposite films as affected by relative humidity. They found that at high relative mugginess, the water diffusion rate indicated higher in the starch nanocomposite films. As indicated by these outcomes, it appeared that it was more water sorption and dispersion in starch matrix because of its initially high swelling capacity and high chain mobility. Besides, the results demonstrated that the oxygen permeability coefficient slightly increased in the range of relative humidity between 30 to 45% and greatly increased at higher relative humidity.

2.4 Development of biodegradable film from starch and their Properties

Kester and Fennema (1986) studied that biodegradable starch films generally provide a good barrier against oxygen at low and intermediate relative humidity, and have good mechanical properties, but their barrier against water vapor is poor due to their hydrophilic nature.

Mali *et al.* (2005) studied the relationships among the composition and physicochemical properties of starches with the characteristics of their films. The physical, molecular, and functional properties of corn, cassava, and yam starches were related to the film properties of these starches. Corn, cassava, and yam starches contained 25%, 19%, and 30% amylose, respectively. Amylose from yam starch showed the smallest molecular weight among the starches and amylopectin from corn starch the smallest molecular weight. Cassava starch presented higher amylopectin content, and its gels and films were less strong, more transparent, and more flexible than corn and yam films. Increase the starch concentration, puncture strength was increased due to starch molecules bind with each other and make a complex structure. This behaviour could be related structural modifications of starch network when plasticizer was incorporated. The matrix of the film becomes less dense, facilitating movement of polymer chains under stress, therefore decreasing the film resistance. Plasticized films of the three starches were more flexible, with a higher strain and lower stress at break when the glycerol content increased. Unplasticized films were brittle and had water vapor permeability values ranging from 6.75 x 10^{-10} to 8.33 x 10^{-10} $g \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$.

Xu *et al*. (2005) studied that rice starch can be used to produce biodegradable films to partially or entirely replace plastic polymers because of its low cost and renewability, as well as possessing good mechanical properties.

Bourtoom and Chinnan (2008b) studied the preparation and properties of rice starch chitosan blend biodegradable film. Biodegradable blend films from rice starch chitosan were developed by casting film-solution on leveled trays. The impact of the proportion of starch and chitosan (2:1, 1.5:1, 1:1, and 0.5:1) on the mechanical properties, water boundary properties, and miscibility of biodegradable mix films was examined. The biodegradable mix film from rice starch chitosan demonstrated an expansion in tensile strength (TS), water vapour permiaility (WVP) lighter color and yellowness and a decreasing elongation at the break (E), and film solubility (FS) after

incorporation of chitosan. The presentation of chitosan expanded the crystalline peak structure of starch film; however, too high chitosan concentration yielded phase separation between starch and chitosan. The amino gathering band of the chitosan atom in the FTIR range moved from 1541.15 cm $^{-1}$ in the chitosan film to 1621.96 cm $^{-1}$ in mix films. These outcomes brought up that there was an atomic miscibility between these two parts. The properties of rice biodegradable mix film and chose biopolymer and manufactured polymer films were thought about; the outcomes exhibited that rice starch chitosan biodegradable mix film had mechanical properties like the other chitosan films. However, the water vapor permeability of rice starch chitosan biodegradable blend film was characterized by relatively lower water vapor permeability than chitosan films but higher than polyolefin.

Talja *et al.* (2008) investigated effect of type and content of binary polyol mixtures on physical and mechanical properties of starch-based edible films. Effects of binary mixtures (1:1) of glycerol, xylitol and sorbitol at various concentrations on physical and mechanical properties of potato starch-based edible films stored at various relative vapor pressures (RVP). Edible films were prepared by casting using suspension of binary polyol mixtures (20–50% of solids), potato starch (50–80% of solids) and distilled water which was heated to gelatinize starch. Water sorption of films was affected by the type and content of binary polyol mixture. Water vapor permeability (WVP) of films was found to increase as the content and plasticization effect of binary polyol mixture as well as RVP gradient increased. Young's modulus of films was observed to decrease with the concurrent increase of elongation at break as the plasticization and the content of binary polyol mixture increased at RVP of 33%. Both tensile strength and elongation at break decreased when films were plasticized at the high content of binary polyol mixtures and stored at the RVP of 54% and 76%.

Xiong *et al*. (2008) investigated structure and properties of starch-based biodegradable film. A starch-based biodegradable film, with a nano silicon dioxide (nano-SiO2) content, was prepared by the coating method. The structure of the film was described by Fourier change infrared spectroscopy (FT-IR), X-beam photoelectron spectroscopy (XPS), X- ray diffraction (XRD), differential scanning calorimeter (DSC), and scanning electron microscopy (SEM). When contrasted with a film without nano-SiO2, the crystallinity of the film was diminished from 41.2 to

32.9%; the elasticity, breaking stretching, and transmittance were expanded by 79.4, 18 and 15%, separately; and the water assimilation was diminished by 70%. A hydrogen bond was framed in nano-SiO_2 and starch/polyvinyl liquor (PVA), and intermolecular hydrogen holding of the starch was diminished at the expansion of nano-SiO2. Meanwhile, the chemical bond C–O–Si was also formed in nano-SiO_2/starch/PVA hybrid materials; therefore, the miscibility and compatibility between starch and PVA were increased.

Lu *et al.* (2009) studied Starch-based completely biodegradable polymer materials. Starch is a characteristic polymer which has numerous extraordinary properties and some weakness all the while. Some synthetic polymers are biodegradable and can be customized without any problem. Hence, by joining the individual preferences of starch and synthetic polymers, starch-based totally biodegradable polymers (SCBP) are potential for applications in biomedical and ecological fields. Therefore it received great attention and was extensively investigated.

Ghanbarzadeh *et al.* (2010) analysed the effect of citric acid (CA) and carboxymethyl cellulose (CMC) in view to improve the barrier and mechanical properties of corn starch-based edible films. The films produced from pure starch are brittle and difficult to handle. Chemical modifications (e.g. crosslinking) and using a second biopolymer in the starch based composite have been studied as strategies to produce low water sensitive and relatively high strength starch based materials. The effects of CA and CMC on the water vapor permeability (WVP), moisture absorption, solubility and tensile properties were investigated. The water vapor barrier property and the ultimate tensile strength (UTS) were improved significantly ($p < 0.05$) as the CA percentage increased from 0 to 10% (W/W). At the level of 15% (W/W) CMC, the starch films showed the lowest WVP values (2.34× G/Pa h m) and UTS increased from 6.57MPa for the film without CMC to 16.11MPa for that containing 20% CMC.

Shinji Ochi (2011) reported durability of starch based biodegradable plastics reinforced with manila hemp fibers. The biodegradability of Manila hemp fiber fortified biodegradable plastics was observed for 240 days in a characteristic soil and 30 days in a fertilizer soil. After biodegradability tests, weights were measured and both tensile strength tests and microscopic observation were performed to evaluate the biodegradation behavior of the composites. The results indicate that the tensile

strength of the composites displays a sharp decrease for up to five days, followed by a gradual decrease. The weight loss and the reduction in tensile strength of biodegradable composite materials in the compost soil are both significantly greater than those buried in natural soil. The biodegradability of these composites is enhanced along the lower portion because this area is more easily attacked by microorganisms.

Dhanapal *et al*. (2012) reviewed edible films from polysaccharides. Consumable films and coatings have gotten significant consideration lately as a result of their favorable circumstances including use as edible packaging materials over synthetic films This could contribute to the reduction of environmental pollution. By functioning as barriers, such edible films and coatings can feasibly reduce the complexity and thus improve the recyclability of packaging materials, compared to the more traditional non-environmental friendly packaging materials and may be able to substitute such synthetic polymer films. New materials have been developed and characterized by scientists, many from abundant natural sources that have traditionally been regarded as waste materials.

Jimenez *et al.* (2012) reviewed edible and biodegradable starch films. Mainly due to environmental aims, petroleum based plastics are being replaced by natural polymers. The impacts of different components included throwing techniques and thermoplastic processes have been likewise broke down. These impacts can be restrained by adding different polymers to the starch framework. Other approaches to improve the starch films' properties are the reinforcement by adding organic or inorganic fillers starch matrix as well as the addition of functional compounds. In this way starch films have improved mechanical and barrier properties and can act as a bioactive packaging material. Physicochemical properties of the starch films demonstrated a great fluctuation relying upon the mixes added to the matrix and the preparing strategy.

Ezeoha and Ezenwanne (2013) studied production of biodegradable plastic packaging film from cassava starch. In this work, a biodegradable plastic film was produced by blending cassava starch and a synthetic biodegradable polymer (PVA). Blend of 1kg powdered cassava starch, 2kg polyvinyl liquor fluid, 100g powder, and 100g urea was required. The subsequent blend was added to 400ml of glycerine. The film delivered was found to have a biodegradability of 41.27% contrasted with 10.33and 85.99% for polythene and paper separately. The film additionally has an

elasticity of 24.87 N/mm^2 compared to 10.86 and 8.29 N/mm^2 for polythene and paper. The capability of creating biodegradable bundling plastic film from cassava starch is empowering and ought to be additionally investigated particularly in a nation with high cassava profitability, similar to Nigeria.

Farahnaky *et al.* (2013) studied effect of glycerol on physical and mechanical properties of wheat starch edible films. Edible films were produced using native wheat starch with different concentrations of glycerol (0, 20, 30, 40 and 50% of starch dry weight basis). Starch films were prepared by casting after gelatinization. The impacts of glycerol on the microstructure, crystallinity, solubility in water, moisture absorption, water vapor permeability, optical and mechanical properties of the films at 25 °C and relative humidity range of 11 to 84% were investigated. The increase of glycerol content led to increase in film solubility, lightness, more compact structures and water absorption at 25 °C due to the higher levels of plasticizer increased the moisture affinity and these results could be attributed to the hydrophilicity of the plasticizers, with accessible hydroxyl groups capable to interact with water by hydrogen bonds. Overall, starch-glycerol films showed higher ability to absorb water at all concentrations and relative humilities. The lowest water vaporpermeabilities were found for the films with 20 and 30% glycerol. Glycerol did not change X-ray patterns of starch films; however, the degree of crystallinity reduced. In general, for all starch films stress at break and Young's modulus decreased and elongation increased when glycerol concentration or relative humidity increased.

Ghasemlou *et al.* (2013) determined physical, mechanical and barrier properties of corn starch films incorporated with plant essential oils. Corn starch-based films are inalienably fragile and lack the necessary mechanical integrity for conventional packaging. In any case, the joining of added substances can possibly improve the mechanical properties and processability of starch films. In this work two essential oils, Zataria multiflora Boiss (ZEO) or Menthapulegium (MEO) at three levels (1, 2 and 3% (v/v)), were incorporated into starch films using a solution casting method to improve the mechanical and water vapor permeability (WVP) properties and to impart antimicrobial activity due to increase the glycerol content, water vapour permeability of film was increased due to hydrophobic or hydrophilic nature of biopolymers and presence of voids in their structure have a considerable influence on the WVP of resulting films. Increasing the content of ZEO or MEO from 2 to 3%

(v/v) increased values for elongation at break from 94.38% to 162.45 % and from 53.34 to 107.71% respectively, but did not significantly change tensile strength values of the films. The WVP properties of the films decreased from 7.79 to 3.37 or 3.19 g mm/m^2 d kPaafter 3% (v/v) ZEO or MEO incorporation respectively. The oxygen barrier properties were unaffected at the 1and 2% (v/v) oil concentration used but oxygen transmission increased with 3% (v/v) for both formulations. Films' color became slightly yellow as the levels of ZEO or MEO were increased although transparency was maintained. Both films demonstrated antimicrobial activity with films containing ZEO more effective against Escherichia coli and Staphylococcus aureus than those containing MEO. These results suggest that ZEO and MEO have the potential to be directly incorporated into corn starch to prepare antimicrobial biodegradable films for various food packaging applications.

Joshi *et al.* (2013) noted that compared to corn and potato starches, lentil starch (30% amylose) possesses a strong gel-forming tendency at a relatively low concentration.Whatever the herbal cause, starch shows a few impediments, for example, solid hydrophilic character (water affectability), which make it unsuitable for certain applications.

Kibar and Us (2013) studied thermal, mechanical and water adsorption properties of corn starch–carboxymethyl cellulose/methylcellulose biodegradable films. The effect of the addition of methylcellulose and carboxymethylcellulose on the thermal, mechanical and water adsorption properties of starch-based films plasticized with glycerol or polyethylene glycol (PEG). Mechanical tests showed that as the methylcellulose and carboxymethyl cellulose proportion increased, starch films became more resistant to break, resulting in higher TS values. As a result, it was suggested that PEG was not as compatible as glycerol with the composite polysaccharide matrix and plasticizer type was the main factor that shaped the thermal profiles of the film samples. Water adsorption isotherm data showed that samples displayed nonlinear sorption profile which is typical for hydrophilic films. In all films tested, equilibrium moisture contents, increased almost linearly up to a a_w of 0.65–0.85

Shekarabi *et al.* (2014) reported effect of glycerol concentration on physical properties of composite edible films prepared from plums gum and carboxy methyl cellulose. Edible film plasticized with glycerol was prepared from plums gum and

carboxy methyl cellulose. The mechanical, gas permeability and thermal properties of blends films Incorporated with four levels of glycerol (5, 10, 15 and 20% w/w) as plasticizer were resolved. The point of this examination was to all the more likely comprehend the chance of getting ready composite edible film based on plums gum, carboxy methyl cellulose (CPG) and glycerol and impact of glycerol substance on the conduct of the physical properties of composite films. Water vapor permeability of the films was found to decrease as the glycerol content increased from 5 to 20% w/w in the formulation, resulted in improvement of films flexibility and significantly lower tensile strength and higher elongation at break. The results of the present study demonstrated the benefit of using plums gum as a natural gum to prepare edible films.

Goswami *et al.* (2015) prepared bio plastics from organic waste. Plastics form an integral part of modern society. The extensive use of this plastic has created a problem world over. Plastic waste is increasing every year and Environmental awareness has driven the development of new biodegradable materials. Tons of potato chips are being consumed every day in the world. Made under different brands potato chips are being prepared at a very high scale, leaving behind the potato peels 10-30% of the weight of potatoes consumed. These peels can be changed over to bio plastic subsequent to separating starch from them in this manner sparing the issue of their removal after the chips have been readied and simultaneously some useable material is made out of it. In the wake of extricating starch the dried starch was blended in with multiple times its weight of distilled water. Subsequently hydrochloric acid, distilled water and 50% glycerol solution is mixed separately and then added to above starch solution. Then this mixture was heated to 200-210 °C with continuous stirring. At last this mass was spread uniformly on aluminium foil to make bio plastic. At that point measure the degradability of plastic both paper and potato strip bio plastic were covered under the dirt at a similar profundity having same states of pH, temperature, moistness, natural issue. These samples were weighed every alternate day for their degradation. Results shows that plastic prepared from potato peel can degrade at a comparable rate with paper.

Muhammed *et al.* (2015) studied effect of plasticizer type and concentration on tensile, thermal and barrier properties of biodegradable films based on sugar palm (*Arenga pinnata*) starch. The effect of different plasticizer types (glycerol (G), sorbitol (S) and glycerol-sorbitol (GS) combination) with varying concentrations (0,

15, 30 and 45, w/w %) on the tensile, thermal and barrier properties of sugar palm starch (SPS) films was evaluated. Regardless of plasticizer types, the tensile strength of plasticized SPS films decreased, whereas their elongation at break (E%) increased as the plasticizer concentrations were raised. However, the E% for G and GS-plasticized films significantly decreased at a higher plasticizer concentration (45% w/w) due to the anti-plasticization effect of plasticizers. Change in plasticizer concentration showed an insignificant effect on the thermal properties of S-plasticized films. The glass transition temperature of SPS films slightly decreased as the plasticizer concentration increased from 15% to 45%. The plasticized films exhibited increased water vapor permeability values from 4.855×10^{-10} to 8.70×10^{-10} $g \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$, irrespective of plasticizer types. This could be related to the strong starch–starch molecule interactions which are dominant at lower plasticizer concentrations, resulting in a dense and more compact starch network and structure. Further increments of plasticizer concentration to promote the mobility and flexibility of starch network chains due to the structural modification of the starch–starch molecular interaction to a looser network. Consequently, the film matrices became less dense and their WVP values eventually increase.

Omotoso *et al.* (2015) prepared biodegradable starch film from cassava, corn, potato and yam. Starch films were set up from starch separated from cassava, corn, potato and yam with and without plasticizers to assess the impacts of the sort and amount of plasticizer on the mechanical properties of the starch films got. The blend was warmed gradually on hot plate to delicate overflowing with persistent mixing for 10minutes. 0.1M sodium hydroxide solution was added drop-wise to the hot product to neutralize the added acid. The above procedures were repeated with different mass percent of glycerol (22, 36, 46, 53 and 59%) and different mass percent of sucrose (22, 36, 46, 53 and 59%) for four types of starch used. Ubbelohde viscometer was used to determine the intrinsic viscosity and molecular weight of the prepared films in dimethyl sulfoxide (DMSO)/water solution (90/10% v/v). The molecular weights of the films were found to range from 2.70×10^{13} - 2.14×10^{17} for films prepared from corn starch, 2.86×10^{10}- 1.01×10^{17} for films prepared from yam starch, 2.69×10^{13} - 3.97×10^{17} for films prepared from potato starch and 4.15×10^{6} - 4.05×10^{8} for films prepared from cassava starch. The molecular weights of the films were found to decrease with increase in the plasticizer content because the bonds between starch-

starch molecules were replaced with the bond between starch-water molecules. This led to the reduction in the chain length of both the amylopectin and amylose, and hence reduction in the molecular weight average. Amylose and amylopectin molecules contribute to mechanical properties and hygroscopic properties of starch. The effects of plasticizer on mechanical properties film prepared from corn starch and yam starch were investigated at six levels of plasticizer content. The tensile strength of the films prepared from corn starch ranged from 0.10 to 5.20MPa, 0.05 to 5.10MPa for films prepared from yam starch, 0.09 to 5.74MPa for films prepared from potato starch and 0.51 to 5.33MPa for films prepared from cassava starch. The value of Young's modulus was found to range from 0.39 to 104.06 MPa for films prepared from corn starch, 0.33 to 49.32MPa for films prepared from yam starch, 0.36 to 191.33 MPa and 3.14 to 97.5MPa for films prepared from cassava starch. The elasticity of movies was impacted by the sort and the substance of plasticizer utilized. By and large, the Young's modulus and the rigidity of the movies diminished with increment in plasticizers content, with simultaneous increment in lengthening at break and tractable strain of the movies because of increment in malleability of the movies. Glycerol exhibited more plasticizing effect on both starches than sucrose due to the hygroscopic or hydrophilic nature of glycerol.

Nguyen and Lumdubwong (2016) reported starch behaviours and mechanical properties of starch blend films with different plasticizers. Mechanical properties and starch behaviours of cassava (CS)/and mungbean (MB) (50/50, w/w) starch blend films containing glycerol or sorbitol at 33% weight content were investigated. It was found that tensile strength TS and elongation of the Glycerol-CSMB films were similar to those of MB films; but elongation of all Sorbitol-films was identical. TS of plasticized films increased when Amylose content and crystallinity increased. When Sorbitol was substituted for Glycerol, crystallinity of starch films and their TS increased. The CSMB and MB films had somewhat a similar molecular profile and comparable mechanical properties. Therefore, it was proposed the starch molecular profile containing amylopectin with high Molecular weight (Mw) low Mw of amylose, and the small size of intermediates may impart the high TS and elongation of starch films.

Sonam and Aditya (2016) studied the development of biodegradable packaging film using potato starch. This examination work manages the chance of

extraction of starch from the potato and joining this starch to make biodegradable packaging film utilizing glycerol as a plasticizer. This work report manages the procedure of readiness of film by casting methods and the after effects of tests which were performed to the mechanical property of the film. The test included tensile strength of the film, puncture strength of the film, moisture permeability of film, water absorbed by the film and elongation at break. The films were formed by making a filmogenic solution of potato starch (7.5gm, 11.25gm), glycerol (1.5ml, 2ml) and citric acid (1 gm each). Two samples of films were made i.e. sample 1 (7.5 gm starch,1.5ml glycerol and 1gm citric acid),sample 2 (11.25 gm starch, 2ml glycerol and 1gm citric acid).The consequence of this investigation showed that the film made by sample 1 were having good elastic and cut quality contrasted with sample 2. While considering other parameters in combination with tensile and puncture strength such as moisture permeability, water absorbed by film and elongation at break the results showed that the film made by sample 1 showed better results than sample 2 due to the films became more extendible when the concentration of plasticizer was increased which results in the reduction of puncture strength film. The reduction of the puncture force was consequences of the incorporation of plasticizers, and to water molecules absorbed by the samples. The sample 1 was found more appropriate in enhancing the shelf life of the food as it showed lesser amount of moisture permeability and water absorption. Both the samples were used to test the performance evaluation of film by packing chikki in it. Moisture content, ash content and total plate count method were used to perform microbial and physio- chemical analysis of chikki. The results of moisture content, ash content and total plate count of sample 1 were more suitable and acceptable in comparison to sample2.The films was found appropriate and suitable for packaging of chikki and similar bakery products.

Sujuthi and Liew (2016) studied properties of bioplastic sheets made from different types of starch incorporated with recycled newspaper pulp. The use of biodegradable material based on natural polysaccharides, particularly starch helps to reduce the usage of non-degradable materials. In this study, three types of starch were used to produce the bioplastic sheets (cassava, corn and potato). The sheets were produced with the mixture of bioplastic (B) incorporated with recycled newspaper pulp fibre (N) at four different ratios (newspaper pulp fibres: bioplastic) N50%:B50%, N30%:B70%, N10%:B90% and N0%:B100%. Water absorption and tensile

properties were investigated for these bioplastic sheets which were done in room temperature. Cassava-based bioplastic sheet had the worst water repellent while corn starch-based bioplastic sheets had the lowest water absorption percentage. Based on the ratios, bioplastic sheet N30%: B70% shows the lowest percentage of water absorption. Result also showed that as the amount of bioplastic ratio increase, the tensile strength decrease. The optimum mixture of fibres/bioplastic was N50%:B50% which obtained highest percentage of tensile strength. Elongation at break was increased as the bioplastic increased.

Basiaka *et al.* (2017) studied the effect of starch type on the physico-chemical properties of edible films. Physical and chemical properties were assessed and reflect the role of the starch type (wheat, corn or potato) and thus that of the amylose/amylopectin ratio, which influences thickness, colour, moisture, wettability, thermal, surface and mechanical properties.Wheat (25% amylose), corn (27% amylose) and potato (20% amylose) starches three sample was taken. 5 g of starch powder (wet basis) in 100 ml of distilled water. The solutions were heated in a water bath at 85 °C for 30 min under 700 rpm stirring to obtain complete solubilisation and gelatinization of starch. Then, film-forming solutions were cooled down to 40 °C. The plasticizer was added at a weight ratio of 0.3:1 glycerol: starch under stirring at 150 rpm. A defined volume of film-forming solution was poured into a Petri dish and films were dried at 25 °C for 48 h in a climatic chamber. Thickness of film in range of 74 to 139 μm. Higher amylose content in films induces higher moisture sensitivity, and thus affects the mechanical and barrier properties. Films made from potato starch constitute a greater barrier for oxygen and water vapour though they have weaker mechanical properties than wheat and corn starch films. Starch species with higher amylose content have lower wettability properties, and better mechanical obstruction, which emphatically relies upon the water content because of the hydrophilic idea of starch films, so they could be utilized for items with higher water movement, for example, cheddar, foods grown from the ground. The starch starting point impacts optical properties and thickness: with more amylose, films are opalescent and thicker; with less, they are straightforward and more slender.

Eterigho *et al*. (2017) investigated the physical properties and biodegradability of potato-starch based plastics. This research work focused on the synthesis and characterization of potato starch based plastics (biodegradable) using polyvinyl

alcohol (PVA) as cross linker. PVA was varied in mass ratios of 15, 30, 45 and 80% in the thermoplastic starch (TPS)/PVA blend. Mechanical properties (such as tensile strength, percentage elongation, young modulus) and specific gravity of the blends were studied. The results showed that 80% PVA plastic had the highest tensile strength, elongation and lowest young modulus of 384.47 kPa, 347.27 % and 310.10 kPa respectively. The specific gravity of the whole blends was 1.2. The elongation at break increases with increasing concentration of PVA, having the least value of 0% and highest value of 481.82% for 15 % and 80 % PVA plastic respectively. In addition, the results obtained showed increase in the values of the properties of the samples with respect to thermal conductivity, acid, base and water resistance with increase in composition of PVA. Biodegradability test was done via soil-burial method and the PVA/TPS blend was noted to be biodegradable.

Thakur *et al.* (2017) used response surface methodology (RSM) to optimize pea starch-chitosan novel edible film formulation. Three independent variables were assigned comprising chitosan (1-2%), pea starch (0.5-1.5%) and glycerol (0.5-1%) to design an empirical model best fit in physical, mechanical and barrier attributes. Impacts of independent variables on thickness, moisture content (MC), solubility, tensile strength (TS), elastic modulus (EM), elongation at break (EB) and water vapor permeability (WVP) of films were evaluated. All the parameters were found to have significant effects ($p<0.05$) on physical and mechanical properties of film. The optimal formulation for preparation of edible film from chitosan, pea starch and glycerol was 1% chitosan, 1.5% pea starch and 0.5% glycerol. An edible film with good physical and mechanical properties can be prepared with this formulation and thus this formulation can be further applied for testing on coating for fruit and vegetables.

Xu *et al.* (2017) studied preparation of starch-based biodegradable film and the application in agriculture. The film was prepared with polyethylene grafted and blended by epichlorohydrin modified-corn starch, linear low density polyethylene, low density polyethylene and maleic anhydride. Epoxy soybean oil was served as plasticizer and a small amount of ethylene propylene diene monomer was used as compatibilizer. 0.012 mm thickness degradable films were prepared with various blow molding methods. FTIR was used to characterize the modified starch and SEM was used to observe the morphology of the film before and after the modification. The

outcomes indicated that the film could keep up a fantastic presentation on mechanical properties and corruption until the substance of altered starch expanded to 70% and have a decent future in horticultural application.

Keziah *et al.* (2018) prepared biodegradable plastic production from corn starch. Bioplastic was produced by using the mixture of 0.5 to 1.5 g glycerol, 1.5 g corn starch, 1 ml vinegar, and 1–2 drops of food color. The mixture was heated to give a good yield. Bioplastics or in other words green plastics are obtained in 2 days.

Khairunnisa *et al.* (2018) studied the effect of glycerol concentration as a plasticizer on edible films made from alginate towards its physical characteristic. The effects of the addition of the best glycerol concentration as a plasticizer on edible film alginate for tensile strength test, thickness test, percent elongation, and transparency test. The research method used was experimental with a Completely Randomized Design consisting of five treatments and three replications. The treatment of the addition of glycerol concentration was 0.3% based on the volume of solution, 0.5% based on the volume of solution, 0.7% based on the volume of solution, 0.9% based on the volume of the solution, and 1.1% based on the volume of the solution. Observations on the physical characteristics of edible films include thickness, tensile strength, percent elongation, and transparency. The results of the study of physical characteristics of thickness, tensile strength, percent elongation, and transparency concluded that the addition of glycerol concentration in edible films as much as 0.9% was the best treatment based on Japanesse Industrial Standard (JIS) with an average value of 0.094 mm thickness, tensile strength 8.25 MPa, elongation percent value 10.83%, and transparency value is 1.86. The result was found that increase in the addition of glycerol percentage caused the transparency value of the film to increase as well. The greater the concentration of glycerol in making edible film was reduce the brightness of edible films. The higher the concentration of glycerol was cause the color of the edible film to become blurred so that the brightness level decreases. This is related to the increase in the amount of solids by glycerol which is greater which causes the thickness of edible films to increase. The higher the thickness value of the edible film will increase the diffusion of light so that the edible film object will appear more turbid and the brightness will be lower. The thicker the edible film will give a color that is not transparent and looks less attractive. This is due to the increase in thickness which will reduce its translucency because the transparency is reduced.

Jangong *et al.* (2019) studied fabrication and characterization starch/chitosan reinforced polypropylene as biodegradable. The production of bioplastic from starch/chitosan reinforced polypropylene with different ratio from 35/65, 50/50 and 65/35. Bioplastic was investigated by using tensile strength test, X-Ray diffraction (XRD), and Fourier transform infra-red (FTIR) spectroscopy, respectively. XRD analysis shows that the sample have amorphous phase structure with the main broad peaks 18^{o} to 30^{o}. FT-IR used to investigate functional group and the result analysis show that the main bonding is of O-H hydrogen bonds (carboxylic acid), CH alkanes, C=C alkenes and C-O alcohols. The tensile strength obtained for bioplastic were 68.41Mpa at ratio 65/35, respectively. These bio plastics have exhibited mechanical properties with high biodegradability that makes them a suitable alternative for the existing conventional plastics.

Mayra Sapper *et al.* (2019) reported improving functional properties of cassava starch-based films by incorporating xanthan, gellan, or pullulan gums. Starch (2% w/w) was dispersed in distilled water at 95°C for 30 min and glycerol was added to the aqueous gelatinized starch at 0.25 g/g of starch. Aqueous solutions of pullulan (2% w/w), xanthan (1% w/w), and gellan (2% w/w) gums were prepared in distilled water and heated while magnetically stirred (400 rpm) at 90 °C for 60 min until the complete dissolution. The tensile properties, barrier capacity to water vapour, and oxygen and water sorption isotherms of the samples were analysed.The mix of starch with gellan gum was powerful to diminish the dampness sorption limit of starch films while lessening water fume penetrability, improving the film quality and protection from break and safeguarding films against starch retrogradation all through the capacity time. Xanthan gum improved the tensile behaviour of the starch films, but did not reduce their water sorption capacity and water vapour permeability. Pullulan did not notably improve the functional properties of the starch films. Gellan gum at 10 and 20 % in the blend could be used to obtain starch films with more adequate properties for food packaging purposes.

Buso-Rios *et al.* (2020) studied the effect of the concentration of starch and clove essential oil on the physicochemical properties of biodegradable films. Biodegradable films were obtained with the casting method using different concentrations of purple sweet potato starch and clove essential oil. Mechanical properties (tensile strength, percentage of elongation and Young's modulus), water

vapor permeability and film adsorption isotherms were evaluated. The statistical analysis showed a significant difference in the mechanical properties of the composite films ($p < 0.05$). The GAB model was used to adjust the experimental data of the adsorption isotherms and a higher moisture content of the films containing the highest concentration of starch and essential oil was observed. The (starch 3.5%, clove essential oil 60 mg/L) formulation showed the lowest water vapor permeability values ($1x10^{-8}$ g/ m s Pa) and the lowest moisture content in the monolayer, which is considered to be the material with the best characteristics for its possible application, avoiding the excessive use of raw material during its preparation.

CHAPTER – III

MATERIALS AND METHODS

This chapter deals with the selection of raw materials, method of developing starch based biodegradable plastic film, procedures followed for determination of physical properties (water absorption and water solubility index) of starch (potato, corn & rice) powder and physical properties (thickness), biochemical properties (moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology), mechanical properties (tensile and puncture strength) and biodegradation properties (reduction in weight) of developed starch based biodegradable plastic film and process flow chart for formation of starch based biodegradable plastic film. The details of machineries, instruments and materials used in the experiments are also described hereunder.

3.1 Location

Investigation on "Development of starch based biodegradable plastic" was carried out in the Processing and Food Engineering Department, College of Agricultural Engineering and Technology, Junagadh and Department of Biochemistry, College of Agriculture, Junagadh in the year 2019-20.The procedure used during the experimentation, methodology adopted and the instrumentations are reported in the chapter.

3.2 Materials required

Potato, corn and rice starch powder (individual), glycerol, acetic acid shown in Plate 3.1 and all other chemicals in analytical grade were procured.

3.3 Development of film casting frame

For the formation of biodegradable plastic film, casting frame is necessary. The 5 mm thick and glossy glass was used to make casting frame (Plate 3.2(A)). The size of casting film was decided as 50x 33 x5 cm based on required bag size. Another small frames dimension was 38x23x5 cm. The frame had made border with 20 mm wide glass strip to prevent spreading outside of the glass and confined within required space. The surface of glass was kept very smooth and plain to make ease in peeling of plastic film. The dimension of casting frame was decided based the dimension of bag.

(A) Potato starch

(B) Corn starch

(C) Rice starch

(D) Glycerol

(E) Acetic acid

Plate 3.1: Material required for development of starch based biodegradable plastic

3.4 Physical properties of starch (Potato, Corn & Rice) powder

The physical properties of starch (Potato, Corn & Rice) powder, viz., WAI (Water Absorption Index) and WSI (Water Solubility Index) were measured with standard methods and instruments.

3.4.1 WAI (Water Absorption Index)

Water absorption index was determined by using method stated by Anderson and Griffin (1969). One gram of sample was dispersed in 10 ml of distilled water in 15 ml measuring cylinder. Solution was centrifuged at 3000 rpm for 30 minutes as shown in Plate 3.2(B). Volume of supernatant was measured accurately. Water absorption index was then expressed as number of grams of water held by 1 g of starch.

$$\text{Water absorption index} = \frac{\text{Grams of water held by starch powder}}{\text{Weight of starch powder}} \times 100$$

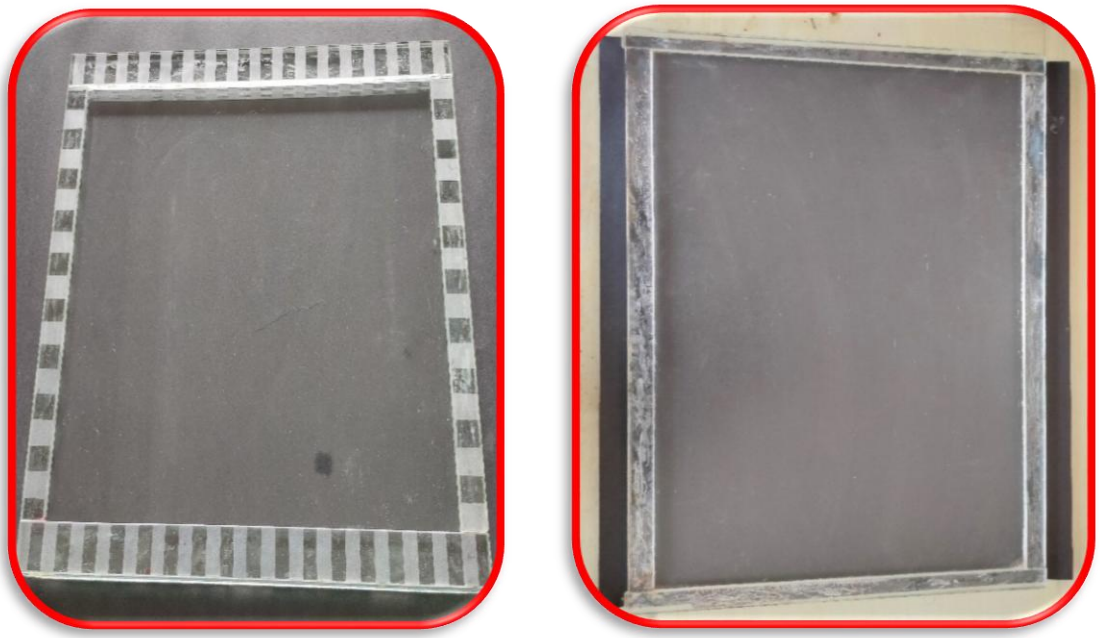
--------(3.1)

3.4.2 WSI (Water Solubility Index)

Water solubility index was determined by the method of Anderson and Griffin (1969). One gram of isolate was suspended in 10 ml of distilled water in a tare centrifuge tube. Isolate was allowed to stand for 30 minutes and then centrifuged at 3000 rpm for 10 minutes as shown in Plate 3.2(B). The water solubility index was then calculated by using supernatant by using formula,

$$\text{Water solubility index} = \frac{\text{Weight of dissolved powder in supernatant}}{\text{Weight of starch powder taken}} \times 100$$

--------(3.2)



(A) Developed film casting frame

(B) Measurement of WAI and WSI

Plate 3.2: Developed film casting frame and Measurement of WAI and WSI of starch

3.5 Experimental procedure

The process of development of starch based biodegradable plastic was carried out in the Department of Processing and Food Engineering, College of Agricultural Engineering and Technology, JAU, Junagadh. The sequence of different processing operations carried out during development of starch based biodegradable plastic as shown in Fig. 3.1 and Plate 3.3.

3.5.1 Procedure for development of starch based biodegradable plastic film

The films were prepared by casting technique using a film-forming solution containing potato, corn and rice starch individual. Glycerol was used as plasticizer. Starch concentration (Potato, Corn and Rice starch individual) (5, 6.5, 8, 9.5 & 11 % (W/V)) and Glycerol concentration (0.5, 0.875, 1.25. 1.625 & 2 % (V/V)) was taken as variable parameter.

Starch was dispersed in distilled water in order to obtain polysaccharide suspensions. The mixture of dry starch and water was taken in a beaker. Accordingly, the dispersion was mixed with glycerol as a plasticizer. Starch and glycerol were dissolved in distilled water with continuous stirring to obtain a film-forming solution. Further addition of 1 ml of acetic acid in film forming solution. To achieve complete dispersion, the mixture was heating with stirring on magnetic stirrer (REMI EQUIPMENTS) at 40°C for 5 minutes.

This suspension was transferred to a water bath (Nova Digital Constant Temperature Water Bath, NV 8550 E) at 85°C temperature for 15 minutes and continuous agitated by glass road. Gelatinization of starch starts at 60 °C and make a film forming solution. Now a cast was prepared and the entire solution was poured on the cast and was left for drying at room temp for 24 hrs. After drying the films were peeled off. Peeling of film was done very carefully to prevent film from brake and crake. Films were kept in poly bags away from moisture.

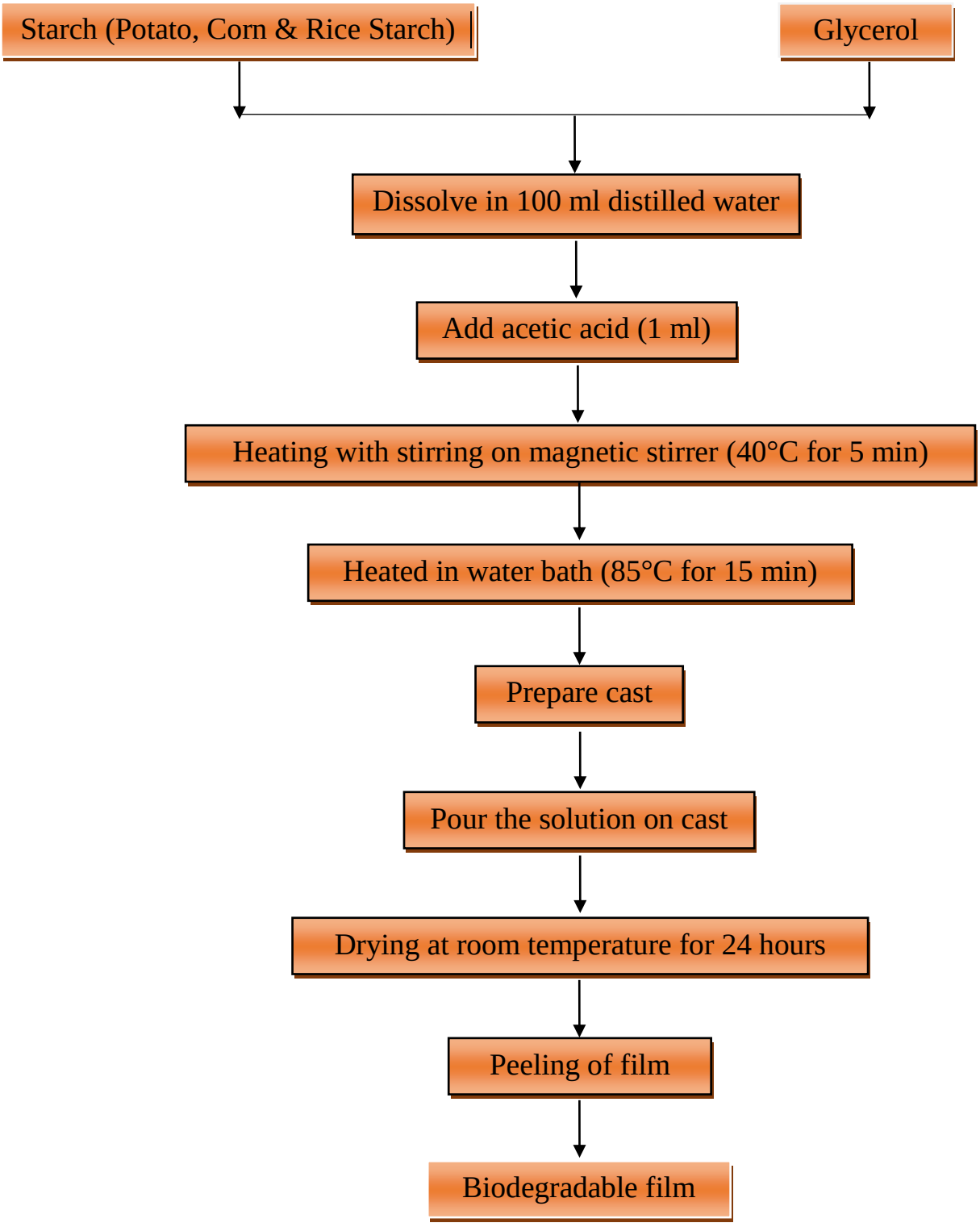


Fig. 3.1 Process flow chart for formation of starch based biodegradable film

Plate 3.3: Pictorial process flow chart for development of biodegradable plastic

3.5.2 Treatment details

3.5.2.1 Independent variable

1 First factor **:** Starch Concentration (Potato, Corn and Rice starch individual)
(5, 6.5, 8, 9.5 & 11 % (W/V))

2 Second factor **:** Glycerol Concentration
(0.5, 0.875, 1.25. 1.625 & 2 % (V/V))

3 Experimental design **:** Central Composite Rotatable Design (Response Surface Methodology)

3.5.2.2 Dependent variables

1. Moisture content, %
2. Tensile strength, MPa
3. Puncture strength, MPa
4. Water absorption capacity, %
5. Water vapour permeability, g.mm/m^2dayKPa
6. Transparency, %

Table 3.1: Treatments combinations

Run	Starch Concentration, % (W/V)	Glycerol Concentration, % (V/V)
1	8.0	1.250
2	8.0	1.250
3	8.0	1.250
4	9.5	1.625
5	5.0	1.250
6	6.5	0.875
7	9.5	0.875
8	8.0	1.250
9	11.0	1.250
10	8.0	1.250
11	8.0	1.250
12	8.0	2.000
13	6.5	1.625
14	8.0	0.500

3.6 Observations recorded

Physical properties (water absorption and water solubility index) of starch (potato, corn & rice) powder were measured as per the following standard methods. Physical properties (thickness), physico-chemical properties (moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology), mechanical properties (tensile and puncture strength) and biodegradation properties (reduction in weight) of developed starch based biodegradable plastic films were measured as per the following standard methods.

Table 3.2: Observations to be taken:

Sr. No.	Observation	Method/ Reference
Physical properties of starch (Potato, Corn & Rice) powder		
1	Water absorption index, %	Anderson and Griffin (1969)
2	Water solubility index, %	Anderson and Griffin (1969)
Physical Properties of developed biodegradable plastic film		
1	Thickness, mm	By using digital Vernier calliper
Physico-chemical properties of developed biodegradable plastic film		
1	Moisture content, %	AOAC (2005)
2	Transparency, %	By using Spectrophotometer
3	Water absorption capacity, %	By using ASTM D570-98 (1998) method
4	Water vapour permeability, g.mm/m^2dayKPa	By using ASTM E96-95 (1995) method
5	Surface morphology	By using scanning electron microscope
Mechanical properties of developed biodegradable plastic film		
1	Tensile strength, MPa	By using texture analyser
2	Puncture strength, MPa	By using texture analyser
Biodegradation properties of developed biodegradable plastic film		
1	Reduction in weight, %	$\frac{W_o - W_t}{W_o} \times 100$ (Azahari *et al.*, (2011))

3.7 Observations recorded for developed biodegradable plastic film

The different physical and physico-chemical properties of developed biodegradable packaging film were measured, determined and estimated with standard analytical methods described in following sub-sections.

3.7.1 Physical properties of developed biodegradable plastic film

The physical properties, viz., thickness of developed biodegradable packaging film were measured with standard methods and instruments.

3.7.1.1 Film Thickness

Film thickness was measured with the help of digital Vernier Caliper (Mitutoyo corporation, Japan made, model- CD-12"), having a least count of 0.01mm as shown in Plate 3.4 (A). Measurements were carried out at different film locations and the mean thickness value was used to calculate the thickness of the films. The observations were recorded in mm.

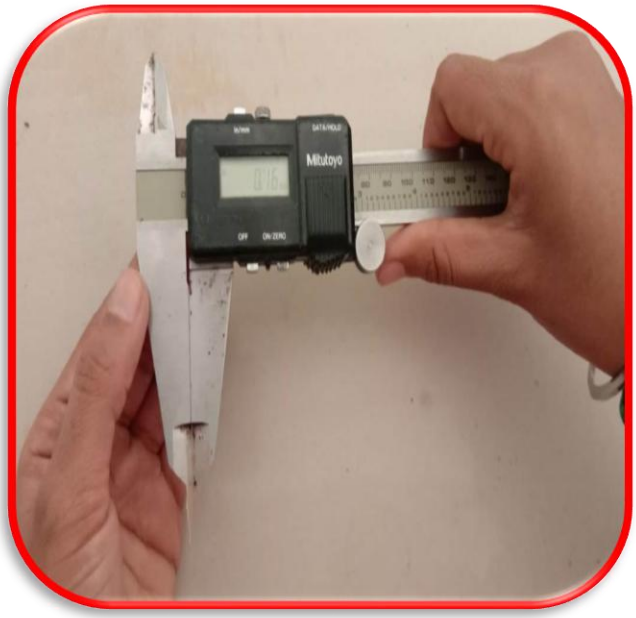
3.7.2 Physico-chemical properties of developed biodegradable plastic film

The physico-chemical properties viz., moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology of developed biodegradable packaging film were determined using standard analytical methods as described in following sub-sections.

3.7.2.1 Moisture content

The moisture content of film was measured by hot air oven method (Plate 3.4(B)) as suggested by AOAC (2005). Film samples were trimmed into small strips. The samples in a petri dish were placed inside the hot air oven (Scientronic-instruments, SON 45) at 100 ± 1 °C till it attains constant weight. The samples were cooled in a desiccator and weighed. The difference in the initial and the final weights of the sample was taken as the weight of water removed and the moisture content was determined using following formula. Moisture content of film was expressed in percent wet basis.

$$\text{Moisture content, \% (wb)} = \frac{\text{Weight of water removed (g)}}{\text{Weight of sample taken (g)}} \times 100 \quad \text{---------- (3.3)}$$



(A) Digital vernier caliper for measuring film thickness

(B) Hot air oven used for determination of moisture content

Plate 3.4: Measurement of thickness and moisture content of biodegradable film

3.7.2.2 Transparency

The transparency of films was determined using a UV-VIS spectrophotometer (GNESYS 50, Thermo Fisher Scientific). The film samples were cut into rectangles and placed on the internal side of the spectrophotometer cell (Plate 3.5 (B)).The transparency of films was determined at 600 nm as described by Han and Floros (1997).

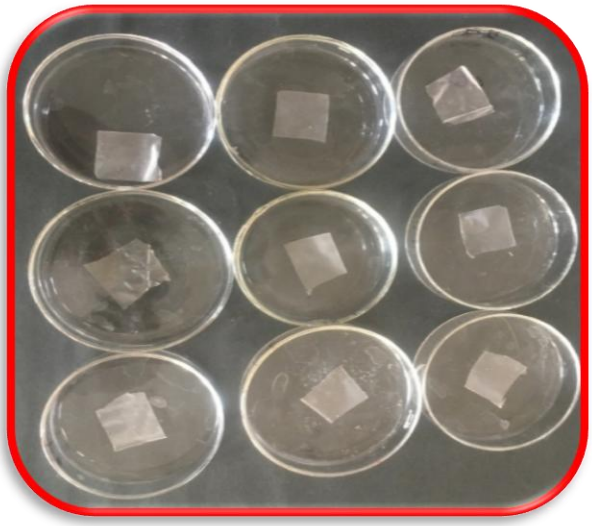
3.7.2.3 Water absorption capacity

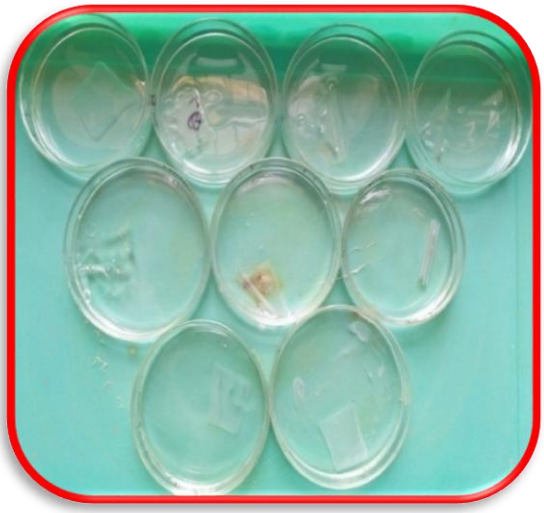
The measurement hydrophilicity of polymeric film was evaluated by measuring the water absorption capacity of the film surface (Plate 3.5 (A)). Water absorption capacity of the film was carried out according to ASTM D570 method. Film pieces 20 mm×20 mm were conditioned in a hot air oven for 2 hour at a temperature of 60 °C and weighed (W_{dry}). Dried films were kept in distilled water at room temperature for 24 hours. After that, samples were removed, dried by wiping gently by blotting paper and weighed (W_{wet}) to determine the water absorbed by the films. The water absorption capacity (W_a) was determined by:

$$\% W_a = \frac{Wwet - Wdry}{Wdry} \times 100 \qquad \text{----------(3.4)}$$

3.7.2.4 Water vapour permeability (WVP)

WVP tests of biodegradable film were carried out by ASTM method E96-95 (1995) with some modifications (Mali *et al.*, 2006). Special cups, with an average diameter of 2 cm and a depth of 4.5 cm were utilized to determine WVP of films (Plate 3.5(C)). Films were cut into discs with a diameter slightly larger than the diameter of the cup. After placing 3 g of anhydrous $CaSO_4$ in each cup, they were covered with edible films of varying composition. RH 0 was maintained using anhydrous $CaSO_4$ in the cup. Each cup was placed in a desiccators containing saturated K_2SO_4 solution in a small beaker at the bottom. A small amount of solid K_2SO_4 was left at the bottom of the saturated solution to ensure that the solution remained saturated at all times. Saturated K_2SO_4 solution in the desiccator provides a constant RH of 97% at 25 °C. The desiccator was kept in an incubator 25.0±0.1 °C. In first, cups were weighed every 2 h (along one day) and then measurement carried out every 12 h for 2 days and water vapour transport was determined by the weight gain of the cup. Changes in the





(A) Measurement of water absorption capacity

(B) Measurement of transparency by using spectrophotometer

(C) Measurement of water vapour permeability

Plate 3.5: Measurement of physico-chemical properties of biodegradable film

weight of the cup were recorded as a function of time. Slopes were calculated by linear regression (weight change vs. time). The water vapor transmission rate (WVTR) was defined as the slope (g/h) divided by the transfer area (m^2). WVP (g. $Pa^{-1}h^{-1}m^{-1}$) was calculated as:

$$WVP = \frac{WVTR}{P(R1- R2)}X$$

---------- (3.5)

Where, P is the saturation vapor pressure of water (Pa) at the test temperature (25°C), R_1 is the RH in the desiccator, R_2, the RH in the cup and X is the film thickness (m). Under these conditions, the driving force is 3073.93 Pa. All measurements were performed in three replicates.

3.7.2.5 Surface Morphology

Morphological investigations were performed on thermoplastic starch films of corn potato and rice starch by using SEM machine model (HITACHI S-3400N). A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample SEM is a type of the electron microscope that images a sample by scanning it with a beam of electrons in a raster scan pattern. The entire film sample was mounted on the aluminium stub using graphite filled tape which was vacuum coated with platinum. Films were cryogenically frozen in liquid nitrogen. After that, the samples were manually fractured after removal from the liquid nitrogen. The fractured surfaces were vacuum-coated with gold. An emission current of 58 μA was used while operating the SEM instrument. The acceleration voltage was kept as 5 kV, and the working distance was fixed to 7.5 mm. Samples were layered with gold before the SEM analysis.

3.7.3 Mechanical properties of developed biodegradable plastic film

The mechanical properties viz., tensile strength and puncture strength of developed biodegradable packaging film were determined using standard methods and instruments as described in following sub-sections.

3.7.3.1 Tensile strength

Tensile strength measurement was used to quantify the mechanical strength of the starch films produced. Tensile strength of the starch based films were measured by the universal compression-tension testing machine (RM 50, TA.XT plus texture analyser, Stable Micro Systems, London) as shown in Plate 3.6 (A). According to the standard ISO 527, forces were applied to the samples from both sides, until a change in shape or a fracture took place.

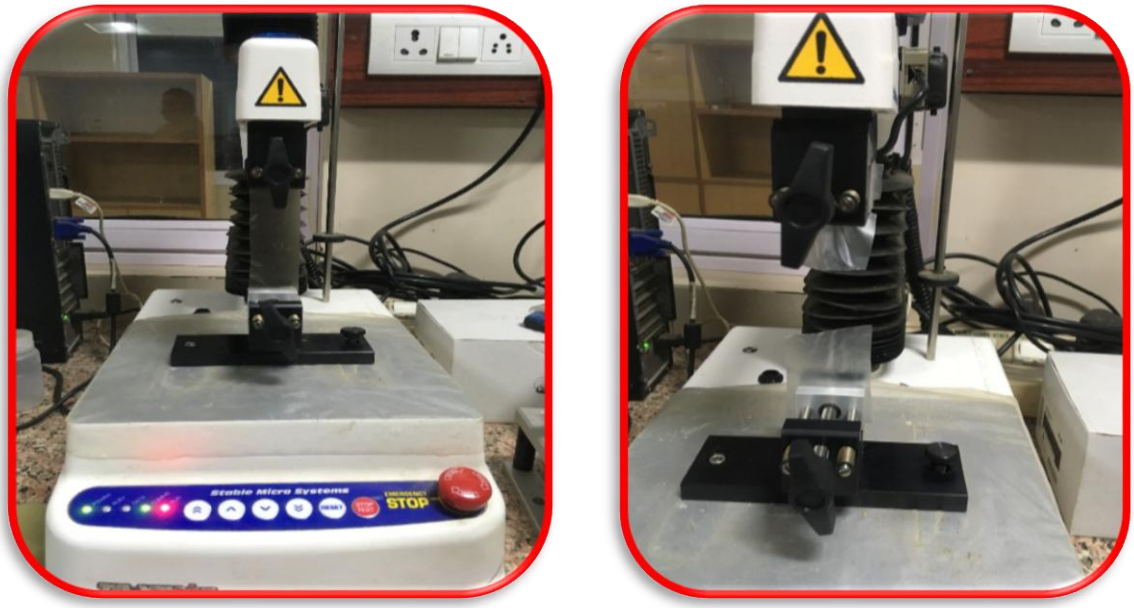
The cast films were cut into strips of 15 mm width and a length of 70 mm. Thin strip of film was cut out from each solidified film on white tile with razor blade. In order to measure the tensile properties as a function of the thickness, the thicknesses of the films were measured. The width and thickness of each strip was determined using the digital vernier callipers. The two ends of the strip were secured with two C-clamps, one end was hung on the suspended spring balance supported with support stand and long ruler. The other clamp was held and pulled gently and slowly (1N per second) to obtain a maximum force required to break the film along it cross-sectional area.

Force (N) and deformation (mm) were recorded during extension at 50 mm min^{-1} and with an initial distance between the grips of 50 mm. The parameters determined were:tensile Stress (MPa). The specimen was stretched using a testing speed of 100 mm/min.

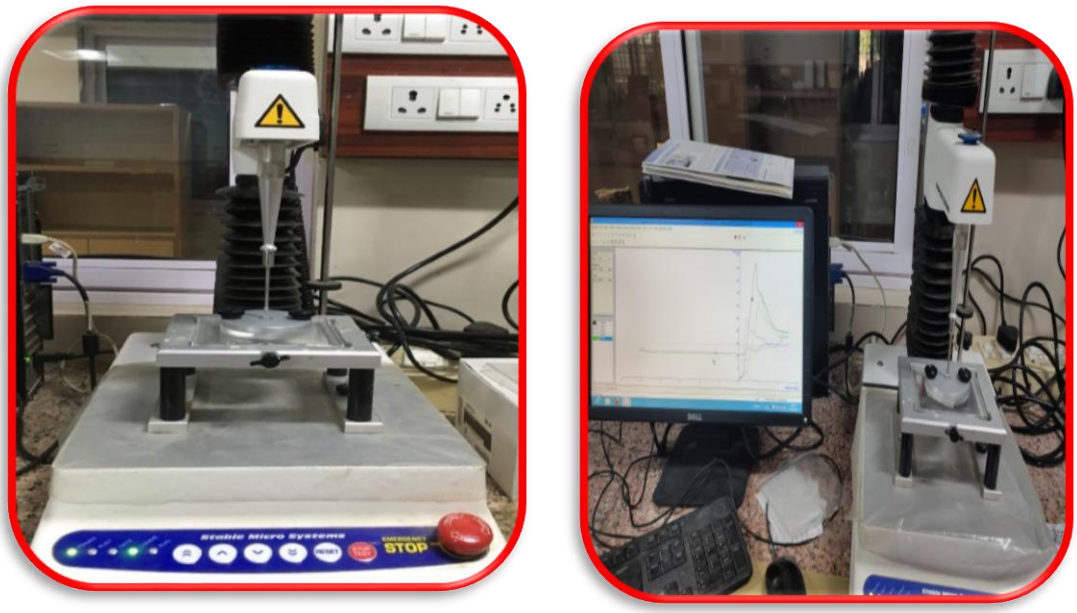
3.7.3.2 Puncture strength

The puncture test method ASTM D7192-08 covers the determination of puncture properties of plastic films over a range of test velocities. Puncture strength of the starch based films were measured by the universal compression-tension testing machine (RM 50,TA.XT plus texture analyser, Stable Micro Systems, London) as shown in Plate 3.6 (B).

The test outlined in the standard is designed to provide load versus deformation responses of plastic film under multi-axial deformation conditions at various impact velocities. By testing at various impact velocities a measure of the rate sensitivity of the film to impact events may be gathered. Recommended in the test standard are speeds of 2.5, 25, 125, 200 and 250 m/min. (0.137, 1.367, 6.835, 10.936 and 13.670 ft/s). The machine used for the test shall consist of two assemblies, one fixed, the other driven by suitable method to achieve the required impact velocity, a specimen clamping assembly with an unsupported region of 76 ± 3.0 mm.



(A) Measurement of tensile strength of biodegradable film



(B) Measurement of puncture strength of biodegradable film

Plate 3.6: Measurement of mechanical properties of biodegradable film

The film shall be held taut but not stretched so tightly as to cause damage to the specimen prior to test. The impulse data acquisition system provides the means for measuring and recording the load, displacement and velocity of the test event simultaneously. In addition values for energy at peak load and total energy absorbed.

The biodegradation properties viz., reduction in weight of developed biodegradable packaging film were determined using standard methods as described in following sub-sections.

3.7.4.1 Reduction in weight

Biodegradability test was done based on the soil burial method as reported by Azahari *et al.*, (2011). The soil was collected from the garden of college campus and treatment of soil was done in the laboratory. Samples of sizes 3 x 3 cm were taken. Weight of the samples was weighed (W_o). These samples were then buried in soil for one month as shown in Plate 3.7. The samples were buried 6.5 cm below under the soil. The moisture content of the soil was maintained at approximately 20%. In every seven days interval films were taken away from the soil. Later than clean-up through water and exposure to air at room temperature, changes in weight (W_t) were calculated. Thus, the % reduction in weight and the rate of biodegradation of each sample were calculated using Equations 3.6

$$\text{Reduction in weight, \%} = \frac{W_o - W_t}{W_o} \qquad \text{----------(3.6)}$$

Where, W_o = original weight of samples and W_t = weight of samples after degradation time.

3.8 Performance evaluation of starch based biodegradable plastic film

3.8.1 Sealing Properties of bio-plastics

Sealing properties of bioplastic observed that no single temperature is accepted for the heat-sealing process. This may occur due to the reason that the plastic could be sealed at moderately molten or melting conditions. A range of temperatures is set as an acceptable sealing temperature, in which a good seal will be formed if it is being prepared within this temperature range. The interval of time that the coated film is brought into close contact with the heated film is the dwelling time. The sealing of bio-plastic was shown in Plate 3.8.

Plate 3.7: Measurement of biodegradation properties of biodegradable film

Plate 3.8: Sealing of bioplastic

3.9 Experimental design

The Response Surface Methodology (RSM) is an empirical statistical modelling technique employed for multiple regression analysis using quantitative data obtained from properly designed experiments. The Central Composite Rotatable Design (CCRD) was used for designing the experiments using design expert 10 software (Khuri and Cornell, 1987).

CHAPTER IV

RESULTS & DISCUSSION

This chapter deals with the results and discussion of the experiments, which was carried out for development of starch based biodegradable plastic. The results obtained from various experiments conducted during investigation which is reported under different subsection as physical, physico-chemical, mechanical and biodegradation properties of starch based biodegradable films. The observation on Design (CCRD), Response Surface Methodology with two factors are presented. The variations in the parameters are presented in tables and depicted graphically. The results have been presented and interpreted under suitable headings and sub-headings. The performance evaluation and cost analysis of developed starch based biodegradable plastic film are discussed here under.

4.1 Physical properties of starch (Potato, Corn & Rice) powder

The physical properties of starch (potato, corn & rice) powder were determined on the basis of WAI (Water Absorption Index) and WSI (Water Solubility Index). Three samples of starch powder were randomly taken for the experiment. The various physical properties of starch (Potato, Corn & Rice) powder were determined as per the standard methods reported in section 3.4 of Chapter III.

4.1.1 WAI (Water Absorption Index)

Water absorption index of potato, corn and rice starch powder powder with their standard deviation was found as 139 ± 1.53%, 155 ± 2 % and 130 ± 2.51% respectively (Table 4.1).

4.1.2 WSI (Water Solubility Index)

Water solubility index of potato, corn and rice starch powder with their standard deviation was found as 82 ± 1.52%, 86 ± 2.50 % and 79 ± 2.08 % respectively (Table 4.1).

4.2 Observations recorded for developed biodegradable plastic film

The observation recorded for developed corn, potato and rice starch based biodegradable plastic film was carried out on the basis of various physico-chemical properties, viz., moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology.

Table 4.1 Physical properties of starch (Potato, Corn & Rice) powder

Sr. No.	Starch source	Water absorption index (%)	
		Mean (n=3)	**SD**
1	Potato starch	139	± 1.53
2	Corn starch	155	± 2.00
3	Rice starch	130	± 2.51
Sr. No.	**Starch**	**Water solubility index (%)**	
		Mean(n=3)	**SD**
1	Potato starch	82	± 1.52
2	Corn starch	86	± 2.50
3	Rice starch	79	± 2.08

The mean values of various physico-chemical and mechanical properties of developed corn, potato and rice starch film are reported in Table 4.3, 4.5 and 4.7 respectively.

4.2.1 Physical properties of developed biodegradable plastic

Potato, corn and rice starch based biodegradable plastic was developed as per the process flow chart and casted in glass frame. The thickness of each film was measured at different places, once in the center of the film and in four places along its perimeter, and an average value was used in the calculations and results were presented in the Table 4.2. Thickness of potato, corn and rice starch film ranged from 0.11 to 0.16 mm, 0.12 to 0.16 mm and 0.08 to 0.15 mm respectively. Table 4.2 revealed that the mean value of thickness with their standard deviation was found as 0.14± 0014 mm, 0.14 ± 0.013 mm and 0.11 ±0.019 mm respectively. Thickness dependes on the poured volume in the casting frame. Large size frame was use for making film and requirement of volume is 200 ml. The result was agreement with Thakur *et al*. (2017) and they reported that increasing the starch in the formulation, the films were thicker.

Table 4.2 Physical properties (Thickness) of developed biodegradable plastic

Run	Potato starch film	Corn starch film	Rice starch film
	Thickness (mm)		
1	0.14	0.13	0.09
2	0.15	0.15	0.14
3	0.12	0.13	0.10
4	0.14	0.14	0.13
5	0.11	0.12	0.08
6	0.16	0.16	0.15
7	0.16	0.15	0.10
8	0.12	0.16	0.11
9	0.14	0.15	0.13
10	0.15	0.13	0.11
11	0.14	0.16	0.12
12	0.12	0.15	0.15
13	0.15	0.15	0.13
14	0.15	0.14	0.13
Mean	**0.14**	**0.14**	**0.11**
SD	**± 0.014**	**± 0.013**	**± 0.019**

4.3 Physico-chemical properties of corn starch biodegradable plastic

The different physico-chemical properties of corn starch biodegradable plastic were analysed viz., moisture content, transparency, water absorption capacity and water vapour permeability were carried out as per methods described in Chapter III. Results were tabulated in Table.4.3. Explanation on effect of different independent variables on response parameters and their graphical presentation are given here under.

Table 4.3 Different physico-chemical properties of corn starch biodegradable plastic.

Std. run	Starch concentration, %	Glycerol concentration,%	Moisture content, %	Transparency %	Water absorption capacity,%	Water vapour permeability, g mm/m^2 day KPa	Tensile strength, MPa	Puncture strength, MPa
1	6.5	0.88	18.96	71.4	142	0.00225	8.21	7.1
2	9.5	0.88	21.46	61.5	170	0.00393	13.62	11.56
3	6.5	1.63	20.20	68.2	148	0.00261	6.86	6.61
4	9.5	1.63	22.66	57.5	173	0.00408	12.55	10.25
5	5	1.25	18.38	72.96	138	0.002	5.93	5.12
6	11	1.25	22.83	54	180	0.00454	14.78	12.1
7	8	0.50	18.95	65.8	154	0.0027	12.35	9.36
8	8	2.00	22.44	62.3	166	0.00328	9.65	7.35
9	8	1.25	21.28	70.2	157	0.00287	10.42	8.18
10	8	1.25	20.53	66.56	164	0.00312	10.4	8.26
11	8	1.25	21.6	66.9	162	0.00284	9.87	8.67
12	8	1.25	21.87	67.5	168	0.00305	10.97	8.29
13	8	1.25	20.72	67.7	153	0.00278	10.25	8.34
14	8	1.25	19.87	67.1	158	0.00291	10.49	7.75

4.3.1 Effect of starch and glycerol concentration on moisture content of corn starch based biodegradable plastic

Moisture content of corn starch biodegradable plastic was ranged from 18.38 to 22.83 %.The maximum moisture content was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum moisture content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on moisture content of corn starch biodegradable plastic film is presented in Table 4.3.while the response surface curves and contour plots of moisture content of corn starch biodegradable plastic films are shown in the Fig. 4.1.

The response surface curve of variation in the moisture content of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.1(a). It represents the interactive effect of starch concentration and glycerol concentration on the, moisture content of corn starch biodegradable plastic film. The contour plot for moisture content of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.1(b) which indicated the increase in moisture content as the starch concentration was increased up to maximum level and increase in glycerol concentration up to maximum level. At this combination, moisture content of corn starch biodegradable plastic film was predicted 24.15%. The result was agreement with the results reported by Kibar and Us (2013). Similar results were reported by Buso- Rios *et al.* (2020) in purple sweet potato starch film and Talja *et al*. (2008) in potato starch film.

The significant F-value and non-significant lack of fit indicates the fitness and reliability of the model for a given response. However, the adequacy of the model needed to be further checked by the coefficient of regression (R^2), which is the ratio of the explained variation to the total variation and is a measure of the degree of fit (Haber and Runyon, 1977). It is also the proportion of the variability in the response variables which is accounted for the regression analysis (Mclaren *et al*., 1977). The closer the value of R^2 to unity, the better is the empirical model fits the actual data. The smaller the value of R^2, the less relevant the dependent variables in the model have in explaining the behaviour variation (Little and Hills, 1978; Mendenhall, 1975).

The value of R^2greater than 0.8 implies that the model indicates a good fit. (Joglekar and May, 1987). Nevertheless, some researchers suggested that a large value of R^2 does not always imply that the regression model is a good one. Increasing R^2 can be obtained by adding a variable to the model. Thus, it is preferred to use an adjusted R^2 to evaluate the model adequacy and it should be over 0.8 (Koocheki *et al.,* 2010). Moreover, other parameters, namely predicted R^2 which should be closer to 1 and adequate precision which should be greater than 4 are supportive of the significance of the model (Akesowan and Choonhahirun, 2013).

The regression analysis and ANOVA results for the moisture content of corn starch plastic film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed positive linear effect which there were significant at $p<0.001$ and glycerol concentration showed positive linear effect on moisture content which was significant at $p<0.01$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration and glycerol concentration was negatively non-significant on moisture content.

The derived model, giving the empirical relation between the moisture content and the test variables in coded units, was obtained as under:

Moisture Content $=+20.98 +1.15\times A +0.78\times B -1.00\times E -002\times AB -0.093\times A^2- 0.071 \times B^2$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for moisture content (12.64) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for moisture content was fitted and reliable. The R^2 value and Adj- R^2 value for the moisture content were 0.8877 and 0.8174, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.7715) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for moisture content. The small value of coefficient of variation (2.94%) for moisture content explained that the experimental results were precise and reliable (Table 4.4).

Table 4.4 Analysis of variance (ANOVA) table and regression coefficients for response surface quadratic model of different physico-chemical properties of corn starch biodegradable plastic.

Source	Moisture content	Transparency	Water absorption capacity	Water vapour permeability	Tensile strength	Puncture strength
Intercept	+20.98	+67.41	+159.94	2.973E-003	+10.35	+8.37
Linear terms						
A(X_1)	+1.15***	-4.90***	+11.42***	+6.858E-004***	+2.40***	+1.84***
B(X_2)	+0.78**	-1.18*	+2.75	+1.392E-004	-0.65***	-0.49*
Interaction terms						
AB (X_1 X_2)	-1.00E -002	-0.20	-0.75	-5.250E-005*	+0.070	-0.20
Quadratic terms						
A^2(X_1^2)	-0.093	-1.06**	-0.38	+9.096E-005	-0.018	+0.11
B^2(X_2^2)	-0.071	-0.93**	-0.13	+2.096E-005	+0.14	+0.042
Indicators for model fitting						
R^2	0.8877	0.9593	0.9047	0.9608	0.9785	0.9689
Adj-R^2	0.8174	0.9339	0.8452	0.9364	0.9651	0.9495
Pred-R^2	0.7715	0.8285	0.7431	0.7734	0.8625	0.8252
Adeq Precision	11.53	22.13	14.92	23.79	32.37	26.80
F-value	12.64	37.74	15.20	39.27	72.92	49.90
Lack of fit	NS	NS	NS	NS	NS	NS
C.V. %	2.94	2.05	2.93	5.74	4.33	4.93

A or X_1 = Starch Concentration, B or X_2 = Glycerol Concentration, ***Significant at p<0.001, **Significant at p<0.01, *Significant at p<0.05, NS = Non-significant

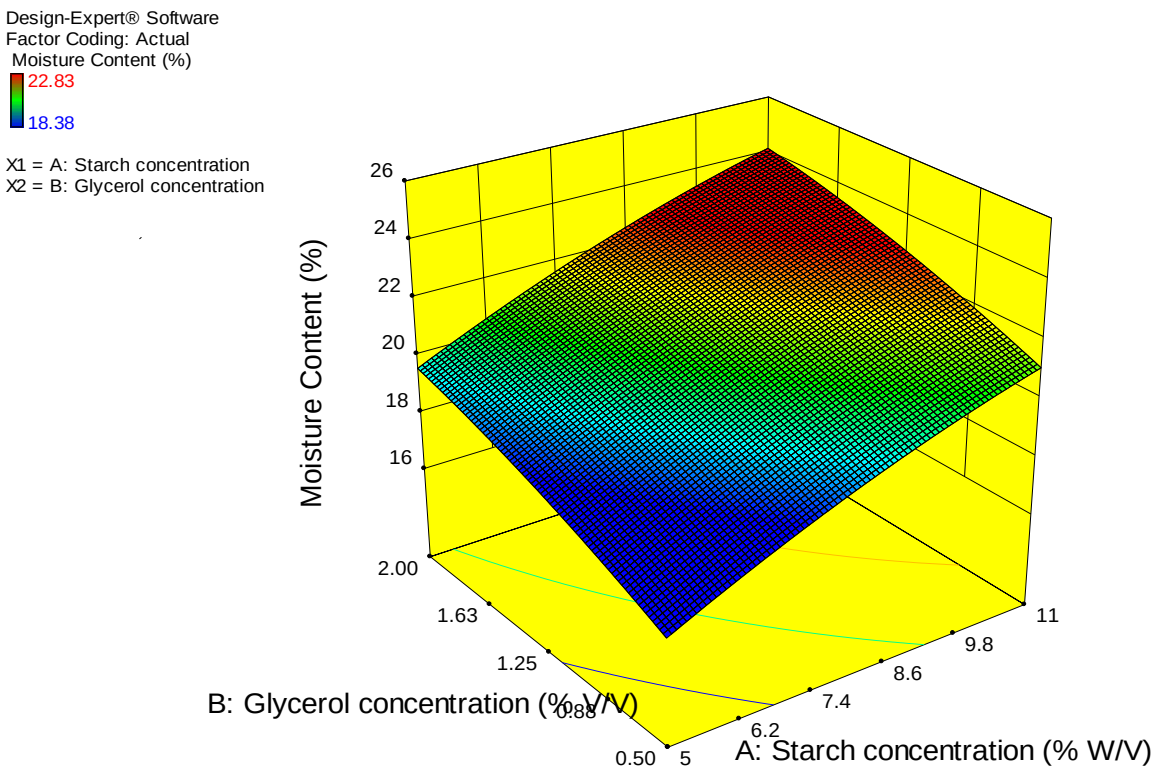


Fig. 4.1(a) Response surface plot for moisture content of corn starch biodegradable plastic

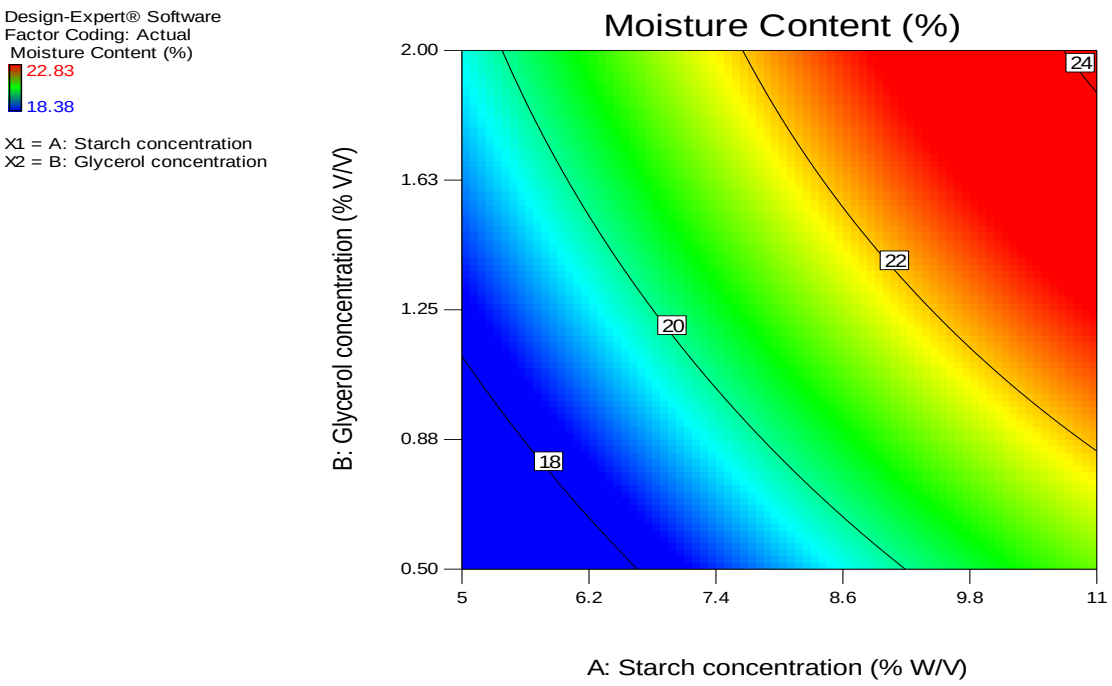


Fig. 4.1(b) Contour plot for moisture content of corn starch biodegradable plastic

4.3.2. Effect of starch and glycerol concentration on transparency of corn starch based biodegradable plastic

Transparency of corn starch film was ranged from 54 to 72.96 %. The maximum transparency was observed for the combination of 5g starch concentration and 1.25 ml glycerol concentration and minimum transparency content was found for the combination of 11g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on transparency of corn starch film is presented in Table 4.3. While the response surface curves and contour plots of transparency of corn starch biodegradable plastic films are shown in the Fig. 4.2.

The response surface curve of variation in the transparency of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.2(a). It represents the interactive effect of starch concentration and glycerol concentration on the, transparency of corn starch film. The contour plot for transparency of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.2(b) which indicated the increase in transparency as the starch concentration was decreased up to minimum level and transparency was increased with an increase in glycerol concentration up to 1.14 ml then further increase in glycerol concentration transparency was decreased. Transparency at the combination of 5 g starch concentration and 1.25 ml glycerol concentration may be observed 73 %. The result was agreement with the result reported by Dai *et al*. (2010). Similar result was found by Khairunnisa *et al.* (2018) in film made from alginate.

The regression analysis and ANOVA results for the transparency of corn starch film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed negative linear effect on transparency which there were significant at $p<0.001$ also glycerol concentration showed negative linear effect on transparency which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration and glycerol concentration was negatively significant at $p<0.01$ on transparency.

The derived model, giving the empirical relation between the bulk density and the test variables in coded units, was obtained as under: B^2

$$\text{Transparency} = +67.41 - 4.90 \times A - 1.18 \times B - 0.20 \times AB - 1.06 \times A^2 - 0.93 \times B^2$$

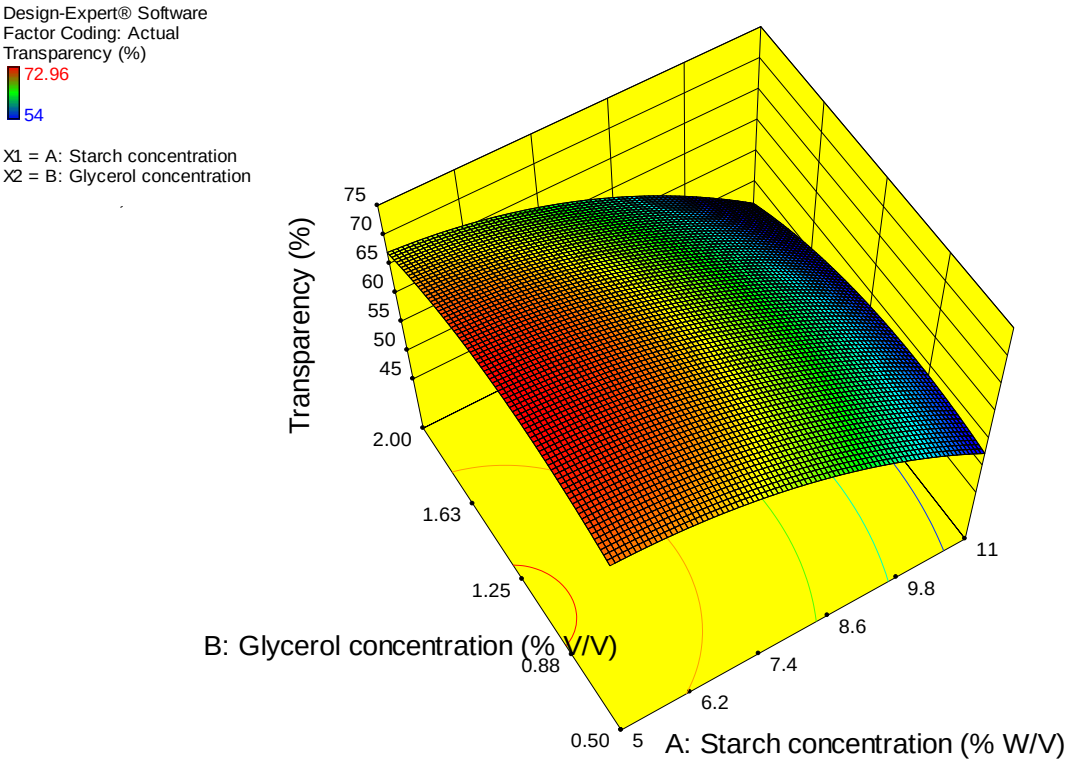


Fig. 4.2(a) Response surface plot for transparency of corn starch biodegradable plastic

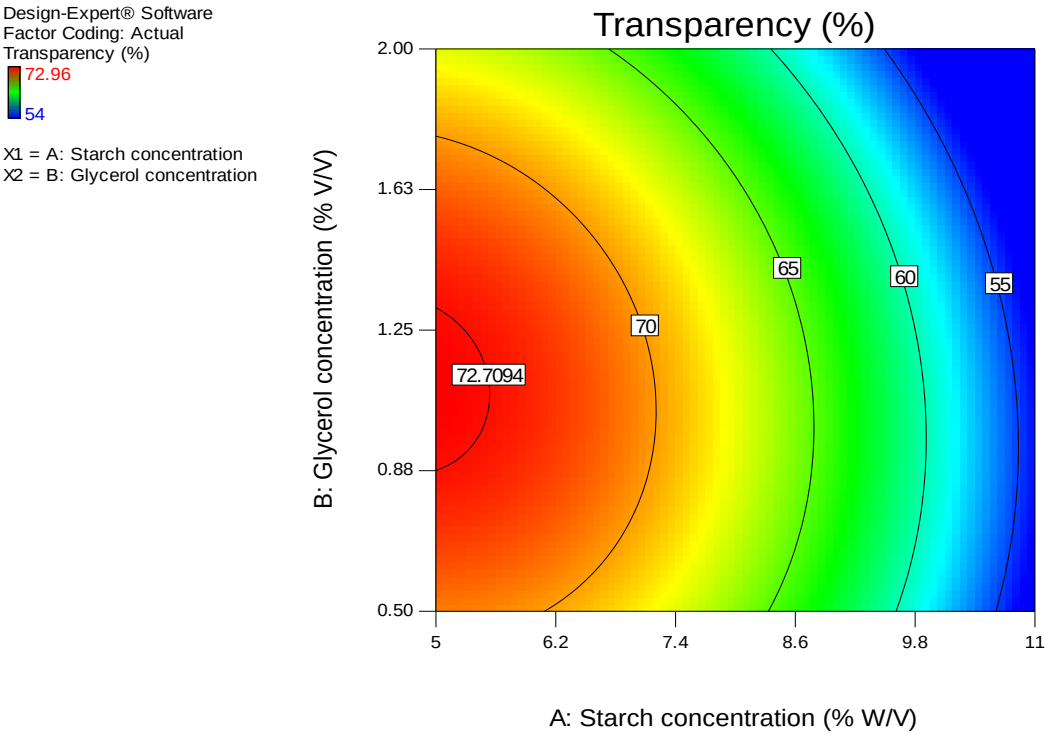


Fig. 4.2(a) Contour plot for transparency of corn starch biodegradable plastic

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for transparency (37.74) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for transparency was fitted and reliable. The R^2 value and Adj- R^2 value for the transparency were 0.9593 and 0.9339, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8285) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for transparency. The small value of coefficient of variation (2.05 %) for transparency explained that the experimental results were precise and reliable (Table 4.4).

4.3.3 Effect of starch and glycerol concentration on water absorption capacity of corn starch based biodegradable plastic

Water absorption capacity of corn starch film was ranged from 138 to 180%. The maximum water absorption capacity was observed for the combination of 11g starch concentration 1.25 ml glycerol concentration and minimum water absorption capacity content was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on water absorption capacity of corn starch film is presented in Table 4.3. While the response surface curves and contour plots of water absorption capacity of corn starch biodegradable plastic films are shown in the Fig. 4.3.

The response surface curve of variation in the water absorption capacity of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.3(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water absorption capacity of corn starch film. The contour plot for water absorption capacity of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.3(b) which indicated the increase in water absorption capacity as the starch and glycerol concentration was increased up to maximum level. At this combination, the water absorption capacity of corn starch film was predicted up to 183 % at that time value of starch concentration was 11 g and glycerol concentration was 2 ml. The result was agreement with the result reported by similar result was found by Bourtoom and Chinnan (2008a) in rice starch-chitosan film.

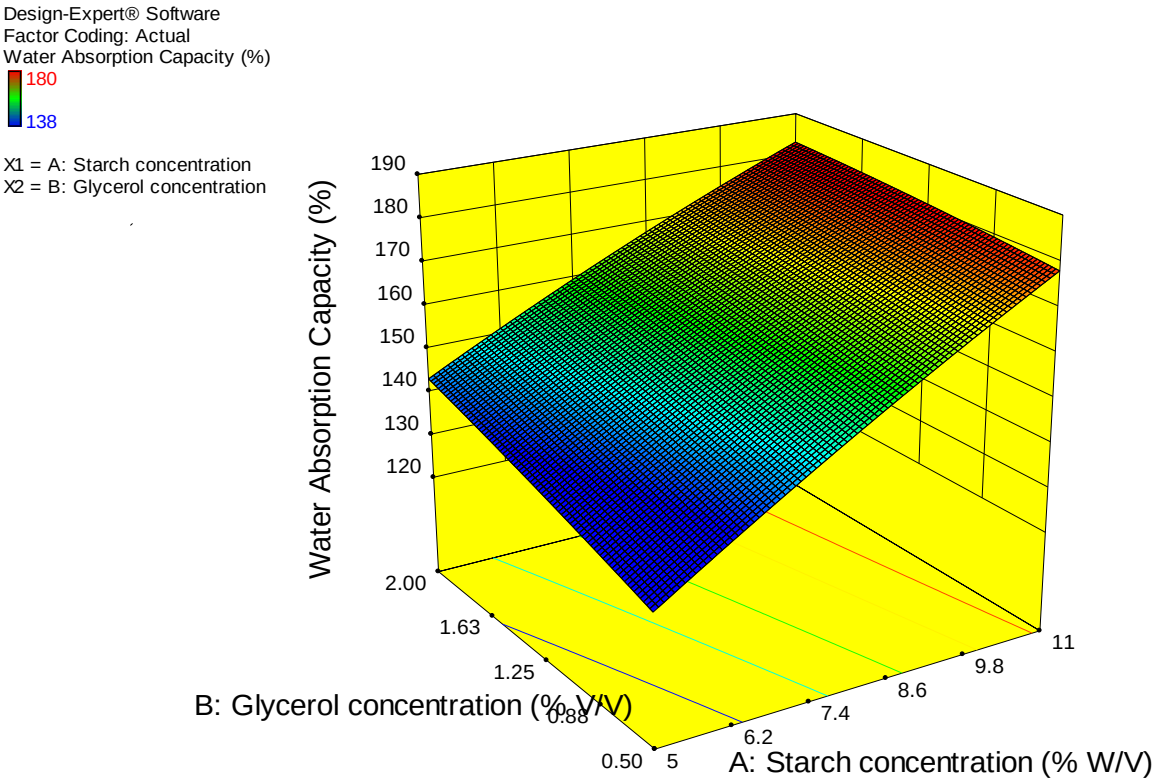


Fig. 4.3(a) Response surface plot for water absorption capacity of corn starch biodegradable plastic

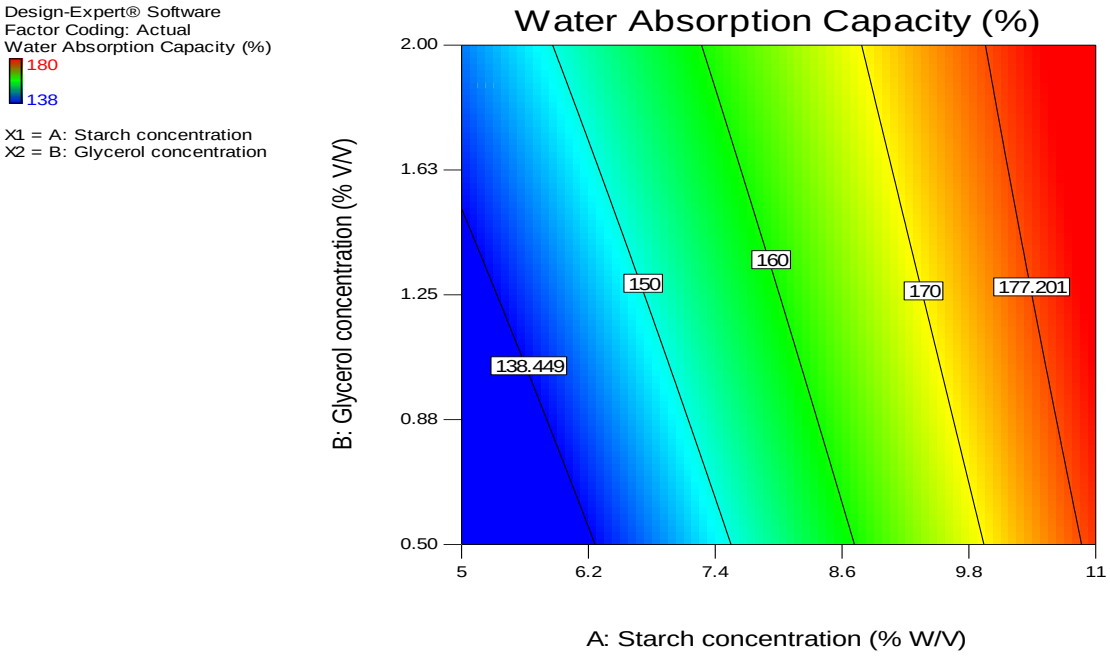


Fig. 4.3(b) Contour plot for water absorption capacity of corn starch biodegradable plastic

The regression analysis and ANOVA results for the water absorption capacity of corn starch film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed positive linear effect on water absorption capacity which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water absorption capacity which was non-significant. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration and glycerol concentration was negatively non-significant on water absorption capacity.

The derived model, giving the empirical relation between the water absorption capacity and the test variables in coded units, was obtained as under:

$$\text{Water Absorption Capacity} = +159.94 +11.42\times A +2.75\times B -0.75\times A -0.38\times A^2 -0.13\times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for water absorption capacity (15.20) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water absorption capacity was fitted and reliable. The R^2 value and Adj- R^2 value for the water absorption capacity were 0.9047 and 0.8452, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.7431) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for water absorption capacity. The small value of coefficient of variation (2.93 %) for water absorption capacity explained that the experimental results were precise and reliable (Table 4.4).

4.3.4 Effect of starch and glycerol concentration on water vapor permeability of corn starch based biodegradable plastic

Water vapor permeability of corn starch film was ranged from 0.002 to 0.0045 g.mm/m^2dayKPa The maximum water vapor permeability was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on water vapor permeability of corn starch film is presented in Table 4.3. While the response surface curves and contour plots water vapor permeability of corn starch biodegradable plastic films are shown in the Fig. 4.4.

The response surface curve of variation in the water vapor permeability of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.4(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water vapor permeability of corn starch film. The contour plot for water vapor permeability of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.4(b) which indicated the decrease the starch and glycerol concentration, water vapor permeability was decreased up to minimum level. At this combination, the water vapor permeability of corn starch film was predicted up to 0.00156 g mm/m^2dayKPa. The result was agreement with the result reported by Ghasemlou *et al.* (2013) in corn starch films incorporated with plant essential oils. Similar result was found by Farahnaky *et al*. (2013) in films made of wheat starch and glycerol and Muhammed *et al.* (2015) in sugar palm starch film incorporated with glycerol and sorbitol.

The regression analysis and ANOVA results for the water vapor permeability of corn starch film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed positive linear effect on water vapor permeability which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water vapor permeability which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration was positively significant at $p<0.05$ and glycerol concentration was positively non-significant on water vapor permeability

The derived model, giving the empirical relation between the water vapor permeability and the test variables in coded units, was obtained as under:

$$\text{Water Vapor Permeability} = +2.973E\text{-}003 + 6.858E\text{-}004 \times A + 1.392E\text{-}004 \times B - 5.250E\text{-}005E \times AB + 9.096E\text{-}005 \times A^2 + 2.096E\text{-}005 \times B^2$$

The calculated F-value for water vapor permeability (39.27) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water vapor permeability was fitted and reliable. The R^2 value and Adj- R^2 value for the water vapor permeability were 0.9608 and 0.9364, respectively, which were higher than the 0.8, indicating the

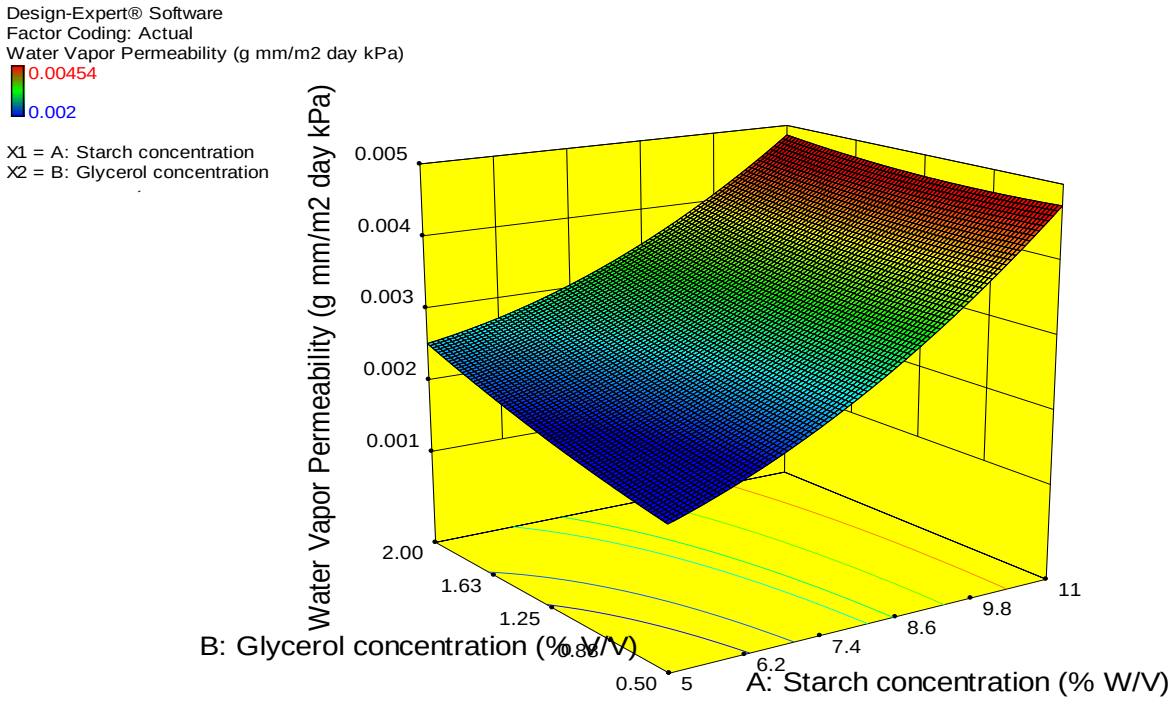


Fig. 4.4(a) Response surface plot for water vapor permeability of corn starch biodegradable plastic

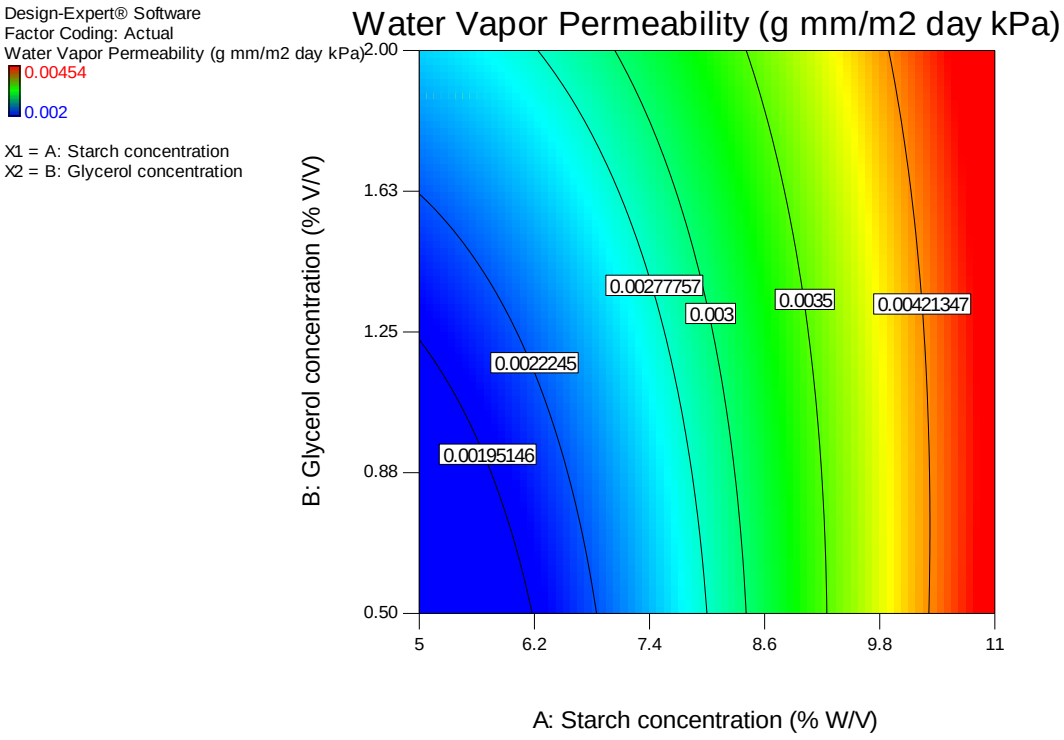


Fig. 4.4(b) Contour plot for water vapor permeability of corn starch biodegradable plastic

adequacy, good fit and high significance of the model. The Pred- R^2 (0.7734) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for water vapor permeability. The small value of coefficient of variation (5.74 %) for water vapor permeability explained that the experimental results were precise and reliable (Table 4.4).

4.3.5 Effect of starch and glycerol concentration on tensile strength of corn starch based biodegradable plastic

Tensile strength of corn starch film was ranged from 5.93 to 14.78 MPa. The maximum tensile strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on tensile strength of corn starch film is presented in Table 4.3. while the response surface curves and contour plots of tensile strength of corn starch biodegradable plastic films are shown in the Fig. 4.5.

The response surface curve of variation in the tensile strength of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.5(a). It represents the interactive effect of starch concentration and glycerol concentration on the, tensile strength of corn starch film. The contour plot for tensile strength of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.5(b) which indicated the increase the tensile strength, starch concentration was increased up to maximum level and tensile strength decrease with increases the glycerol concentration up to maximum level. At this combination, the tensile strength of corn starch film was predicted up to 16.64 MPa. The result was agreement with the result reported by Muhammed *et al.* (2015) and Muscat *et al.* (2012). Similar result was reported by Sonam and aditya (2016) in potato starch film, increase in the plasticizer concentration causes a reduction of the tensile strength due to the decrease in the intermolecular interactions.

The regression analysis and ANOVA results for the tensile strength of corn starch film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed positive linear effect on tensile strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on tensile strength which was significant at $p<0.001$. Whilst, the interaction effect of starch and

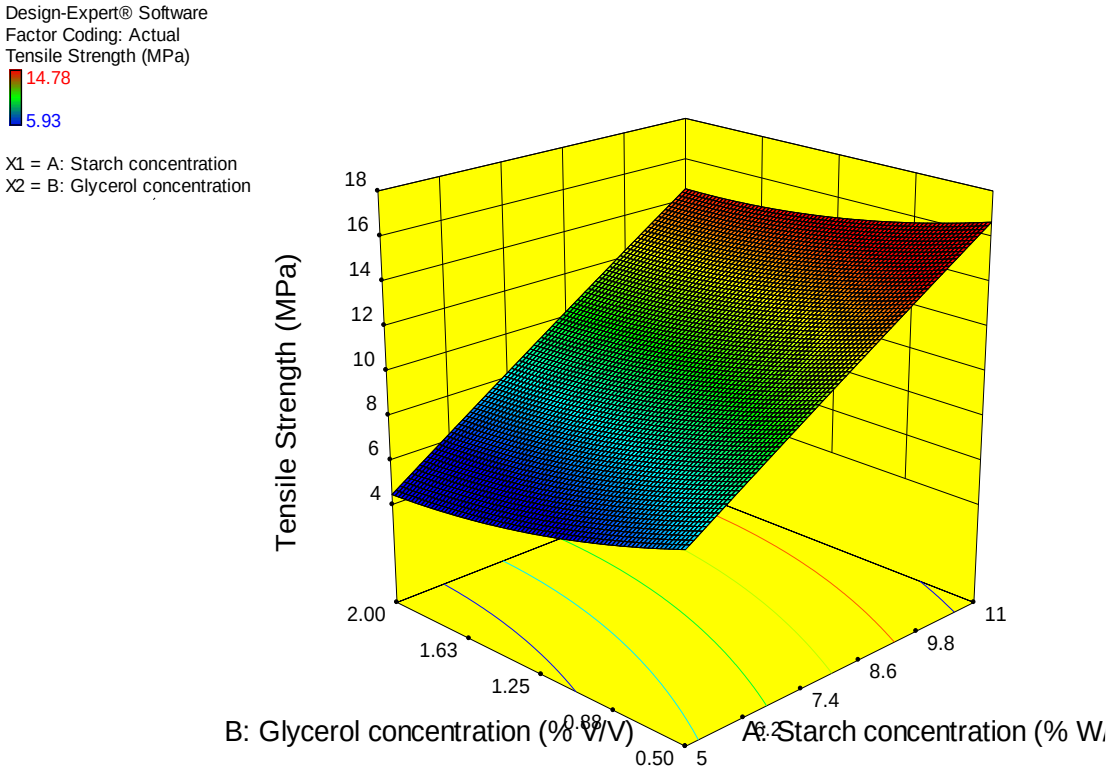


Fig. 4.5(a) Response surface plot for tensile strength of corn starch biodegradable plastic

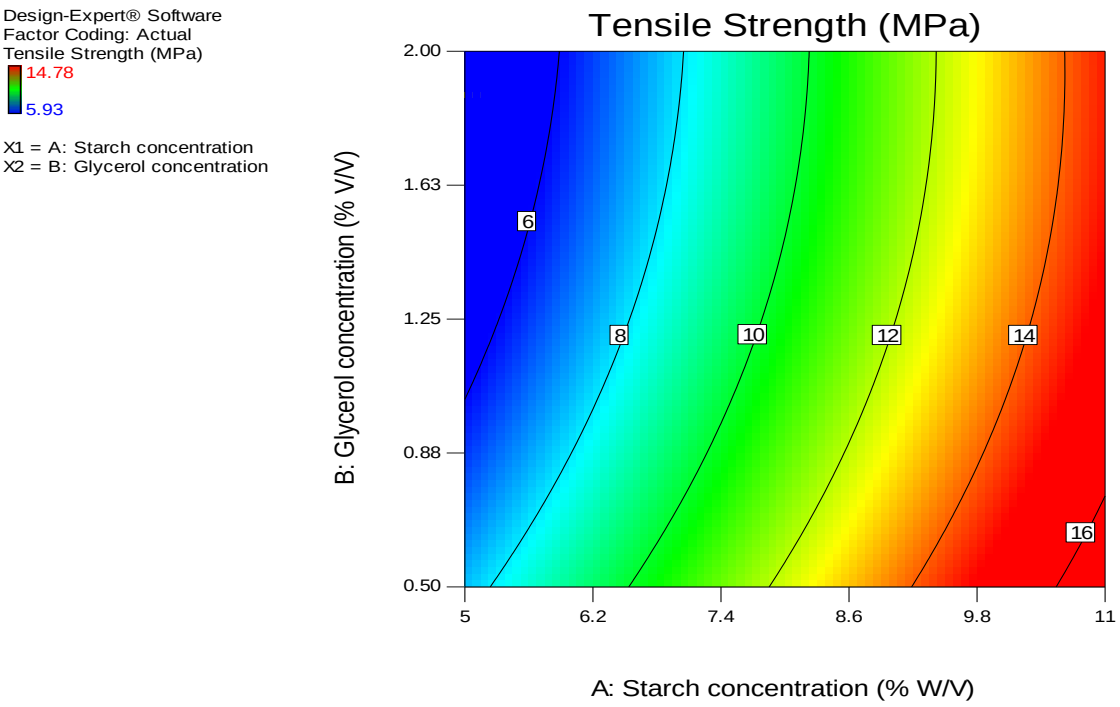


Fig. 4.5(b) Contour plot for tensile strength of corn starch biodegradable plastic

glycerol concentration was positively non -significant and the quadratic effect of starch concentration was positively non-significant on tensile strength. The derived model, giving the empirical relation between the tensile strength and the test variables in coded units, was obtained as under:

$$\text{Tensile Strength} = +10.35 + 2.40 \times A - 0.65 \times B + 0.070 \times AB - 0.018 \times A^2 + 0.14 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for tensile strength (72.92) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for tensile strength was fitted and reliable. The R^2 value and Adj- R^2 value for the tensile strength were 0.9785 and 0.9651, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8625) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for tensile strength. The small value of coefficient of variation (4.33%) for tensile strength explained that the experimental results were precise and reliable (Table 4.4).

4.3.6. Effect of starch and glycerol concentration on puncture strength of corn starch based biodegradable plastic

Puncture strength of corn starch film was ranged from 5.12 to 12.1 MPa. The maximum puncture strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on puncture strength of corn starch film is presented in Table 4.3. While the response surface curves and contour plots of tensile strength of corn starch biodegradable plastic films are shown in the Fig. 4.6.

The response surface curve of variation in the puncture strength of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.6(a). It represents the interactive effect of starch and glycerol concentration on the, puncture strength of corn starch film. The contour plot for puncture strength of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.6(b) which indicated the increase the puncture strength, starch concentration was increased up to maximum level and puncture strength decrease with increases the glycerol

concentration. At this combination, the puncture strength of corn starch film was predicted up to 14.40 MPa. The result was agreement with the result reported by Sonam and Aditya (2016) in potato starch film. Similar result was reported by Mali *et al*. (2005) in corn, cassava, and yam starch films.

The regression analysis and ANOVA results for the puncture strength of corn starch film are shown in the Table 4.4. It can be seen from the table, that starch concentration showed positive linear effect on puncture strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on puncture strength which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration was negatively non-significant at on tensile strength. The derived model, giving the empirical relation between the puncture and the test variables in coded units, was obtained as under:

$$\text{Puncture Strength} = +8.37 + 1.84 \times A - 0.49 \times B - 0.20 \times AB + 0.11 \times A^2 + 0.042 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for puncture strength (49.90) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for puncture strength was fitted and reliable. The R^2 value and Adj- R^2 value for the puncture strength were 0.9689 and 0.9495, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8252) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for puncture strength. The small value of coefficient of variation (4.93 %) for puncture strength explained that the experimental results were precise and reliable (Table 4.4).

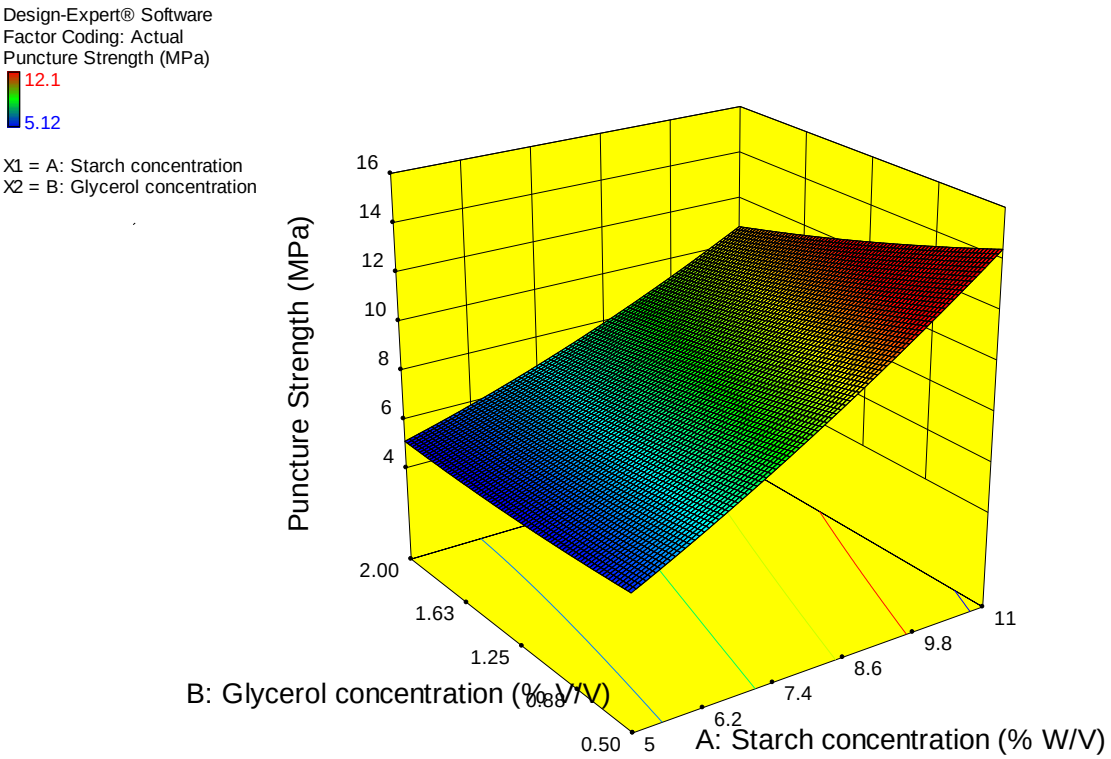


Fig. 4.6(a) Response surface plot for puncture strength of corn starch plastic

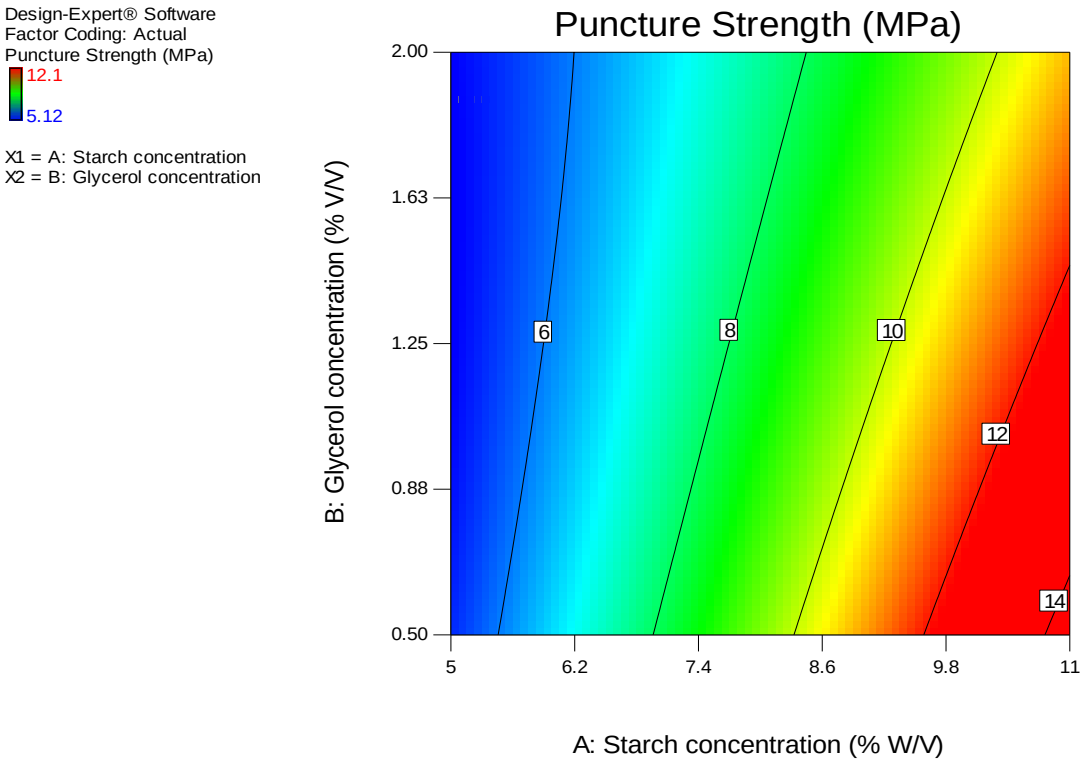


Fig. 4.6(b) Contour plot for puncture strength of corn starch biodegradable plastic

4.4 Physico-chemical properties of potato starch biodegradable plastic

The different physico-chemical properties of potato starch biodegradable plastic were analysed and studied viz., moisture content, transparency, water absorption capacity, water vapour permeability and surface morphology were carried out as per methods described in Chapter III. Results were tabulated in Table.4.5. The explanation on effect of different independent variables on response parameters and their graphical presentation are given here under.

4.4.1 Effect of starch and glycerol concentration on moisture content of potato starch based biodegradable plastic

Moisture content of potato starch film was ranged from 18.19 to 23.1%. The maximum moisture content was observed for the combination 11 g starch concentration and 1.25 ml glycerol concentration and minimum moisture content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on moisture content of potato starch biodegradable plastic is presented in Table 4.5. While the response surface curves and contour plots of moisture content of potato starch biodegradable plastic are shown in the Fig. 4.7.

The response surface curve of variation in the moisture content of potato starch biodegradable plastic as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.7(a). It represents the interactive effect of starch concentration and glycerol concentration on the, moisture content of potato starch film. The contour plot for moisture content of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.7(b) which indicated the increase in moisture content as the starch concentration was increased up to maximum level and increase in glycerol concentration up to maximum level. At this combination, moisture content of potato starch biodegradable plastic film was predicted 24.55 %. The result was agreement with the results reported by Talja *et al*. (2008). Similar results were reported by Buso- Rios *et al.* (2020) in purple sweet potato starch film and Kibar and Us (2013) in corn starch film.

The regression analysis and ANOVA results for the moisture content of potato starch plastic are shown in the Table 4.6. It can be seen from the table, that starch and glycerol concentration showed positive linear effect which significant at $p<0.001$.Whilst, the interaction effect of starch concentration and glycerol

Table 4.5 Different physico-chemical properties of potato starch biodegradable plastic.

Std. run	Starch concentration,%	Glycerol concentration,%	Moisture content, %	Transparency %	Water absorption capacity,%	Water vapour permeability, g mm/m^2 day KPa	Tensile strength, MPa	Puncture strength, MPa
1	6.5	0.88	19.22	67.31	156	0.004	7.01	6.35
2	9.5	0.88	21.4	59.63	178	0.0052	11.76	8.97
3	6.5	1.63	19.72	66.25	161	0.00411	6.23	5.31
4	9.5	1.63	22.32	58.23	183	0.00524	11.07	8.36
5	5	1.25	18.19	69.54	151	0.00371	5.09	4.28
6	11	1.25	23.1	52.86	190	0.0058	13.62	10.45
7	8	0.50	18.73	63.21	165	0.00435	10.58	7.83
8	8	2.00	21.22	60.9	176	0.00473	8.54	6.95
9	8	1.25	20.23	64.23	173	0.00462	10.36	7.47
10	8	1.25	20.43	62.31	167	0.00428	10.47	7.36
11	8	1.25	20.46	62.45	172	0.00458	9.85	7.28
12	8	1.25	19.93	62.85	171	0.00459	10.2	7.08
13	8	1.25	19.95	63.10	168	0.00447	9.50	7.39
14	8	1.25	19.96	64.76	169	0.00465	9.78	7.53

concentration was positively non-significant and the quadratic effect of starch concentration was positively non- significant and quadratic effect of glycerol concentration was negatively non-significant on moisture content.

The derived model, giving the empirical relation between the moisture content and the test variables in coded units, was obtained as under:

$$\text{Moisture Content} = +20.16 + 1.22 \times A + 0.53 \times B + 0.10 \times AB + 0.13 \times A^2 - 0.034 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for moisture content (34.13) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for moisture content was fitted and reliable. The R^2value and Adj-R^2 value for the moisture content were 0.9552 and 0.9272, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2(0.7391) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for moisture content. The small value of coefficient of variation (1.76 %) for moisture content explained that the experimental results were precise and reliable (Table 4.6).

4.4.2. Effect of starch and glycerol concentration on transparency of potato starch based biodegradable plastic

Transparency of potato starch film was ranged from 52.86 to 69.54 %. The maximum transparency was observed for the combination of 5g starch concentration and 1.10 glycerol concentration and minimum transparency was found for the combination of 11g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on transparency of potato starch film is presented in Table 4.5. While the response surface curves and contour plots of transparency of potato starch biodegradable plastic films are shown in the Fig. 4.8

The response surface curve of variation in the transparency of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.8(a). It represents the interactive effect of starch concentration and glycerol concentration on the, transparency of potato starch film. The contour plot for transparency of potato starch biodegradable plastic film as a function starch concentration (X_1) and glycerol concentration (X_2) is presented in

Table 4.6 Analysis of variance (ANOVA) table and regression coefficients for response surface quadratic model of different physico-chemical properties of potato starch biodegradable plastic.

Source	Moisture content	Transparency	Water absorption capacity	Water vapor permeability	Tensile strength	Puncture strength
Intercept	+20.16	+63.38	+169.09	+4.543E-003	+9.86	+7.32
Linear terms						
$A(X_1)$	+1.22***	-4.09***	+10.17***	+5.425E-004***	+2.22***	+1.50***
$B(X_2)$	+0.53***	-0.59*	+2.67**	+7.583E-005	-0.46*	-0.28***
Interaction terms						
$AB(X_1 X_2)$	+0.10	-0.085	+2.82878E-014	-1.750E-005	+0.023	+0.11
Quadratic terms						
$A^2(X_1^2)$	+0.13	-0.51**	+0.10	+5.724E-005*	-0.19	-1.103E-004
$B^2(X_2^2)$	-0.034	-0.30	+0.10	+3.493E-006	-0.14	+6.140E-003
Indicators for model fitting						
R^2	0.9552	0.9744	0.9751	0.9686	0.9628	0.9892
Adj-R^2	0.9272	0.9585	0.9595	0.9489	0.9395	0.9825
Pred-R^2	0.7391	0.9470	0.9316	0.9054	0.7923	0.9346
Adeq Precision	20.81	29.97	30.16	27.11	24.61	46.92
F-value	34.13	61.01	62.55	49.29	41.36	146.90
Lack of fit	NS	NS	NS	NS	NS	NS
C.V. %	1.76	1.33	1.21	2.66	5.76	2.67

A or X_1 = Starch Concentration, B or X_2 = Glycerol Concentration, ***Significant at $p<0.001$, **Significant at $p<0.01$,*Significant at $p<0.05$, NS = Non-significant

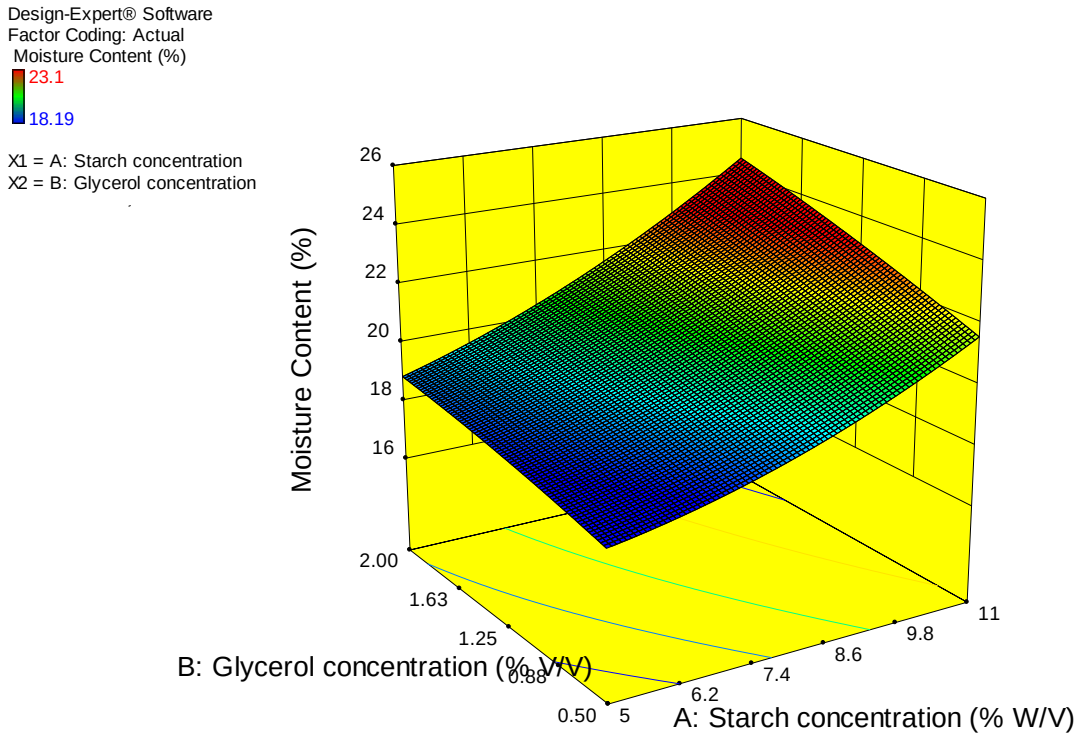


Fig. 4.7(a) Response surface plot for moisture content of potato starch biodegradable plastic

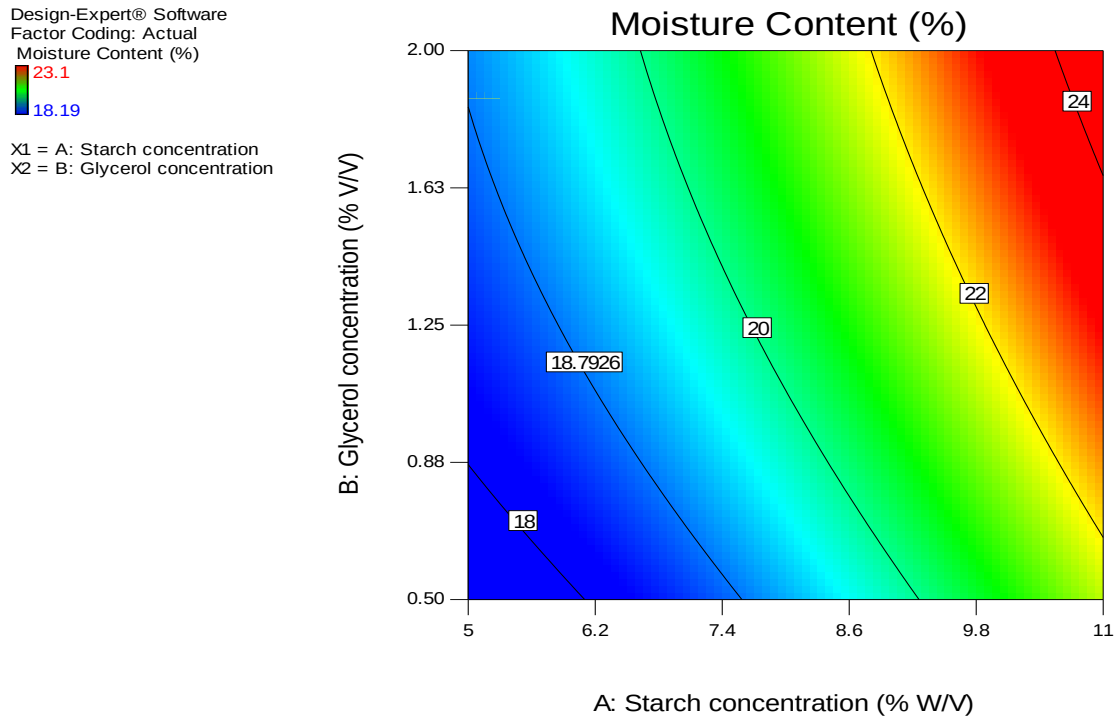


Fig. 4.7 (b) Contour plot for moisture content of potato starch biodegradable plastic

the Fig. 4.8(b) which indicated the increase in transparency as the starch concentration was decreased up to minimum level and transparency was increased with an increase in glycerol concentration up to 1.10 ml then further increase in glycerol concentration transparency was decreased. Transparency at the combination of 5 g starch concentration and 1.02 ml glycerol concentration may be observed 69.65 %. The result was agreement with the result reported by Dai *et al*. (2010). Similar result was found by Khairunnisa *et al.* (2018) in film made from alginate.

The regression analysis and ANOVA results for the transparency of potato starch film are shown in the Table 4.6. It can be seen from the table, that starch concentration showed negative linear effect on transparency which there were significant at $p<0.001$ also glycerol concentration showed negative linear effect on transparency which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration showed negatively significant at $p<0.01$ and glycerol concentration was negatively non-significant on transparency.

The derived model, giving the empirical relation between the bulk density and the test variables in coded units, was obtained as under:

$$\text{Transparency} = +63.38 - 4.09 \times A - 0.59 \times B - 0.085 \times AB - 0.51 \times A^2 - 0.30 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for transparency (61.01) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for transparency was fitted and reliable. The R^2 value and Adj- R^2 value for the transparency were 0.9744 and 0.9585, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.9470) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for transparency. The small value of coefficient of variation (1.33 %) for transparency explained that the experimental results were precise and reliable (Table 4.6).

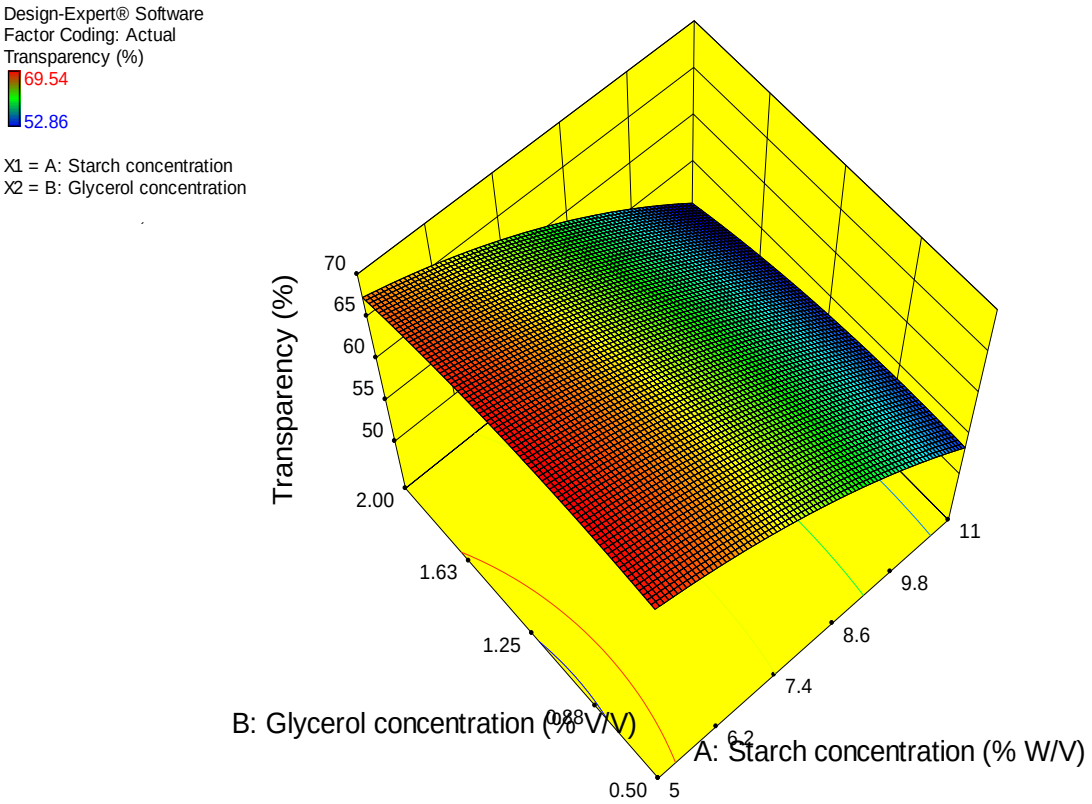


Fig. 4.8(a) Response surface plot for transparency of potato starch biodegradable plastic

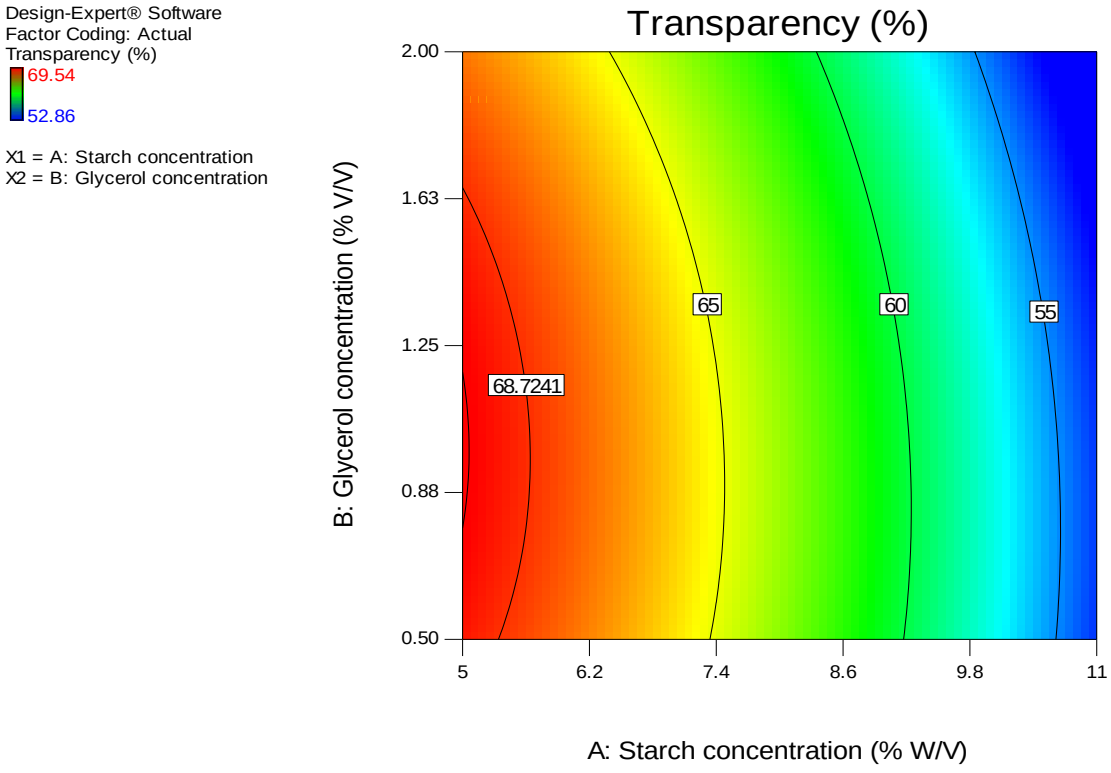


Fig. 4.8(b) Contour plot for transparency of potato starch biodegradable plastic

4.4.3 Effect of starch and glycerol concentration on water absorption capacity of potato starch based biodegradable plastic

Water absorption capacity of potato starch film was ranged from 151 to 190%. The maximum water absorption capacity was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum water absorption capacity content was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration.

The response surface curve of variation in the water absorption capacity of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.3(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water absorption capacity of potato starch film. The contour plot for water absorption capacity of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.3(b) which indicated the increase in water absorption capacity as the starch and glycerol concentration was increased up to maximum level. At this combination, the water absorption capacity of potato starch film was predicted up to 145 %. The result was agreement with result reported by Farahnaky *et al.* (2013) in wheat starch film and similar result was found by Bourtoom and Chinnan (2008a) in rice starch-chitosan film.

The regression analysis and ANOVA results for the water absorption capacity of potato starch film are shown in the Table 4.6. It can be seen from the table, that starch concentration showed positive linear effect on water absorption capacity which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water absorption capacity which was significant at $p<0.01$. Whilst, the interaction effect of starch concentration and glycerol concentration was positively non-significant and the quadratic effect of starch concentration and glycerol concentration was positively non-significant on water absorption capacity.

The derived model, giving the empirical relation between the water absorption capacity and the test variables in coded units, was obtained as under:

Water Absorption Capacity=+169.82 +10.17×A+2.67×B+0.000 ×AB +0.10×A^2+0.10×B^2

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for water absorption capacity (62.55) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water absorption capacity was fitted and reliable. The R^2value and Adj- R^2 value for the water absorption capacity were 0.9751 and 0.9595, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2(0.9316) was in reasonable agreement with the Adj-R^2. The high Adequate Precision value (>4) again supported the significance of the model for water absorption capacity. The small value of coefficient of variation (1.21 %) for water absorption capacity explained that the experimental results were precise and reliable (Table 4.6).

4.4.4 Effect of starch and glycerol concentration on water vapor permeability of potato starch based biodegradable plastic

Water vapor permeability of potato starch film was ranged from 0.00371 to 0.00580 g.mm/m^2dayKPa The maximum water vapor permeability was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on water vapor permeability of potato starch film is presented in Table 4.5. While the response surface curves and contour plots of water vapor permeability of potato starch biodegradable plastic films are shown in the Fig. 4.10.

The response surface curve of variation in the water vapor permeability of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.10(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water vapor permeability of potato starch film. The contour plot for water vapor permeability of potato starch biodegradable plastic film as a function of starch concentration (X_1)and glycerol concentration (X_2) is presented in the Fig. 4.10(b) which indicated the decrease the starch and glycerol concentration, water vapour permeability was decreased up to minimum level. At this combination, the water vapour permeability of potato starch film was predicted up to 0.00348 g.mm/m^2dayKPa. The result was agreement with the result reported by Ghasemlou *et al.* (2013) in corn starch films incorporated with plant

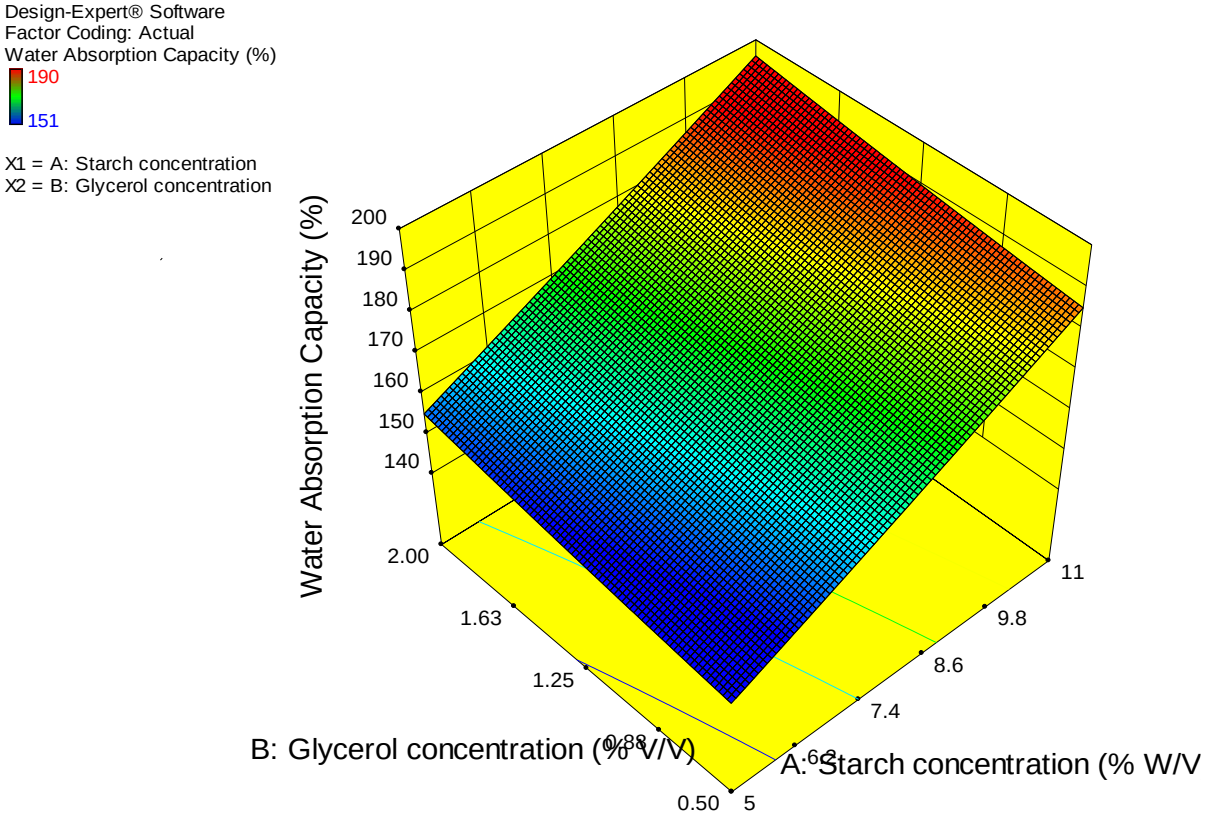


Fig. 4.9(a) Response surface plot for water absorption capacity of potato starch biodegradable plastic

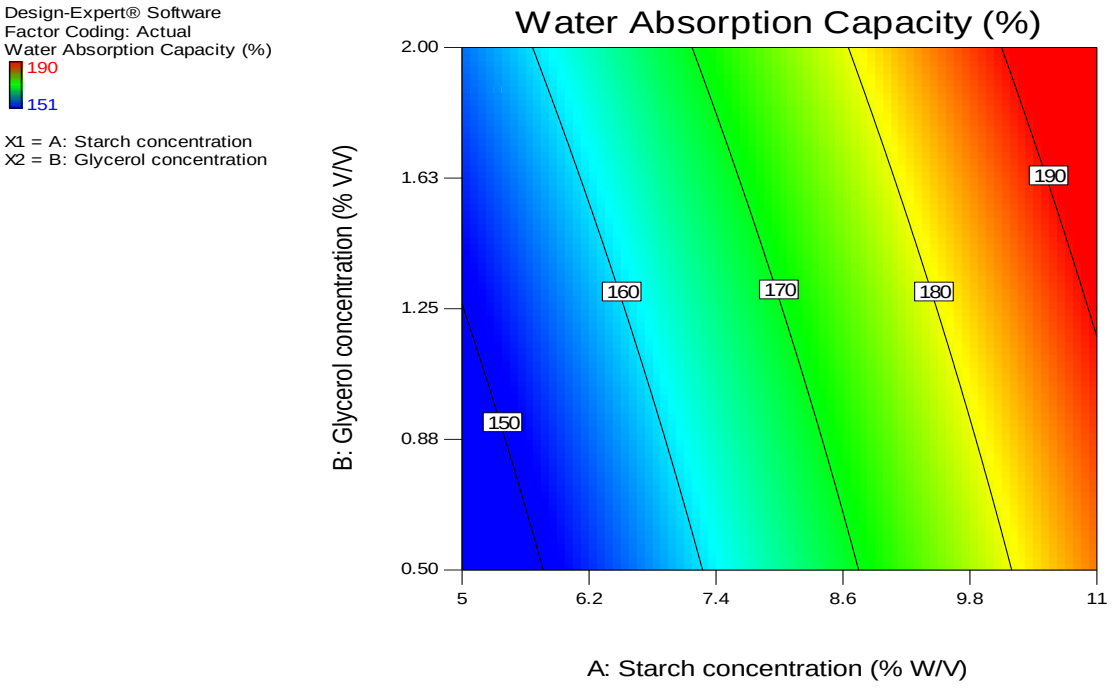


Fig. 4.9 (b) Contour plot for water absorption capacity of potato starch biodegradable plastic

essential oils. Similar result was found by Farahnaky *et al*. (2013) in films made of wheat starch and glycerol and Muhammed *et al.* (2015) in sugar palm starch film incorporated with glycerol and sorbitol.

The regression analysis and ANOVA results for the water vapor permeability of potato starch film are shown in the Table 4.6. It can be seen from the table, that starch concentration showed positive linear effect on water vapor permeability which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water vapor permeability which was non-significant. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration was positively significant at $p<0.05$ and glycerol concentration was positively non-significant on water vapor permeability.

The derived model, giving the empirical relation between the water vapor permeability and the test variables in coded units, was obtained as under:

$$\text{Water Vapor Permeability} = +4.543E\text{-}003 +5.425E\text{-}004\times A +7.583E\text{-}005\times B -1.750E\text{-}005 \times AB +5.724E\text{-}005\times A^2+3.493E\text{-}006\times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for water vapor permeability (49.29) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water vapor permeability was fitted and reliable. The R^2 value and Adj- R^2 value for the water vapor permeability were 0.9686 and 0.9489, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.9054) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for water vapour permeability. The small value of coefficient of variation (2.66 %) for water vapor permeability explained that the experimental results were precise and reliable (Table 4.6).

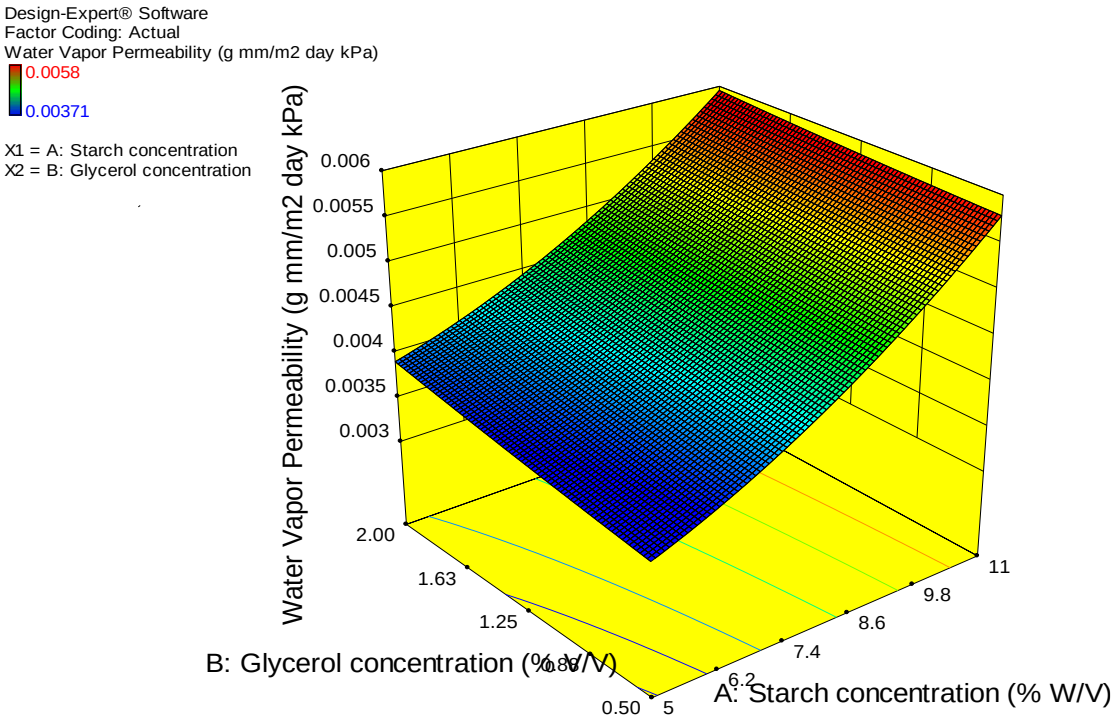


Fig. 4.10 (a) Response surface plot for water vapor permeability of potato starch biodegradable plastic

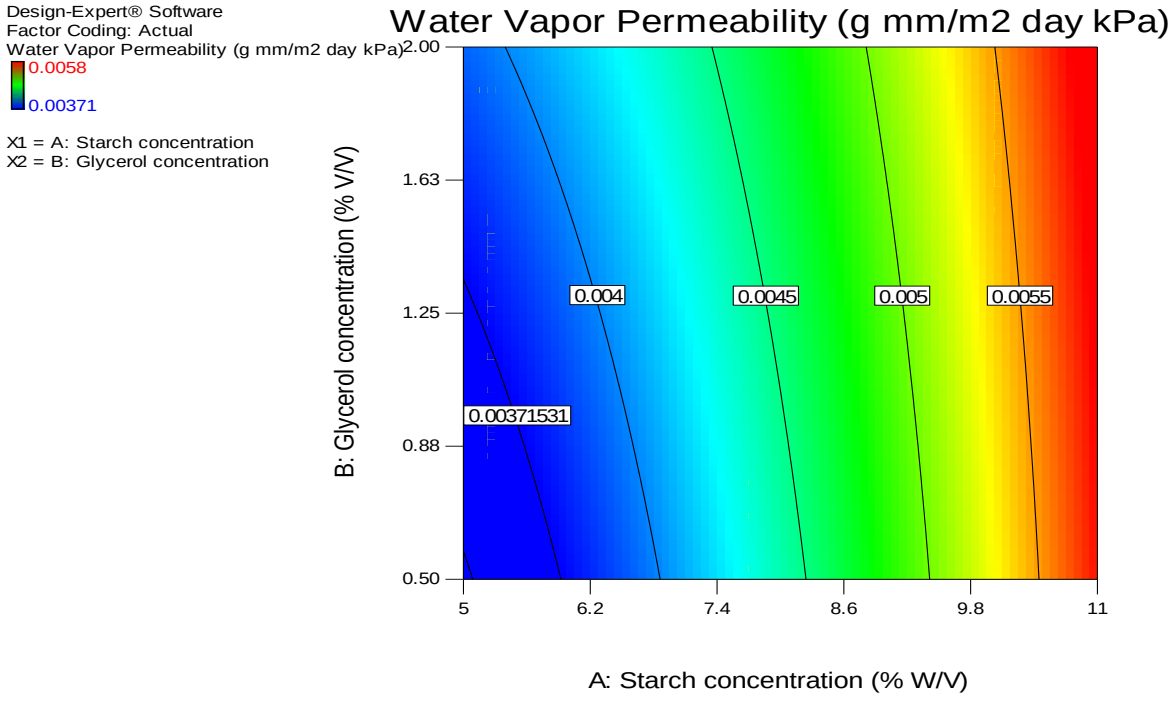


Fig. 4.10 (b) Contour plot for water vapor permeability of potato starch biodegradable plastic

4.4.5 Effect of starch and glycerol concentration on tensile strength of potato starch based biodegradable plastic

Tensile strength of potato starch film was ranged from 5.09 to 13.62 MPa. The maximum tensile strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on tensile strength of potato starch film is presented in Table 4.5. while the response surface curves and contour plots of tensile strength of potato starch biodegradable plastic films are shown in the Fig. 4.11.

The response surface curve of variation in the tensile strength of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.11(a). It represents the interactive effect of starch concentration and glycerol concentration on the, tensile strength of potato starch film. The contour plot for tensile strength of corn starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.11(b) which indicated the increase the tensile strength, starch concentration was increased up to maximum level and tensile strength decrease with increases the glycerol concentration. At this combination, the tensile strength of potato starch film was predicted up to 13.82 MPa. The result was agreement with the result reported by Muhammed *et al.* (2015) and Muscat *et al.* (2012). Similar result was reported by Sonam and aditya (2016) in potato starch film, increase in the plasticizer concentration causes a reduction of the tensile strength due to the decrease in the intermolecular interactions.

The regression analysis and ANOVA results for the tensile strength of potato starch film are shown in the Table 4.6. It can be seen from the table, that starch concentration showed positive linear effect on tensile strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on tensile strength which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was positively non-significant and the quadratic effect of starch and glycerol concentration was negatively non-significant at tensile strength.

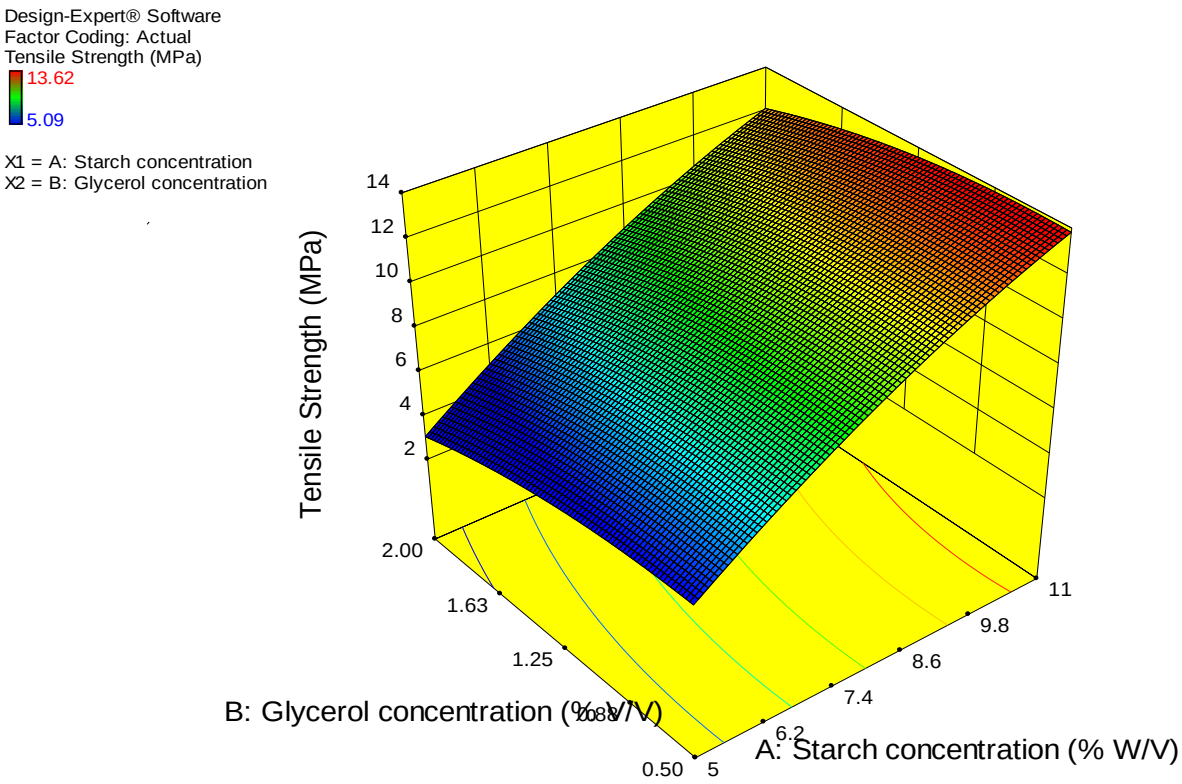


Fig. 4.11 (a) Response surface plot for tensile strength of potato starch biodegradable plastic

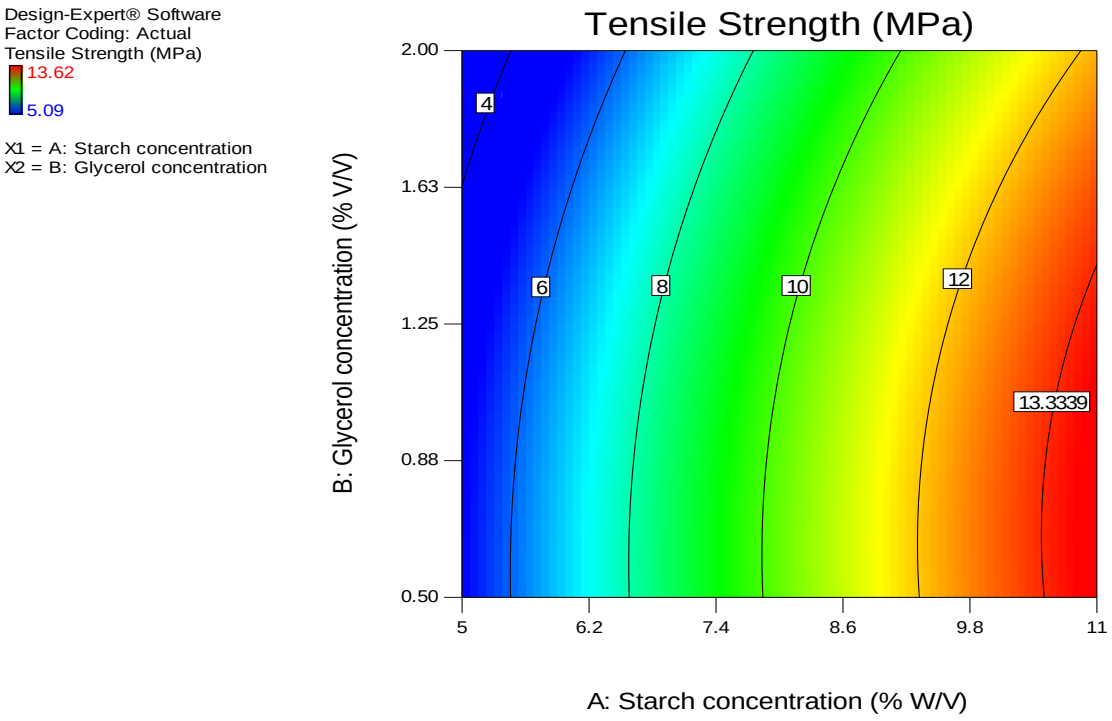


Fig. 4.11 (b) Contour plot for tensile strength of potato starch biodegradable plastic

The derived model, giving the empirical relation between the tensile strength and the test variables in coded units, was obtained as under:

$$\text{Tensile Strength} = +9.86 + 2.22 \times A - 0.46 \times B + 0.023 \times AB - 0.19 \times A^2 - 0.14 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for tensile strength (41.36) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for tensile strength was fitted and reliable. The R^2 value and Adj- R^2 value for the tensile strength were 0.9628 and 0.9395, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.7923) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for tensile strength. The small value of coefficient of variation (5.76 %) for tensile strength explained that the experimental results were precise and reliable (Table 4.6).

4.4.6. Effect of starch and glycerol concentration on puncture strength of potato starch based biodegradable plastic

Puncture strength of potato starch film was ranged from 4.28 to 10.45 MPa. The maximum puncture strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on puncture strength of potato starch film is presented in Table 4.5. While the response surface curves and contour plots of tensile strength of potato starch biodegradable plastic films are shown in the Fig. 4.12.

The response surface curve of variation in the puncture strength of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.12(a). It represents the interactive effect of starch concentration and glycerol concentration on the, puncture strength of potato starch film. The contour plot for puncture strength of potato starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.12(b) which indicated the increase the puncture strength, starch concentration was increased up to maximum level and puncture strength decrease with increases the glycerol concentration. At this combination, the

puncture strength of potato starch film was predicted up to 10.48 MPa. The result was agreement with the result reported by Sonam and Aditya (2016) in potato starch film. Similar result was reported by Mali *et al*. (2005) in corn, cassava, and yam starch films.

The regression analysis and ANOVA results for the puncture strength of potato starch film are shown in the Table 4.6. It can be seen from the table, that starch concentration showed positive linear effect on puncture strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on puncture strength which was significant at $p<0.001$. Whilst, the interaction effect of starch and glycerol concentration was positively non-significant and the quadratic effect of starch concentration was negatively non-significant and glycerol concentration was positively non-significant on puncture strength.

The derived model, giving the empirical relation between the puncture strength and the test variables in coded units, was obtained as under:

$$\text{Puncture Strength} = +7.32 +1.50\times A -0.28\times B +0.11\times AB -1.103E\text{-}004\times A^2 +6.140E\text{-}003 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for puncture strength (146.90) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for puncture strength was fitted and reliable. The R^2 value and Adj- R^2 value for the puncture strength were 0.9892 and 0.9825, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.9346) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for puncture strength. The small value of coefficient of variation (2.67 %) for puncture strength explained that the experimental results were precise and reliable (Table 4.6).

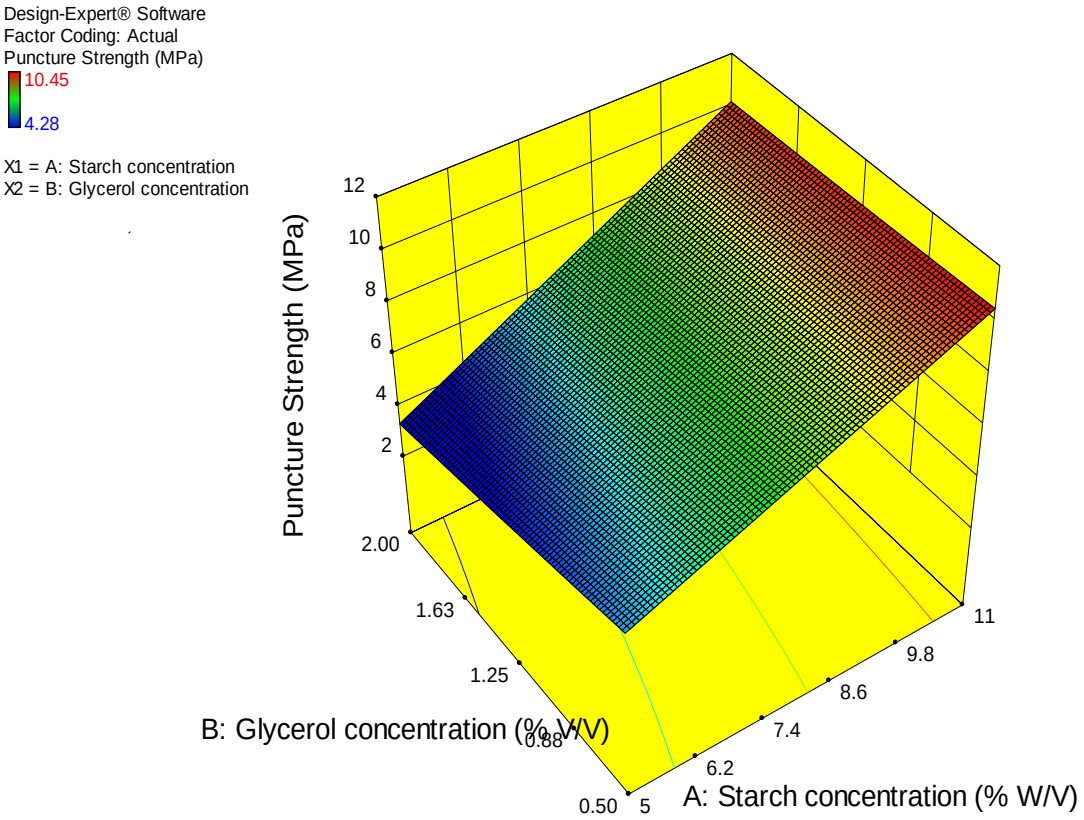


Fig. 4.12 (a) Response surface plot for puncture strength of potato starch biodegradable plastic

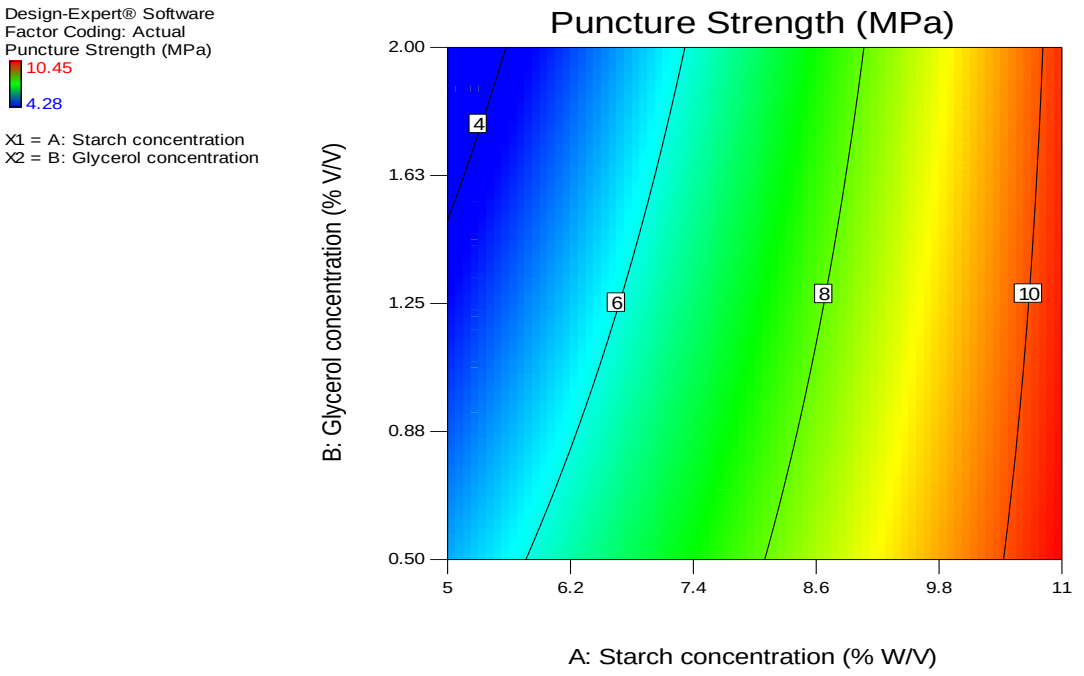


Fig. 4.12 (b) Contour plot for puncture strength of potato starch biodegradable plastic

4.5 Physico-chemical properties of rice starch biodegradable plastic

The different physico-chemical properties of rice starch biodegradable plastic were analysed and studied viz., moisture content, transparency, water absorption capacity and water vapour permeability were carried out as per methods described in Chapter III. The explanation on effect of different independent variables on response parameters and their graphical presentation are given here under.

4.5.1 Effect of starch and glycerol concentration on moisture content of rice starch based biodegradable plastic

Moisture content of rice starch film was ranged from 14.53 to 24.12%. The maximum moisture content was observed for the combination 11 g starch concentration and 1.25 ml glycerol concentration and minimum moisture content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on moisture content of rice starch biodegradable plastic is presented in Table 4.7. While the response surface curves and contour plots of moisture content of rice starch biodegradable plastic are shown in the Fig. 4.13.

The response surface curve of variation in the moisture content of rice starch biodegradable plastic as a function of starch concentration (X_1) and glycerol concentration (X2) is shown in Fig 4.13(a). It represents the interactive effect of starch concentration and glycerol concentration on the, moisture content of rice potato starch film. The contour plot for moisture content of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_1) is presented in the Fig. 4.13(b) which indicated the increase in moisture content as the starch concentration was increased up to maximum level and increase in glycerol concentration up to maximum level. At this combination, moisture content of potato starch biodegradable plastic film was predicted 25 %. The result was agreement with the results reported by Kibar and Us (2013). Similar results were reported by Buso-Rios *et al.* (2020) in purple sweet potato starch film and Talja *et al*. (2008) in potato starch film.

The regression analysis and ANOVA results for the moisture content of rice starch plastic are shown in the Table 4.6. It can be seen from the table, that starch concentration and glycerol concentration showed positive linear effect which there were significant at $p<0.001$. Whilst, the interaction effect of starch and glycerol concentration was positively non significant and the quadratic effect of starch

Table 4.7 Different physico-chemical properties of rice starch biodegradable plastic.

Std. run	Starch concentration, %	Glycerol concentration,%	Moisture content, %	Transparency %	Water absorption capacity,%	Water vapour permeability, g mm/m^2 day KPa	Tensile strength, MPa	Puncture strength, MPa
1	6.5	0.88	15.23	51.9	161	0.00483	5.96	4.12
2	9.5	0.88	21.56	41.4	182	0.00632	9.87	7.46
3	6.5	1.63	16.12	52.7	164	0.00503	5.48	3.48
4	9.5	1.63	21.56	43.3	187	0.00651	9.26	7.32
5	5	1.25	14.53	53.6	149	0.00426	3.36	2.31
6	11	1.25	24.12	39.2	195	0.00711	11.23	8.63
7	8	0.50	17.85	46.2	166	0.00512	7.87	6.46
8	8	2.00	20.21	48.3	179	0.00612	6.32	4.67
9	8	1.25	18.12	46.6	169	0.00554	6.95	5.86
10	8	1.25	17.96	47.5	172	0.00587	6.89	4.95
11	8	1.25	18.78	47.9	169	0.00557	7.38	5.42
12	8	1.25	18.73	49.1	175	0.00545	7.35	5.36
13	8	1.25	18.62	47.4	171	0.00573	7.22	5.48
14	8	1.25	18.35	48.6	173	0.00565	7.16	5.56

concentration was positively non-significant and quadratic effect of glycerol concentration was negatively non-significant on moisture content.

The derived model, giving the empirical relation between the moisture content and the test variables in coded units, was obtained as under:

Moisture Content = $+18.3 +2.58\times A +0.47\times B -0.22\times AB +0.22\times A^2 +0.15\times B^2$

The calculated F-value for moisture content (73.85) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for moisture content was fitted and reliable. The R^2 value and Adj- R^2 value for the moisture content were 0.9788 and 0.9655 respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8480) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for moisture content. The small value of coefficient of variation (2.55 %) for moisture content explained that the experimental results were precise and reliable (Table 4.8).

4.5.2. Effect of starch and glycerol concentration on transparency of rice starch based biodegradable plastic

Transparency of rice starch film was ranged from 39.20 to 53.6 %. The maximum transparency was observed for the combination of 5 g starch concentration and 1.25 ml glycerol concentration and minimum transparency was found for the combination of 11 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on transparency of rice starch film is presented in Table 4.7 while the response surface curves and contour plots of transparency of rice starch biodegradable plastic films are shown in the Fig. 4.14.

The response surface curve of variation in the transparency of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.14(a). It represents the interactive effect of starch concentration and glycerol concentration on the, transparency of rice starch film. The contour plot for transparency of rice starch biodegradable plastic film as a function plot for transparency of rice starch biodegradable plastic film as a function of starch (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.14(b) which

Table 4.8 Analysis of variance (ANOVA) table and regression coefficients for response surface quadratic model of different physico-chemical properties of rice starch biodegradable plastic.

Source	Moisture content	Transparency	Water absorption capacity	Water vapour permeability	Tensile strength	Puncture strength
Intercept	+18.3	+47.85	+171.68	+5.642E-003	+7.27	+5.47
Linear terms						
A(X_1)	+2.58***	-4.06***	+10.17***	+7.225E-004***	+1.9***	+1.65***
B(X_2)	+0.47**	-0.57*	+ 2.83**	+1.992E-004*	-0.35*	-0.36**
Interaction terms						
AB (X_1 X_2)	-0.22	+0.28	+0.50	-2.500E-006	-0.032	+0.13
Quadratic terms						
A^2(X_1^2)	+0.22	-0.36	+1.022*	+1.335E-005	+0.048	+0.011
B^2(X_2^2)	+0.15	-0.15	+ 0.27	-2.904E-006	-2.132E-003	+0.035
Indicators for model fitting						
R^2	0.9788	0.9575	0.9733	0.9742	0.9822	0.9781
Adj-R^2	0.9655	0.9309	0.9566	0.8581	0.9710	0.9644
Pred-R^2	0.8480	0.7346	0.9053	0.8118	0.8984	0.8914
Adeq Precision	33.04	23.22	31.05	29.53	36.41	32.50
F-value	73.85	36.01	58.27	60.40	88.11	71.39
Lack of fit	NS	NS	NS	NS	NS	NS
C.V. %	2.55	2.25	1.25	2.64	4.48	5.64

A or X_1= Starch Concentration, B or X_1 = Glycerol Concentration, ***Significant at p<0.001, **Significant at p<0.01,*Significant at p<0.05, NS = Non-significant

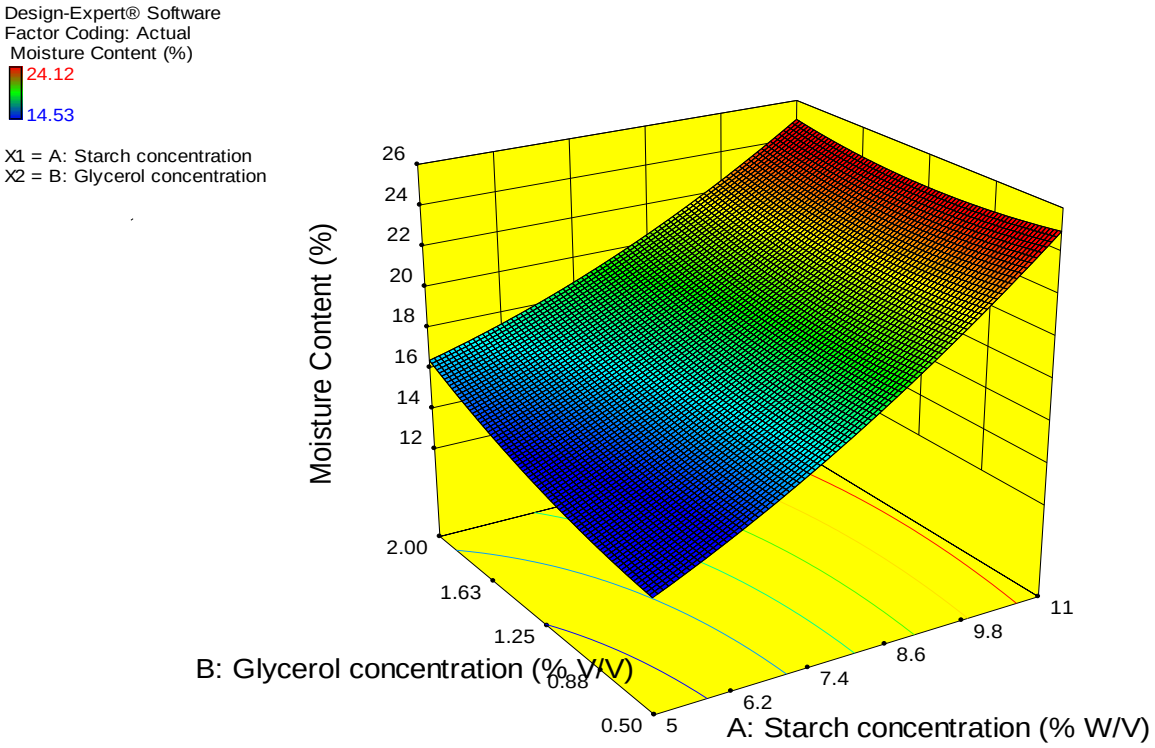


Fig. 4.13(a) Response surface plot for moisture content of rice starch biodegradable plastic

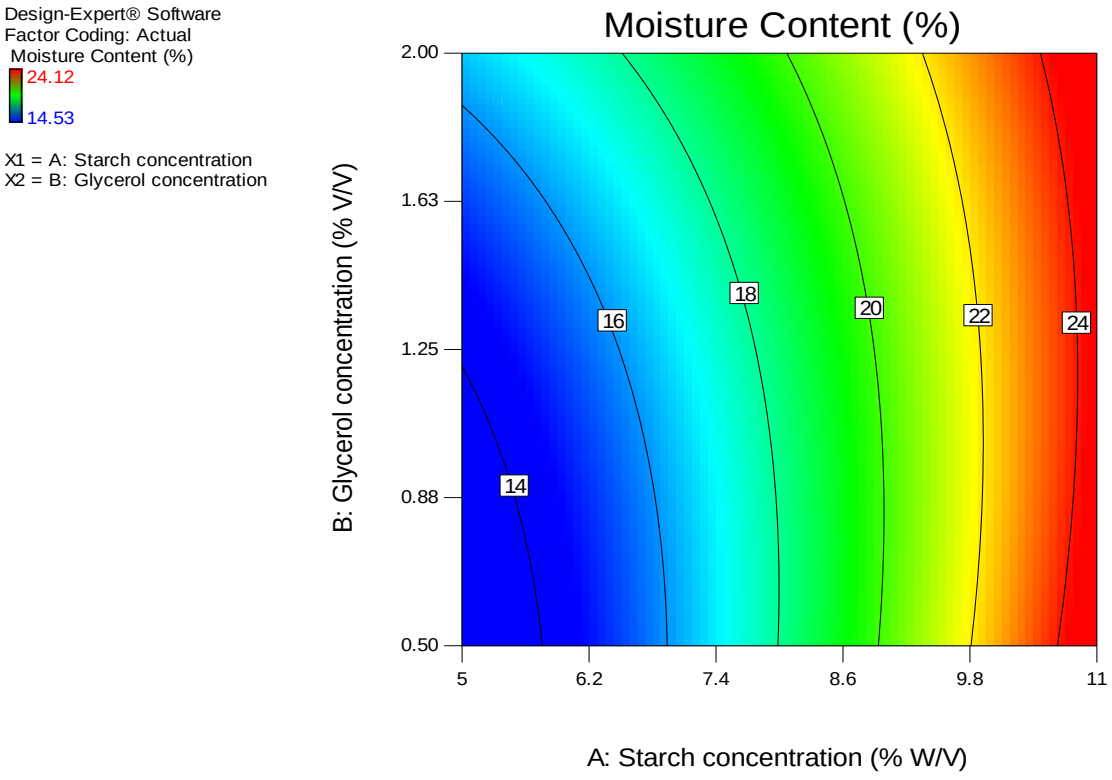


Fig. 4.13(b) Contour plot for moisture content of rice starch biodegradable plastic

indicated the increase in transparency as the starch concentration was decreased up to minimum level and transparency was increased with an increase in glycerol concentration up to 1.24 ml then further increase in glycerol concentration transparency was decreased. Transparency at the combination of 5 g starch concentration and 1.24 ml glycerol concentration may be observed 54.50 %. The result was agreement with the result reported by Dai *et al*. (2010) in corn starch film. Similar result was found by Khairunnisa *et al.* (2018) in film made from alginate.

The regression analysis and ANOVA results for the transparency of rice starch film are shown in the Table 4.8. It can be seen from the table, that starch concentration showed negative linear effect on transparency which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on transparency which was non-significant. Whilst, the interaction effect of starch concentration and glycerol concentration was positively non-significant and the quadratic effect of starch and glycerol concentration showed negatively non-significant.

The derived model, giving the empirical relation between the bulk density and the test variables in coded units, was obtained as under:

$$\text{Transparency} = +47.85 - 4.06 \times A - 0.57 \times B + 0.28 \times AB - 0.36 \times A^2 - 0.15 \times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for transparency (36.01) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for transparency was fitted and reliable. The R^2 value and Adj- R^2 value for the transparency were 0.9575 and 0.9309, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.7346) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for transparency. The small value of coefficient of variation (2.25 %) for transparency explained that the experimental results were precise and reliable (Table 4.8).

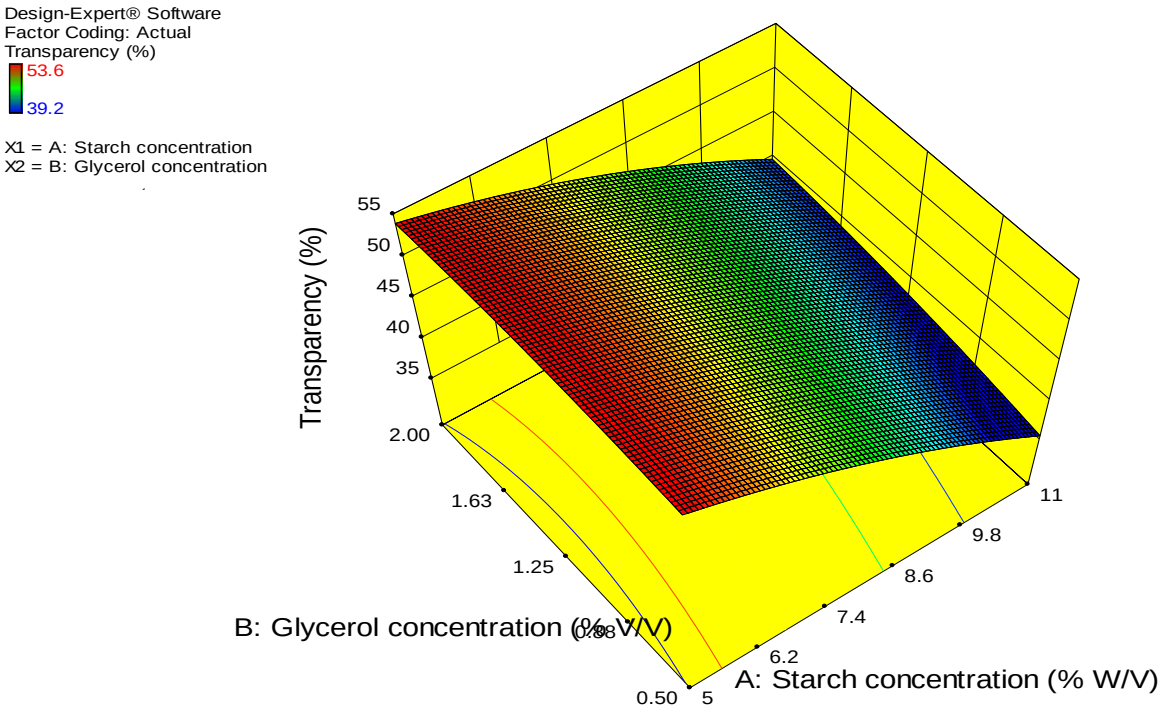


Fig. 4.14(a) Response surface plot for transparency of rice starch biodegradable plastic

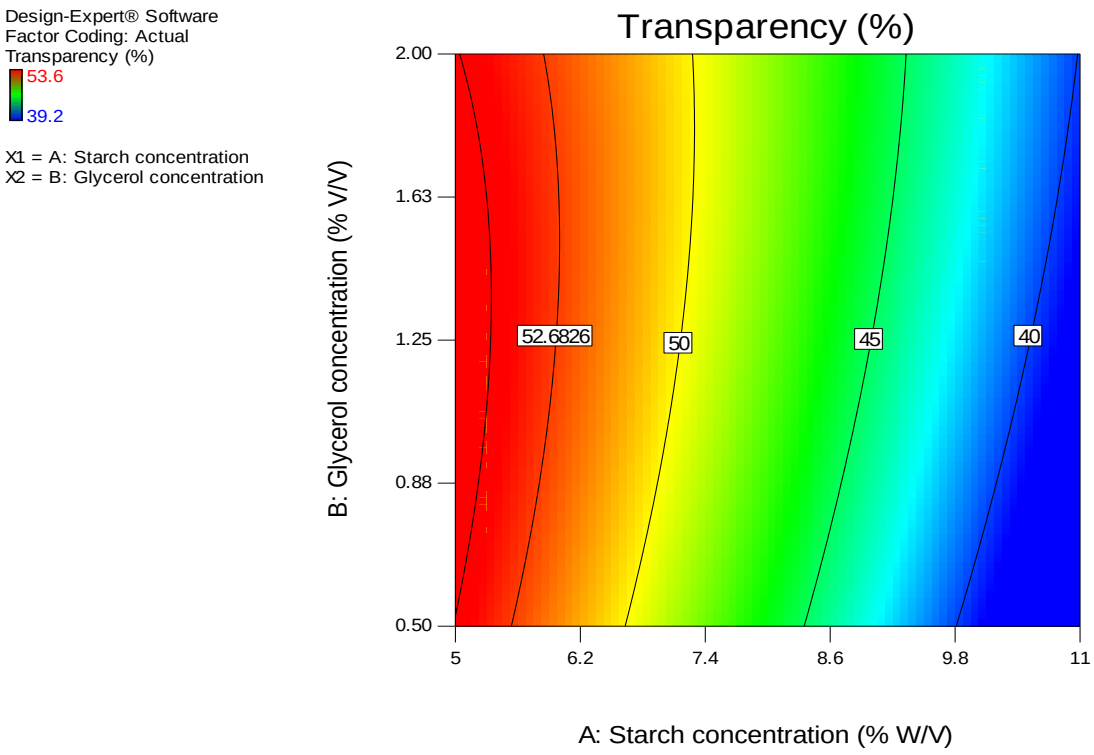


Fig. 4.14(b) Contour plot for transparency of rice starch biodegradable plastic

4.5.3 Effect of starch and glycerol concentration on water absorption capacity of rice starch based biodegradable plastic

Water absorption capacity of rice starch film was ranged from 149 to 195%. The maximum water absorption capacity was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum water absorption capacity content was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on water absorption capacity of rice starch film is presented in Table 4.7 while the response surface curves and contour plots of water absorption capacity of rice starch biodegradable plastic films are shown in the Fig. 4.15.

The response surface curve of variation in the water absorption capacity of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.15(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water absorption capacity of rice starch film. The contour plot for water absorption capacity of potato starch biodegradable plastic film as a function of starch concentration (X_1) ation and glycerol concentration (X_2) is presented in the Fig. 4.15(b) which indicated the increase in water absorption capacity as the starch and glycerol concentration was increased up to maximum level. At this combination, the water absorption capacity of corn starch film was predicted up to 203 %. The result was agreement with result reported by Farahnaky *et al.* (2013) in wheat starch film and similar result was found by Bourtoom and Chinnan (2008a) in rice starch-chitosan film.

The regression analysis and ANOVA results for the water absorption capacity of rice starch film are shown in the Table 4.8. It can be seen from the table, that starch concentration showed positive linear effect on water absorption capacity which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water absorption capacity which was significant at $p<0.01$. Whilst, the interaction effect of starch concentration and glycerol concentration was positively non-significant and the quadratic effect of starch concentration was positively significant at $p<0.05$ and glycerol concentration was positively non-significant on water absorption capacity.

The derived model, giving the empirical relation between the water absorption capacity and the test variables in coded units, was obtained as under:

Water Absorption Capacity = +171.68 +10.17×A+ 2.83×B +0.50×AB +1.022×A^2+ 0.27×B^2
Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for water absorption capacity (58.27) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water absorption capacity was fitted and reliable. The R^2 value and Adj- R^2 value for the water absorption capacity were 0.9733 and 0.9566, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.9053) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for water absorption capacity. The small value of coefficient of variation (1.25 %) for water absorption capacity explained that the experimental results were precise and reliable (Table 4.8).

4.5.4 Effect of starch and glycerol concentration on water vapor Permeability of rice starch based biodegradable plastic

Water vapor permeability of rice starch film was ranged from 0.00426 to 0.00711 g.mm/m^2dayKPa The maximum water vapor permeability was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on water vapor permeability of rice starch film is presented in Table 4.7 while the response surface curves and contour plots of water vapor permeability of rice starch biodegradable plastic films are shown in the Fig. 4.16.

The response surface curve of variation in the water vapor permeability of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.16(a). It represents the interactive effect of starch concentration and glycerol concentration on the, water vapor permeability of rice starch film. The contour plot for water vapor permeability of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.16(b) which indicated the decrease the starch and glycerol concentration, water vapor permeability was decreased up to minimum level. At this combination, the water vapor permeability of rice starch film was predicted up to 0.00384 g.mm/m^2dayKPa.

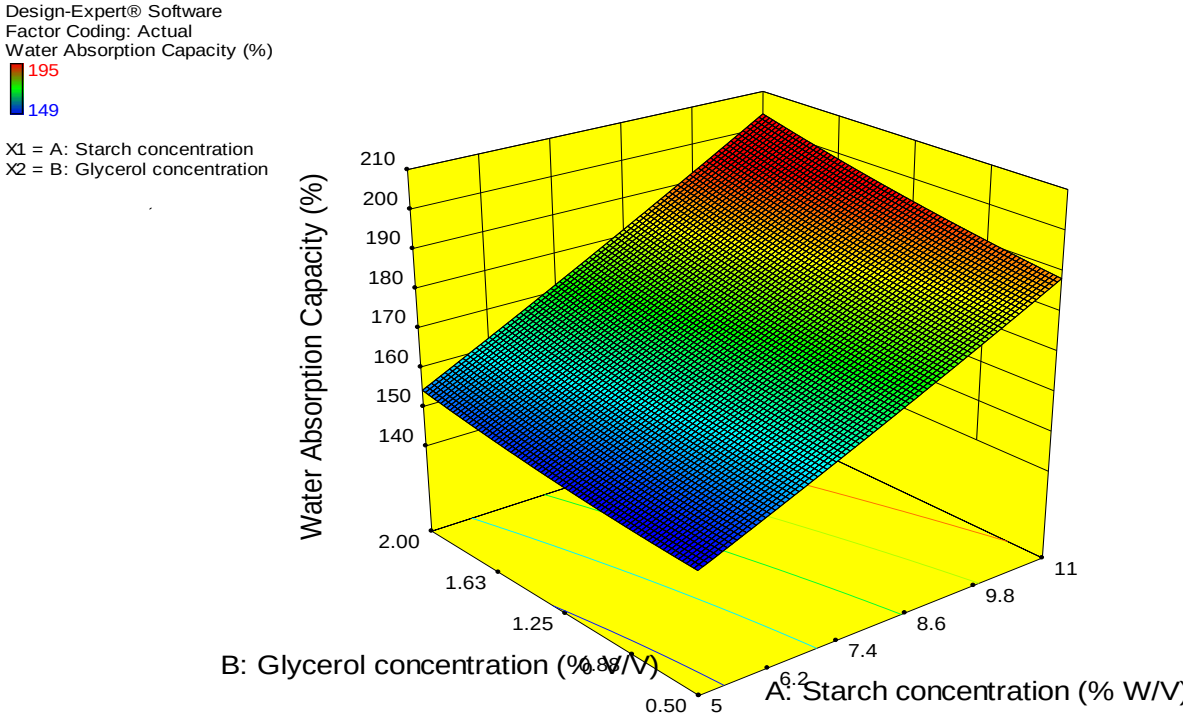


Fig. 4.15(a) Response surface plot for water absorption capacity of rice starch biodegradable plastic

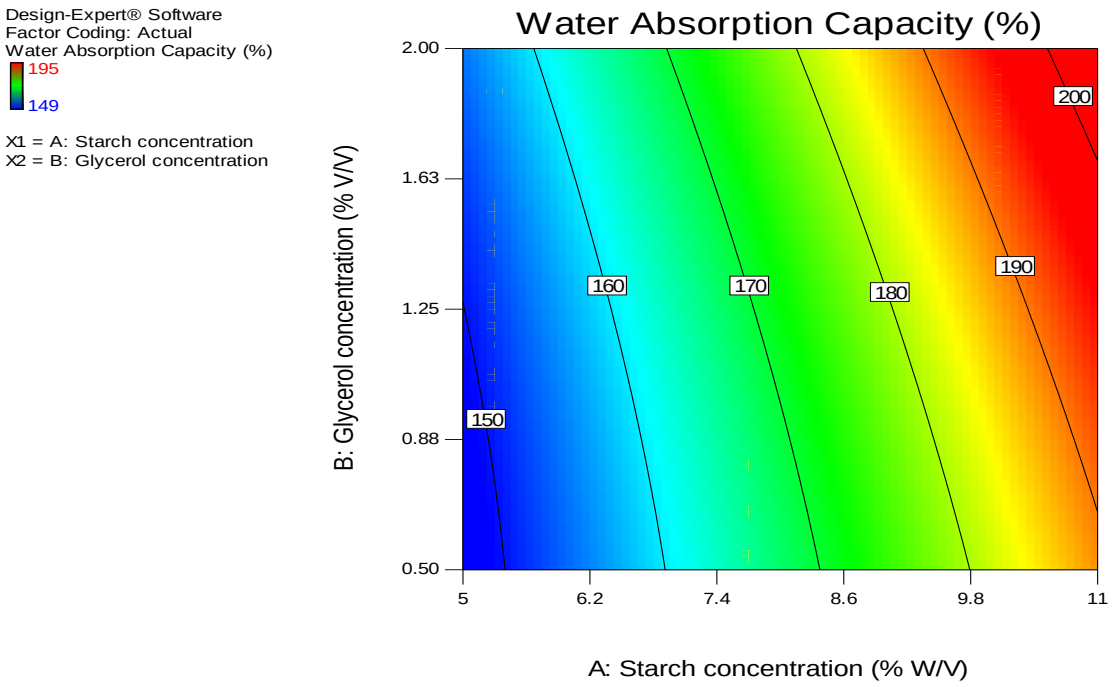


Fig. 4.15(b) Contour plot for water absorption capacity of rice starch biodegradable plastic

The result was agreement with the result reported by Ghasemlou *et al.* (2013) in corn starch films incorporated with plant essential oils. Similar result was found by Farahnaky *et al*. (2013) in films made of wheat starch and glycerol and Muhammed *et al.* (2015) in sugar palm starch film incorporated with glycerol and sorbitol.

The regression analysis and ANOVA results for the water vapor permeability of rice starch film are shown in the Table 4.8. It can be seen from the table, that starch concentration showed positive linear effect on water vapor permeability which there were significant at $p<0.001$ also glycerol concentration showed positive linear effect on water vapor permeability which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration was positively non-significant and glycerol concentration was nagetively non-significant on water vapor permeability.

The derived model, giving the empirical relation between the water vapour permeability and the test variables in coded units, was obtained as under:

$$\text{Water Vapour Permeability} = +5.642E\text{-}003 +7.225E\text{-}004\times A +1.992E\text{-}004\times B -2.500E\text{-}006\times AB +1.335E\text{-}005\times A^2 -2.904E\text{-}006\times B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for water vapor permeability (60.40) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for water vapor permeability was fitted and reliable. The R^2 value and Adj- R^2 value for the water vapor permeability were 0.9742 and 0.8581, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8118) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for water vapor permeability. The small value of coefficient of variation (2.64 %) for water vapor permeability explained that the experimental results were precise and reliable (Table 4.8).

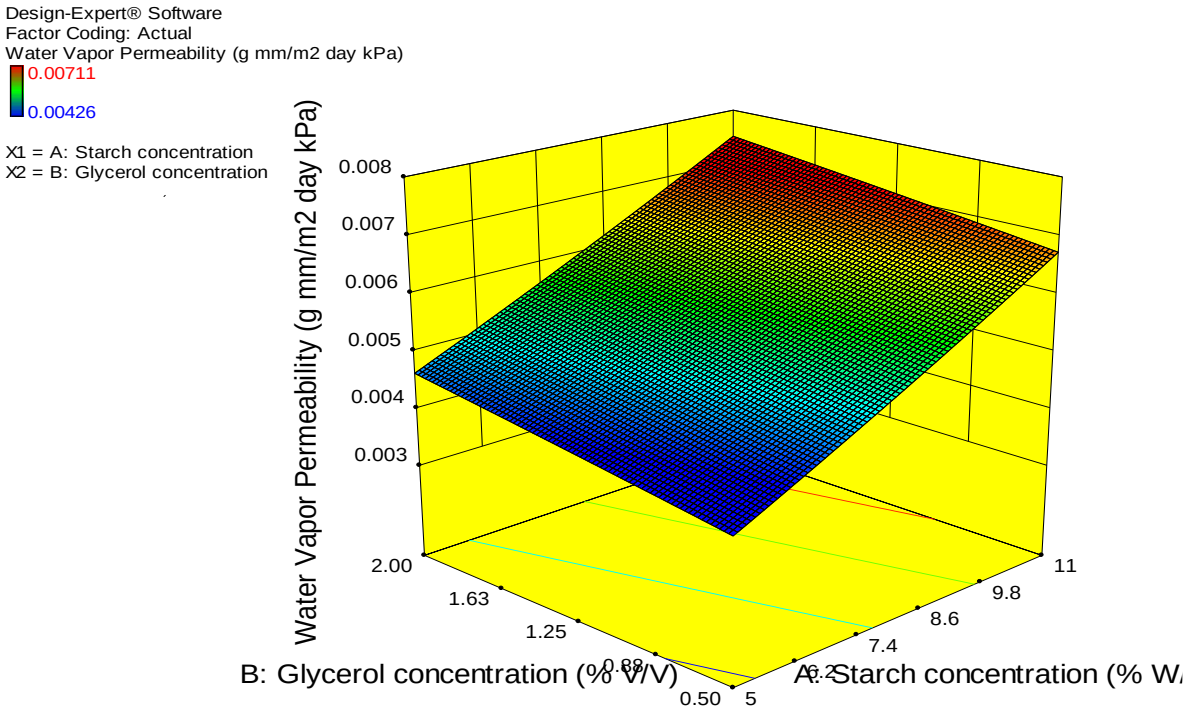


Fig. 4.16(a) Response surface plot for water vapor permeability of rice starch biodegradable plastic

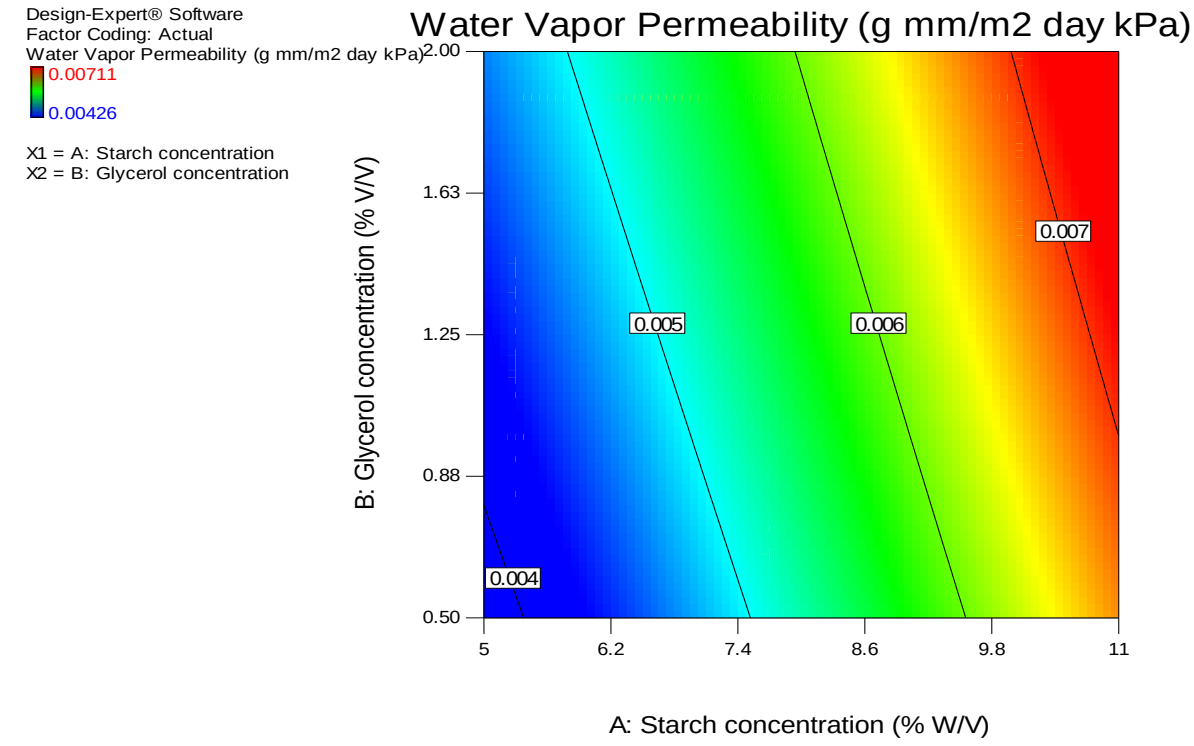


Fig. 4.16(b) Contour plot for water vapor permeability of rice starch biodegradable plastic

4.5.5 Effect of starch and glycerol concentration on tensile strength of rice starch based biodegradable plastic

Tensile strength of rice starch film was ranged from 3.36 to 11.23 MPa. The maximum tensile strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on tensile strength of rice starch film is presented in Table 4.7 while the response surface curves and contour plots of tensile strength of rice starch biodegradable plastic films are shown in the Fig. 4.17.

The response surface curve of variation in the tensile strength of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.17(a). It represents the interactive effect of starch concentration and glycerol concentration on the, tensile strength of rice starch film. The contour plot for tensile strength of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.17(b) which indicated the increase the tensile strength, starch concentration was increased up to maximum level and tensile strength decrease with increases the glycerol concentration. At this combination, the tensile strength of potato starch film was predicted up to 12.17 MPa. The result was agreement with the result reported by Muhammed *et al.* (2015) in sugar palm starch and Muscat *et al.* (2012). Similar result was reported by Sonam and aditya (2016) in potato starch film, increase in the plasticizer concentration causes a reduction of the tensile strength due to the decrease in the intermolecular interactions.

The regression analysis and ANOVA results for the tensile strength of rice starch film are shown in the Table 4.8. It can be seen from the table, that starch concentration showed positive linear effect on tensile strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on tensile strength which was significant at $p<0.05$. Whilst, the interaction effect of starch concentration and glycerol concentration was negatively non-significant and the quadratic effect of starch concentration was positively non-significant and glycerol concentration was negatively non-significant on tensile strength.

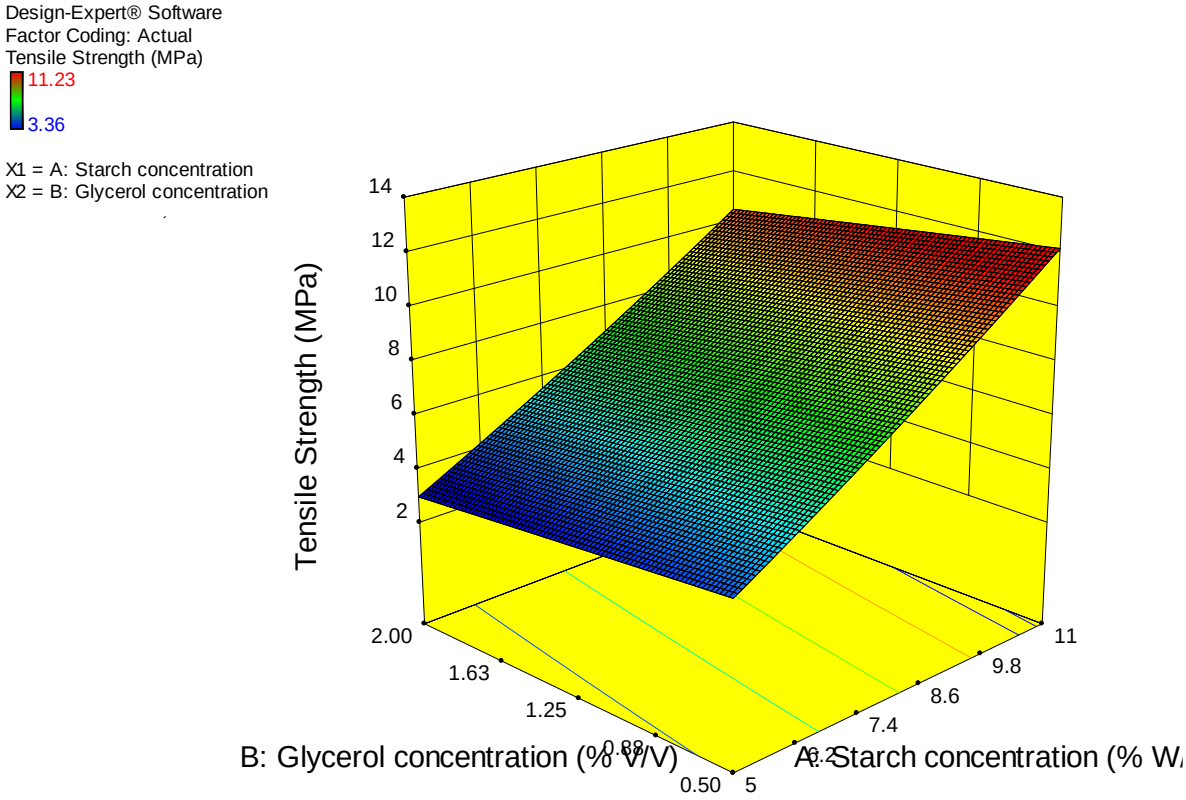


Fig. 4.17(a) Response surface plot for tensile strength of rice starch biodegradable plastic

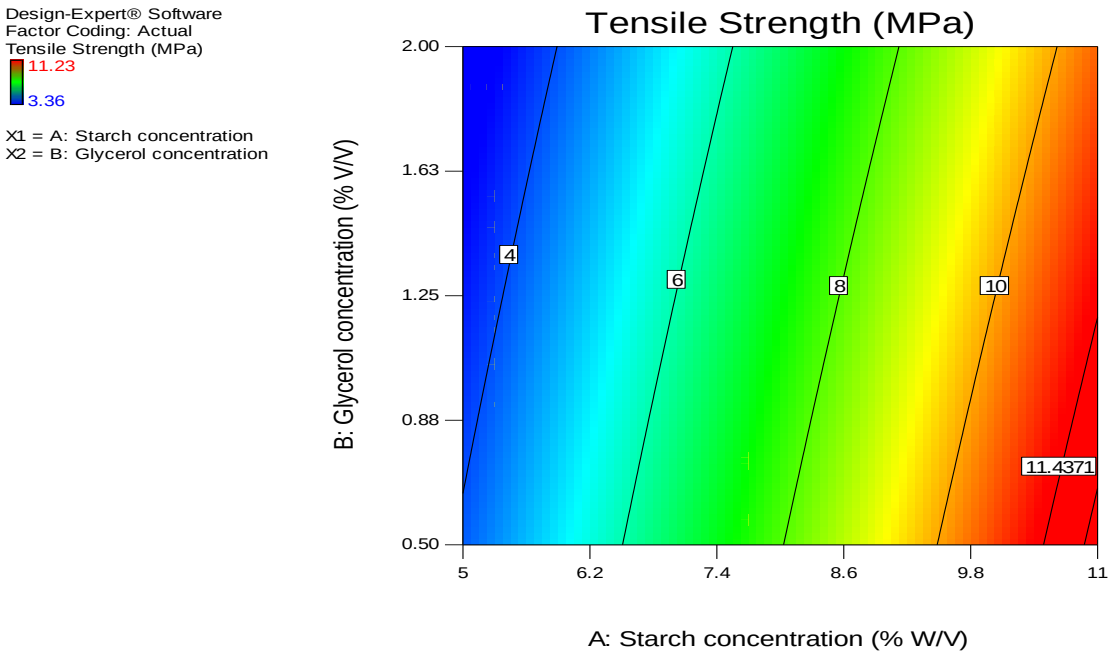


Fig. 4.17(b) Contour plot for tensile strength rice starch biodegradable plastic

The derived model, giving the empirical relation between the tensile strength and the test variables in coded units, was obtained as under:

Tensile Strength =+7.27 +1.9×A -0.35×B -0.032×AB +0.048×A^2-2.132E-003× B^2

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for tensile strength (88.11) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for tensile strength was fitted and reliable. The R^2 value and Adj- R^2 value for the tensile strength were 0.9822 and 0.9710, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8984) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for tensile strength. The small value of coefficient of variation (4.48 %) for tensile strength explained that the experimental results were precise and reliable (Table 4.8)

4.5.6. Effect of starch and glycerol concentration on puncture strength of rice starch based biodegradable plastic

Puncture strength of rice starch film was ranged from 2.31 to 8.63 MPa. The maximum puncture strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration. The effect of starch and glycerol concentration on puncture strength of rice starch film is presented in Table 4.7 while the response surface curves and contour plots of tensile strength of rice starch biodegradable plastic films are shown in the Fig. 4.18.

The response surface curve of variation in the puncture strength of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is shown in Fig 4.18(a). It represents the interactive effect of starch concentration and glycerol concentration on the, puncture strength of rice starch film. The contour plot for puncture strength of rice starch biodegradable plastic film as a function of starch concentration (X_1) and glycerol concentration (X_2) is presented in the Fig. 4.18(b) which indicated the increase the puncture strength, starch concentration was increased up to maximum level and puncture strength decrease with increases the glycerol concentration. At this combination, the puncture strength

of rice starch film was predicted up to 9.17 MPa. The result was agreement with the result reported by Sonam and Aditya (2016) in potato starch film. Similar result was reported by Mali *et al*. (2005) in corn, cassava, and yam starch films.

The regression analysis and ANOVA results for the puncture strength of rice starch film are shown in the Table 4.8. It can be seen from the table, that starch concentration showed positive linear effect on puncture strength which there were significant at $p<0.001$ glycerol concentration showed negative linear effect on puncture strength which was significant at $p<0.01$. Whilst, the interaction effect of starch and glycerol concentration was positively non-significant and the quadratic effect of starch and glycerol concentration was positively non-significant on puncture strength.

The derived model, giving the empirical relation between the puncture strength and the test variables in coded units, was obtained as under:

$$\text{Puncture Strength} = +5.47 + 1.65 \times A - 0.36 \times B + 0.13 \times AB + 0.011 \times A^2 + 0.035\ B^2$$

Where, A and B are the coded factors of starch and glycerol concentration, respectively.

The calculated F-value for puncture strength (71.39) was significant at $p<0.001$. At the same time, it possessed non-significant lack of fit ($p>0.05$). These values indicated that the model for puncture strength was fitted and reliable. The R^2 value and Adj- R^2 value for the puncture strength were 0.9781 and 0.9644, respectively, which were higher than the 0.8, indicating the adequacy, good fit and high significance of the model. The Pred- R^2 (0.8914) was in reasonable agreement with the Adj- R^2. The high Adequate Precision value (>4) again supported the significance of the model for puncture strength. The small value of coefficient of variation (5.64 %) for puncture strength explained that the experimental results were precise and reliable (Table 4.8).

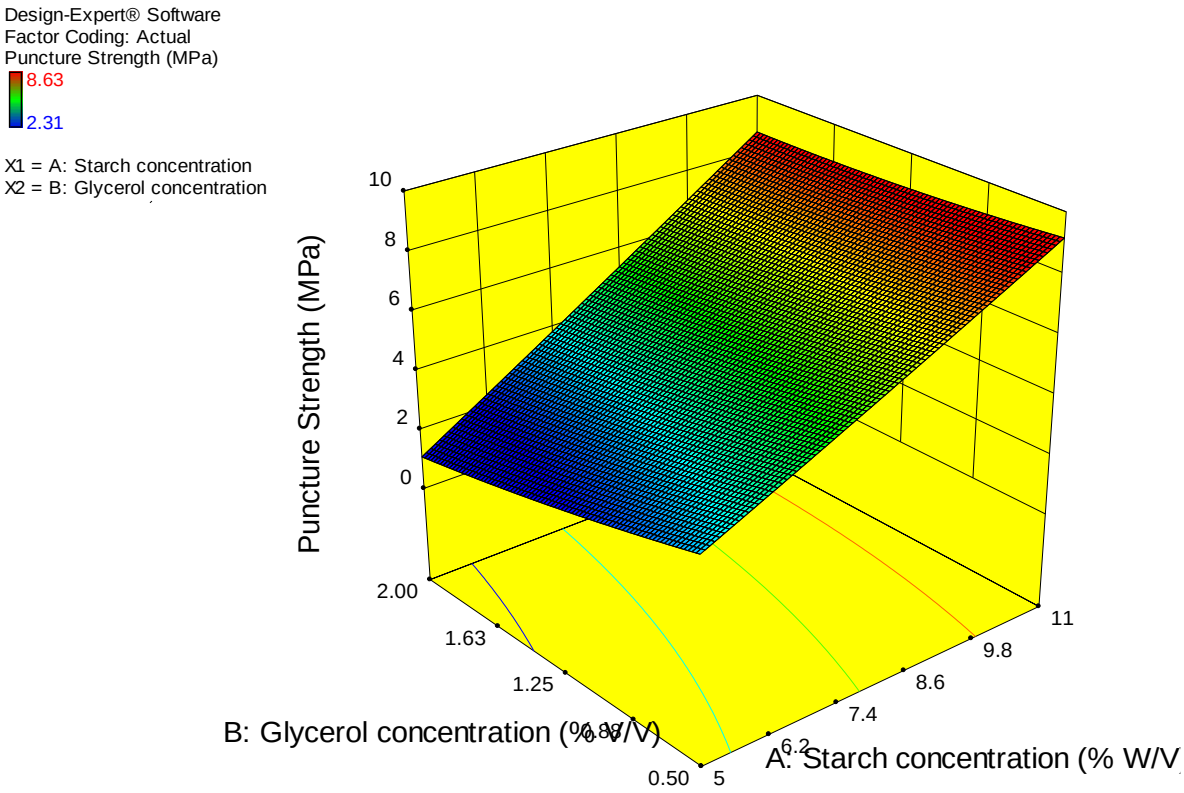


Fig. 4.18(a) Response surface plot for puncture strength of rice starch biodegradable plastic

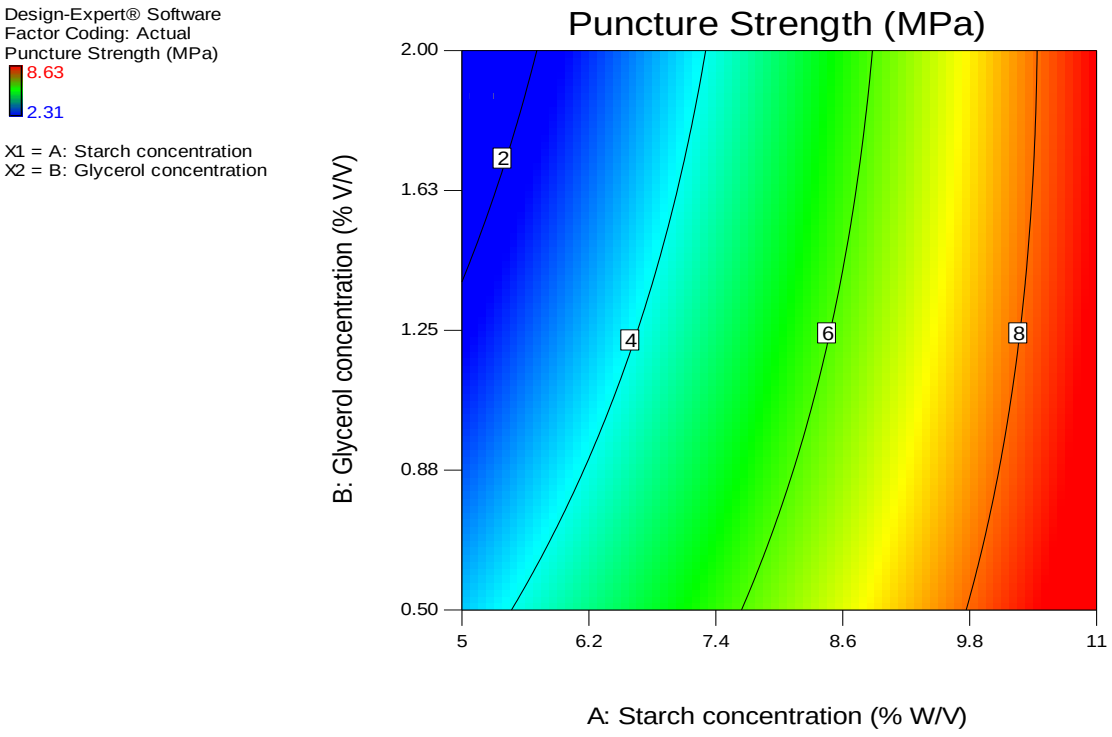


Fig. 4.18(b) Contour plot for puncture strength of rice starch biodegradable plastic

4.6 Optimization and validation of process variables

The optimum condition for the development of starch based biodegradable plastic was determined by the numerical optimization technique, using Design Expert software version 10 (State-Ease Inc., Minneapolis, MN, USA). The main criteria applied for constraints optimization in the study were: (a) moisture content: minimum (b)transparency: maximum (c)water absorption capacity: minimum (d) water vapour permeability : minimum (e) tensile strength : maximum (f) puncture strength : maximum. Accordingly, the goals that were set for variables and responses to obtain the best combination are illustrated in the Table 4.9. All the independent variables and responses were given an equal importance during optimization process. Under these constraints, the optimum treatment conditions for corn starch film were found to be, 7.090 g starch concentration and 0.5 ml glycerol concentration. The analysis showed that at this combination of starch and glycerol concentration, it would be possible to produce a corn starch based biodegradable plastic film with a moisture content of 18.38 %, transparency 68.36, water absorption capacity 145.93%, water vapour permeability 0.002 g mm/m^2 day KPa, tensile strength 10.84 MPa and puncture strength 8.18 MPa with a desirability of 0.71 (Table 4.9). The optimum treatment conditions for potato starch film were found to be, 7.1 g starch concentration and 0.5 ml glycerol concentration. The analysis showed that at this combination of starch and glycerol concentration, it would be possible to produce a potato starch based biodegradable plastic film with a moisture content of 18.52 %, transparency 65.47%, water absorption capacity 159 %, water vapour permeability 0.004 g mm/m^2 day KPa, tensile strength 8.90 MPa and puncture strength 7.17 MPa with a desirability of 0.68 (Table 4.10). Under these constraints, the optimum treatment conditions for rice starch film were found to be, 6.59 g starch concentration and 0.5 ml glycerol concentration. The analysis showed that at this combination of starch and glycerol concentration, it would be possible to produce a rice starch based biodegradable plastic film with a moisture content of 15.39 %, transparency 50.08, water absorption capacity 157.90 %, water vapour permeability 0.005 g mm/m^2 day KPa, tensile strength 6.11 MPa and puncture strength 5.03 MPa with a desirability of 0.65 (Table 4.11). Using these optimized conditions the experiments were again conducted to find the variation in the different response variables. The results revealed that the experimental values of conducted experiments were very close to the predicted values (Table 4.9 to 4.11). This implied that there was a high degree of fit between the

observed and predicted values from the regression models and each model was quite accurate in prediction. The closeness of the observed and predicted responses indicated the validity of developed model.

4.6.1 Improved method suggested for development of starch (potato, corn & rice) based biodegradable plastic

As a result of studies conducted on various aspects of development of starch based biodegradable plastic an improved process for production of better quality of biodegradable plastic is suggested through a flow diagram given in Fig. 4.19.

Table 4.9 Constraints, criteria and output for numerical optimization of corn starch plastic

Variables				
Constraint	**Goal**	**Importance**	**Optimum value**	**Experimental value**
(1)	(2)	(3)	(4)	(5)
Starch Concentration (g)	In the range	3	7.090	7
Glycerol concentration (ml)	In the range	3	0.5	0.5

Responses					
Constraint	**Goal**	**Importance**	**Predicted value**	**Experimental value**	**Deviation (%)**
(1)	(2)	(3)	(4)	(5)	(6)
Moisture content	Minimum	3	18.38	17.56	4.66
Transparency	Maximum	3	68.36	67.25	1.65
Water absorption capacity	Minimum	3	145.93	147.21	0.87
Water vapor permeability	Minimum	3	0.0020	0.0018	1.1
Tensile strength	Maximum	3	10.84	11.24	3.69
Puncture strength	Maximum	3	8.18	8.05	1.61

Table 4.10 Constraints, criteria and output for numerical optimization of potato starch plastic

Variables				
Constraint	**Goal**	**Importance**	**Optimum value**	**Experimental value**
(1)	(2)	(3)	(4)	(5)
Starch Concentration (g)	In the range	3	7.13	7.1
Glycerol concentration (ml)	In the range	3	0.5	0.5

Responses					
Constraint	**Goal**	**Importance**	**Predicted value**	**Experimental value**	**Deviation (%)**
(1)	(2)	(3)	(4)	(5)	(6)
Moisture content	Minimum	3	18.52	17.69	4.04
Transparency	Maximum	3	65.47	64.10	2.09
Water absorption capacity	Minimum	3	159.04	163.2	2.54
Water vapor permeability	Minimum	3	0.004	0.0039	2.5
Tensile strength	Maximum	3	8.90	9.30	4.30
Puncture strength	Maximum	3	7.17	7.05	1.67

Table 4.11 Constraints, criteria and output for numerical optimization of rice starch plastic

Variables				
Constraint	**Goal**	**Importance**	**Optimum value**	**Experimental value**
(1)	(2)	(3)	(4)	(5)
Starch Concentration (g)	In the range	3	6.59	6.6
Glycerol concentration (ml)	In the range	3	0.5	0.5

Responses					
Constraint	**Goal**	**Importance**	**Predicted value**	**Experimental value**	**Deviation (%)**
(1)	(2)	(3)	(4)	(5)	(6)
Moisture content	Minimum	3	15.39	14.9	3.29
Transparency	Maximum	3	50.08	52.1	4.03
Water absorption capacity	Minimum	3	157.9	156.2	1.09
Water vapor permeability	Minimum	3	0.005	0.0048	4.16
Tensile strength	Maximum	3	6.11	6.35	3.92
Puncture strength	Maximum	3	5.033	4.905	2.61

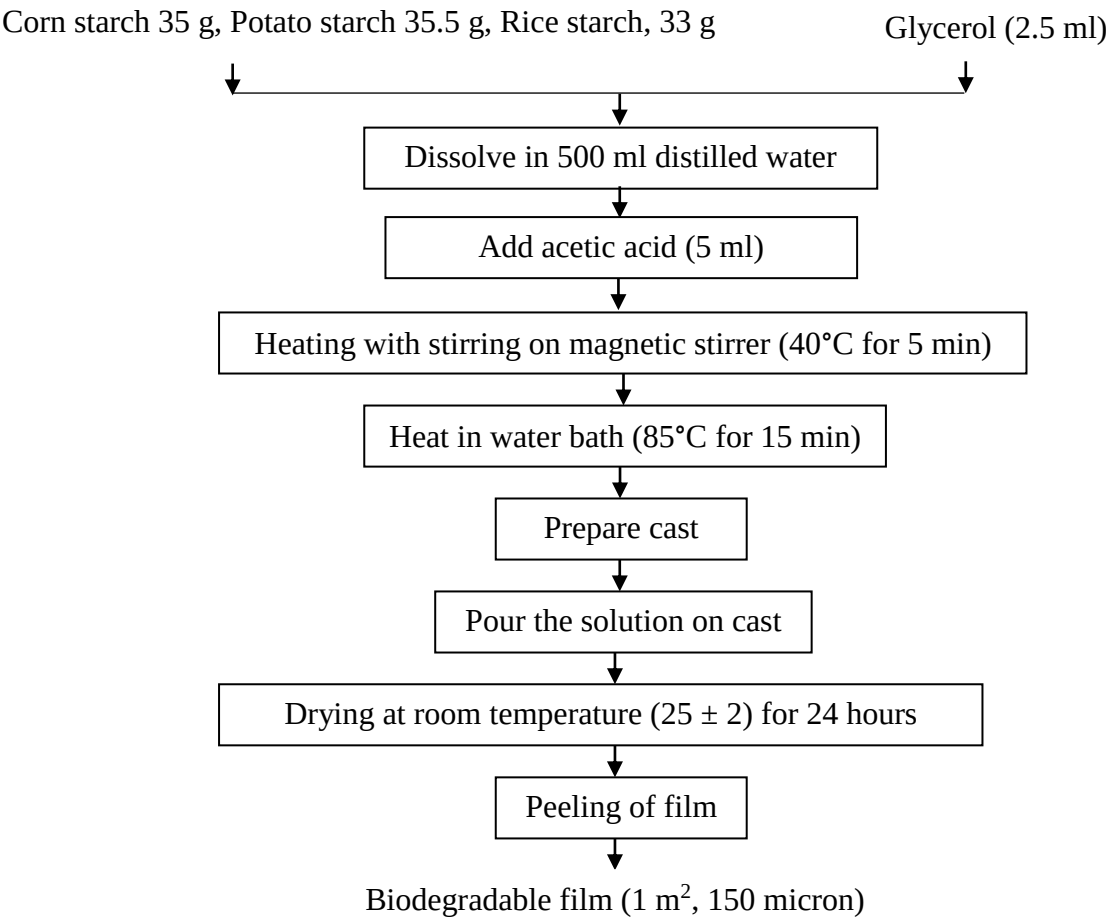


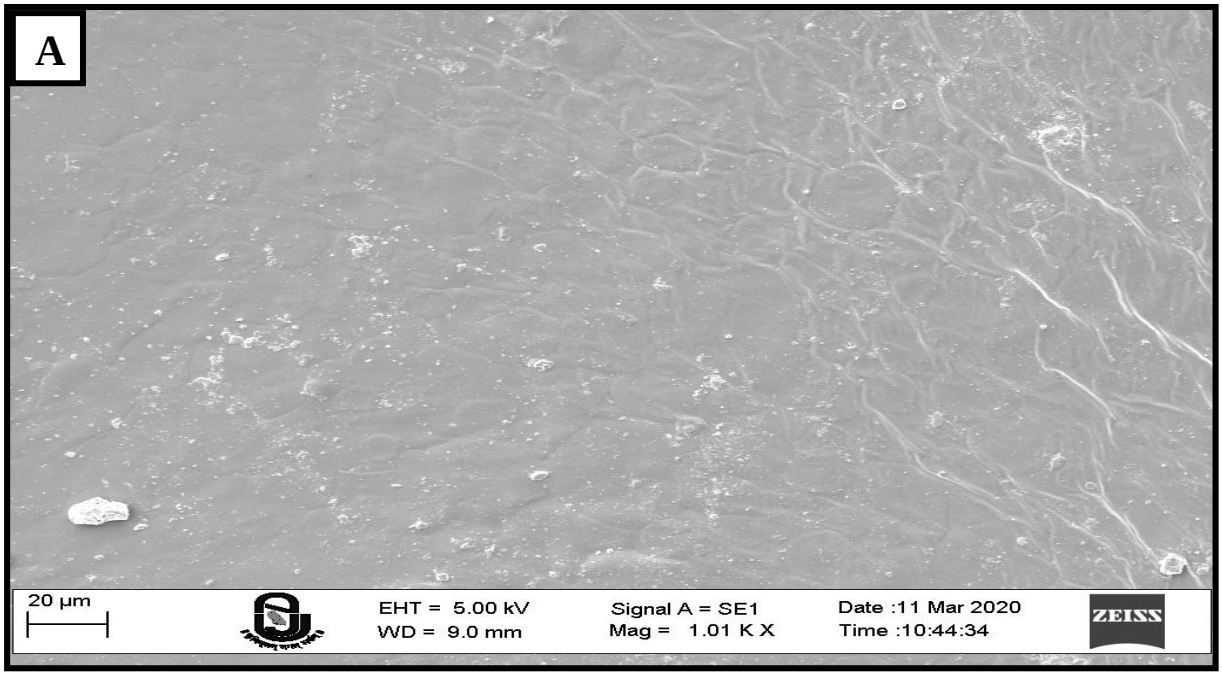
Fig. 4.19 Recommended process flow chart for development of starch based biodegradable plastic

4.7 Surface morphology of developed corn, potato and rice starch plastic

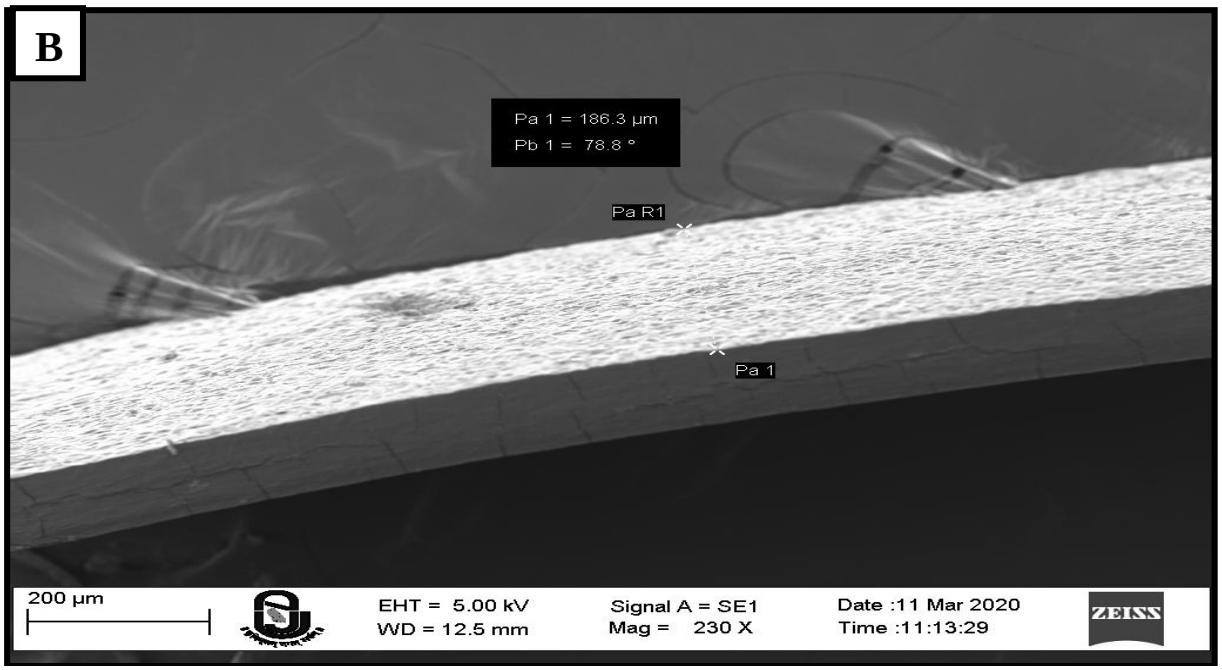
The surface morphology of corn starch, potato starch, and rice starch/glycerol films was studied with SEM was seen in Fig. 4.20, 4.21 and 4.22 respectively. In order to have the conducting impact, the samples were gold plated and the scanning was synchronized with microscopic beam so as to maintain the small size over a large distance relative to the specimen. The resulting images had a great depth of the field. A remarkable three dimensional appearance with high resolution was obtained in case of cross linked matrix.

The corn starch films were smooth and compact as compared to the potato starch and rice starch films, while the potato starch films had several unequalized holes, suggesting that the miscibility and compatibility in each component in potato starch films were increased. The rice starch film contained voids, which indicate that the bubble was trapped in the sample during the casting so, that rice starch film has rougher surface compared to potato and corn starch film. Rice starch caused the cryo-fractured surface to become rougher and more brittle. With increase in glycerol content the surface of the film gets smoother.

From the SEM micrograph, it can be concluded that the corn starch granules dispersed well in the glycerol. This dispersion helps to improve the mechanical properties of the film and shows an agreement with the tensile property results.

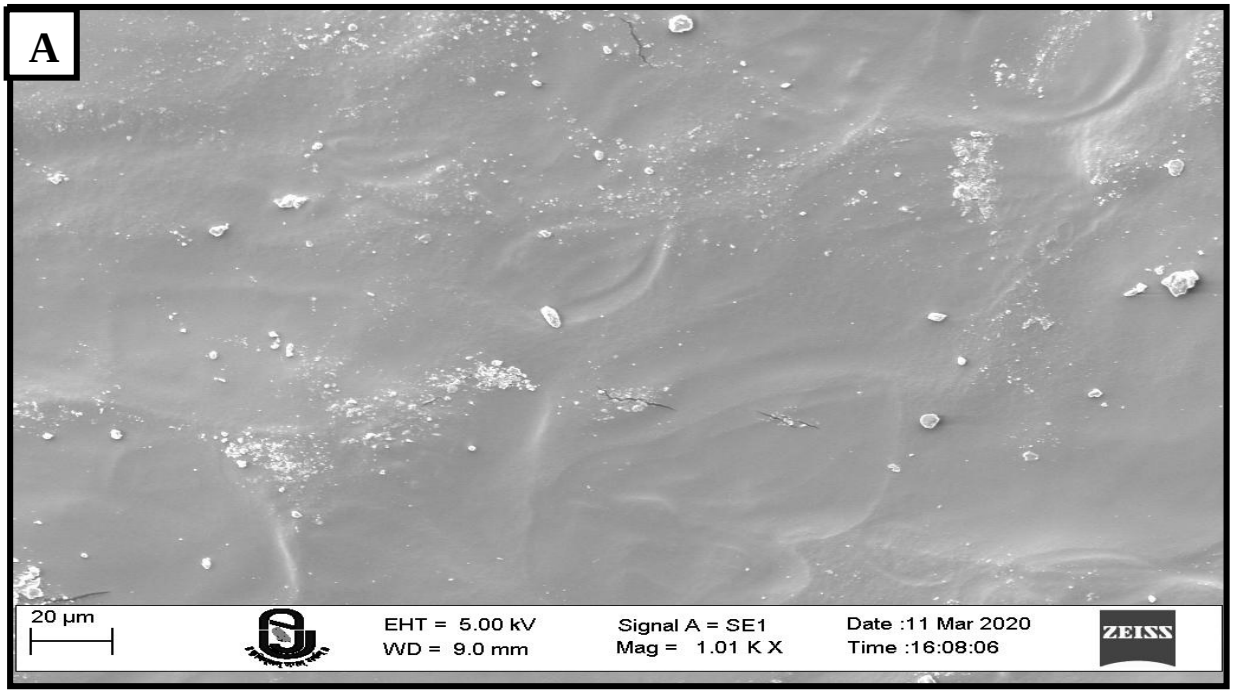


(A) Surface or top view

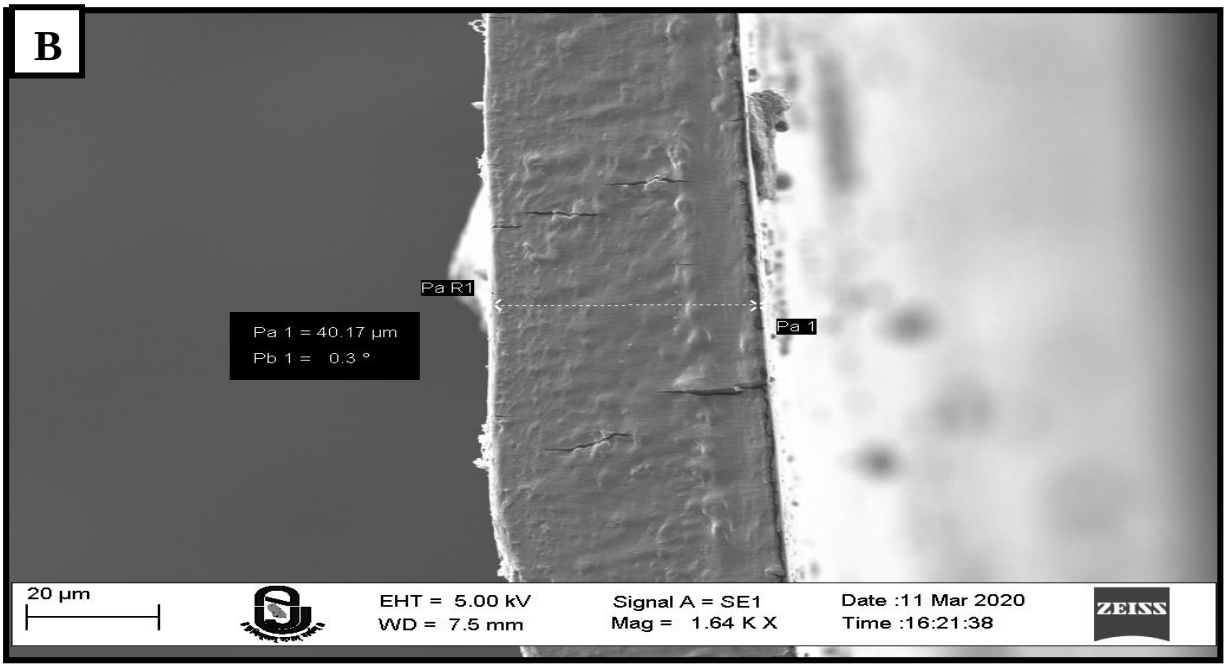


(B) Side view

Fig 4.20 Scanning electron microscopic images of cross-section of corn starch films

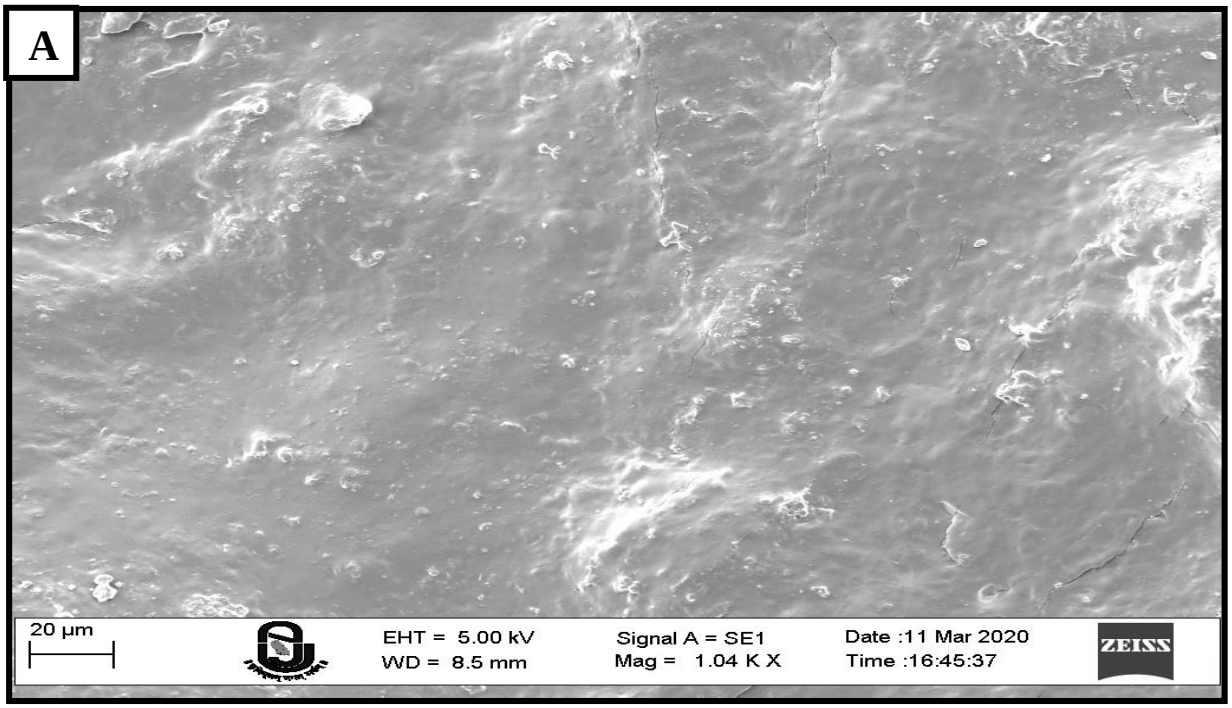


(A) Surface or top view

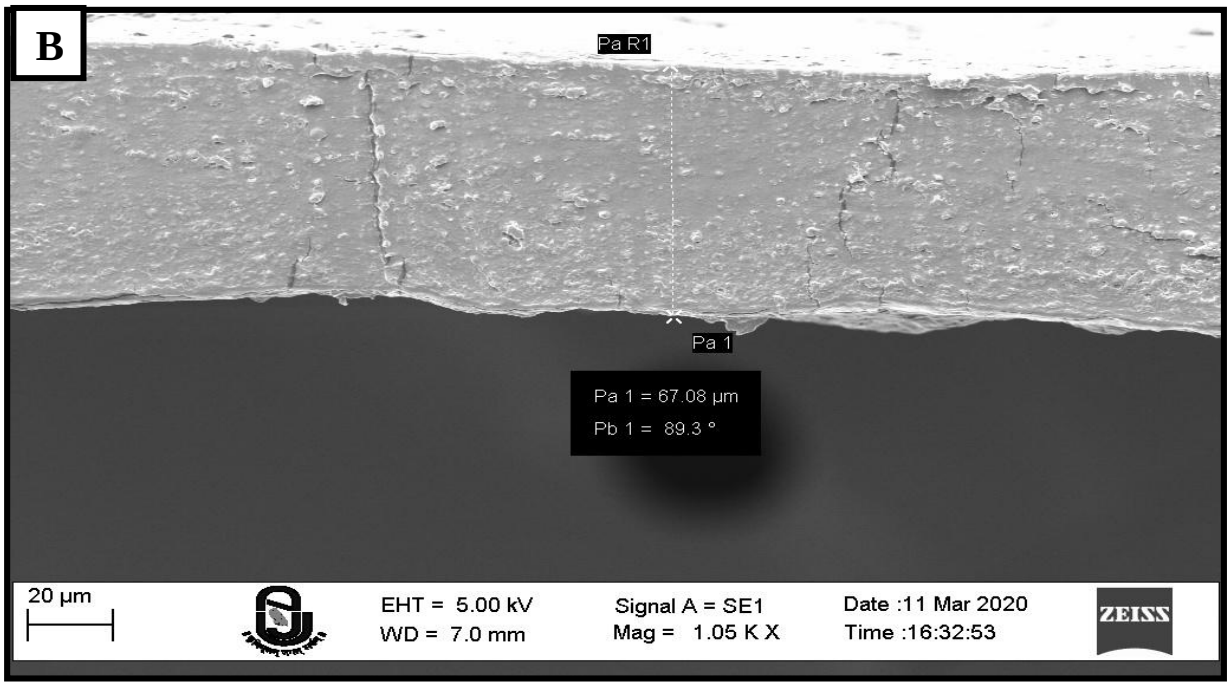


(B) Side view

Fig. 4.21 Scanning electron microscopic images of cross-section of potato starch films



(A) Surface or top view



(B) Side view

Fig. 4.22 Scanning electron microscopic images of cross-section of rice starch film

4.8 Biodegradation properties of developed corn, potato and rice starch biodegradable plastic

The biodegradation properties viz., reduction in weight of developed potato, corn and rice starch based biodegradable film were measured and results were presented in Table 4.6.These samples were then buried in soil for one month as shown in Plate 3.7. In every seven days interval films were taken away from the soil. Later than clean-up through water and exposure to air at room temperature, changes in weight were calculated. Based on these findings, it is obvious that starch based plastic is biodegradable as shown in Plate 3.7. Similar results were reported by Eterigho *et al.,* (2017) for biodegradation of potato starch based plastic film and the results were shown under here.

Biodegradation is the biochemical material conversion process in the water, biomass, carbon dioxide or methane in terms of the action of microorganisms. The process of biodegradation of the polymer consists of two steps. First, the process of reducing the polymer chain breaking of carbon bonds in terms of the effect of heat (degradation rate depends on temperature), humidity and the presence of microorganisms. Second, part of the process of biodegradation process begins when shorter chains become energy sources of microorganisms (bacteria, fungi or algae). This process is in full sense confirmed as biodegradation only when carbon compounds become food and microorganisms are transformed into water, biomass or carbon dioxide (Barone and Arikan, 2007).

Biodegradation properties of developed potato, corn and rice starch based biodegradable packaging films were measured by soil burial method. The result shown that approximately 25, 48, 75 and 95 % weight loss was found after 7, 14, 21, 28 days respectively. The biodegradability increased up to 95 % as the burial time increased. Corn starch film was rapidly degrading due to more amylose content present in corn starch. Potato and rice starch film takes more time to 100 % degradation.

Rice starch takes more time for degradation then potato and corn starch film. Concentration of starch and glycerol was also affecting on biodegradation rate of developed biodegradable packaging film. Starch and glycerol concentration increases with biodegradation rate was increases due to starch is hydrophilic nature of starch starch and easily interact with the moisture of soil and easily degrade in soil. In a biodegradation test carried out on starch based bioplastic, an increase of degradation

Table 4.12 Biodegradation properties of developed starch (corn, potato & rice) based biodegradable plastic

Run	Corn starch film biodegradation, %				Potato starch film biodegradation, %				Rice starch film biodegradation, %			
	After 7 days	After 14 days	After 21 days	After 28 days	After 7 days	After 14 days	After 21 days	After 28 days	After 7 days	After 14 days	After 21 days	After 28 days
1	19	42	70	86	19	44	69	90	15	36	62	75
2	20	44	71	88	20	45	72	90	16	36	63	80
3	18	42	69	85	18	43	67	87	14	34	58	73
4	22	49	74	93	24	49	75	95	19	43	70	85
5	14	36	60	80	15	38	62	82	11	28	59	68
6	20	43	71	87	20	47	74	93	16	37	62	77
7	19	45	69	89	22	45	72	91	17	38	64	79
8	22	47	73	90	22	47	73	92	18	40	67	82
9	16	39	64	82	17	41	66	85	13	32	62	71
10	23	50	75	94	25	52	76	98	20	45	72	87
11	21	45	70	89	21	46	74	91	17	38	65	78
12	19	45	68	89	21	45	72	92	16	39	63	81
13	21	42	70	86	21	43	71	92	15	38	65	80
14	21	48	72	92	23	48	74	93	18	41	68	83

with increasing starch concentration was observed and a degradation of about 95% was found. The biodegradation was accelerated by the presence of water that led to faster break down and allowed further attack of microorganisms. The result was agreement with the result reported by Jangong *et al.* (2019).

4.9 Performance evaluation of starch based biodegradable film for packaging

Packaging is any product that is used to hold, protection, handling, delivery and presentation of goods, from raw materials to finished products, from producers to consumers. The ability of developed biodegradable film to seal at sides and corners to form plastic carry bag was evaluated. The results indicated that the prepared bioplastic samples have good sealing capabilities. The principal purpose of the sealing crimp is to "squeeze" the two layers of film to achieve as great a molecular contact over as much of the sealing area as possible, within the constraints of the bag/pouch design. The heat-sealing feature was estimated through visual inspection. The sample was inspected manually. The sealed sample seems have excellent sealing properties. Developed plastic sealing was carried to measure sealability at different temperature and timings for particular thickness. Since sealing properties are important for preparing plastic bags, hence, it is concluded that the bioplastics produced in this project can be used to manufacture bioplastic carry bags. A sample bag produced from the starch is used for packaging is shown in Plate. 4.1

(A) Pigeon pea dhal packed in corn starch film and (B) Wheat packed in potato starch

(C) Pigeon pea packed in corn starch film and (D) Maida flour packed in rice starch film

Plate 4.1: Biodegradable plastic film used for packaging

4.10 Cost analysis of process for starch based biodegradable packaging film

4.10.1 Cost analysis of process for corn starch biodegradable packaging film

The production cost for development of corn starch biodegradable packaging film was estimated by carrying out the cost analysis with following assumptions.

Assumptions:

1. Cost of corn starch powder = Rs. 600/kg
2. Cost of glycerol = Rs. 1000/liter
3. Cost of acetic acid = = Rs. 1000/liter
4. Time required to develop 1 m^2 biodegradable film = 35 minutes.
5. Material required of for 1 square meter film
   = 35 g corn starch, 2.5 ml glycerol and 5 ml acetic acid
6. Quantity of biodegradable packaging film developed/h = 1.7 m^2
7. Quantity of biodegradable packaging film developed/day = 13.6 m^2

The cost analysis was carried out as per the assumptions described in the above section. The cost of development of 1 m^2 corn starch biodegradable packaging film is reported. The cost of 1 kg corn starch is Rs. 600, glycerol 1000 Rs/lit and acetic acid 1000/litre. Therefore cost of material for producing 1 m^2 corn starch biodegradable packaging film is Rs. 28.5.

Working capital = (12 × 600) + (0.84 × 1000) + (1.7× 1000) = 9740 Rs/month

(0.06 × 8 × 25 = 12 kg corn starch per month

(0.0042 × 8 × 25 =0.84 litre Glycerol per month)

(0.0085 × 8 × 25 =1.7 litre acetic acid per month)

The cost of material (Rs/h):

P = Cost of biodegradable material x Quantity of film produced in an hour

i.e. P = (0.035 × 600 + 0.0025 × 1000 + 0.005× 1000) X 1.7 = 48.45 Rs/h

Cost of corn starch biodegradable film of 1m^2 = 28.5 Rs.

4.10.2 Cost analysis of process for potato starch biodegradable packaging film

The production cost for development of potato starch biodegradable packaging film was estimated by carrying out the cost analysis with following assumptions.

Assumptions:

1. Cost of potato starch powder = Rs. 800/kg
2. Cost of glycerol = Rs. 1000/liter
3. Cost of acetic acid = = Rs. 1000/liter
4. Time required to develop 1 m^2 biodegradable film = 35 minutes.
5. Material required of for 1 square meter film
   = 35.5 g potato starch, 2.5 ml glycerol and 5 ml acetic acid
6. Quantity of biodegradable packaging film developed/h = 1.7 m^2
7. Quantity of biodegradable packaging film developed/day = 13.6 m^2

The cost analysis was carried out as per the assumptions described in the above section. The cost of development of 1 m^2 potato starch biodegradable packaging film is reported. The cost of 1 kg potato starch is Rs. 800, glycerol 1000 Rs/lit and acetic acid 1000/litre. Therefore cost of material for producing 1 m^2 potato starch biodegradable packaging film is Rs.35.9

Working capital = (12 × 800) + (0.84 × 1000) + (1.7× 1000) = 12140 Rs/month

(0.06 × 8 × 25 = 12 kg corn starch per month

(0.0042 × 8 × 25 =0.84 litre Glycerol per month)

(0.0085 × 8 × 25 =1.7 litre acetic acid per month)

The cost of material (Rs/h):

P = Cost of biodegradable material x Quantity of film produced in an hour

i.e. P = (0.035 × 800 + 0.0025 × 1000 + 0.005× 1000) X 1.7 = 61.03 Rs/h

Cost of potato starch biodegradable film of 1m^2 = 35.9 Rs.

4.10.3 Cost analysis of process for rice starch biodegradable packaging film

The production cost for development of rice starch biodegradable packaging film was estimated by carrying out the cost analysis with following assumptions.

Assumptions:

1. Cost of rice starch powder = Rs. 1400/kg
2. Cost of glycerol = Rs. 1000/liter
3. Cost of acetic acid = = Rs. 1000/liter
4. Time required to develop 1 m^2 biodegradable film = 35 minutes.

5. Material required of for 1 square meter film
   = 33 g rice starch, 2.5 ml glycerol and 5 ml acetic acid
6. Quantity of biodegradable packaging film developed/h = 1.7 m^2
7. Quantity of biodegradable packaging film developed/day = 13.6 m^2

The cost analysis was carried out as per the assumptions described in the above section. The cost of development of 1 m^2 rice starch biodegradable packaging film is reported. The cost of 1 kg rice starch is Rs. 1400, glycerol 1000 Rs/lit and acetic acid 1000/litre. Therefore cost of material for producing 1 m^2 rice starch biodegradable packaging film is Rs. 53.7

Working capital = (11.22 × 1400) + (0.84 × 1000) + (1.7 × 1000) = 18248 Rs/month

(0.056 × 8 × 25 = 11.22 kg corn starch per month

(0.0042 × 8 × 25 =0.84 litre Glycerol per month)

(0.0085 × 8 × 25 =1.7 litre acetic acid per month)

The cost of material (Rs/h):

P = Cost of biodegradable material x Quantity of film produced in an hour

i.e. P = (0.033 × 1400 + 0.0025 × 1000 + 0.005× 1000) X 1.7 = 91.3 Rs/h

Cost of rice starch biodegradable film of 1m^2 = 53.7 Rs.

CHAPTER V

SUMMARY AND CONCLUSIONS

Petrochemical based plastics films such as polyolefin, polyesters and polyamides have been increasingly used as packaging materials because of their availability in large quantities at low cost and functionality characteristics such as good tensile and tear strength, good barrier properties to oxygen and aroma compounds and heat seal ability. However, these plastics are made of petroleum-based materials that are not readily biodegradable and therefore lead to environmental pollution, the most obvious form of pollution associated with plastic packaging is waste plastic dump to the landfills.

Notable biopolymers incorporate starch, proteins and peptides, DNA, and RNA. These are biodegradable, eco-accommodating and are gotten from normal sources. Biopolymers are regularly biodegradable, and not lethal to deliver. Biopolymers are an alternative to petroleum-based polymers (traditional plastics).This biopolymer is also called a biodegradable "green plastic", as it is developed from plants and microorganisms. Biodegradable plastics are those that can be totally degraded in landfills, composters or sewage treatment plants by the activity of naturally occurring micro-organisms. Really biodegradable plastics leave no dangerous, obvious or discernable deposits following degradation.

Starch and starch derivatives are considered to be promising candidates for the development of biopolymer based environment friendly packaging materials mainly due to their renewability, abundance, low cost, film forming properties, bland taste and color, low solubility, biodegradability etc. The actual tendency in packaging research is to develop and promote the use of “bio-plastics” which are useful in reducing waste disposal and are good replaces of petroleum, a non-renewable resource with diminishing quantities.

To extend the shelf-life of all types of foods with increasing the preservation and protection from oxidation and microbial spoilage the tendency is to use more natural compounds. The use of synthetic films has led to big ecological problems because these materials are non-biodegradable. The common polymers that are utilized in food packaging have the favorable circumstances to be accessible from replenishable resources, biocompatible, biodegradable, and these attributes prompted natural well-being.

The principal function of packaging is protection and preservation of food from external contamination. This function involves retardation of deterioration, extension of shelf life, and maintenance of quality and safety of packaged food. Biodegradable polymers are the one which fulfil all these functions without causing any threat to the environment. The belief is that biodegradable polymer materials will reduce the need for synthetic polymer production (thus reducing pollution) at a low cost, thereby producing a positive effect both environmentally and economically.

Keeping above facts in view, the present research work was undertaken to standardize the process parameter for starch based biodegradable plastic with the following objectives.

1. To study the physical properties of potato, corn and rice starch powder.
2. To optimize the process parameters for development of starch based biodegradable plastic using response surface methodology.
3. To study the physico-chemical and mechanical properties of developed starch based biodegradable plastic.
4. To evaluate starch based biodegradable plastic film for packaging.
5. To study the biodegradability of developed starch based biodegradable plastic.
6. To carry out the cost analysis of developed starch based biodegradable plastic.

The raw materials for conducting the research work like potato, corn and rice starch powder (individual), glycerol, acetic acid and all other chemicals in analytical grade were procured.

The process is carried out in the biochemical laboratory. The required quantity of starch (corn, potato & rice) powder was taken as per the level mentioned. The films were prepared by casting technique using a film-forming solution. The water was added to the starch. Then after add glycerol and acetic acid in the solution. After mixing the mixture was heated with stirring on magnetic stirrer. This suspension was transferred to a water bath and continuous agitated by glass road. Then cast was prepared and the entire solution was poured on the cast and was left for drying at room temp for 24 hrs. After drying the films were peeled off. Films were kept in poly bags away from moisture.

The physical properties of starch (potato, corn & rice) powder were measured as per the standard methods. Physical properties (thickness), physico-chemical properties (moisture content, transparency, water absorption capacity, water vapor permeability and surface morphology), mechanical properties (tensile and puncture strength) and

biodegradation properties (reduction in weight) of developed starch based biodegradable plastic films were measured as per the standard methods.

The Response Surface Methodology (RSM) was used in designing the experiment. A two factor six-level Central Composite Rotatable Design (CCRD) with quadratic model was employed to study the combined effect of two independent variables, *viz.*, starch concentration (X_1), glycerol concentration (X_2)on different response variables, (2)to create mathematical models between the variables and (3) to determine the effect of these variables to optimize the selected response variables.

The following's are the salient conclusions of the investigation.

Physical properties of starch (Potato, Corn & Rice) powder

1) The physical properties of starch powder *viz.*, water absorption index of potato, corn and rice starch powder with their standard deviation was found as 139 ± 1.53%, 155 ± 2 % and 130 ± 2.51% respectively and water solubility index of potato, corn and rice starch powder with their standard deviation was found as 82 ± 1.52%, 86 ± 2.50 % and 79 ± 2.08 % respectively.

Physical properties of developed (Potato, Corn & Rice) biodegradable plastic

1) The Physical properties (Thickness) of developed potato, corn, &rice starch biodegradable plastic were ranged from 0.11 to 0.16 mm, 0.12 to 0.16 mm and 0.08 to 0.15 mm respectively.

Physico-chemical properties of corn starch biodegradable plastic

1) Moisture content of corn starch biodegradable plastic was ranged from 18.38 to 22.83 %.The maximum moisture content was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum moisture content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
2) Transparency of corn starch film was ranged from 54 to 72.96 %. The maximum transparency was observed for the combination of 5 g starch concentration and 1.25 ml glycerol concentration and minimum transparency content was found for the combination of 11 g starch concentration and 1.25 ml glycerol concentration.
3) Water absorption capacity of corn starch film was ranged from 138 to 180 %. The maximum water absorption capacity was observed for the combination of 11 g starch concentration 1.25 ml glycerol concentration and minimum water absorption capacity was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.

4) Water vapor permeability of corn starch film was ranged from 0.002 to 0.0045 g.mm/m^2daykPa. The maximum water vapor permeability was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
5) Tensile strength of corn starch film was ranged from 5.93 to 14.78 MPa. The maximum tensile strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
6) Puncture strength of corn starch film was ranged from 5.12 to 12.1 MPa. The maximum puncture strength was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5g starch concentration and 1.25 ml glycerol concentration.
7) The response surface quadratic model for corn starch film optimized the treatment condition as 7 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 18.38 %, transparency 68.36 %, water absorption capacity 145.93 %, water vapor permeability 0.002 g.mm/m^2daykPa, tensile strength 10.84 MPa and puncture strength 8.18 MPa.
8) The production cost for development of corn starch biodegradable packaging film of 1m^2 was estimated by 28.5 Rs.

Physico-chemical properties of potato starch biodegradable plastic

1) Moisture content of potato starch film was ranged from 18.19 to 23.1 %. The maximum moisture content was observed for the combination 11 g starch concentration and 1.25 ml glycerol concentration and minimum moisture content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
2) Transparency of potato starch film was ranged from 52.86 to 69.54 %. The maximum transparency was observed for the combination of 5 g starch concentration and 1.10 glycerol concentration and minimum transparency was found for the combination of 11 g starch concentration and 1.25 ml glycerol concentration.

3) Water absorption capacity of potato starch film was ranged from 151 to 190 %. The maximum water absorption capacity was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum water absorption capacity was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
4) Water vapor permeability of potato starch film was ranged from 0.0037 to 0.0058 g.mm/m^2daykPa The maximum water vapor permeability was observed for the combination of 11g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
5) Tensile strength of potato starch film was ranged from 5.09 to 13.62 MPa. The maximum tensile strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
6) Puncture strength of potato starch film was ranged from 4.28 to 10.45 MPa. The maximum puncture strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
7) The response surface quadratic model for potato starch film optimized the treatment condition as 7.1 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 18.52 %, transparency 65.47%, water absorption capacity 159 %, water vapour permeability 0.004 g.mm/m^2dayKPa, tensile strength 8.90 MPa and puncture strength 7.17 MPa
8) The production cost for development of potato starch biodegradable packaging film of 1m^2 was estimated by 35.9 Rs.

Physico-chemical properties of rice starch biodegradable plastic

1) Moisture content of rice starch film was ranged from 14.53 to 24.12 %. The maximum moisture content was observed for the combination 11 g starch concentration and 1.25 ml glycerol concentration and minimum moisture

content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.

2) Transparency of rice starch film was ranged from 39.52 to 53.6 %. The maximum transparency was observed for the combination of 5 g starch concentration and 1.25 ml glycerol concentration and minimum transparency was found for the combination of 11 g starch concentration and 1.25 ml glycerol concentration.
3) Water absorption capacity of rice starch film was ranged from 149 to 195 %. The maximum water absorption capacity was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum water absorption capacity content was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
4) Water vapor permeability of rice starch film was ranged from 0.0043 to 0.0071 g.mm/m^2daykPa. The maximum water vapor permeability was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum water vapor permeability was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
5) Tensile strength of rice starch film was ranged from 3.36 to 11.23 MPa. The maximum tensile strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
6) Puncture strength of rice starch film was ranged from 2.31 to 8.63 MPa. The maximum puncture strength was observed for the combination of 11 g starch concentration and 1.25 ml glycerol concentration and minimum tensile strength was found for the combination of 5 g starch concentration and 1.25 ml glycerol concentration.
7) The response surface quadratic model for rice starch film optimized the treatment condition as 6.6 g starch concentration and 0.5 ml glycerol concentration which gave the predicted values of moisture content of 15.39 %, transparency 50.08, water absorption capacity 157.90 %, water vapor

permeability 0.005 g.mm/m^2 day kPa, tensile strength 6.11 MPa and puncture strength 5.03 MPa.

8) The production cost for development of rice starch biodegradable packaging film of 1m^2 was estimated by 53.7 Rs.

Surface morphology of developed corn, potato and rice starch plastic

1) Surface morphology of developed corn, potato and rice starch plastic was observed that, corn starch films were smooth and compact as compared to the potato and rice starch films. From the SEM micrograph, it can be concluded that the corn starch granules dispersed well in the glycerol. This dispersion helps to improve the mechanical properties of the film and shows an agreement with the tensile property results.

Biodegradation properties of developed corn, potato and rice starch biodegradable plastic

1) Biodegradation properties of developed plastic were 25, 48, 75, 95 % weight loss was found after 7, 14, 21, 28 days respectively. The biodegradability increased up to 95 % as the burial time increased and increase of degradation with increasing starch concentration was observed and a degradation of about 95 % was found.

Finally, concluded that corn starch biodegradable film is better for packaging because it has lower moisture content, higher transparency, lower water absorption capacity, lower water vapour permeability and higher tensile and puncture strength. However, surface morphology of corn starch film is compact and smooth and biodegradability is higher due to the higher amylose content present in corn starch and low cost as compared to potato and rice starch film.

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