



UNIVERSITI
TEKNOLOGI
PETRONAS

FINAL YEAR PROJECT: PROJECT DISSERTATION

**PHYSICAL AND MECHANICAL PROPERTIES STUDY OF
STARCH/PVA BLEND BIODEGRADABLE FOAM**

PREPARED BY

ZURAIMI BIN ADZHAR

14393

SUPERVISOR

ASSOC. PROF. DR. KU ZILATI KU SHAARI

A project dissertation submitted in partial fulfilment of the requirement for the
Bachelor of Engineering (Hons)

(Chemical)

SEPTEMBER 2014

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh,

Perak Darul Ridzuan

CERTIFICATION

CERTIFICATION OF APPROVAL

PHYSICAL AND MECHANICAL PROPERTIES STUDY OF STARCH/PVA BLEND BIODEGRADABLE FOAM

Prepared by

ZURAIMI BIN ADZHAR

14393

A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
Bachelor of Engineering (Hons)
(Chemical)

SEPTEMBER 2014

Approved by,

.....

(Assoc. Prof. Dr Ku Zilati Ku Shaari)

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

.....

(Zuraimi Adzhar)

ABSTRACT

Mixing biodegradable polymers with each other could improve their fundamental properties. Starch is one of the most widely used materials to be fused/mixed with other biodegradable polymers. Starch/PVA combination compared to pure starch materials has shown improved tensile strength, elongation and processability. PVA is a biodegradable material however PVA has a low degree of biodegradation and as well it is not a renewable material. The study of physical and mechanical properties of starch and PVA blend studied the effect of mass ratio between starch and PVA. The mixture of starch and PVA are prepared with 4 different ratios with the aim of decreasing the amount of PVA in the mixture. The blends are prepared using a constant stirring as well as constant foaming speed. The time of blending, foaming and curing is also constant. The curing temperature varied from 90, 100 and 110 °C. After 4 hours of curing, the samples are taken out to determine its weight and thickness to find its density as well as the test for tensile strength. The foam with equal ratio of starch and PVA and cured with temperature 100 °C shown the lowest density. The tensile strength of the sample could not be completed as the samples have failed the tensile strength test where samples are too soft and delicate to be tested on the tensile strength machine.

ACKNOWLEDGEMENT

First of all, I would like to express the utmost gratitude to Allah S.W.T for giving the chance to finish this Final Year Project (FYP) dissertation successfully. Special thanks to my supervisor, Assoc. Prof Dr. Ku Zilati Ku Shaari who had given the golden opportunity to do this FYP and her informative supervision towards the author from the beginning until to the end of the semester was really meaningful. Without her guidance and persistent help, this dissertation would not have been possible.

With love and thanks to both the author's parents for giving a fully support until I managed to finish this FYP. Not to forget, highest gratitude to FYP coordinator, Dr. Asna Mohd Zain and all lecturers in Chemical Engineering Department for their unlimited support toward the final year students who are taking FYP. All the knowledge they supplied to me and combined with what I had gained during conducting the FYP sure will drive the author to a bright future, InshaAllah. Last but not least, I would like to say thanks to everybody who involved during conducting of the FYP period and the co-operation and advises given.

TABLE OF CONTENTS

CERTIFICATION OF APPROVAL	i
CERTIFICATION OF ORIGINALITY	ii
ABSTRACT	iii
ACKNOWLEDGEMENT	iv
LIST OF FIGURES	vi
LIST OF TABLES.....	vii
CHAPTER 1: INTRODUCTION	1
1.1. Background	1
1.2. Problem Statement	3
1.3. Objective	4
1.4. Scope of Study	4
CHAPTER 2: LITERATURE REVIEW AND THEORY	6
2.1. Biodegradable Polymers	6
2.2. Thermoplastic Starch	7
2.3. Polyvinyl Alcohol (PVA).....	8
2.4. Starch/PVA Blend Films Containing Citric Acid	9
2.5. Film Preparation.....	12
2.6. Mechanical Properties.....	13
CHAPTER 3: METHODOLOGY	15
3.1. Experiment Methodology	15
3.2. Project Flowchart	16
CHAPTER 4: RESULTS AND DISCUSSIONS	17
4.1. Physical Properties	17
4.2. Foam Weight, Thickness and Density	19
4.3. Mechanical Properties.....	24
CHAPTER 5: CONCLUSION AND RECOMMENDATIONS	26
REFERENCES	28
APPENDICES	30

LIST OF FIGURES

Figure 1: Solubility as a function of degree of hydrolysis.....	9
Figure 2: Tensile strength (TS) and elongation (%E) of starch/PVA blend film	10
Figure 3: Tensile strength (TS) of starch/PVA blend films with additives	11
Figure 4: Elongation (%E) of starch/PVA blend films with additives	11
Figure 5: Variation of the tensile strength versus concentration.	13
Figure 6: Variation of elongation at break versus concentration.....	14
Figure 7: Process methodology of the study.....	16
Figure 8: Ratio A cured at 100 °C	17
Figure 9: Ratio B, C, D cured at 100 °C.....	17
Figure 10: Ratio A, B, C, D cured at 90 °C.....	18
Figure 11: Ratio A, B, C, D cured at temperature 110 °C.....	19
Figure 12: Graph of Density (g/cm ³) against Ratio of Foam at 100 °C and 110 °C	24
Figure 13: Placement of foam strip with the tensile strength machine.....	25

LIST OF TABLES

Table 1: Types of biodegradable polymers	2
Table 2: Wt% of each materials and the codification of the independent variables.....	12
Table 3: Starch:PVA:Citiric Acid Ratio	15
Table 4: Weight and thickness of Ratio A cured at 100 °C.....	19
Table 5: Weight and thickness Ratio B cured at 100 °C	20
Table 6: Weight and thickness Ratio C cured at 100 °C	20
Table 7: Weight and thickness Ratio D cured at 100 °C	21
Table 8: Weight and thickness Ratio A cured at 110 °C	21
Table 9: Weight and thickness Ratio B cured at 110 °C	22
Table 10: Weight and thickness Ratio C cured at 110 °C	22
Table 11: Weight and thickness Ratio C cured at 110 °C	22
Table 12: Density of foam with Ratio A, B, C, D cured at 100 °C and 110 °C	23

CHAPTER 1: INTRODUCTION

1.1. Background

The use of synthetic polymer materials has caused considerable environmental problems. Solid waste from these resources is a major contributor to environmental pollution because it can take up to a thousand years to degrade. One of the solutions taken to overcome this serious problem is by developing various biodegradable materials (Azhari, Othman, & Ismail, 2011). There are two categories of environmental friendly polymer based on the raw material used which are biodegradable polymers derived from petroleum resources and biodegradable polymers derived from renewable resources. Biodegradable polymers derived from petroleum resources are divided into many different classes which are; polymers with additives, synthetic polymers with hydrosylable backbones and synthetic polymers with carbon backbones. Biodegradable polymers derived from renewable resources are also divided into two different classes which are natural polymers or agro-polymers and bacterial polymers (Vroman & Tighzert, 2009).

Mixing biodegradable polymers with each other could improve their fundamental properties. Starch is one of the most widely used materials to be fused/mixed with other biodegradable polymers due to its low cost. One other factor that starch is used in blending with other materials is despite its high receptivity to water it has a relatively poor mechanical properties compared to other petrochemical polymers. The materials that are usually used to be blended with starch are as follows (Vroman & Tighzert, 2009):

- a) Starch/poly(ethylene-co-vinyl alcohol)
- b) Starch/polyvinyl alcohol (PVA)
- c) Starch/polylactide (PLA)
- d) Starch/polycaprolactone (PCL)
- e) Starch/poly(butylene succinate)
- f) Starch/poly(hydroxybutyrate) (PHB)

Starch/PVA combination compared to pure starch materials has shown improved tensile strength, elongation and processability. The content of PVA influenced the rate of

degradation of this blend where the increasing amount of PVA will decrease the rate of degradation (Vroman & Tighzert, 2009).

Plastics foams are used in packaging majorly as cushioning materials and as containers. The most often material used as foam is polystyrene and there are also a considerable use of polyethylene, polypropylene, and urethane foams, along with some other materials. Foams are characterized as a very light weight material, good insulating capacity, and the ability to absorb shocks and protect the enclosed products. There are two major types of foams. *Open cell* foams and *closed cell* foams. In an open cell foams, liquids and gases could travel through the foam by pass through the channels between the cells hence therefore these foams tend to be absorbent; sponge is a common example. Contradict with the closed cell foams where liquid or gas could only diffuse through the polymer at the boundaries between the cells hence these type of foams provide better barrier and non-absorbent (Selke, Culter, & Hernandez, *Plastics Packaging; Properties, Processing, Applications, and Regulations*, 2004).

Starch was not traditionally recognized as a plastic but there are techniques for producing starch based foams that naturally involve plasticizing the starch with plasticizing agents (urea is used in this project). The major advantage of starch-based foams is that they are considered as an environmental friendly material (Selke, Culter, & Hernandez, *Plastics Packaging; Properties, Processing, Applications, and Regulations*, 2004).

According to Smith, biodegradable polymers are categorized as follows:

Table 1: Types of biodegradable polymers according to Smith (2005)

Base Polymer	Source Type	Advantages	Disadvantages	Potential Applications
Starch	Renewable	<ul style="list-style-type: none"> • Low Cost • Fast biodegradation 	<ul style="list-style-type: none"> • Poor mechanical properties • Hydrophilicity 	<ul style="list-style-type: none"> • Foams • Films and bags • Moulded items
Polyhydroxyalkanoates (PHA)	Renewable	<ul style="list-style-type: none"> • Rapid biodegradation 	<ul style="list-style-type: none"> • High cost 	<ul style="list-style-type: none"> • Moulded items

		<ul style="list-style-type: none"> • Water stable 		
Cellulose and cellulose acetates	Renewable	<ul style="list-style-type: none"> • High strength • Water stable 	<ul style="list-style-type: none"> • Difficult to process • Very low biodegradability 	<ul style="list-style-type: none"> • Composites • Fibre-board
Fatty acid (triglyceride oil based) polymers	Renewable	<ul style="list-style-type: none"> • High strength 	<ul style="list-style-type: none"> • Brittle • Low biodegradability 	<ul style="list-style-type: none"> • Composites • Adhesives • Compatibilisers
Lignin polymers	Renewable	<ul style="list-style-type: none"> • High strength 	<ul style="list-style-type: none"> • Brittle • Low biodegradability 	<ul style="list-style-type: none"> • Composites • Adhesives • Compatibilisers
Collagen/gelatine polymers	Renewable	<ul style="list-style-type: none"> • High strength 	<ul style="list-style-type: none"> • Non-reproducible properties 	<ul style="list-style-type: none"> • Films
Polyactic acid (PLA)	Renewable and non-renewable	<ul style="list-style-type: none"> • High strength 	<ul style="list-style-type: none"> • Brittle 	<ul style="list-style-type: none"> • Injection moulding • Fibres
Polyglycolic acid (PGA)	Non-renewable	<ul style="list-style-type: none"> • High strength 	<ul style="list-style-type: none"> • Brittle • Soluble in water 	<ul style="list-style-type: none"> • Fibres • Sutures
Polycaprolactone (PCL)	Non-renewable	<ul style="list-style-type: none"> • Water stable • Hydrolysable 	<ul style="list-style-type: none"> • Low melting point 	<ul style="list-style-type: none"> • Compost bags • Cold packaging
Polyvinyl alcohol (PVA)	Non-renewable	<ul style="list-style-type: none"> • Good barrier properties 	<ul style="list-style-type: none"> • Low biodegradability • Solubility in water 	
Synthetic polyesters	Non-renewable	<ul style="list-style-type: none"> • High strength • Good processing 	<ul style="list-style-type: none"> • Relatively high cost 	<ul style="list-style-type: none"> • Films • Moulded items

1.2. Problem Statement

Petroleum based plastics have been widely used throughout the world. With increased applications, the disposal of waste plastics has become a major problem. Therefore, development of new plastics that could be degraded by microorganisms in soil and seawater has recently been attracting much attention (Park, Chough, Yun, &

Yoon, 2005). Starch, as a profuse and inexpensive raw material, has been applied in the field of biomaterials. However, packaging films (composed entirely of starch) lack the strength and rigidity to withstand the stresses to which many packaging materials are subjected (Parvin, Rahman, & Islam, 2010).

Thermoplastic starch seems to be a perfect solution to obtain commercial packaging material produced from pure starch and to exclude synthetic polymers from the formulation. However, to obtain thermoplastic starch, thermal and mechanical processing should disrupt semi crystalline starch granules. As the melting temperature of pure starch is substantially higher than its decomposition temperature, there is a necessity to use plasticizers like glycerol. Influence of both, temperature and shear forces, create the disruption of the natural crystalline structure of starch granules; and polysaccharides form a continuous polymer phase (Mitrus & Moscicki, 2013).

One other approach of improving the mechanical properties of starch is by blending and the most recent research has focused on pure starch-based materials and starch/degradable polymer blend materials such as starch/cellulose, starch/PVA (Park, Chough, Yun, & Yoon, 2005). PVA is widely used because of its solubility in water and it can be easily biodegraded by microorganisms as well as enzymes. However, PVA is categorized under the biodegradable polymers derived from petroleum sources hence it is not a renewable source (Vroman & Tighzert, 2009).

1.3. Objective

The main objectives of this study are:

- 1) To study the effect of mass ratio of starch and PVA to the physical and mechanical properties of the foam.
- 2) To study the effect of curing temperature of starch and PVA to the physical and mechanical properties of the foam.

1.4. Scope of Study

The study will involve in researching the potential of reducing polyvinyl alcohol (PVA) in the foam blend. At first, PVA will be added at optimum quantity and tested. Then the quantity of the PVA will be reduced to study its parameters change. The

parametric of the foam fabricated will be analysed. The parametric study will focus more on:

- 1) Physical properties
- 2) Tensile strength
- 3) Elongation.

CHAPTER 2: LITERATURE REVIEW AND THEORY

2.1. Biodegradable Polymers

Plastic in a form of cellulose nitrate was the first man made plastic and it was prepared by A. Parker in 1838 and was publically shown at the Great International Exhibition in London in 1862. It was called “parkesine” and intended to replace natural materials such as ivory. Later in 1860 a procedure known as “vulcanization” was developed by Goodyear and Hancock to eliminate tackiness and to add elasticity to natural rubber. It was not until 1920 the exact nature of plastics, rubber, and similar natural materials was known. H. Staudinger was the person that proposed a revolutionary idea: all plastics, rubber, and materials such as cellulose were polymers, or macromolecules. Before Staudinger’s theory, the scientific community was very confused about the exact nature of plastics, rubbers and other materials of very high molecular weight (Selke, Culter, & Hernandez, *Plastics Packaging; Properties, Processing, Applications, and Regulations*, 2004).

“Biodegradable polymers” or “compostable polymers” were first commercially introduced in 1980s. The first generation of these biodegradable products were made from a conventional polymer (polyolefin e.g. polyethylene) mixed together with starch or some other organic materials. The idea was when starch was eaten by microorganism, the products were broken down and leaving only small fragments of polyolefin (Rudnik, 2008). According to Narayan et al. “The U.S. biodegradable industry fumbled at the beginning by introducing starch filled (6-15%) polyolefin as true biodegradable materials. These at best were only biodisintegratable and not completely biodegradable. Data showed that only the surface starch biodegraded, leaving behind a recalcitrant polyethylene material”. Consumers and government regulators were confused by the statement of Narayan et al and created misunderstanding about what was and what was not biodegradable/compostable. Additionally, there were no scientifically based test methods or standards exist to support claims made by plastics manufacturers for the biodegradability and compostability of their products (Rudnik, 2008).

More recently, international standard bodies such as International Organization for Standardization (ISO) and American Society for Testing and Materials (ASTM) have

developed definitions related to degradation of plastics and even describing in detail the purpose of “biodegradable” and “compostable”.

According to ISO/DIS 17088, the definitions of compostability:

- Compostable plastics
A plastics that undergoes degradation by biological processes during composting to yield CO₂, water, inorganic compounds, and biomass at a rate consistent with other known compostable materials and leaves no visible, distinguishable or toxic residue.
- Composting
The autothermic and thermophilic biological decomposition of biowaste (organic waste) in the presence of oxygen and under controlled conditions by the action of micro- and macro-organisms in order to produce compost.
- Disintegration
The physical breakdown of material into very small fragments.

2.2. Thermoplastic Starch

Thermoplastic starch or also known as plasticized starch offers an interesting alternative for synthetic polymers in specific applications. Significant research has been done in developing a new class of fully biodegradable “green” composites called bio-composites. They consist in biodegradable plastics with reinforcements of biodegradable natural fibres. Starch can be used as the biodegradable polymeric compound. However, the problems with starch-based products are that it suffers from water receptivity, brittleness and poor mechanical properties (Vroman & Tighzert, 2009).

The thermoplastic starch mechanical properties depend on the temperature of starch processing, water content as well as number and kind of added plasticizers and assistance materials. Rise of the plasticizer content brings concerning a lost in tensile strength of thermoplastic starch; whereas, the elongation at break increases. Starch is a usual polymer consisting of countless hydrogen bonds among the hydroxyl radicals in its molecules; consequently it manifests comprehensive tensile strength values. Glycerol, sorbitol or glycol behave like diluents and cut the inter-action amid molecules and subsequently, they diminish tensile strength. At the same time, they act as plasticizers that enhance macromolecular mobility and leads to a development in elongation at break.

Addition of filler materials like cellulose fibres, flax, kaolin or pectin increases the tensile strength but decreases the elongation at break. In turn, urea or boric acid addition improves the elongation at break but decreases the tensile strength (Mitrus & Moscicki, 2013).

2.3. Polyvinyl Alcohol (PVA)

Polyvinyl alcohol (PVA) has a relatively simple chemical structure with a pendant hydroxyl group. The monomer, vinyl alcohol does not exist in a stable form rearranging to its tautomer, acetaldehyde. Hence, PVA is produced by the polymerization of vinyl acetate to polyvinyl acetate followed by its hydrolysis to PVA. The hydrolysis reaction does not go to completion resulting in polymers with a certain degree of hydrolysis that depends on the extent of reaction. Basically, PVA is always a copolymer of PVA and polyvinyl acetate. Commercial PVA grades are available with a high degree of hydrolysis up to 98.5%. The degree of hydrolysis or the content of acetate group in the polymer has a general effect on its chemical properties, solubility and crystallizability of PVA (Hassan & Peppas, 2000).

The degree of hydrolysis and polymerization affect the solubility of PVA in water and has been shown that PVA grades with high degree of hydrolysis have low solubility in water. The figure below shows the solubility of a PVA sample with a number average molecular weight of $M=77000$ as a function of the degree of hydrolysis as dissolution temperatures of 20 and 40 °C. Residual hydrophobic acetate groups weaken the intramolecular and intermolecular hydrogen bonding of adjoining hydroxyl groups. The temperature must be increased well above 70 °C for dissolution to occur. The occurrence of acetate groups also affects the ability of PVA to crystallize upon heat treatment. PVA grades with a high degree of hydrolysis are more difficult to crystallize (Hassan & Peppas, 2000).

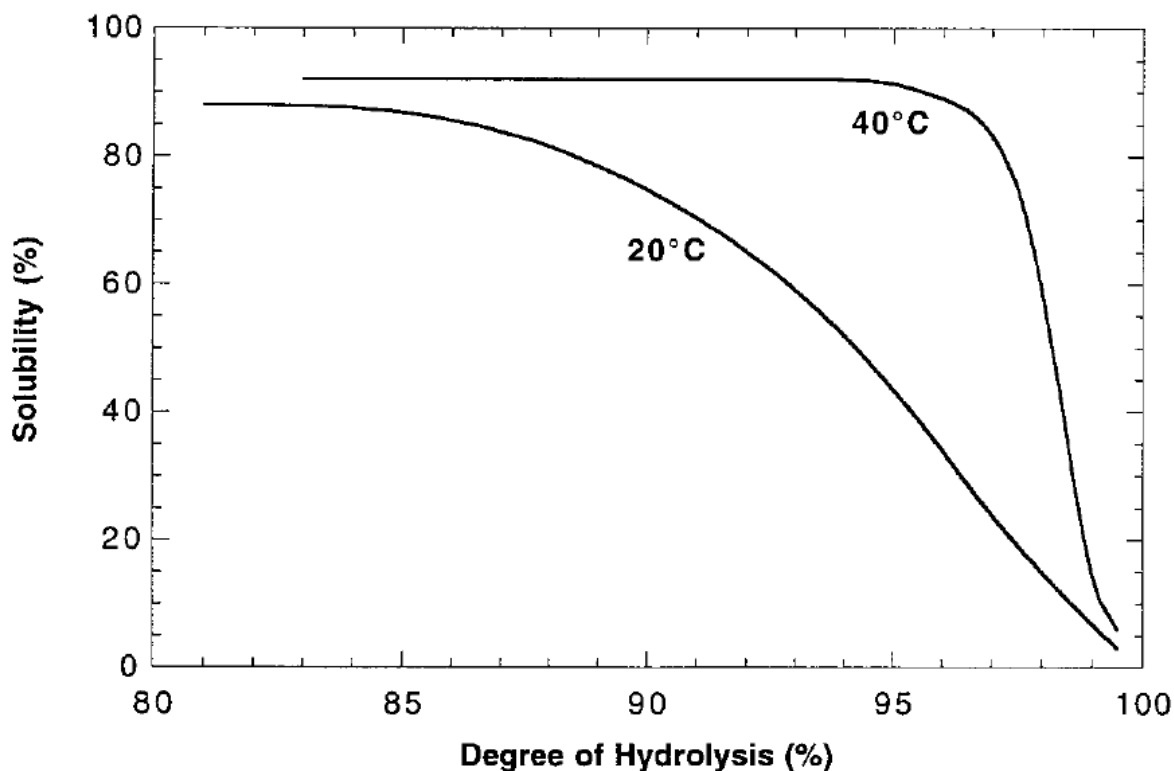


Figure 1: Solubility as a function of degree of hydrolysis at dissolution temperatures of 20 and 40 degree Celsius (Hassan & Peppas, 2000)

Polyvinyl alcohol (PVA) is a biodegradable synthetic material which has the advantages of excellent film forming, strong conglutination, and high thermal stability. In recent years, PVA has widely used in the materials industry and preparation of starch-based polymer has been reported. In one study, polymer films of rice starch/PVA were produced and different physico-mechanical and biodegradable properties were studied. The prepared films were farther grafted with acrylic monomer using UV radiation and the physico-mechanical and biodegradable properties of the treated films were studied (Parvin, Rahman, & Islam, 2010).

2.4. Starch/PVA Blend Films Containing Citric Acid

In a study, starch-based plastic has been prepared by extraction and irradiation process. The study prepared starch/PVA/glycerol blend using extraction process and reported on its mechanical properties and thermodynamics (Mao, Imam, Gordon, Cinelli, & Chienelli, 2000). In another study, the blend was prepared on irradiation technology and investigated its mechanical properties, thermal analyses and water adsorption (Zhai,

Yoshii, & Kume, 2003). Starch-based plastics prepared on extraction and irradiation technology were shown to be sensitive moisture and temperature. The influence of starch sources, starch components, starch molecular mass and plasticizer, such as water and polyols, has been studied to improve the mechanical properties of starch plastics. However, poor mechanical properties are still one of the major unresolved problems. Although their tensile strength may be rather high (30-60 MPa), these materials are fragile with low elongation at break and poor in water resistance. After absorbing water, they are too weak to be used (Hulleman, Janssen, & Feil, 1998).

In another study, starch/PVA blend film was prepared by a mixing process without the extraction and irradiation process. Additives were made use of glycerol, sorbitol, and citric acid. Sorbitol is an alcohol sugar extensively used in the food industry, not only as a sweetener but also as a humectants, texturizer, and softener. Citric acids exist in a natural way in foods like citrus fruits and pineapples where it is the main organic acid. Citric acid is utilized as multi-functional food additive in the processes of producing different foods because it presents antibacterial and acidulant effect, reinforces the antioxidant action of other substances, and improves the flavours of juices, soft drinks and syrups. Synthesized starch/PVA blend films using glycerol, sorbitol and citric acid as additives were investigated for their mechanical properties; tensile strength and elongation (Park, Chough, Yun, & Yoon, 2005).

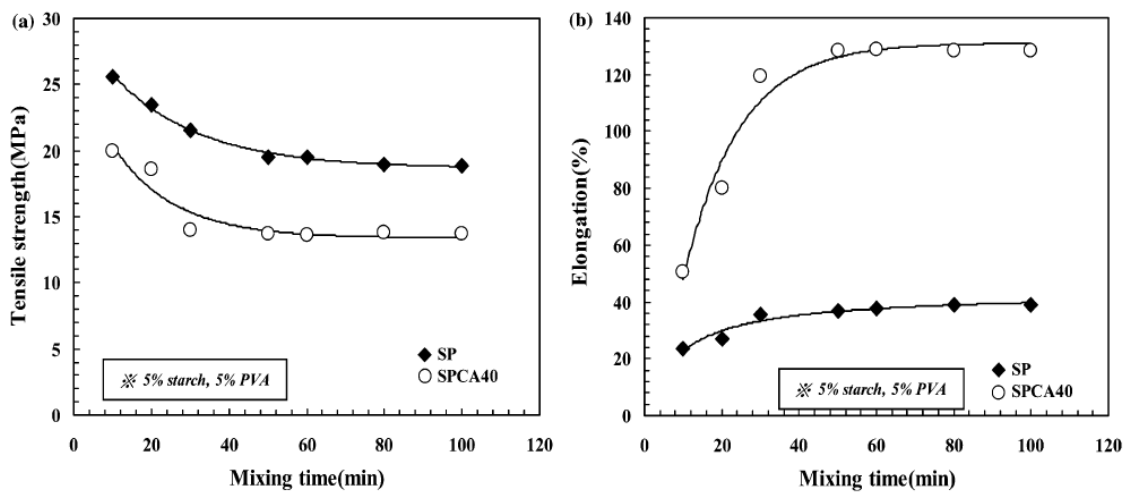


Figure 2: Tensile strength (TS) and elongation (%E) of starch/PVA blend film versus mixing time (a) Tensile strength (TS) of starch/PVA blend film adding with/without additive (b) Elongation (%E) of starch/PVA blend film adding with/without additive (Park, Chough, Yun, & Yoon, 2005)

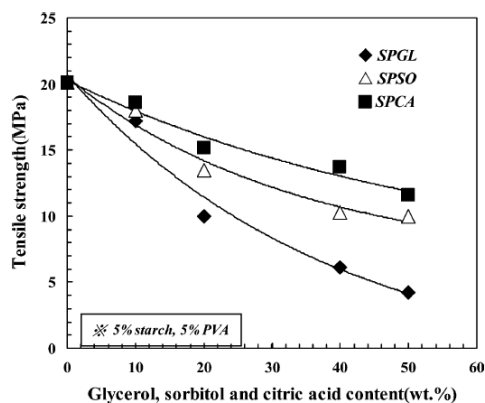


Figure 3: Tensile strength (TS) of starch/PVA blend films adding glycerol (GL), sorbitol (SO) and citric acid (CA) as additives (Park, Chough, Yun, & Yoon, 2005)

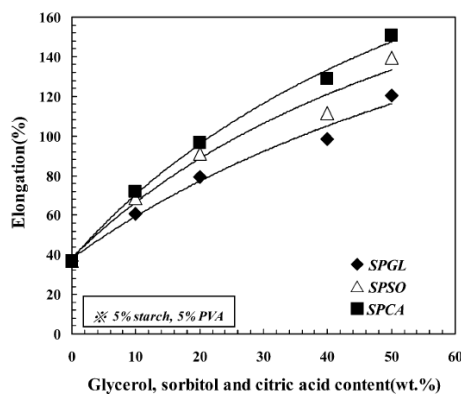


Figure 4: Elongation (%E) of starch/PVA blend films adding glycerol (GL), sorbitol (SO) and citric acid (CA) as additives (Park, Chough, Yun, & Yoon, 2005)

Parvin et al. prepared the blend by casting method. Starch/PVA and starch/PVA/10% sugar were blended in hot water at about 150 °C for about 1 hour to form a homogeneous solution. Several formulations were prepared with this solution, varying the concentration of starch and polyvinyl alcohol (PVA). Polymer films were prepared by casting on silicon cloth. Mechanical properties; tensile strength (TS) and percent elongation at break (Eb) of the blend films were measured with Universal Testing Machine. The blend was also undergo morphological analysis which to observe the physical structure of the blend film and also water uptake test. Soil burial test was also done to the blend to find out its biodegradability (Parvin, Rahman, & Islam, 2010).

2.5. Film Preparation

Tudorachi et al. had completed a similar experiment with difference parameters. The materials used in the experiments are industrial corn starch, a white fine powder with 2% by weight moisture content, acidity 2.0 (cm³ 0.01 N NaOH solution) and around 1% by weight proteins and lipids was provided by Amidex-Tg, Secuiesc, Romania. The PVA polymer, with hydrolysis degree 88%, polymerization degree 1200, saponification index 140±30 mg KOH/g, K value 65±5 and 2% by weight ash, was obtained from Romacryl-Rasnov, Romania. Other materials used in this study were urea, glycerine, NaCl, Na₂HPO₄, analytical grade reagents being obtained from Reactivul-Bucharest, Romania, and agar supplied by Fluka, Switzerland.

The films are prepared by casting some mixtures of starch, PVA, glycerine and urea firstly with the watery solutions of PVA (40% by weight) and starch (60% by weight). The mixtures are mixed together at temperature of 70 °C for 2 hours. Then urea (used as a microorganism nitrogen source and/or plasticizer) dissolved in glycerine (used as a plasticizer) was introduced into the flask and the obtained mixture was stirred for 1 h. The films obtained with dimensions 300'100 mm in size and 250±5 mm thick were dried in air at 30–40 °C.

The compositions of the mixtures are listed in the table;

Table 2: Wt% of each materials and the codification of the independent variables

Blend components	Independent variables	Codification level				
		-2	-1	0	1	2
		Component content (g)				
Starch	X1	20	30	40	50	60
PVA	X2	60	50	40	30	20
Glycerine	X3	10	12	14	16	18
Urea	X4	10	8	6	4	2

The films were then tested its tensile strength and elongation at break.

2.6. Mechanical Properties

The results of the tests conducted for tensile strength and elongation were tabulated and presented in a graph.

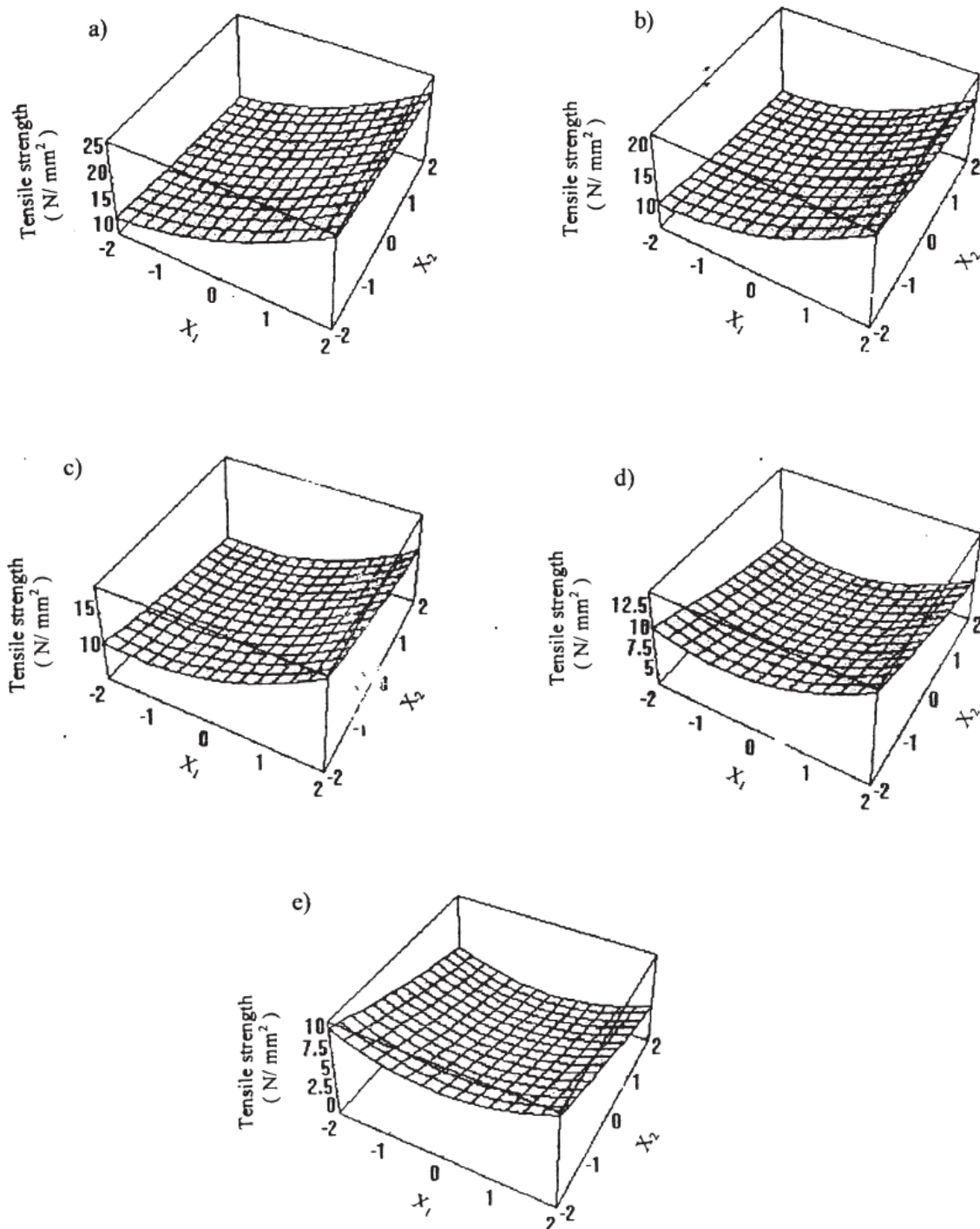


Figure 5: Variation of the tensile strength versus concentration in starch (X₁) and in PVA (X₂), in conditions when the X₃ and X₄ are maintained constantly: (a) X₃ = -2 and X₄ = 2; (b) X₃ = -1 and X₄ = 1; (c) X₃ = 0 and X₄ = 0; (d) X₃ = 1 and X₄ = -1; (e) X₃ = 2 and X₄ = -2.

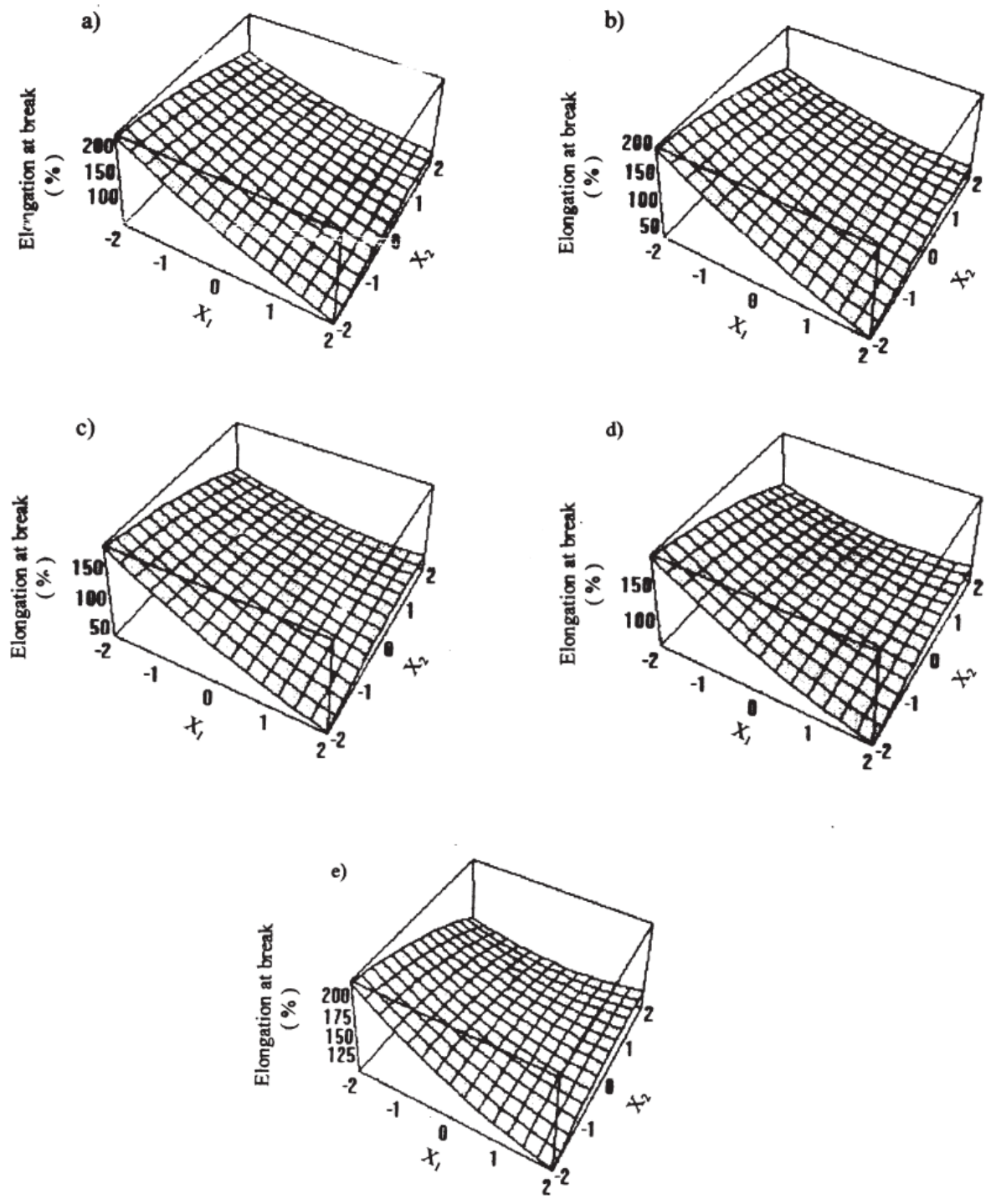


Figure 6: Variation of elongation at break versus concentration in starch (X1) and in PVA (X2), in the same conditions as those mentioned in Figure 5

CHAPTER 3: METHODOLOGY

3.1. Experiment Methodology

Start the mixture preparations by weighing the materials as follow:

Table 3: Starch:PVA:Citric Acid Ratio

Ratio	Starch	PVA	Citric Acid
A	5.00 g	5.00 g	20.00 g
B	6.25 g	3.75 g	20.00 g
C	7.50 g	2.50 g	20.00 g
D	8.75 g	1.25 g	20.00 g

The weight of urea (plasticizers) used in all mixtures is a constant of 3.00 g which is 10% of the total weight of the mixture.

Pour PVA into a flask and add 100 ml of water with it. Starch and urea are mixed inside a beaker with 50 ml of distilled water before the mixture is poured into the flask along with the PVA. The flask is set up immersed in a water bath as shown in Figure 7. The flask is then heated up to 90 °C and stirred with 300 rpm for 45 minutes. The mixture will be a gel-like fluid after 45 minutes of mixing the materials together. Leave the mixture to cool down to room temperature before starting to foam it.

After the mixture has cool down to the room temperature, add the 20.00 g of citric acid into the solution and stir the mixture with a mechanical stirrer at 1500 rpm for 45 minutes. The mixture will foamed after being stirred for 45 minutes.

While the mixture is being foamed, pre-heat the mould with the oven temperature of 100 °C. After 45 minutes of foaming, pour the foam into the pre-heated mould. The weight of foam poured into the mould would be around 35.00 to 40.00 g. The mould then put back into the oven and left to be cured for 4 hours.

After 4 hours of curing, take out the mould and examine the foam. The properties studied are weight, density and tensile strength.

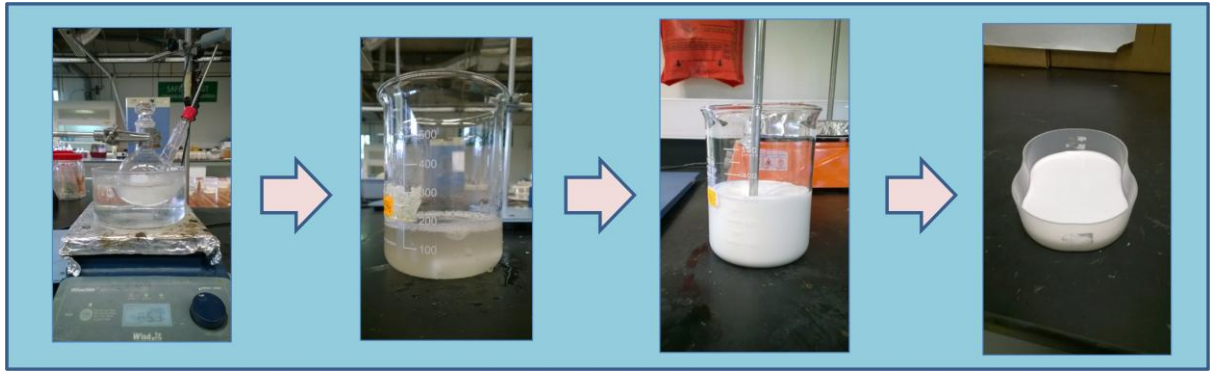
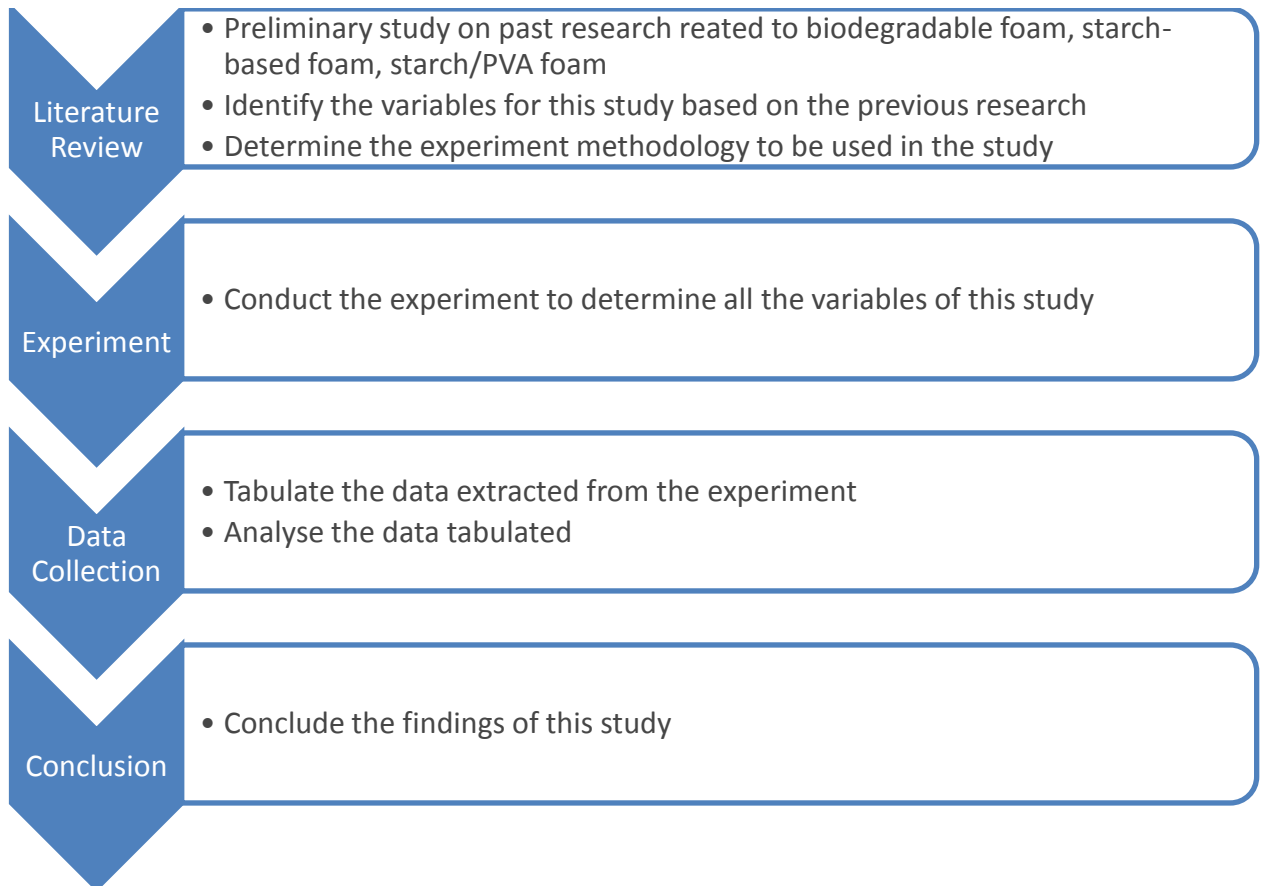


Figure 7: Process methodology of the study (From left to right: (1) Apparatus set up. (2) Gel-like fluid after stirring for 45 minutes. (3) Foaming process. (4) Foam poured into the mould.

3.2. Project Flowchart



CHAPTER 4: RESULTS AND DISCUSSIONS

4.1. Physical Properties



Figure 8: Ratio A cured at 100 °C

Figure 8 shows the foam condition when mixed with Ratio A and cured at 100 °C. The surface of the foam does not show any visible defects.



Figure 9: Ratio B, C, D cured at 100 °C

Figure 9 shows the foam condition when mixed with Ratio B, C and D and cured at 100 °C. The surface of the foam shows defect which is holes. The sample does not foamed properly and is not suitable to be tested for tensile strength.



Figure 10: Ratio A, B, C, D cured at 90 °C

Figure 10 shows the foam condition when mixed with Ratio A, B, C and D and cured at 90 °C. The surface of the foam does not dried completely. There are still some parts of the foam that are still wet/not cured properly. The sample does not foamed properly and is not suitable to be tested for tensile strength.

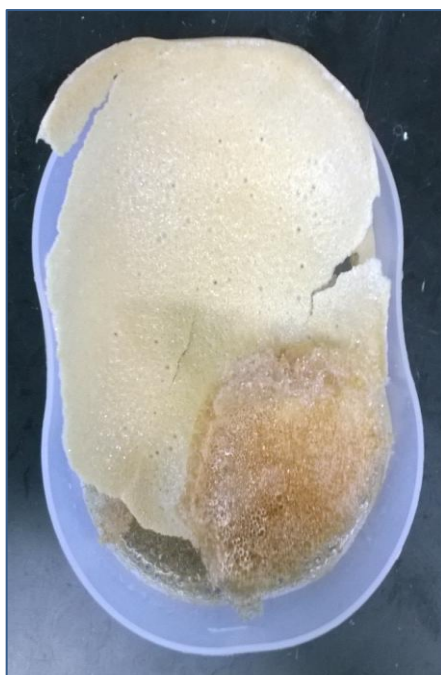


Figure 11: Ratio A, B, C, D cured at temperature 110 °C

Figure 11 shows the foam condition when mixed with Ratio A, B, C and D and cured at 110 °C. The surface of the foam is completely dried and brittle. The sample does not foamed properly and is not suitable to be tested for tensile strength.

4.2. Foam Weight, Thickness and Density

To study the weight, thickness and density of the foam, the samples are cut down to strips. The dimensions of the strips are 10 mm in width and 80 mm in length. The thickness of the strips varies. Ratio A sample cured at 100 °C shows the thickest foam. The data is as shown below:

Table 4: Weight and thickness of Ratio A cured at 100 °C

Sample	Weight (g)	Thickness (cm)
1	0.689	0.5
2	0.660	0.5
3	0.680	0.5
4	0.679	0.5
5	0.679	0.5
Average	0.677	0.5

The density for Ratio A cured at 100 °C is:

$$\text{Average weight} = 0.677 \text{ g}$$

$$\text{Volume} = 0.5 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 4.0 \text{ cm}^3$$

$$\text{Density} = \text{Weight (g)} / \text{Volume (cm}^3) = 0.1693 \text{ g/cm}^3$$

Table 5: Weight and thickness Ratio B cured at 100 °C

Sample	Weight (g)	Thickness (cm)
1	0.680	0.3
2	0.675	0.3
3	0.660	0.2
4	0.673	0.3
5	0.671	0.3
Average	0.672	0.28

The density for Ratio B cured at 100 °C is:

$$\text{Average weight} = 0.672 \text{ g}$$

$$\text{Volume} = 0.28 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 2.24 \text{ cm}^3$$

$$\text{Density} = \text{Weight (g)} / \text{Volume (cm}^3) = 0.3 \text{ g/cm}^3$$

Table 6: Weight and thickness Ratio C cured at 100 °C

Sample	Weight (g)	Thickness (cm)
1	0.659	0.2
2	0.662	0.2
3	0.664	0.2
4	0.669	0.3
5	0.671	0.3
Average	0.665	0.22

The density for Ratio C cured at 100 °C is:

$$\text{Average weight} = 0.665 \text{ g}$$

Volume = $0.22 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 1.76 \text{ cm}^3$

Density = $\text{Weight (g)} / \text{Volume (cm}^3) = 0.38 \text{ g/cm}^3$

Table 7: Weight and thickness Ratio D cured at 100 °C

Sample	Weight (g)	Thickness (cm)
1	0.657	0.2
2	0.660	0.2
3	0.659	0.2
4	0.658	0.3
5	0.666	0.3
Average	0.660	0.22

The density for Ratio D cured at 100 °C is:

Average weight = 0.660 g

Volume = $0.22 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 1.76 \text{ cm}^3$

Density = $\text{Weight (g)} / \text{Volume (cm}^3) = 0.375 \text{ g/cm}^3$

Table 8: Weight and thickness Ratio A cured at 110 °C

Sample	Weight (g)	Thickness (cm)
1	0.687	0.3
2	0.662	0.3
3	0.681	0.3
4	0.680	0.3
5	0.682	0.3
Average	0.678	0.3

The density for Ratio A cured at 110 °C is:

Average weight = 0.678 g

Volume = $0.3 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 2.4 \text{ cm}^3$

Density = $\text{Weight (g)} / \text{Volume (cm}^3) = 0.283 \text{ g/cm}^3$

Table 9: Weight and thickness Ratio B cured at 110 °C

Sample	Weight (g)	Thickness (cm)
1	0.679	0.3
2	0.680	0.3
3	0.675	0.3
4	0.672	0.3
5	0.671	0.3
Average	0.675	0.3

The density for Ratio B cured at 110 °C is:

$$\text{Average weight} = 0.675 \text{ g}$$

$$\text{Volume} = 0.3 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 2.4 \text{ cm}^3$$

$$\text{Density} = \text{Weight (g)} / \text{Volume (cm}^3) = 0.281 \text{ g/cm}^3$$

Table 10: Weight and thickness Ratio C cured at 110 °C

Sample	Weight (g)	Thickness (cm)
1	0.656	0.2
2	0.660	0.2
3	0.660	0.2
4	0.659	0.3
5	0.662	0.3
Average	0.659	0.22

The density for Ratio C cured at 110 °C is:

$$\text{Average weight} = 0.659 \text{ g}$$

$$\text{Volume} = 0.22 \text{ cm} \times 1 \text{ cm} \times 8 \text{ cm} = 1.76 \text{ cm}^3$$

$$\text{Density} = \text{Weight (g)} / \text{Volume (cm}^3) = 0.374 \text{ g/cm}^3$$

Table 11: Weight and thickness Ratio C cured at 110 °C

Sample	Weight (g)	Thickness (cm)
--------	------------	----------------

1	0.654	0.2
2	0.659	0.2
3	0.658	0.2
4	0.658	0.3
5	0.664	0.3
Average	0.659	0.22

The density for Ratio D cured at 110 °C is:

Average weight = 0.659 g

Volume = 0.22 cm × 1 cm × 8 cm = 1.76 cm³

Density = Weight (g) / Volume (cm³) = 0.374 g/cm³

Samples from curing temperature 90 °C are not taken into consideration because the physical conditions of the foams that are not completely dried. The densities of all the samples above are tabulated in the following table:

Table 12: Density of foam with Ratio A, B, C, D cured at 100 °C and 110 °C

Ratio	Curing Temperature (°C)	Density (g/cm ³)
A	100	0.169
B		0.300
C		0.380
D		0.375
A	110	0.283
B		0.281
C		0.374
D		0.374

From the table, a graph is drawn to compare the density of the foam.

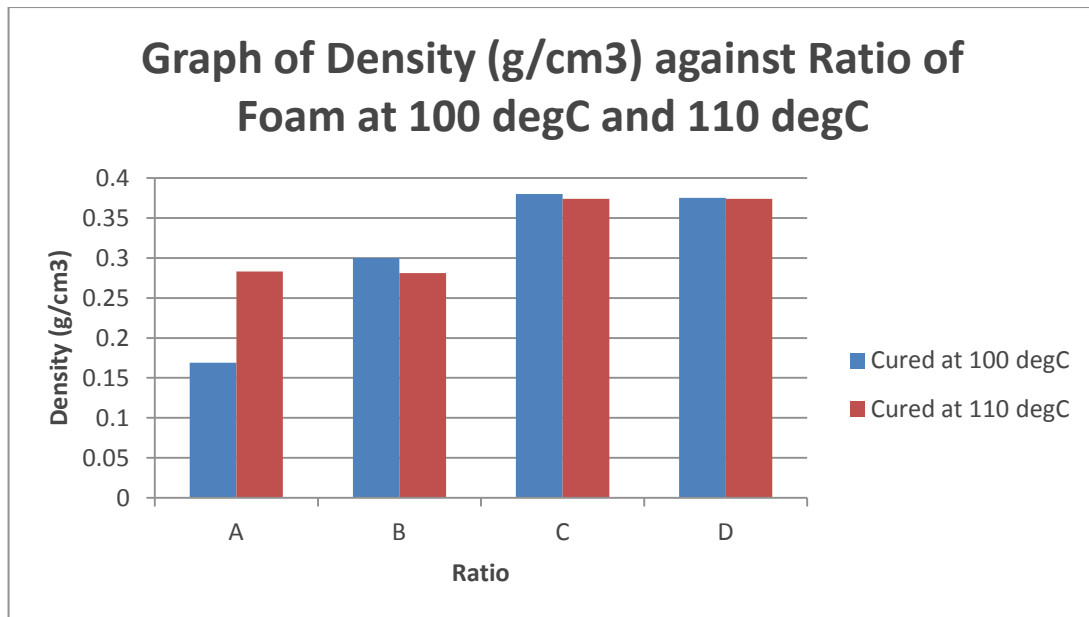


Figure 12: Graph of Density (g/cm³) against Ratio of Foam at 100 °C and 110 °C

From the graph, the density of foam with Ratio A cured at 100 °C shows the lowest density. As the amount of starch increases, the density of the foam also increases. The same trend also applied to the sample cured at 110 °C although the density of Ratio A foam cured at 100 °C is much more lower than Ratio A foam cured at 110 °C.

4.3. Mechanical Properties

To study the tensile strength of the foam, the foam needs to be cut down into strips. The dimensions of the strips are; 80 mm in length, 10 mm in width and thickness varies. The strips are placed in the tensile strength machine as shown in Figure 13.



Figure 13: Placement of foam strip with the tensile strength machine

However the tensile test could not be completed as the foams tend to break where the gauge was clipped. It is concluded that the tensile test could not be done with the foam as the foam is categorized as being too soft and delicate. Hence the data of tensile strength could not be obtained.

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

The sample made with Ratio A cured at 100 °C has shown the most suitable properties of a foam. The physical condition of Ratio A sample does not show any defects such as holes in the surface and the foam expands appropriately based on the recorded thickness of Ratio A foam which is 0.5 cm while other samples average thickness is 0.3 cm. When the curing temperature is increased to 110 °C, the foam formed tends to expand creating a void space in between the foam. When cut into strips, the void space collapses hence making the thickness of the sample less than Ratio A cured at 100 °C. When the curing temperature decreased to 90 °C, the sample does not cure properly; does not dry properly hence the sample could not be examined its thickness and density.

The density of Ratio A cured at 100 °C is 0.169 g/cm³ and was the lowest recorded density compared to other foam samples. As the ratio of starch is increased in the sample, the density of the foam also increased. The highest density of foam recorded was Ratio C sample cured at 100 °C.

The mechanical properties of the foam could not be taken as the sample foams are categorized as being too soft and delicate. The foam strips tore at where the gauge clip was clipped. Hence the reading of the tensile strength could not be measured. To overcome this problem in the future, the sample should be made in a bigger size using a bigger mould. This will decrease the chances of the sample to tear down where it being clipped.

There are some recommendations and adjustments that could be made if the study should be repeated and improvised. One of them is to change the variables. The variables that could be changed are the additives and plasticizers ratio as well as the mixing time and the mixing temperature. This to ensure the elasticity and texture of the foam hence could withstand tensile strength machine. The biodegradation rate should be added as the measured parameters. Another recommendation is to produce a bigger sample. Bigger sample could withstand the tensile strength machine.

In conclusion, the physical properties of the foam sample has been studied and foam sample with Ratio A cured at 100 °C has shown the best physical properties compared to other samples. Foam sample with Ratio A cured at 100 °C has the lowest

density which is 0.169 g/cm^3 compared to other samples. The surface condition of the sample also does not show any significant defects such as holes and bloating. Hence the first objective of the study is achieved. However, the second objective of this study; to study the effect of curing temperature of starch and PVA to the physical and mechanical properties of the foam, could not be achieved as the foam strip failed the tensile strength test.

REFERENCES

- Ahmed, J., Tiwari, B. J., Imam, S. H., & Rao, M. A. (2012). *Starch-Based Polymeric Materials and Nanocomposites*. Boca Raton: CRC Press.
- Azhari, N. A., Othman, N., & Ismail, H. (2011). Biodegradation Studies of Polyvinyl Alcohol/Corn Starch Blend Films in Solid and Solution Media. *Journal of Physical Science, Vol. 22(2)*, 15-31.
- Bhatnagar, S., & HANNA, M. A. (1996). Starch-Based Plastic Foams From Various Starch Sources. *Cereal Chem. 73(5)*, 601-604.
- Ghanbarzadeh, B., & Almasi, H. (2013). Biodegradable Polymers. In R. Chamy, & F. Rosenkranz, *Biodegradation - Life of Science* (pp. 141-185). InTech.
- Giles, G. A., & Bain, D. R. (2000). *Materials and Development of Plastics Packaging for The Consumer Market*. London: Sheffield Academic Press.
- Giles, G. A., & Bain, D. R. (2001). *Technology of Plastics Packaging for The Consumer Market*. London: Sheffield Academic Press.
- Hassan, C. M., & Peppas, N. A. (2000). Structure and Applications of Poly(vinyl alcohol) Hydrogels Produced by Conventional Crosslinking or by Freezing/Thawing Methods. *Advances in Polymer Science, Vol. 153*, 37-65.
- Hulleman, S. H., Janssen, F. H., & Feil, H. (1998). *Polymer*, 2043-2048.
- International Organization for Standardization. (2012). *ISO 17088:2012 - Specifications for compostable plastics*. Retrieved from www.iso.org:
http://www.iso.org/iso/catalogue_detail.htm?csnumber=57901
- Kaewtatip, K., Tanrattanakul, V., & Phetrat, W. (2013). Preparation and Characterization of Kaolin/Starch Foam. *Applied Clay Science 80-81*, 413-416.
- Kolybaba, M., Tabil, L. G., Panigrahi, S., Crerar, W. J., Powell, T., & Wang, B. (2003). *Biodegradable Polymers: Past, Present, and Future*. North Dakota: The Society for Engineering in Agriculture, Food, and Biological System.
- Mao, M., Imam, S., Gordon, S., Cinelli, P., & Chienelli, E. (2000). *Journal of Polymers and Environments*, 205-211.
- Mitrus, M., & Moscicki, L. (2013). Extrusion-Cooking of Starch Protective Loose Fill-Foams. *Chemical Engineering Research and Design*.
- Narayan, R., Doi, Y., & Fukada, K. (1994). Impact of Government Policies, Regulations, and Standards Activities on an Emerging Biodegradable Plastics Industry. *Biodegradable Plastics and polymers*, 261.

- Park, H.-R., Chough, S.-H., Yun, Y.-H., & Yoon, S.-D. (2005). Properties of Starch/PVA Blend Films Containing Citric Acid as Additive. *Journal of Polymers and the Environment*, Vol. 13, No. 4, 375-382.
- Parvin, F., Rahman, M. A., & Islam, J. M. (2010). Preparation and Characterization of Starch/PVA Blend for Biodegradable Packaging Material. *Advanced Materials Research*, Vols 123-125, 351-354.
- Polat, S., Uslu, M.-K., Aygun, A., & Certel, M. (2013). The Effects of The Addition of Corn Husk Fibre, Kaolin and Beeswax On Cross-linked Corn Starch Foam. *Journal of Food Engineering* 116, 267-276.
- Rudnik, E. (2008). *Compostable Polymer Materials*. Oxford: Elsevier.
- Selke, S. E. (1997). *Understanding Plastics Packaging Technology*. Munich: Carl Hanser Verlag.
- Selke, S. E., Culter, J. D., & Hernandez, R. J. (2004). *Plastics Packaging: Properties, Processing, Applications, and Regulations*. Munich: Hanser Publishers.
- Shogren, R. L., Lawton, J. W., & Tiefenbacher K., F. (2002). Baked starch foams: starch modifications and additives improve process parameters, structure and properties. *Industrial Corps and Products* 16, 69-79.
- Smith, R. (2005). *Biodegradable Polymers for Industrial Applications*. Cambridge: Woodhead Publishing Limited.
- Tang, X., & Alaavi, S. (2001). Recent Advances in Starch, Polyvinyl Alcohol Based Polymer Blends, Nanocomposites and Their Biodegradability. *Carbohydrated Polymers* 85, 7-16.
- Tudorachi, N., Cascaval, C. N., Rusu, M., & Pruteanu, M. (2000). Testing of Polyvinyl Alcohol and Starch Mistures As Biodegradable Polymeric Materials. *Polymer Testing* 19, 785-799.
- Vasile, C., & Zaikov, G. E. (2009). *Environmentally Degradable Materials Based on Multicomponent Polymeric System*. Leiden: VSP.
- Vroman, I., & Tighzert, L. (2009). Biodegradable Polymers. *Materials*, 307-344.
- Zhai, M., Yoshii, F., & Kume, T. (2003). *Carbohydrate Polymers*, 311-317.

APPENDICES



Appendix 1: Foam strip cut down to 80 mm length and 10 mm width



Appendix 2: Determination of strip thickness using a vernier calliper



Appendix 3: Tensile strength machine used in Block 17, Universiti Teknologi PETRONAS

BRITISH STANDARD

Plastics — Determination of tensile properties —

Part 1: General principles

BS EN ISO
527-1:1996
BS 2782-3:
Method 321:
1994
ISO 527-1:
1993
*Incorporating
Amendment No. 1*

The European Standard EN ISO 527-1:1995 has the status of a
British Standard

IMPORTANT NOTE. Before reading this method it is essential to read BS 2782-0 Introduction,
issued separately.

ICS 83.080

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW



Appendix 4: Standard for tensile strength determination