

BIODEGRADABLE FILMS FROM POLY (LACTIC ACID) (PLA)-
CHITOSAN-POLYETHYLENE GLYCOL (PEG): FABRICATION AND
EVALUATION OF MECHANICAL PROPERTIES

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POLYETHYLENE GLYCOL (PEG): FABRICATION AND EVALUATION OF
MECHANICAL PROPERTIES

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this project is my own except for quotations and summaries which have been duly acknowledged. The project has not been accepted for any degree and is not concurrently submitted for award of other degree.

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BIODEGRADABLE FILMS FROM POLY (LACTIC ACID) (PLA)-CHITOSAN-POLYETHYLENE GLYCOL (PEG): FABRICATION AND EVALUATION OF MECHANICAL PROPERTIES

ABSTRACT

In this research, the production of biodegradable plastic food packaging from biopolymers poly (lactic acid) (PLA), chitosan and polyethylene glycol (PEG) was investigated. In addition, the objective for this research also wants to study the mechanical properties of biofilms at different percentage of weight of polylactic acid, chitosan, and polyethylene glycol (PEG). The biodegradable plastic food packaging was prepared by blending the films based on these four main chemicals (PLA, chitosan, PEG) and 30 ml of silver nanoparticles for every blend of biofilms. Then tested the films with universal testing machine according to American Standard Test Method (ASTM D882) to investigate the mechanical properties (tensile and elongation) with 1 mm thickness film. Then, the mechanical properties of biodegradable food packaging films optimized by changing the parameters of percentage polyethylene glycol (PEG) and ratio of chitosan to PLA using Response Surface Method (RSM) in order to obtain the good biodegradable food packaging films. Degradation rate test has been determined by using the soil burial degradation rate method by burial the blend films for 14 days. The results show that, at the optimum condition, the tensile strength reached the maximum value of 10.573 MPa when the PEG is 17.6 % and PLA is 71.21%. For the elongation at break point, the optimum condition for the elongation at the break point reached the maximum value of 68% when the PEG is 22.07% and PLA is 50%. For the biodegradation rate indicate that the highest value of the chitosan will have the highest degradation rate among the prepared biofilms. As a conclusion the blend films produces will have a good mechanical properties that and can degraded easily.

**BIODERADASI FILEM DARIPADA POLILAKTIK ASID (PLA) KITOSAN-
POLYETHYLENE GLYCOL: FABRIKASI DAN PENILAIAN CIRI
MEKANIKAL**

ABSTRAK

Dalam kajian ini, penghasilan biodegradasi plastik bungkusan makanan telah dihasilkan daripada biopolimer polilaktik asid (PLA), kitosan dan polietilena glikol (PEG) .Di samping itu, objektif kajian ini juga adalah untuk mengkaji sifat-sifat mekanikal biofilm pada komposisi yang berbeza sama ada melalui perbezaan komposisi terhadap Poli laktik asid/kitosan, dan polietilena glikol (PEG). Bungkusan plastik makananan yang mesra alam ini telah dihasilkan dengan mencampurkan empat bahan kimia utama iaitu (PLA, kitosan, PEG) dan 30 ml nanoparticle perak untuk setiap campuran biofilm. Kemudian file mini diuji dengan mesin ujian sejagat menurut Kaedah Ujian American Standard (ASTM D882) untuk menyiasat sifat-sifat mekanikal (tegangan dan pemanjangan) dengan 1 mm ketebalan filem. Kemudian, sifat-sifat mekanikal filem pembungkusan makanan terbiodegradasi dioptimumkan dengan menukar parameter peratusan polietilena glikol (PEG) dan nisbah Chitosan untuk PLA menggunakan Response Kaedah Permukaan (RSM) dalam usaha untuk mendapatkan pembungkusan yang baik dalam menghasilkan filem makanan terbiodegradasi. Degradasi ujian kadar telah ditentukan dengan menggunakan teknik kambusan tanah dan menunjukkan kadar degradasi oleh kambusan tanah telah dikaji selama 14 hari. Keputusan menunjukkan bahawa, pada kondisi optimum, kekuatan tegangan mencapai nilai maksimum 10,573 MPa apabila PEG adalah 17.6% dan PLA adalah 71,21%. Untuk pemanjangan pada titik filem itu terputus,, keadaan optimum untuk pemanjangan filem itu putus mencapai nilai maksimum sebanyak 68% apabila PEG adalah 22,07% dan PLA adalah 50%. Bagi kadar biodegradasi menunjukkan bahawa nilai tertinggi kitosan akan mempunyai kadar degradasi tertinggi daripada biofilm yang telah dihasilkan. Sebagai kesimpulan filem campuran menghasilkan akan mempunyai sifat mekanikal yang baik dan boleh dengan mudah terurai.

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LIST OF SYMBOLS

°C	Degree Celsius
g	grams
h	hour
L	Liter
mL	milliliter
mm	millimeter
min	minutes
% w/w	ratio of weight percentage

LIST OF ABBREVIATIONS

CH ₃ COOH	Acetic Acid
DD	Degree Deacetylation
PCL	Poly (ε-caprolactone)
PEK	Polyethylketone
PGA	Poly (glycolic acid)
PLA	Poly (lactic acid)
PHB	Poly (3-hydroxybutyrate)
PHBV	Polyhydroxyvalerate
PVC	Polyvinyl chloride

CHAPTER 1

INTRODUCTION

1.1 Background of propose study

Development of plastic food packaging that made from fossil fuels can cause harmful to the human bodies and environmental because it have the carcinogen characteristic and take time to degradable maybe hundred year. According to Doi Fukuda (1994), the word “biodegradable” means the materials which can be degrade by the enzymatic action of living organism such as bacteria, fungi, yeast, and the ultimate end-product of the degradation process. Biomass also act as supported degraded agent and can be divided into two that’s biomass under aerobic condition and hydrocarbon, methane and biomass under anaerobic conditions.

The most dangerous thing in plastic food packaging to human bodies when it can cause carcinogen because the material made from the Polyvinyl chloride (PVC). Producing the polyvinyl chloride (PVC) making the vinyl chloride released in to the air and wastewater. Vinyl chloride is known as the human carcinogen chemical and has been linked to increased mortality from liver and breast cancer among workers that involved in its manufacturing. In addition, it can cause the migration between plastic food packaging and food content. Besides that, there are another chemical that has been reported for having a migration when expose to food for example di(2-ethylhexyl) adipate(DEHA). The specific migration limit (SML) set for di(2-ethylhexyl) adipate (DEHA) by the EU is 18 mg/kg food and according to EC (2002), migration values for packaging films must be expressed in term of surface area (mg/dm^2). Because of the adverse impact on the use of plastic food packaging, recent research and development efforts succeeded in producing of biodegradable polymers which can be found from renewable resources.

The plastic that have both characteristics and the biofilms that have less impact on the environment, human bodies is needed. Biopolymers can be defined from the input point of view as polymers that mean biopolymer is polymers produced by living organism. Biopolymers are polymers that have the monomers, which are totally or mainly contained in the biomass, or which can be made from biomass using biotechnological processes. Biodegradable polyester can be divided into four different group based on their chemical structure. Polylactic acid and polyglycolic acid are the example of biodegradable polyester generally used to make scaffolds.

In addition due to the biodegradable plastic food packaging, PLA and chitosan are used because the chitosan itself have a good antimicrobial activity, and according by S.Hirano (1996). Chitosan is a monocompenant of microbial agent that has advantages of biodegradability and excellent film forming ability. To make chitosan better as an antimicrobial agent some modification must be made into chitosan with the addition of organic metal such as argentum (Ag), and zinc oxide (ZnO). Therefore it will give the good characteristic on the tensile properties, the moisture of the biofilms and the antimicrobial properties.

1.2 Problem statement

The producing of plastic food packaging by synthetic polymers are not easily degrade and it's produce harmful gases that can cause harmful to human bodies because of the carcinogen characteristic and harmful to environmental pollutants because the degrade of synthetic polymers take hundred years to degrade. To overcome this problem biopolymer has been introduced from polylactic acid combine with polyethylene glycol (PEG) and chitosan in order to improve the strength towards any barrier and have well degradable after disposal.

1.3 Objectives

1. To produce the biodegradable plastic food packaging from PLA, Chitosan and polyethylene glycol (PEG).
2. To investigate the effect of tensile strength and elongation at break point by considering the different percentage of PEG and PLA/chitosan in blending the biodegradable food packaging biofilms.
3. To evaluate the tensile strength properties of the synthesized plastic.

1.4 Research questions

1. What is the advantage of biodegradable plastic food packaging from PLA, chitosan and Ag-nanoparticles?
2. What methods are used to producing the biofilms?
3. What are the parameter needed to be considered in optimize the performance of biofilms?

1.5 Scope of studies

To achieve the objective of the research scope has been identified:

1. Preparing the biofilms from different concentration of PLA/Chitosan(range in 20 to 80 %) and polyethylene glycol (PEG)(ranged in between 5 to 25 % of PEG) by using the fabrication method.

2. Testing the tensile strength properties of the produced biofilms plastic by using the ASTM D882 method.

1.6 Significance of the study

The significant of this study is to produce biodegradable plastic food packaging from PLA and also modified those biopolymers with chitosan and polyethylene glycol to make it resistance to any moisture and also increase the mechanical properties of the biodegradable plastic

CHAPTER 2

LITERATURE RIVIEW

2.1 Conventional Plastic

The demanding for plastics is increasing due to the wide application for human being. Production of plastic from petroleum base increasing with an annual world production of approximately 140 million ton (Masayuki 2001). In other hand, the production of plastic from natural or synthetic materials also can be produced for example the polyvinylchloride (PVC), polylactic acid (PLA) and polyhydroxybutyrate (PHB). There is the conventional plastic made from petroleum that can be degraded by environment and a few of companies trying harder to produces the plastic from the petroleum-based product. The fragments plastic leaves small pieces take hundred years to degrade or can take many decades to fully disappear.

The plastic can be divided into many categories such as polyethylene terephthalate (PET), poly vinyl chloride (PVC), polyester (PET) and others and every category have different risk. The usage of the high-density polyethylene to produce a grocery bag also known as HDPE. PET is used widely to produce plastic because of the manipulation on its size, colour and shape. The usage of plastic can give the impact to environment including air, land and water pollution. The disposal of plastic material also can affect the human and animal health. According to the Badami (2005), at least 1000 death of Mumbai people and additional people suffering injuries when the huge monsoon flooding occurs in the city of Mumbai been blamed by the destructive floods on plastic bags which clogged gutters and drains that prevent the rainwater from leaving the city through underground system. When the drains are blocked by the plastic material cause the water in stagnant position and the water cannot flow freely and the stagnant water will produces the ideals habitat for mosquitoes and other parasites.

In other hand, the disposal of plastics to the ocean will make the animal extinct from the earth for example over a billions seabirds and mammals including turtles die annually from ingestion of plastic (Andrady 2011). It also reported that, the plastic groceries bag have the potential to resulting the health of human being in danger condition with the neurological problems and cancer because of the leaching of the chemical component into our water resources and soil. To overcome this problem many researcher come out with new invention about the biodegradable and antimicrobial of plastic.

2.2 Biodegradable Polymers

The synthetic polymers increasing the demanding due to the need plastics in daily life and the problem is how to dispose or to manage the disposal after it just used. These scenarios become a serious problem and due to this the few researchers try to find alternative about the way to remove, replace or to overcome this problem. When the recycling the synthetic polymers, the plastic food packaging cannot be recycle back into plastic food packaging again and another option left by produce the biodegradable plastic food packaging and sustainability. According to Brundtland commission defined the word sustainability as “social economic advance to assure human beings a healthy and productive life, but one that did not compromise the ability of future generations to meet their needs. Then, the society just starting to understand what the earth needs to avoid any pollution and the biopolymers been increase for the last two decades (Vilaplana, 2010).

Biodegradable polymer can be defined as the polymer that able to undergo hydrolysis at temperature 50°C (Gupta and Kumar 2007). Besides the biodegradable characteristics the polymer also must be non-toxic. The biodegradable can be divided into two types which are natural polymer or based on renewable starch. These include polysaccharide (starch, cellulose, chitin, chitosan and etc.), proteins (gelatin, silk, and wood) and polyester produced from microorganisms, polyester derived from bioderived monomers including polylactic acid, polyhydroxyalkanoate and polyhydroxybutyrate. Petroleum can be used to produce the biodegradable polymer and a few companies try to make the biodegradable plastic from the petroleum since it consumed by microbes and

biodegrade in compost environment. For the example the BASFTM just used the aliphatic-aromatic and ϵ -caprolactam to make the biodegradable plastic. Polyester is one of the biodegradable polymer and according by (Müller *et al.*, 2001) the structure of polyester can be undergo hydrolysis under certain condition and it can be divided into two major group of polyester, that's aliphatic polyester and aromatic polyester. However, the aliphatic polyester show excellent result in degradation process compare with aromatic polyester because of the stability of the aromatic ring. To break the chain of aromatic will need more energy and time because the ring is stronger and more stable compare to linear chain. By the way, both of them can show the fully biodegradable (Witt *et.al* 2001).

2.3 Chitosan

In order to produce the excellent biopolymers some chemical improvement must be considered by adding the chitosan into biofilms. According to (George *et al.*, 2011) chitin's is the one of polymeric materials that is easier to get and have a lower cost to purchase and it is one of the most abundance polysaccharides and can be found in various invertebrates and lower plants. Chitins also are one of the most abundance polysaccharide that is syntheses in lower animal such as crab shell or shrimp after cellulose. According by (Muzzarelli, 1997), the structural of chitin is quite similar to cellulose but having a different in acetamide group at the C-2 positions in place of hydroxyl group in amino polysaccharide. The different are shown in Figure 2.1.

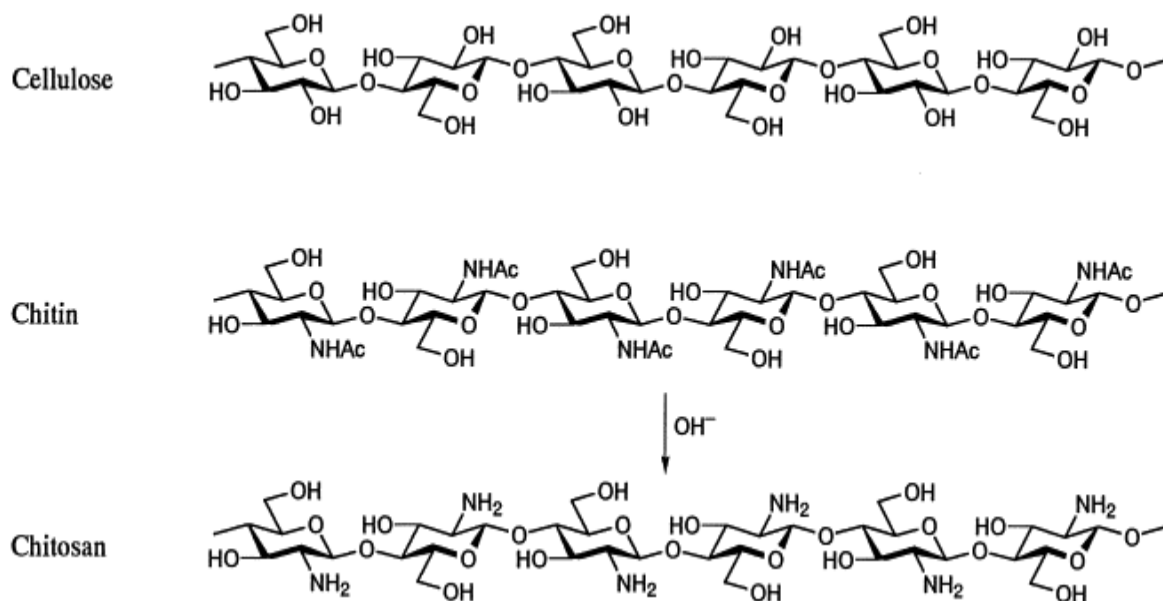


Figure 2.1 The structural formula for cellulose, chitin, and chitosan

For this study, chitosan will be chosen as a polysaccharide to making the biofilms even though it is the second polysaccharide most abundance compare to cellulose because it have the excellent biodegradable, bioactivity and biocompatibility. Chitosan can be prepared by deacetylating α -chitin and the study by Horton (1956) chitosan is commonly prepared by deacetylating α -chitin by using 40-50% aqueous alkali at 100-160°C for a few hours and the process as shown in Figure 2.2.

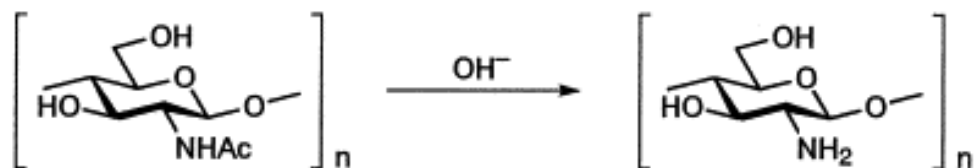


Figure 2.2 The mechanism of chitosan from α -chitin

Chitosan have similar chemical properties with chitin that a most stable substances against acid and alkali and this component also not soluble in ordinary solvent. When chitosan mixed with polylactic acid (PLA), the toxicity of the PLA will be reduced because chitosan can generate acidic degradation product as an implanted site reaction that raised the unwanted product for the tissue reactions.

According to Gandini (2008), chitosan is one of the most important polymer in biomedical application because of the characteristic like biodegradability, biocompatibility and low toxicity, and all the characteristic have led to the development of numerous application in biomedicine like surgical sutures, biodegradable sponge and bandages. According by (Amidi *et al.*, 2010), the important of chitosan in biomedicine including microcapsules and the delivery of drug (glaucoma treatment) and the best thing about chitosan in biomedical is due to excellent properties when interacting in human bodies. For the bandage that come from the chitosan have a ability to accelerate the wound healing effect and due to its biodegradability characteristic, it has been applied in tissue regeneration and tissue restoration. Chitosan also can be improved its properties by combine it with silver nanoparticle or any inorganic metal such as argentine(Ag), zinc oxide (ZnO), silicon dioxide (SiO₂), and titanium dioxide (TiO₂) for improving the degradation rate, mechanical strength and antimicrobial activities.

2.4 Polylactic Acid (PLA)

Biodegradable polymer such as poly (lactic acid) (PLA), poly (glycolic acid) (PGA) and poly (3-hydroxybutyrate) (PHB) are classified as a biodegradable polymer and among of these three, the PLA will give more attention due to its renewable resources, biocompatibility, biodegradation, excellent thermal and mechanical properties and superior transparency biopolymers. PLA can behave likes PET and also performs a lot like polypropylene (PP), a polyolefin. PLA also has good organoleptic characteristics and best for food contact and related food packaging application.

Nowadays, PLA can be a successful material in replacing petroleum based plastic in commodity application, with most initial uses limited to biomedical applications such as suture (Sinclair,1986). PLA have been produced by from renewable resources and it is a linear aliphatic thermoplastic polyester and due to the easily to degraded through enzymatic pathways and hydrolytic. It also can be synthesized by using the condensation polymerization of the monomer lactic acid. To obtain the monomer lactic acid through the fermentation process of lower cost material starch including corn, potato, sugar beat and sugar cane. PLA can be produced for a large scale production through injection molding, blow molding and extrusion. To avoid the PLA from undergo degradation, the polymer must undergoes thermal degradation at temperature above 200°C (Jhamshidi *et al.*, 1988). Catalyst also helping in the degradation processes by decreases the degradation temperature and increase the degradation rate of PLA.

Lactic acid (2-hydroxy propanoic acid) is the simplest hydroxyl acid with an asymmetric carbon atom and exist in two optical configurations. Human bodies and wild mammals will produce the L (+)-isomer, meanwhile the bacterial system will produce the L (+)-enantiomers and D(-). The organisms that predominantly yield the positive – isomers are *Lactobacilli amylophilis* and *L. salivarius* and both bacteria are used to fermentation to produce the lactic acid. The physical properties of PLA by addition of clay have been extensively reported by (Ahmed *et al.*, 2010). The fabrication of PLA with clay nanocomposites were mixed with the PLA matrix and originally-modified clay using melt blending the result indicates the fabricated nanocomposites with the various surface modification of clay in improving the mechanical testing and the degradation rate.

CHAPTER 3

METHODOLOGY

3.1 Materials

Chitosan flakes from crab shells were purchased from Fisher Scientific, Polyethylene glycol (PEG) 400 and Polylactic acid (PLA) (pellet form) were supplied form by Sigma Aldrich. The acetic acid was obtained from Sigma Aldrich and chloroform was provided by Maersk Supplier Company. The preparation of silver nanoparticles from tea extract solution was used by using the tea BOH and mix with the silver nitrate.

3.2 Preparation of silver nanoparticles

1. 5g of tea BOH grounds has been added with 500 ml of water. Make the solution to blend for 1 hour and leave the solution to settle for a day after it mixes.
2. The solution then was filter to remove the tea levees by using the filter tea.
3. The process just been repeated by using filter paper to remove the particulate tea from the solution.
4. Lastly the solution will be filter again by using the filter membrane of 0.2 micrometer and the type of the membrane filter is (cronus membrane nylon).
5. Prepare the solution of 0.1 M AgNO_3 with dilute the 1.6987g of AgNO_3 in 100 ml distilled water.
6. The extract tea then will be mix with the solution of AgNO_3 with 10 ml of extract plus with 2 ml of 0.1M solution AgNO_3 .
7. Then the solution will be stirrer for 1 day for every solution.
8. Lastly the 12 ml solution consist the extract tea and 2 ml of extract solution will be undergo washing process to remove the poly phenol that consist in the silver nanoparticles by using the centrifugal machine for 10 minutes with speed of 10000 rpm. The washing process will repeat for 4 times by washing the solution with DI water.

3.3 Preparation of Biodegradable Food Packaging Films.

1. Solution of chitosan in 1% w/w of acetic acid was prepared by adding 1 gram of chitosan in 100 ml of acetic acid in conical flask. After that, this mixture solution was poured together in a beaker while heated and stirred using magnetic stirrer simultaneously for 90 minutes.
2. Solution of PLA in 1% w/w of chloroform was prepared by adding 1 gram of PLA in 100 ml of chloroform in conical flask. After that, this was poured together in a beaker while heated and stirred using magnetic stirrer simultaneously for 90 minutes. The preparation of PLA was handled in fume chamber to avoid direct inhalation of chloroform
3. . Then, PEG was added into the mixture solution of PLA and chitosan until the percentage of PEG was about 15% of solution. However the chitosan solution was filtered first before adding to. In the end, the solution was heated and stirred for 1 hour.
4. Then, the films were prepared according to the weight ratio of PLA and chitosan of 50/50 and then the solution was stirred about 30 minutes before added into Petri dish. The stirring was used to homogenize the mixture prior to adding the solution into the dish. Make sure the thickness of films must be 1mm.
5. Then the casting was left dried in the oven at the room temperature of 30°C for 1 day.
6. The experiment was repeated with different weight ratio of PLA and chitosan and with different percentage amount of PEG used towards PLA.

The Table 3.1 below shown the preparation of films with different weight ratio of PLA with chitosan and with different of percentage PEG used towards PLA.

Table 3.1 The different composition to produce biofilms

Std	Run	Variable 1 PEG content %	Variable 2 PLA content %	response 1 Tensile MPa	response 2 Elongation at break point %
10	1	15.00	50.00		
7	2	15.00	20.00		
9	3	15.00	50.00		
2	4	22.07	28.79		
6	5	25.00	50.00		
4	6	22.07	71.21		
1	7	7.93	28.79		
8	8	15.00	80.00		
11	9	15.00	50.00		
12	10	15.00	50.00		
3	11	7.93	71.21		
5	12	55.00	50.00		

3.4 Soil Burial Degradation

The soil burial degradation test was carried to evaluate the biodegradation rate of blends of chitosan and poly (lactic) acid (PLA) (CS/PLA) film at different composition of PEG and different composition of PLA. In addition, every blend was blended with 30 ml of silver nanoparticle. The garden pots were filled with soil taken from field around the Universiti Malaysia Pahang. The films were cut into 2x2 cm pieces and weight to obtain the initial weight (W1). The films were buried in the soil at depth of 5 cm. The pots were placed in an uncovered place near the laboratory. The soil was kept moist by sprinkling water at regular time interval to maintain humidity. The excess water was drained

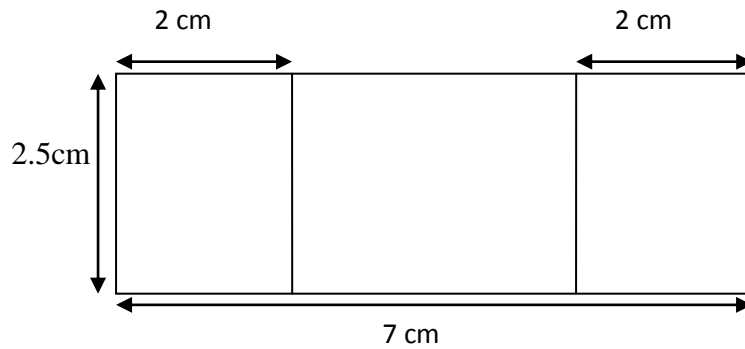
through the hole of the bottom of the pot. The degradation of the films was determined at a regular time interval (14 days) by taking the films carefully from the soil and washing it gently with distilled water to remove the soil. The films were dried in the oven at temperature 30°C for 20 minutes until the constant weight was obtained (W2). Weight loss of the sample with time was used to indicate the degradation rate in the soil burial test. The degradation rate was calculated as equation (3.1)

$$\text{Degradation rate} = (W2-W1/W1) \times 100 \quad (\text{Eq. 3.1})$$

3.5 Mechanical testing machine test

Tensile strength and elongation of biofilms were determined by using the universal testing machine according to the American Standard Test Method of ASTM D882. The testing the tensile properties were followed by t the following procedure:

1. A film sample was cut into rectangular specimens of 2.5 x 7 cm.
2. For the sample size, about 2 cm above and below the sample is needed for the grip when the tensile strength is run.



3. The initial grip separation and cross-head speed was set on universal testing machine. Then the cross-head at the machine was set at 1 mm/min of speed.
4. For each sample the mechanical properties were reported as mean and \pm standard deviant.
5. Tensile strength is calculated by dividing the maximum load to break the film by the cross-sectional area of the film.
6. Elongation was obtained by dividing film elongation at rupture by the initial gauge length of the specimen and multiplying by 100. In this case the elongation at the rupture will be divided by 30 mm (initial length of the films before undergoes mechanical testing).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Soil burial degradation test

In this study, the biodegradation rate for the bio film loaded with chitosan, polylactic acid (PLA) and silver nanoparticle has been test for 2 weeks. The sample has been leaves in environmental condition where as the temperature test is under room temperature. The soil test biodegradation is test by using the acidic soil at pH 4.96 (soil is retrieved at laboratory of civil engineering University Malaysia Pahang).

Table 4.1 Data for analysis of the degradation rate for the biofilms samples.

Sample films	Weight before degradation process (W ₁)g	Weight after degradation process after 2 weeks (W ₂)g	% of degradation rate $(w_2-w_1/w_1) \times 100$
PEG 15% with PLA 50%	0.0165	0.0139	15.76
PEG 15% with PLA 80%	0.0184	0.0164	10.89
PEG 22.07% with PLA 71.21 %	0.0193	0.0169	12.44
PEG 22.07 % with PLA 28.79 %	0.0236	0.0193	18.22
PEG 7.93 % with PLA 71.21 %	0.0133	0.0116	12.78
PEG 7.93 % with PLA 28.79 %	0.0171	0.0142	16.96

In this study, the evaluation of weight loss of the sample of the biofilms containing the different percentage value of polylactic acid (PLA) and polyethylene glycol (PEG). In this study, the 6 samples were used for different percentage of PEG with PLA (PEG 15% with PLA 50%, PEG 15% with PLA 80%, PEG 22.07% with PLA 71.21 %, PEG 22.07 % with PLA 28.79 %, PEG 7.93 % with PLA 71.21 %, and PEG 7.93 % with PLA 28.79 %). for every blend of films that consist of 30 ml of silver nanoparticles appear no bacteria or microorganism to growth or inhibit at the surface of the biofilms. In this case, the Ag⁺ will be released it ion in controlled manner which in turn leads to a powerful antibacterial activity against a wide spectrum of bacteria (Fortunati *et al.*, 2010). Therefore the silver nanoparticles will decrease the degradation

rate of the biofilms due to the ion released compared with the film without the modification with silver nanoparticles.

For Table 4.1 the degradation rate for PEG 22.07 % with PLA 28.79 % has the highest degradation rate compared to all biofilms samples. It's because the low percentage of PLA towards chitosan degrade the biofilms faster. PLA is a polymer that have lower degradation rate but when it was blended with chitosan, the degradation rate faster due to the chitosan characteristic that have the hydrophilic functional group which can absorb more water from the surrounding and the moisture also can affect the degradation rate of the PLA/Chitosan blend (Peesan *et al.*, 2005). The presence of PEG 22.07 % in the blend films of PEG 22.07% with PLA 28.79% cause the biofilms degrade more faster even though the PEG content increase the plasticizer of the biofilms and the chain of the plastic extended into longer molecular weight(Zhao *et al.* 2006). This is because of the hydroxyl group at least one hydroxyl group of the PEG content that can make the biofilms degrade faster (Chiellini *et al.* 2003). For the degradation rate of PEG 15 % with PLA 80 % indicate the lowest rate of degradation rate with 10.89 % lowest compared with other samples. For the 80 % PLA, the percentage remaining for the blend films is 20 % of chitosan in 30 ml of blending solution of biofilms. The data shown in Table 4.1 has the lowest rate of degradation due to the amount of PLA in the biofilms. PLA is a polymer that has a lowest degradation rate and has a higher tensile strength (Tokiwa *et al.* 2009). More percentage of PLA in the blend film makes the plastic less easy to degrade. Moisture, microorganism, temperature and whether also play a role important factor in degradation rate.

4.2.1 OPTIMIZATION OF BIODEGRADABLE FILMS

This section was conducted to study the optimization of the biodegradable films from PLA, PEG, chitosan and silver nanoparticles. In this study the blend film was tested on two factors by using the universal testing machine to indicate the value for tensile strength and elongation at break point. To maximize the tensile strength and the elongation at break point, optimization processes are performed by using the response surface methodology (RSM) with central composite design in order to attain optimum value of the tensile strength and elongation at break point. RSM is a collection of statistical and mathematical technique which is very hands for developing, improving, and optimizing processes. RSM has the ability to predict the influence and interaction between many variables that could affect the performance measure of effect. RSM is one of the software that has a technique to design the experiment by providing the adequate and reliable measurement of the response. It can develop the mathematical model having the best fit to the data obtained from the experimental design and can determine the optimum value of the independent variables that produces maximum or minimum value for the response.

4.2.2 Experimental Design Using Response Surface Methodology (RSM)

In this study, the response surface methodology (RSM) was utilized to determine the optimum of the biodegradable films by selected two parameters that can affect the elongation at break point and the tensile strength reading.

The two parameters which are;

1. The percentage of the Poly ethylene glycol (PEG)
2. The percentage of the poly lactic acid (PLA)

Experiment design was performed by using the Central Composite Design (CCD) and the design consist of 12 experiments. Two responses will be choosing for the experimental modelings which are tensile strength and the elongation at break point. In CCD, the variance of the predicted response Y at the same point of X (process variable) is only a function of the distance from the point to the design centre. The basic theoretical aspect, the fundamental assumptions and the experimental implication of RSM was discussed elsewhere (Rahman *et al.* 2007).

4.2.3 Statistical Modelling

Experiment range and level of independent process variables, X_1 is a percentage of PEG and X_2 is the percentage of PLA and to fit the response function and experimental data, regression analysis was performed and the second order model was evaluated by (ANOVA) for dependant variables on response Y where (Y_1 is a tensile strength and Y_2 is a elongation at break point) which are presented in Table 4.2.

Table 4.2 Data for design RSM method for tensile strength and elongation at break point

Std	Run	Variable 1 PEG content %	Variable 2 PLA content %	response 1 Tensile MPa	response 2 Elongation at break point %
10	1	15.00	50.00	7.786	65.21
7	2	15.00	20.00	3.468	58.56
9	3	15.00	50.00	7.835	61.89
2	4	22.07	28.79	4.325	66.08
6	5	25.00	50.00	7.151	73.16
4	6	22.07	71.21	11.113	64.12
1	7	7.93	28.79	4.447	55.99
8	8	15.00	80.00	12.376	60.79
11	9	15.00	50.00	7.450	58.19
12	10	15.00	50.00	7.955	68.25
3	11	7.93	71.21	9.485	53.68
5	12	5.00	50.00	7.100	48.14

4.2.4 Tensile strength and elongation at break point

The result obtained from Table 4.2 indicate that at 15% PEG blend with 80% of PLA give the highest value of tensile strength that is 12.365 MPa due to the present of PLA that higher than the amount of chitosan. PLA is a polymer that has a high mechanical strength, thermoplastic and high modulus polymer and is considered biodegradable and compostable (Sébastien *et al.* 2006). In this study, the hydrophobic characteristic of PLA can make chitosan blend film to reduces its hydrophobic nature and consequently improve their moisture barrier properties. For the blend of biofilm at standard 4 (PEG 22.07 with PLA 71.21%) and standard 3 (PEG 7.93 % with PLA71.21%) from Table 4.2 also have a good tensile strength that are 11.105 MPa and 9.485 MPa respectively. It is because when the blend solution of the PLA and chitosan has the higher composition of the PLA it will give the higher value of tensile strength due to the PLA characteristic that have a good mechanical properties. The similar results

also was observed by Suyatma *et al.*, (2004) on the mechanical properties study. When increasing the PLA in the blend solution increasing the tensile properties due to PLA polymer that have a good mechanical properties compared to chitosan.

For the elongation at break point in Table 4.2 indicate that the highest elongation is at standard 6 (PEG 25% with PLA 50 %) which is 73.16% and the lowest elongation at break point is at standard 5 (PEG 5% with PLA 50 %) which is 48.14 %. It is because the PEG itself is a plasticizer which can improve the elongation properties for the blending PLA/chitosan film. Decreases the amount of PEG in the blends film, increases the tensile and the elongation at break point. The same results also found by Sébastien *et al.*, (2006). In his study when increasing the percentage of PEG in blend solution of PLA/chitosan the mechanical properties decreases due to the influence of the plasticizer on the mechanical properties of PLA films led to the same tendency. These results are obviously due to the plasticization of films, decreasing the rigidity and the brittleness of materials, thus improving their mechanical properties and their recovery. As specified before, potential degradation of the mechanical properties could be due to hydrolytic reactions by water showed that hydration of amorphous PLA resulted in a dramatic change in the development of the craze structure on deformation, but with little effect on the bulk mechanical properties. In crystalline annealed PLA, absorbed water plasticized the movement of crystallites resulting in a change of the bulk mechanical properties

4.2.5 Response surface contour and interaction analysis on each variables

Based on models, numerical optimization was carried out with help of design expert v.7.1.6 considering each value of response. Estimation of the tensile strength (Y_1) and elongation at break point (Y_2) over independent variables: the percentage of PEG (X_1) and percentage of PLA(X_2) in terms of 3D response surface and interaction graph in Figure 4.1, 4.2, 4.3, and 4.4. The figures show the optimum condition for the blending of the biofilms that consist of 30 ml of silver nanoparticles for every blending of PLA with chitosan at 30 ml of blending solution.

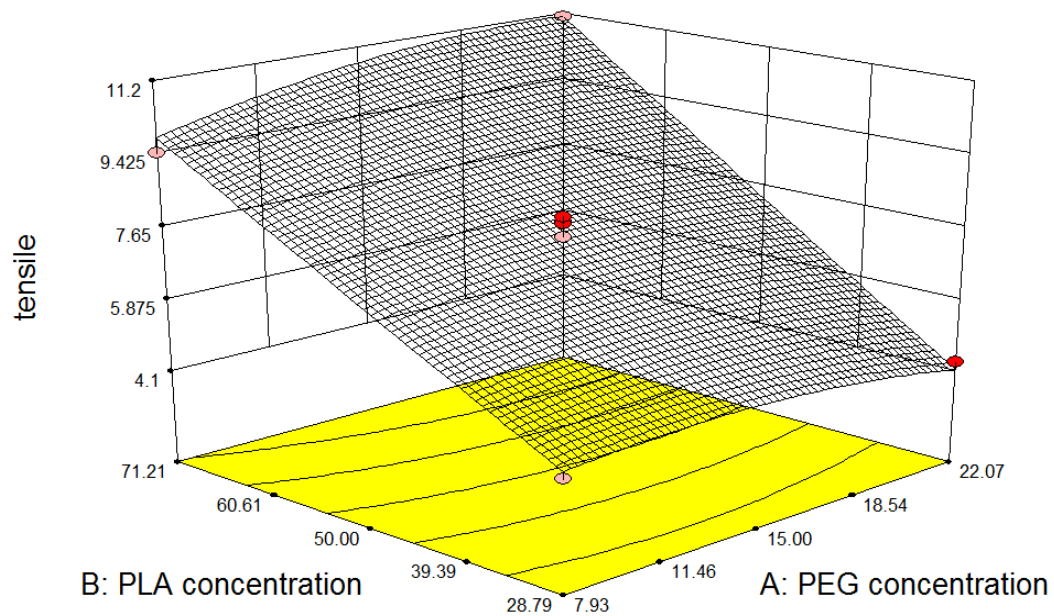


Figure 4.1 3D response surface plots between percentage of PEG concentration and percentage of PLA concentration to tensile strength.

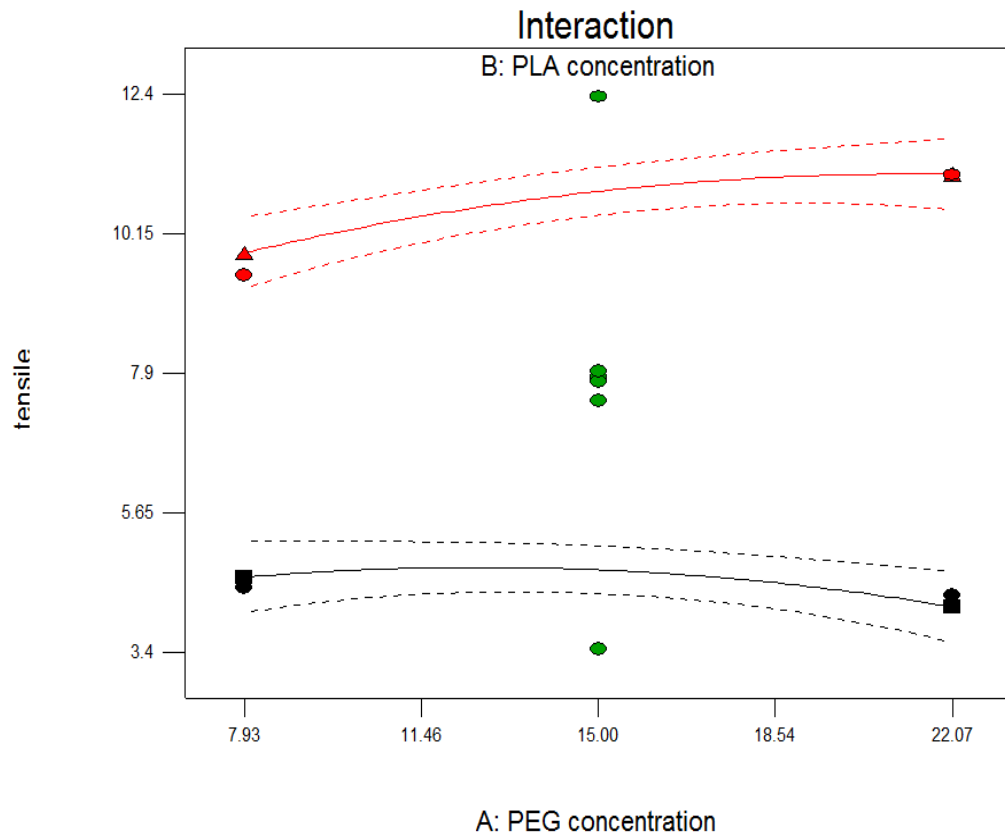


Figure 4.2 Interaction graph between percentage of PEG concentration and percentage of PLA concentration to tensile strength reading.

Based on Figure 4.1 the interaction between the tensile strength indicated that when the percentage PEG concentration increases as a percentage of PLA concentration increases which lead to the increasing of tensile strength. However, from the elliptical contour shows the percentage of the PEG concentration was the major factor for increasing of the tensile strength compared to the percentage of PEG concentration.

Figure 4.2 shows the interaction between the concentration of PEG and percentage of PLA concentration. It indicated that when the percentage PEG concentration increases as a percentage of PLA concentration increases which lead to the increasing of tensile strength but not achieving to the maximum value. The graph described the stimulated data generated by the software which showed the optimum condition to the blending biofilm at 30 ml of blend solution. At this optimum condition, the tensile strength reached the maximum value of 10.573 MPa when the PEG is 17.6 % and PLA is 71.21%.

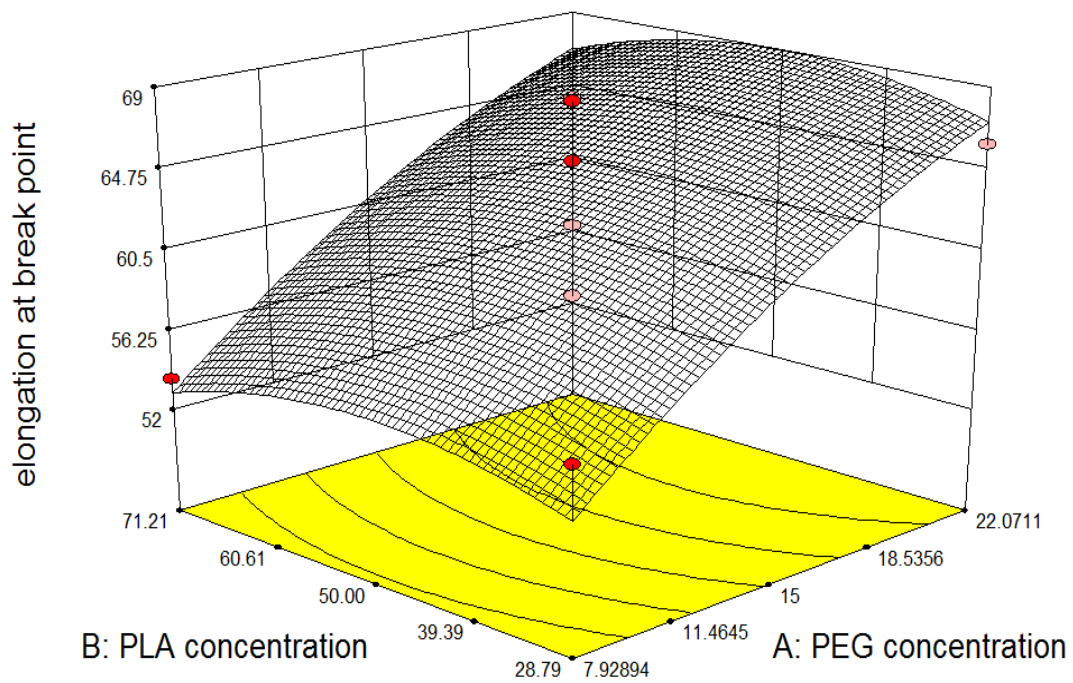


Figure 4.3 3D response surface plots between percentage of PEG concentration and percentage of PLA concentration to elongation at break point.

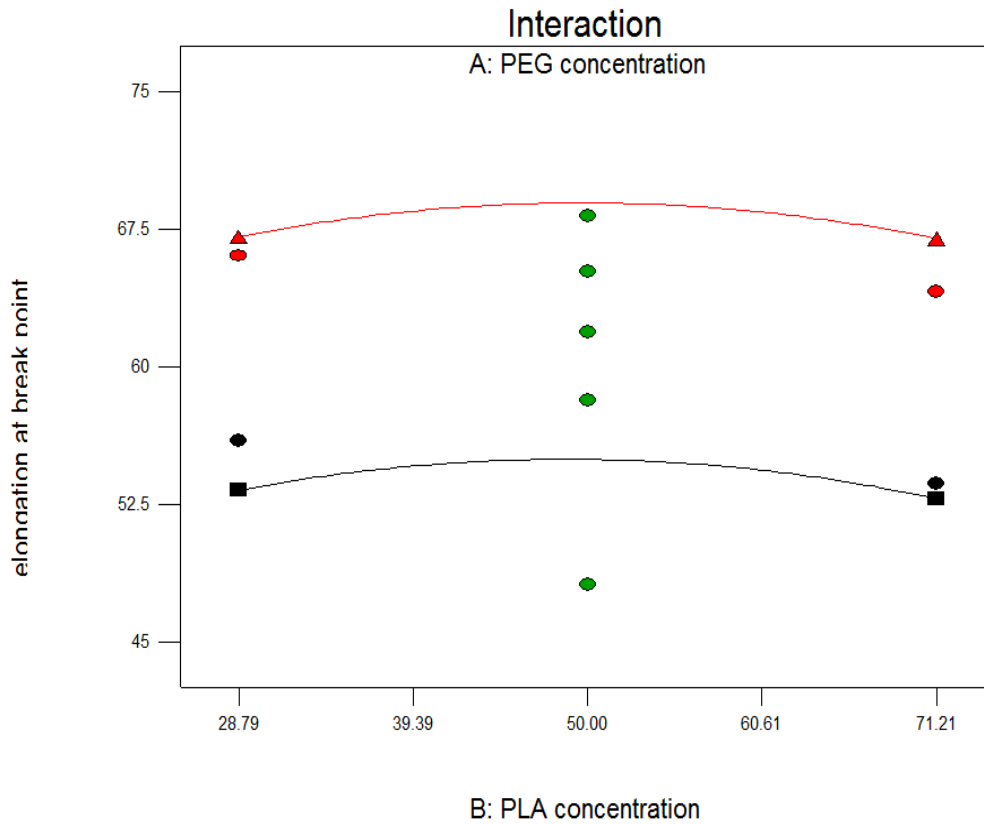


Figure 4.4 Interaction graph between percentage of PEG concentration and percentage of PLA concentration to elongation at break point.

Based on Figure 4.3 the interaction between elongations at break point indicated that when the percentage PEG concentration increases as a percentage of PLA concentration increases which lead to the increasing of elongation at break point of blend films. However, the elliptical contour show the percentage PEG concentration was the major factor for the elongation at the break point compared with concentration of PLA.

Figure 4.4 shows the interaction between the percentage of PLA concentration and percentage of PEG concentration. It indicated that when the percentage PEG concentration increases as a percentage of PLA concentration increases which lead to the increasing of elongation at the break point but not achieving to the maximum value. The graph described the stimulated data generated by the software which showed the optimum condition to the blending biofilms at 30 ml of blend solution. At this optimum condition, the elongation at the break point reached the maximum value of 68% when the PEG is 22.07% and PLA is 50%.

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusions

As a conclusion, the objectives of this research were achieved by using biopolymer materials from poly (lactic acid) (PLA) loaded with chitosan and polyethylene glycol (PEG). The chitosan and poly (lactic acid) (PLA) exhibited interesting qualities in term of good mechanical and moisture barrier properties associated with liquid water resistance of PLA films. The mechanical properties can be modified by controlling the concentration ratio PEG-chitosan to PLA thus can give the biodegradable food packaging films high properties of tensile strength. For the biodegradation rate in Table 4.1 indicate that the degradation rate for PEG 22.07 % with PLA 28.79 % has the highest degradation rate among the prepared biofilms because of the presence of more chitosan because the plastic to decay faster due to the hydrophilic characteristic of

chitosan can undergo hydrolysis degradation. In the other hand, bio plastic sample with PEG 15 % and PLA 80 % indicate the lowest rate of degradation with 10.89 % lowest compared to the other samples. For the RSM method, the optimization of the blend sample was determined by using central composite design (CCD) and the result shows that, at the optimum condition, the tensile strength reached the maximum value of 10.573 MPa when the PEG is 17.6 % and PLA is 71.21%. For the elongation at break point, the optimum condition for the elongation at the break point reached the maximum value of 68% when the PEG is 22.07% and PLA is 50%.

5.2 Recommendation

For the recommendation, some modifications must be made to make sure the biofilm could have a good mechanical properties, degradation rate, water absorption and solubility. Chitosan have the higher solubility and it also degraded when it's oppose to the water because it can undergo hydrolysis degradation due to the OH component in the chitosan. Even though by making the chitosan blend with PEG will improve some weakness of the chitosan, it does not improve the solubility towards water. Through the Millard reaction, the chitosan can overcome the solubility limitation when adding the reducing sugar (glucose or glucosamine). Glucosamine, like chitosan, contains active amino and hydrophilic a relatively wide pH range.

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APPENDIX

Table A.1 Design summary

Design Summary											
Study Type	Response Surface		Runs	12							
Initial Design	Central Composite		Blocks	No Blocks							
Design Model	Quadratic										
Factor	Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded	Mean	Std. Dev.		
A	PEG concentrati %		Numeric	7.93	22.07	-1.000	1.000	15.000	5.774		
B	PLA concentrati %		Numeric	28.79	71.21	-1.000	1.000	50.000	17.321		
Response	Name	Units	Obs	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Trans	Model
Y1	tensile	MPa	12	Polynomial	3.465	12.365	7.53833	2.64464	3.56854	None	Quadratic
Y2	elongation at bre %		12	Polynomial	48.14	73.16	61.1717	6.823	1.51973	None	RQuadratic

Table A.2 Point prediction

Factor	Name	Level	Low Level	High Level	Std. Dev.	Coding		
A	PEG concentra	15.00	7.93	22.07	0.000	Actual		
B	PLA concentra	50.00	28.79	71.21	0.000	Actual		
Response	Prediction	SE Mean	95% CI low	95% CI high	SE Pred	95% PI low	95% PI high	
tensile	7.755	0.15	7.39	8.12	0.33	6.94	8.57	
elongation at b	63.385	1.80	59.13	67.64	4.03	53.86	72.91	

Table A.3 ANOVA design for tensile strength

Use your mouse to right click on individual cells for definitions.

Response	1	tensile				
ANOVA for Response Surface Quadratic Model						
Analysis of variance table [Partial sum of squares - Type III]						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	76.41	5	15.28	172.92	< 0.0001	significant
<i>A-PEG concent</i>	0.31	1	0.31	3.54	0.1088	
<i>B-PLA concent</i>	74.43	1	74.43	842.22	< 0.0001	
<i>AB</i>	0.76	1	0.76	8.61	0.0261	
<i>A²</i>	0.83	1	0.83	9.35	0.0223	
<i>B²</i>	7.562E-003	1	7.562E-003	0.086	0.7797	
Residual	0.53	6	0.088			
<i>Lack of Fit</i>	0.40	3	0.13	2.94	0.1998	not significant
<i>Pure Error</i>	0.13	3	0.045			
Cor Total	76.94	11				

The Model F-value of 172.92 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case B, AB, A² are significant model terms.

Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 2.94 implies the Lack of Fit is not significant relative to the pure error. There is a 19.98% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table A.4 ANOVA for elongation at break point

Response		2		elongation at break point		
ANOVA for Response Surface Reduced Quadratic Model						
Analysis of variance table [Partial sum of squares - Type III]						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	421.29	4	105.32	8.12	0.0091	significant
<i>A-PEG concen</i>	390.79	1	390.79	30.13	0.0009	
<i>B-PLA concen</i>	0.16	1	0.16	0.012	0.9158	
<i>A</i> ²	12.84	1	12.84	0.99	0.3530	
<i>B</i> ²	23.20	1	23.20	1.79	0.2230	
Residual	90.80	7	12.97			
<i>Lack of Fit</i>	34.57	4	8.64	0.46	0.7656	not significant
<i>Pure Error</i>	56.22	3	18.74			
Cor Total	512.09	11				

The Model F-value of 8.12 implies the model is significant. There is only a 0.91% chance that a "Model F-Value" this large could occur due to noise.

Values of "Prob > F" less than 0.0500 indicate model terms are significant.

In this case A are significant model terms.

Values greater than 0.1000 indicate the model terms are not significant.

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 0.46 implies the Lack of Fit is not significant relative to the pure error. There is a 76.56% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Appendix B

Table B.1 Table for force analysis

PLA %	PEG %	Force 1	Force 2	Force 3	Force 4	Force 5	Average force (N)
50.00	15.00	6.780	8.255	9.125	7.340	9.730	8.246
20.00	15.00	4.255	5.985	3.890	5.120	4.785	4.807
50.00	15.00	9.250	11.560	10.675	7.150	7.675	9.262
28.79	22.07	5.430	5.790	5.420	4.380	7.985	5.801
50.00	25.00	8.400	7.675	8.925	8.225	10.535	8.752
71.21	22.07	10.890	11.760	11.970	12.210	12.345	11.835
28.79	7.930	6.960	6.140	6.360	10.130	8.500	7.618
80.00	15.00	12.250	13.165	9.830	7.900	10.505	10.73
50.00	15.00	7.840	8.560	6.655	5.575	7.575	7.241
50.00	15.00	6.750	8.450	7.845	8.980	8.425	8.090
71.21	7.930	8.550	7.575	10.055	11.110	12.235	9.905
50.00	5.000	8.255	7.150	8.455	7.825	7.450	7.827

Calculation for tensile strength

Tensile strength (MPa)

$$= \frac{\text{average force}}{\text{Area of force exerted}}$$

$$= \frac{8.246 \text{ N}}{30 \text{ mm} \times 0.0353 \text{ mm}}$$

$$= 7.786 \text{ MPa}$$

Table B.2 Data for the tensile strength

PLA %	PEG %	Average force (N)	Average thickness (mm)	Tensile strength MPa
50.00	15.00	8.246	0.0353	7.786
20.00	15.00	4.807	0.0462	3.468
50.00	15.00	9.262	0.0394	7.835
28.79	22.07	5.801	0.0447	4.325
50.00	25.00	8.752	0.0408	7.151
71.21	22.07	11.835	0.0355	11.113
28.79	7.930	7.618	0.0571	4.447
80.00	15.00	10.73	0.0289	12.376
50.00	15.00	7.241	0.0324	7.450
50.00	15.00	8.090	0.0339	7.955
71.21	7.930	9.905	0.0348	9.485
50.00	5.000	7.827	0.0367	7.1

Table B.3 Data for the elongation at rupture

PLA %	PEG %	Data 1	Data 2	Data 3	Data 4	Data 5	Average elongation (mm)
50.00	15.00	19.890	20.156	20.443	19.323	18.003	19.563
20.00	15.00	18.767	15.332	14.567	17.119	22.055	17.568
50.00	15.00	18.946	18.979	19.167	18.744	17.004	18.568
28.79	22.07	20.613	19.788	20.233	17.650	20.831	19.823
50.00	25.00	24.743	19.717	21.747	19.633	23.905	21.949
71.21	22.07	22.451	18.368	19.229	18.767	17.365	19.236
28.79	7.930	12.994	18.313	14.674	17.821	20.178	16.796
80.00	15.00	16.870	17.632	18.822	19.243	18.613	18.236
50.00	15.00	19.974	13.243	20.189	15.713	18.161	17.456
50.00	15.00	19.337	20.246	22.849	18.385	21.568	20.477
71.21	7.930	16.422	17.771	15.994	16.475	13.853	16.103
50.00	5.000	12.889	13.676	18.910	14.849	11.881	14.441

The data in the Appendix B2 has shown the elongation data of the biofilms.

Calculation for the elongation at break point

$$= \frac{\text{elongation at rupture}}{\text{actual length of films}} \times 100\%$$

$$= \frac{19.563}{30.00} \times 100\%$$

$$= 65.21 \%$$

Table B.4 Data for elongation at break point

PLA %	PEG %	elongation at rupture(mm)	Elongation at break point %
50.00	15.00	19.563	65.21
20.00	15.00	17.568	58.56
50.00	15.00	18.568	61.89
28.79	22.07	19.823	66.08
50.00	25.00	21.949	73.16
71.21	22.07	19.236	64.12
28.79	7.930	16.796	55.99
80.00	15.00	18.236	60.79
50.00	15.00	17.456	58.19
50.00	15.00	20.477	68.25
71.21	7.930	16.103	53.68
50.00	5.000	14.441	48.18