OPTIMIZATION STUDY ON THE MECHANICAL PROPERTIES OF CORN COB/POLYVINYL ALCOHOL/GLYCEROL BIODEGRADABLE FILM CONSIDERING THE EFFECT OF CHEMICAL COMPOSITION

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A project report submitted in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons) Petrochemical Engineering

> Faculty of Engineering and Green Technology Universiti Tunku Abdul Rahman

> > **JUNE 2022**

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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ABSTRACT

In this study, the blended film was made from mixture of corncob powder (CCP)/polyvinyl alcohol (PVA)/glycerol by solution casting method. Various mixtures were prepared by adjusting the loading of CCP and composition of glycerol based on the result from statistical optimization tool. The functional group of CCP and every PVA/CCP/glycerol blended film were determined by Attenuated Total Reflectance Spectroscopy (ATR). Besides, the mechanical properties of every PVA/CCP/glycerol blended film were analyzed by using tensile test in order to determine its tensile strength, elongation at break and elastic modulus. Based on the result, the highest tensile strength and elastic modulus obtained were 11.46MPa and 209.9MPa respectively while the minimum elongation at break obtained was 11.78%. The mechanical properties of each of the blended film were then further studied by using statistical optimization tool where Analysis of Variance (ANOVA) and Three Dimension (3D) Surface Plot take place. According to the 3D surface plot, it showed that the result obtained was not optimum as it did not provided a proper dome shape. However, the statistical optimization tool provided a predicted optimum loading of CCP and composition of glycerol to obtain the optimum mechanical properties of blended film, which were 3g of CCP and 1mL of glycerol. After tensile test, all the fracture PVA/CCP/glycerol blended film would undergo Scanning Electron Microscopy (SEM) for analyzing their cross section morphology. Furthermore, biodegradability test was conducted for studying its biodegradability. Each of the blended film were buried in soil and leave it at outdoor for a month. The result showed that the blended films which contain 2g of CCP and 1ml of glycerol has the highest biodegradability rate (43.24%) among other blended film.

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LIST OF SYMBOLS/ABBREVIATIONS

cm^{-1}	Centimetres
kg	Kilogram
ml	Millilitres
mm	Millimetres
МРа	MegaPascal
wt%	Weight percentage
%	Percentage
°C	Degree Celsius (temperature unit)
ANOVA	Analysis of Variance
ATR	Attenuated Total Reflectance Spectroscopy
BC	Bacterial Cellulose
BBD	Box-Behnken Design
CAGR	Compound annual growth rate
CC	Corn Cob
CCC	Central Composite Circumscribed
CCD	Central Composite Design
CCF	Composite Faced Centered
CCI	Central Composite Inscribed
CCP	Corn Cob Powder
CNC	Cellulose nanocrystals
CNF	Cellulose nanofibrils
GHG	Greenhouse Gaseous
HBP	Hydro-Biodegradable Plastic
HDPE	High Density Polyethylene

LDPE	Low Density Polyethylene
LLDPE	Linear Low Density Polyethylene
MCC	Microcrystalline cellulose
MFC	Microfibrillated cellulose
NMOCs	Ammonia and Methane Organic Compound
OBP	Oxo-Biodegradable Plastic
PE	Polyethylene
PET	Polyethylene Terephthalate
PHA	Polyhydroxyalkanoate
PLA	Polyactic acid
PP	Polypropylene
PS	Polyester
PTFE	Polytetrafluorethylene
PVA	Polyvinyl Alcohol
PVAc	Polyvinyl Acetate
PVA/BC	Polyvinyl alcohol/bacterial cellulose
PVA/CCP	Polyvinyl alcohol/corn cob powder
PVA/CNF	Polyvinyl alcohol/cellulose nanofibrils
PVA/MCC	Polyvinyl alcohol/microcrystalline cellulose
PVA/MFC	Polyvinyl alcohol/microfibrillated cellulose
RSM	Response Surface Methodology
SEA	Southeast Asia
SEM	Scanning Electron Microscopy
WWF	Worldwide Fund for Nature

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CHAPTER 1

INTRODUCTION

1.1 Background

Plastics are one of the most well-known versatile materials that can be converted into any universal material. For instance, plastics can be molded into water bottles, container, household furniture, and many other useful tools. Besides, it is a type of synthetic polymer that is similar to natural resins abundantly found in the nature. Plastics are commonly used due to its flexible and plasticity (Rodriguez, 2021). Even though plastics bring convenient to our life, they also cause negative impacts to the environment such as water pollution, soil pollution, and air pollution. Furthermore, plastics required a prolonged period to be completely degraded and decomposed. Hence, such scenario leads to the accumulation of plastics in the environment over the years. In order to overcome the negative impact of plastics to the environment, bioplastics are then be introduced as an alternative to the conventional plastics.

First and foremost, the introduction of bioplastics is able to reduce or even overcome the problems that are caused by the conventional plastics. It is because bioplastics are made of biodegradable materials and thus, it required relatively shorter time to be decomposed into materials that are harmless to the environment, unlike the conventional plastics. Thus, bioplastics help in reducing carbon footprint and non-biodegradable waste that are the main factors for the environment contamination. The use of bioplastics became common nowadays and they consist of natural polymers from the agricultural wastes such as cellulose, potato and corn starch which are available abundantly (Acconia, 2019). However, starch-based bioplastics that are made up of potato and corn starch have some severe limitations They have poor stability and are brittle and

sensitive to the environment moisture. According to the study of Ashter (2016), the addition of polyvinyl alcohol (PVA) into starch-based bioplastics was able to enhance the plastic performance.

Polyvinyl alcohol (PVA) is a colorless and water-soluble synthetic polymer. It is an unique polymer as it is not formed through the polymerization process from a single-unit precursor molecule that is usually known as monomer. Instead, it is made by dissolving polyvinyl acetate (PVAc) in an alcohol solution with an alkaline catalyst to promote the hydrolysis reaction. Due to the hydrolysis reaction of PVAc with an alcohol, the degradability of bioplastics could be enhanced if PVA was added into these starch-based bioplastics. It was found that the water absorption of the starch-based bioplastics was decreased as the content of PVA was increased in the starch-based bioplastics. Such observation is due to the interaction between the starch and PVA that reduces the free-OH groups in the resulting bioplastics (Marta, 2016).

Since bioplastics can be made from starch, corn cob (CC) can be one of the potential materials. Using corn wastes like CC to synthesis bioplastics helps to reduce the disposal of CC waste. Thus, it is able to reduce the impact on the environment as the global corn production was approximately 1.13 Gtonnes. Since CC wastes can be obtain abundantly, it also shows a great potential review as an enhancement to the bioplastic (Chong, 2021). Corn starches are also the potential candidate for plastic matric reinforcement as they are a source for saccharides. The saccharides from the corn starch can be used for bioplastic matrix improvement, while the lignocellulosic fibers by-products are still useful as it can be used as the reinforcement material. The lignocellulosic fibers usually can be obtained from CC, corn husk, corn stalk, and corn stover (Noor, 2022). However, defect might persists the synthesis of pure starch-based bioplastics. Hence, plasticizer has to be added into the starch-based bioplastics in order to have sufficient elasticity (Sanyang, 2015).

Plasticizer had been commonly used in the modification of polymers. It is an additive that can enhance the properties of plastics. For instance, glycerol is usually added into the starch-based bioplastics to make it more elastic. Generally, glycerol and starch are melted at temperatures between 90°C to 180°C. The molten mixture can be used to produce thermoplastic under shear stress through injection, extrusion, and blowing equipment (Bilck, 2015). It helps to improve the workability and durability of bioplastics as there is a reduction of polymer-polymer contact and

cause the rigidity of the 3D structure of bioplastics decrease. Hence, it enhances the deformation ability without rupture (Tyagi, 2022).

Mechanical properties of blended film are the vital properties in plastic making. In order to determine any reproducible feature of the blended film, acknowledge its optimum composition of raw material is crucial for an engineer to obtain the enhanced mechanical properties of the blended film. Therefore, optimization tools like Response Surface Methodology (RSM) is necessary for engineer to obtain optimum results. However, there is a lack of information in using the RSM for the determination of correlation between the chemical composition and mechanical properties in the blended films.

In this study, PVA was blended with different composition of CCP and glycerol using solution casting method and the mechanical properties such as tensile strength, elongation at break, and elastic modulus are being analyzed and optimized through RSM coupled with Central Composite Design (CCD).

1.2 Problem Statement

CC is one of the corn wastes that can be found abundantly. The global production of corn was about 1.13 Gtonnes which was the second highest production in a farm. For every 1kg of corn, it contains 0.15kg of CC, 0.5kg of corn stalks, and 0.22kg of leaves (Chong, 2021). Since the production of corn by farmers and enterprises kept increasing, huge amount of corn waste was generated, whereby these corn wastes could lead to negative impact to the environment if not disposed properly. Generally, corn wastes are disposed through incineration or open burning, releasing the second highest greenhouse gases emissions in the globe (WarmHeartWorldWide, *2018*). Therefore, using corn wastes for the synthesis of bioplastics would help to reduce the negative impact of corn wastes to the environment.

Apart from the greenhouse gases that generated by the incineration, there are also large amount of smog released into the atmosphere that cause severe human health. Once human inhale PM2.5, it will pass through wall of human lung and stick with the bloodstream. Moreover, the smog that contains ammonia and methane organic compound (NMOCs) will affect the aquatic organisms through precipitation (WarmHeartWorldWide, 2018).

Furthermore, the demand of plastics in the society keeps increasing and thus causes the increased plastics production due to the high demand. At the same time, as the consumption of plastics increases, the accumulation of plastic wastes in the environment will be serious issue. In addition, the accumulation of plastic wastes might also cause water pollution. For instance, plastics that thrown into the sea or river could affect the ecosystem of the aquatic life (WBUR, 2014). It might also cause soil pollution as most of the bacteria in soil could not decompose the plastics due to the indestructible polymer long chain of plastics and thus lead to the accumulation of plastics (Tey, 2016). This is also one of the reason that the landfill is not sufficient for the disposal of the increasing number of plastics.

Last but not least, the one-factor-at-time method that commonly used in research is time consuming and ineffective as it can only run with one factor at a time. Therefore, the introduction of an systematic optimization technique is important as it can help to enhance the efficiency of the experiment and obtain a better results. Optimization technique is significant in engineering because engineers always seek for the best possible solution, most economical, and greatest efficiency for their goals (Terlaky, 2017). Hence, optimization technique is preferable and necessary for achieving goals when the system has high complexity.

1.3 Aims and Objectives

The main purpose of this research project is to synthesize, study and evaluate the biodegradable properties of corn cob powder (CCP)/polyvinyl alcohol (PVA) blended film using statistical optimization tool. The sub-objectives are as follows:

- i. To fabricate the corn cob powder (CCP)/polyvinyl alcohol (PVA) and glycerol blended film using solution casting method.
- To evaluate the effect of chemical composition on the mechanical properties of PVA/CCP/glycerol blended film through statistical optimization tool.
- iii. To study the biodegradable properties of PVA/CCP/glycerol blended film.

1.4 Scope of Study

In order to achieve the objectives, there were three scopes that need to be identified. First of all, the blending of PVA/CCP/glycerol blended film was performed using the solution casting method. Next, the characteristics of PVA/CCP/glycerol blended film were analyzed using Attenuated Total Reflectance (ATR) and Scanning Electron Microscopy (SEM).

Furthermore, tensile properties were analyzed. They were then further analyzed through the Response Surface Methodology (RSM). RSM was used to analyze the mechanical properties of PVA/CCP/glycerol blended film that more incorporate of with the added glycerol. Two main parameters were featured on this optimization study, including the composition of CCP and composition of glycerol while the tensile strength, elongation at break, and elastic modulus were the response.

Moreover, the biodegradable properties was analyzed by operating soil burial test. The test would be carried out for one month. During the test, the weight of blended film would be weight and record down with an interval of 1 week.

CHAPTER 2

LITERATURE REVIEW

2.1 Plastics Industry in Malaysia

Plastic was a synthetic material which made from polymer. Polymer was formed when long molecules were built around the chain of carbon atoms which typically with hydrogen, oxygen, sulphur and nitrogen. Plastic was a wide range product because it was low cost, ease of manufacture, versatility and more (Woodford, 2018). *Figure 2.1* showed the major market segments for plastic products in Malaysia. The largest segment of plastic industry in Malaysia was packaging industry which was 48%, and all the products were mostly for single use. Furthermore, plastic can also be used in electronics and electrical industry which comprise about 27%, then followed by automotive, construction, household, agriculture and others. According to the TMR Media Sdn Bhd, Malaysia had high plastic packaging among all the South-east Asia countries which was 16.78 kg/person. Meanwhile, the household plastic packaging consumption was estimated to reach 523,000 metric ton at the end of 2020. All these consumptions were came from small businesses or end users such as schools, hospitals and government buildings (Esther, 2019).



Figure 2.1: Major Market Segments for Plastic Product in Malaysia (Esther, 2019).

Since the consumption of plastic in Malaysia was increasing year by year, the plastic industry in Malaysia was significant. Malaysia plastic market price increased due to most of the plastic products such as plastic films and plastic sheets that produce from Malaysia were exported to other countries. The *Figure 2.2* showed that there were RM5.85 billion of the plastic films and plastic sheets exported to different counties in 2017. The main source was came from Europe and other countries instead of Australia, China, Indonesia, Japan, Singapore and USA. Besides that, the plastic industry also significant to Malaysia economy because it generated about RM31 billion in 2018 (MPMA, 2018). In 2018, there were 47% of the total plastic products, which value around RM14.56 billion from exports. Furthermore, Malaysia plastic market was valued at USD3243 million in 2020. CAGR of Malaysia had increase around 3% during the forecast period which from 2021 to 2026 (Mordor Intelligence, 2021).



Figure 2.2: Export Market for Plastic Films and Sheets by Countries (MPMA, 2018).

There were more than 1300 plastic products which manufactured in Malaysia and these 1300 plastic product included all major market segment (MIDA, 2021). Polyethylene was a major dominate in the traditional plastic industry because it was one of the key products used in our daily life. Normally, polyethylene was used in the packaging industry such as container and bottles, plastic bags as well as plastic films. There were several types of polyethylene such as low-density polyethylene (LDPE), high density polyethylene (HDPE), linear low-density polyethylene (LLDPE), polyethylene terephthalate (PET) and more. Apart from packaging industry and personal care industry, construction industry and pharmaceutical industry also used different types of plastic. However, packaging industry was the largest application segment in Malaysia plastic market because plastic have better wear and chemical resistance, recyclability, ease of molding and high mechanical strength. For example, PET was one of the plastics that normally used for food and beverage industry. It is light weight, ease of cleaning, design flexibility and protection against moisture which is suitable used in food and beverage industry (GlobeNewswire, 2020).

Nevertheless, the economic growth of Malaysia was affected due to the pandemic of COVID-19. Plastic industry also get affected especially construction industry because Malaysia Prime Minister had imposed the restriction of movement order in March 2020 due to the COVID-19 cases increased. This had directly affected the growth of plastic industry as they could not export their goods to other countries. However, during this period, food and beverage packaging helped to rise the demand of the plastic industry (Mordor Intelligence, 2021). In short, the demand of plastic industry still increased even though the pandemic was happened.

2.1.1 Plastic Pollution in Malaysia

The accumulation of synthetic plastic product that had generated issues to the environment including wildlife, habitats, plants, and human was known as plastic pollution (Moore, 2022). In fact, Malaysia had higher percentage of plastic generated in solid wastes, which had ranked second highest in Asia. According to the study from Kaur (2021), Worldwide Fund for Nature (WWF) had stated that Malaysia was the second highest for plastic used per year across Asia in 2019, which is approximate 16.78kg of plastic used per person. The amount of plastic usage in Malaysia had outrank other larger nations like Indonesia, Philippines, Thailand, and Vietnam, where the usage of plastics for the countries were about 12.5kg per person, 12.4kg per person, 15.52kg per

person, and 12.93 kg per person respectively (Meikeng, 2021). This scenario had showed that Malaysia had huge plastic waste problem.

Malaysia considered as one of the largest plastic production industries in the globe as there were 1,300 plastic manufacturing industries in Malaysia (Chen, 2021). The reasons that Malaysia had generated large amount of plastic waste is due to the imperfect plastic waste management. Even though Malaysia considered as one of the developed countries in Southeast Asia, the waste management system in Malaysia was inadequate to deal with the huge amount of plastic waste that had been generated. Besides, Malaysia also imported plastic waste from other nation. According to CTN News (2019), it stated that Malaysia had significantly increased the volume of plastic waste that Malaysia imported in the year between 2016 to 2018. Moreover, the volume of plastic waste that Malaysia imported was the highest among Southeast Asia (SEA) countries. The statistics graph is showed in *Figure 2.3*.



Figure 2.3: Volume of Plastic Waste Import among Southeast Asia (SEA) countries (CTN News., 2019).

Since Malaysia had the ability to generate huge amount of plastic but do not have a proper plastic waste management, the plastic wastes were accumulated. The main ways of Malaysia handling the plastic waste were disposal through landfill and incineration (Chen, 2021). However, both of the disposal methods were not the most suitable method to deal with the plastic waste because landfill method was overflowing with low value plastic packaging and sachets in Malaysia as the expansion of take-away food packaging culture had accelerated the domestic plastic packaging generation while the incineration method would generate air pollution as the domestic burning of plastic waste would generate carbon dioxide and toxic gases to the environment and create environmental issues.

According to the study from Chen (2021), it was stated that solid waste management was one of the main reasons that caused environmental problems in Malaysia, upon to the population growth of the country and development of landfill sites. Apart from that, Malaysian household wastes generation was approximate 1.5kg person, which was higher than other developing countries like Indonesia and Philippines with 0.22kg per person and 0.4kg per person respectively. Based on the study from Lee (2021), Malaysia had produced 0.94 million tons of mismanaged plastic waste in the year of 2010 and around 0.14 to 0.37 million tons of the plastics was leaked into the ocean where it would affect the ecosystem of the aquatic life in the ocean.

2.2 Types of Plastics

2.2.1 Petroleum-based Plastics

Petroleum-based plastics were known as artificial organic polymers which normally obtained from natural gas or oil. It was usually utilized in every aspect of our daily life especially in medicinal, commercial, industrial area, and municipal field. The examples of petroleum-based plastic were polyethylene (PE), polypropylene (PP), nylon, polyester (PS), polytetrafluorethylene (PTFE), and epoxy. According to Suman (2020), global production of petroleum-based plastic in 2015 had reached 322 million tons while the production of petroleum-based plastic in 1950 was only 1.7 million tons, which was much lower. It is due to massive usage and demand of petroleum-based plastic in the contemporary society. Even though petroleum-based plastics bring a lot of convenient to our daily life, but it had caused a significant negative impact to the environment because petroleum-based plastics cannot decompose naturally due to its profound stability.

Besides that, most of the petroleum-based plastic were directly extracted from crude oil. Therefore, the amount of petroleum and energy that required for synthesis of petroleum-based plastic had become significant when its consumption increased (Atiwesh, 2021). Due to that, greenhouse gaseous and heat were released from every plastic industry to the atmosphere and lead to global warming. Hence, the huge amount of production and utilization of petroleum-based plastic had become a serious problem to the world.

2.2.2 Biodegradable Plastics

Biodegradable plastic was a plastic that can be degraded under biological action, which means it able to degrade by microorganisms present in the environment. It was usually manufactured under a controlled conditions of temperature and humidity environment (Omnexus, 2022). However, some of the biodegradable plastics were compostable but some of them were not, which means that those biodegradable plastics that were compostable able to degrade under controlled conditions. The compostable biodegradable plastics only degrade under certain specified and tested condition. For instance, it might be degradable in soil, in wastewater treatment plants and etc (Kerge, 2020).

There were two main types of biodegradable plastics which are Oxo-Biodegradable Plastic (OBP) and Hydro-Biodegradable Plastic (HBP). Both types of biodegradable plastics were considered as compostable plastics and would emitted carbon dioxide when they degraded. However, HBP not only emitted carbon dioxide when it degraded but also emitted methane. HBP were made from petroleum-based sources, bio-based sources such as corn, wheat, sugarcane, or a mix of both bio-based sources and petroleum-based sources while OBP were made by adding a small portion of fatty acid compounds of specific transition metals to traditional plastics. OBP had longer time frame for its carbon to convert to carbon dioxide but the degradation rate of OBP was lower than HBP (Omnexus, 2022). Furthermore, there was a compostability standard for biodegradable plastic to determine its compostability which are ASTM D6400 and EN13432. ASTM D6400 was the US standard where solid material biodegradation required for labelling of plastics designed to be aerobically composted in municipal or industrial facilities while EN13432 was the EU standard where key component needed to recover packaging waste on the basis of industrial composting (Kerge, 2020). Since biodegradable plastics were more eco-friendly.

2.3 Polyvinyl alcohol (PVA)

Polyvinyl alcohol (PVA) was a synthetic polymer that was soluble in water which made up by multiple molecules. It was also known as PVOH, PVA, and PVAL. PVA was manufactured by dissolving polyvinyl acetate (PVAc) in an alcohol such as methanol over with an alkaline catalyst such as sodium hydroxide that in aqueous form whereby PVAc was the monomer of PVA and was prepared through polymerization of vinyl acetate (Saxena, 2004). *Figure 2.4* showed the chemical structure of PVA.



Figure 2.4: Chemical Structure of PVA (Bakr, 2018)

Since PVA was soluble in water, it was effective in film forming, emulsifying, and had the quality of adhesive. Apart from that, it was not toxic, no odor, and resistant to oil, grease, and solvents. Even though PVA was ductile, but it appeared to be strong and flexible and able to function as a high oxygen and aroma barrier as well (Mehta, 2018).

Due to the properties of PVA, it was commonly used for strengthening textile yarn and papers so that the latter can be more resilient to oils and grease. Besides that, PVA was also used as coating agent for food supplements because it did not cause any health risks as it was non-toxic. Other than that, it was used for food packaging as well whereby it can be considered as one of the leading industrial uses of PVA in the globe. Apart from the advantage of non-toxic PVA for food packaging application, the main reason for its usability in food packaging was PVA film created to be thin and water resistant. PVA film can be thin and water resistant due to its crosslinking density and resistance to moisture (Mehta, 2018).

2.4 Agricultural Waste

Agricultural wastes were the residues of the growing and processing of raw agricultural products such as fruits, vegetables, and crops. They can be considered as undesired product of production, but it might still be beneficial because it may contain material that able to reprocess to other product or act as raw material for other product. Agricultural waste also known as agro-waste, which were comprised of animal waste, food processing waste, crop waste and etc. Nowadays, significant increase in agricultural waste globally was estimated as the expansion of agricultural waste production had eventually resulted in quantity of livestock waste, agricultural crop residues, and agro-industrial waste matter to the world. It was estimated that approximately 998 million tons of agricultural wastes were produced annually (Obi, 2016).

2.4.1 Corn Cob Waste

CC waste was an agricultural waste that can be obtained in huge quantities during corn processing (Berber-Villamar, 2018). It was normally being disposed by most of the corn processing industry. According to Chong (2021), global production of corn was approximately 1.13 Gtons, which was the second highest among all agricultural crops. Therefore, CC waste can be accumulated in large quantities if the disposal method was improper. The improper method of disposal of CC waste could cause negative impacts to the environment such as water pollution and soil pollution. In addition, it could cause severe environmental problems as it was the second-highest greenhouse gaseous (GHG) emissions source for landfilling and the highest GHG source for biomass burning in 2017. Hence, disposal of CC waste had to be treated well. Fortunately, the uses of CC waste were discovered where CC waste had the potential to be an alternative source of reinforcement to other natural fibers for bioplastic composites. Based on the study from Chong (2021), the natural fibers from CC waste can reinforced in bioplastics composites as it can enhanced the structural stability of the composite when it act as reinforcement filler. Besides that, it was renewable, cheap, and environmentally friendly. Moreover, the utilization of agro-waste lignocellulosic able to reduce the CC waste. *Figure 2.5* showed the picture of CC waste.



Figure 2.5: Picture of CC Waste (Kaamin, M., 2018)

2.4.2 Banana Peel Waste

Banana was mostly grown in tropical and subtropical countries especially in Malaysia. It is due to tropical and subtropical countries have been sustainably cultivated and thus, banana had become one of the significant crops in the world. Banana peel waste was one of the agricultural waste that can be obtained easily because the amount of banana produced is huge. There were 106 million tons of banana produced in 2013 where 57% of bananas were produced in Asia and 26% of bananas were produced in America. Apparently, banana peel was one of significant agricultural waste. According to ScienceDaily (2016), it stated that the ratio of banana waste and product was 2:1. Since there were a lot of banana wastes, it can caused environmental problems if it did not treated properly because it contained large quantities of nitrogen, phosphorus, and high water content. However, it was discovered that banana peel waste able to utilize for bioplastic synthesis. According to the study from Razak (2020), it showed that can be the main material for bioplastic production due to its renewable nature and biodegradability. *Figure 2.6* showed the picture of banana peel waste.



Figure 2.6: Picture of Banana Peel Waste (Banana, 2011)

2.4.3 Oil Palm Waste

Oil palm can be considered as one of the most important product from Malaysia because it had help to change the scenario of the agricultural and economy. It was reported that Malaysia had produced 135 million tons of oil palm waste every year (Dungani, 2018). The presence of oil palm wastes had created major disposal problem. However, it was discovered that oil palm waste can be an alternative material to fabricate bioplastics due to its mesocarp fibers (Indonesian Palm Oil Association, 2020). Therefore, oil palm waste can be reduced and fully utilized it. Moreover, there were a few research had been done on the oil palm based bioplastics and thus, the development of oil palm based bioplastics would be more mature. *Figure 2.7* showed the picture of oil palm waste.



Figure 2.7: Picture of Oil Palm Waste (Flickr, 2022)

2.5 Plasticizer

Plasticizer was a low molecular weight substance that always being added into a raw polymer so that the flexibility of the particular polymer can be enhanced and allowed it easier to shape and mold. Besides that, It also helped to increase the flow and thermoplasticity of the polymer by reducing the viscosity of the polymer melt, elastic modulus of the finished product, glass transition temperature (Tg), and melting temperature (Tm). It was commonly used as additives in plastic industry because it is cheaper compared to other additives (Daniels, 2022). There were three types of plasticizers that usually used for polymer which are glycerol, lactic acid, and sorbitol.

2.5.1 Glycerol

Glycerol was a colorless and odorless liquid. It was nontoxic in low concentrations and would be vicious at room temperature (BiologyDictionary, 2017). It was widely used in several industrial application such as pharmaceutical industry, plastic industry, cosmetics and personal care products industry, and etc. It acted as a plasticizer for polymer in plastic industry. Among every plasticizer, glycerol was usually selected to use as plasticizer for producing starch based biodegradable films due to its compatibility with amylose. The compatibility of the film with amylose interfered the amylose packing and decreased the intermolecular forces between the starch molecules (Tarique, 2021). Hence, it allowed the film to have better mechanical properties by showing its good flexibility.

2.5.2 Lactic acid

Lactic acid was the monomer of polylactic acid (PLA) whereby PLA was formed through direct condensation. It was a biodegradable hydrolysable aliphatic semicrystalline polyester (Kurokawa, 2017). In fact, it was produced from the fermentation of renewable sugar supply such as starch and polysaccharides. Lactic acid had left-handed characteristics that make itself a good biocompatibility substance. Due to its biocompatibility, it can acted as plasticizer for polymer. Apart from its biocompatibility, the other advantages of using lactic acid as plasticizer were due to its high strength, thermoplastic fabricability, good crease retention, and resistance to grease or oil. According to the research from Li (2014), it showed that the lactic acid able to reduce the usage of phthalates and even enhanced the performance of plasticizing agent that already existed.

2.5.3 Sorbitol

Sorbitol can be considered as a sugar alcohol that usually found in fruits and plants with diuretic, laxative, and cathartic property. It was a colorless and odorless solid which would sink when mixed with water (NIH, 2022). In fact, sorbitol can be one kind of plasticizer that used for enhancing polymer properties. It is due to the active hydroxyl groups of sorbitol caused themselves as an effective plasticizer for some -OH or -NH rich polymers. Besides, sorbitol was non-toxic and

thus, it can also be used for food packaging materials (Tian, 2017). One of the advantages that using sorbitol as plasticizer was more thermally stable. According to the study from Li (2011), the result showed that the vapor pressure of glycerol had increased exotically when the temperature was above 200°C while the vapor pressure of sorbitol was almost negligible. Based on the result, it showed the advantage of using sorbitol as plasticizer in term of limiting losses of plasticizer because sorbitol had better thermal stability when compared to glycerol.

2.6 PVA with Natural Fiber

Natural fibers were the alternative material to replace the inorganic fillers and fibers. It is because natural fibers were renewable and abundantly available resources and able to reduce the serious environmental issues that caused by the use of inorganic fillers and fibers. It was also an occurring polymer in our environment which can be found in grass, leaves, plants, and animals.

In fact, the structure of natural fibers was known as lignocellulose fibers due to its main components were lignin and cellulose. Due to the presence of cellulose in natural fibers, the properties of natural fibers were almost similar to the nature of cellulose and its crystallinity properties. For instance, fibers with higher cellulose content would have better mechanical properties but it tended to be more flammable than those with higher hemicellulose content. While those with higher hemicellulose content would tend to absorb more moisture than those with higher cellulose content. In addition, fibers with higher lignin content would have better char formation as the degradation only occurred at relatively low temperature. *Table 2.1* showed the mechanical properties of several natural fibers. Moreover, cellulose was usually extracted from the natural fibers for reinforcement purpose and mainly used it for composites production due to its hierarchical structure and semicrystalline nature. However, natural fibers were difficult to be manufactured into the desired form because it cannot be melted or dissolved in a common solvent due to its strong intermolecular hydrogen bonding, high degree of crystallinity, and high degree of polymerization. Despite that, it was compatible with biodegradable polymer such as PVA.

Natural fibers	Density (g/cm ³)	Young's modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)
Flax	1.54	27.5-85	345-2,000	1–4
Ramie	1.5-1.56	27-128	400-1,000	1.2-3.8
Hemp	1.47	17-70	368-800	1.6
Jute	1.44	10-30	393-773	1.5-1.8
Sisal	1.45-1.5	9-22	350-700	2-7
Coconut	1.15	4-6	131-175	15-40
Cotton	1.5-1.6	5.5-12.6	287-597	7-8
Kenaf	1.2	14-53	240-930	1.6
Bamboo	0.6-1.1	11–17	140-230	-

Table 2.1: Mechanical Properties of several Natural Fibers (Tan, 2015).

2.6.1 Natural Fiber Reinforced PVA based Composite

The reinforcement of PVA with natural fibers had allowed them to become an economic sustainable composite due to the four key factors as these factors can significantly affect the strength of the PVA composites. The factors were compatibility of reinforcement-matrix, the dispersion of reinforcement in matrix, the properties of matrix, and the properties of reinforcement (Tan, 2015). It was found that the presence of -OH groups and the hydrophilic nature of natural fibers were compatible with PVA and thus, result in good interaction between natural fibers and PVA. Due to the good interaction between natural fibers and PVA, it showed great composite properties such as high tensile, good fiber/matrix bonding, and exhibited Young's modulus with maximum fiber content.

Furthermore, extrusion, injection molding, and solution casting method were the common processing methods for producing natural fibers based composites. However, the processing temperature was preferred to set at low temperature which was usually about 200°C with shorter processing time because natural fibers had lower thermal stability (Tan, 2015). Hence, the degradation of natural fibers can be avoided when the processing temperature was low. Besides, it was found that addition of plasticizer during extrusion processing method able to reduce the melt viscosity, which indicated that the die pressure of extrusion was reduced and thus, processing of composites became easier.
2.6.2 PVA Incorporated with Various Type of Cellulose

Due to the properties of cellulose such as flexibility, recyclability, and environmentally friendly, PVA can also be incorporated with various type of cellulose. There were two main types of cellulose which are micro-cellulose and nano-cellulose.

Micro-celluloses were made up of very short fibers. Therefore, combining micro-cellulose with PVA able to overcome the limitations of PVA such as low strength and low stiffness. Mircrocelluloses were divided into two types which are micro-crystalline cellulose (MCC) and microfibrillated cellulose (MFC). MCC was a crystalline cellulose. It was produced through acid hydrolysis whereby the acid hydrolysis dissolved the amorphous region of the cellulose and thus, separated the cellulose microcrystals (Ashori, 2010). However, these separated cellulose microcrystals tended to combine again due to the strong hydrogen bonding and formed MCC. This reformed MCC was then showed spongy, porous, and random fine structures. Since MCC was a crystalline cellulose, it able to enhance the crystallinity of the PVA when they combined together. The enhanced PVA was then confined crystallization and reduced its melting temperature. Furthermore, MFC was an interconnected web-like structure of tiny fibrils and microfibril bundles, which is difference from MCC. It was obtained through fibrillation process of cellulose fibers (Jovic, 2017). According to the study from Tan (2015), the SEM image of PVA/MFC composites showed that the MFC was distributed uniformly in PVA. These well distributed of MFC in PVA indicated that it restrained the free movement of polymer chains and restricted its ability to fold and hence, reduced the crystallinity of PVA.

Nano-cellulose was a light solid substance that contained nanosized cellulose fibrils where it can be obtained from plant matter (Soutter, 2012). It was categorized into three types with different production and properties, which are cellulose nanocrystals (CNC), cellulose nanofibrils (CNF), and bacterial cellulose (BC). For instance, CNC and CNF were usually produced through destruction method while BC was synthesized from bacteria. However, the advantages of using nano-cellulose based composites were similar, which are having excellent mechanical properties, light weight, and biodegradability.

PVA/nano-cellulose were usually prepared by using solution casting method. According to the research from Tan (2015), PVA/CNC/glycerol composites had showed the enhancement in mechanical and barrier properties due to the superior reinforcement property of CNC with the

presence of glycerol, well distribution of CNC in PVA, and the great adhesion between starch fibers and polymer matrix. Apart from PVA/CNC/glycerol composites, PVA/BC/glycerol nanocomposites also showed excellent mechanical and optical properties. It is because the presence of BC allowed to synthesize and gathered in the presence of glycerol in order to form BC network. The BC network was then restricted the mobility of the PVA chain and resulted in the crystallinity of the matrix in the composites increased. Nevertheless, PVA/CNF/glycerol composites also had the similar enhancement of mechanical properties as CNC. In spite of the improvement of mechanical properties, PVA/CNF/glycerol composites also showed good thermal stability and transparency with a visible light transmittance (Tan, 2015).

2.7 Solution Casting Method

Solution casting method was an easy and versatile method to produce PVA thin film in laboratory scale based on the principle of Stokes' Law (De Azeredo, 2014). It was an alternative method to replace film extrusion so that it can produced high quality films with great mechanical and optical properties. In solution casting method, polymer and solvent were the main components of the solution system where all the necessary material and polymer would dissolved in the solvent to obtain homogeneous solution. Once all the material was completely dissolved, the homogeneous solution was then casted onto a flat glass plate and leave it to evaporate. After that, the dried membrane was then peeled off from the flat glass plate (Galiano, 2014). *Figure 2.8* showed the general procedure of solution casting method. Moreover, the advantages of using solution casting method included the ability to process at low temperature, ability to produce high temperature resistant films from non-thermoplastic, and various material choices with casting from aqueous or solvent based solution (Koomen, 2015).



Figure 2.8: Procedure of Solution Casting Method (De Azeredo, 2014).

2.8 Mechanical Properties of PVA Blended Film

2.8.1 Tensile Strength

Tensile strength was the ability of a material to withstand a pulling force where the strength was refer to the breaking of the material when the force was applied to it (Corrosionpedia, 2020). In the other words, it was the maximum tensile stress that the biodegradable film able to withstand before failure where the failure was refer to the breaking or permanent deformation of the film. It was usually measured in units of force per cross-sectional area. *Equation 2.1* showed the formula of tensile strength:

Tensile Strength,
$$\sigma = \frac{Force \ that \ applied \ in \ the \ film,F}{Cross-sectional \ area \ of \ the \ film,A}$$
 (Equation 2.1)

In fact, there were three types of tensile strength, which are yield strength, ultimate strength, and breaking strength. Yield strength was the stress that the film could withstand without permanent deformation while the ultimate strength indicated the maximum stress of the film able to withstand. Meanwhile, the breaking strength stand for the stress that merged on the stress-strain curve at the rupture point (Corrosionpedia, 2020). *Figure 2.9* showed the typical stress-strain curve.



Figure 2.9: Typical Stress-Strain Curve (Justynar, 2021).

2.8.2 Elongation at Break

Elongation at break was the ratio between the increased length and the initial length after the breakage of the test specimen. It expressed the capability of the biodegradable film to withstand the changes of shape without crack formation or deformation (Djafari, 2017). Elongation at break was also known as fracture strain. It can be determined by using tensile test and it was usually measured in percentage. *Equation 2.2* showed the formula of elongation at break:

Elongation at break,
$$\epsilon = \left(\frac{Final \ length \ of \ specimen, \Delta L}{Initial \ length \ of \ specimene, L}\right) \times 100$$
 (Equation 2.2)

In fact, elongation was inversely proportional to hardness, tensile strength, and modulus, which means that when the hardness, tensile strength, and modulus of the film was greater, the elongation would be lesser (JLWInstrument, 2022). Moreover, a film with good elongation at break usually exhibited a value not more than 5%. However, there were some factors that could affected the elongation at break such as temperature, filler content, velocity of testing, and orientation level. For instance, higher temperature would increase the elongation at break but the elongation at break would decrease when the filler content was increased (Gooch, 2011). Hence, all the parameters was ensured to set properly before measure elongation at break of biodegradable film so that the results would not be affected.

2.8.3 Young's Modulus of Elasticity

Young's modulus was a measure of the film's capability to withstand changes in length whenever a force of tension or compression was applied to it (Encyclopedia, 2019). It can also be known as modulus of elasticity. In the other words, Young's modulus can be expressed as the ratio between stress and strain. It was usually measured in the unit of megapascals, MPa. By measuring the value of Young's modulus, the stiffness of the film can be known where the relationship showed that the larger the value of Young's modulus, the stiffer the biodegradable film (Omnexus, 2022). The value of Young's modulus can be calculated by using *Equation 2.3*. The equation showed the formula of Young's modulus of elasticity:

Young's modulus of Elasticity,
$$E = \frac{Stress,\sigma}{Strain,\epsilon}$$
 (Equation 2.3)

However, there were some factors that could affected the value of Young's modulus. One of the common factors that would affected the value of Young's modulus was the temperature of the film. It is because film with higher temperature could increase the atomic vibration of the film and caused the necessary energy for further separation of the atoms decreased (Omnexus, 2022). Hence, the stress that required to produce a desired strain would be reduced and thus, affected the precision of the result obtained.

2.9 Biodegradation Study

Biodegradation was a natural waste management and recycling system. It was a necessary natural process that helped to keep the environment clean and healthy (Poznyak, 2016). However, the rate of waste produced was too rapid than the rate of natural biodegradation and thus, caused a bunch of environmental issues to the world as the pollution such as air pollution, water pollution, and soil pollution kept increasing. Hence, biodegradability test was introduced to analyze the biodegradability of biodegradable blended film. There were several biodegradability testing methods, which are soil burial test and natural weathering test.

2.9.1 Soil Burial Test

Soil burial test was one of the methods to analyze the biodegradability of the film. This test required the blended film to bury under the soil for a period of time. In order to obtain a better result, fertile soil was more preferable because it was full of nutrients and bacteria (Surip, 2018). Therefore, it able to obtain a better result of biodegradation of the particular blended film. Apart from that, the duration of the test also brought significant effect to the result of biodegradability of blended film. The longer the duration for the blended film to be buried, the better the result as the blended film needed time to degrade. If the time period was longer enough for the blended film to be degraded, the results would become more obvious to be seen and it became easier to distinguish which blended film had better degradation.

2.9.2 Natural Weathering

Natural weathering was another biodegradability testing method. It was conducted by exposing the blended film to the natural environment such as sunlight, rain, humidity moisture, UV radiation and more. Normally, the blended film was hang on a rack and leave it outdoor for a period of time. In order to obtain better result, all the blended film had to be ensure that they were equally expose to the environment by arranged them accordingly with a gap in between. Last but not least, the blended film was checked regularly in order to ensure the blended film was still able to expose to the environment (Surip, 2018).

2.10 Optimization Study

Optimization study was a study that used to seek solution for an objective by minimizing or maximizing the feature parameter of the particular analysis. Meanwhile, it constrained by a set of specified rules within an acceptable range (PTC, 2022). In fact, any optimization process would be achieved by undergo certain phases so that it able to identify the important factors and also determined the factors that was nearest to the optimum. Response surface design was a set of advanced experiment design techniques that used to optimize the response through Response Surface Method (RSM) where the relationship between one or more measured responses and vital

input factor was quantified by RSM (Bhattacharya, 2021). There were two types of response surface design, which are Central Composite Design (CCD) and Box-Behnken Design (BBD). However, CCD was always the choice for such kind of interpretation due to its accuracy was higher compared to the BBD and it did not required a three level of factorial experiment for constructing a second order quadratic model (Minitab, 2022).

2.10.1 Central Composite Design

Central Composite Design (CCD) was also known as Box-Wilson Central Composite Design. It was made up of full factorial or fractional design with a center point that was elevated with a group of axial points which also known as 'star points', where these 'star points' allowed the estimation of curvature. Moreover, the distance from the center of the design space to a star point would be $|\alpha| > 1$, if the distance between the center of the design space and the factorial point was ± 1 unit for each factor. However, the precise value of α was always depends on several properties that desired for the design and the number of factors involved. *Figure 2.10* showed the generation of CCD for two factors involved.



Figure 2.10: Generation of CCD for Two Factors (NIST, 2022).

Furthermore, the k value of CCD was always twice the number of star points in the design. It is because the star points indicated the new extreme values for each of the factors in the design where the value can be high or low (NIST, 2022). Besides that, there were three main types of CCD which depends on the location of the star points placed. The types of CCD included Central Composite Circumscribed (CCC) design, Central Composite Inscribed (CCI) design, and Central Composite Faced centered (CCF) design. *Table 2.2* and *Figure 2.11* showed the description of each type of CCD and the comparison between the three types of CCD respectively.

Types of Central Composite Design	Terminology	Description						
Circumscribed	CCC	Original form of the CCD						
		There is some distance between the star points						
		and the center based on the properties that desired						
		for the design and also the number of factors						
		Star points exhibit new extremes for high and low settings for all factors						
		It is in circular, spherical, or hyper-spherical symmetry design						
		 It required five levels of each factor In order to generate CCC design, existing 						
		factorial or resolution of V fractional factorial						
		design with star points need to be elevated						
Inscribed	CCI	CCI design is a downsize of CCC design with						
		each factor level of CCC design divided by α						
		The factor settings are being used as star points						
		when the situations of limits specified for factor settings are really limited						
		It created a factorial or fractional factorial design						
		within the limits						
		It required five levels of each factor						
Face centered	CCF	The star points are at the center of each face of						
	the factorial space, $(\alpha = \pm 1)$							
		It required three levels of each factor						
	In order to generate CCF design, existing							
		factorial or resolution of V fractional factorial						
		design with star points need to be elevated						

Table 2.2: Description of Each Type of CCD (Central Composite Designs, 2022).



Figure 2.11: Comparison Between the Three Types of CCD (NIST, 2022).

CHAPTER 3

METHODOLOGY

3.1 Process Flow Chart



Figure 3.1: Overall Process Flow Chart

3.2 List of Materials and Chemicals

Table 3.1 listed all the materials and chemicals during the research.

Table 3.1: List of Materials & C	Chemicals
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Materials & Chemicals	Description			
Corn Cob Powder (CCP)	It is used as filler during the film production			
Polyvinyl alcohol (PVA)	It is used as the main polymer during the film production			
Glycerol	It is used as plasticizer during the film production			

3.3 List of Equipment & Apparatus

The list of equipment and apparatus were listed in *Table 3.2*.

Equipment & Apparatus	Description			
Beaker	It is used to place the mixture or chemicals			
Glass Plate	It is used to cast the PVA/CCP/glycerol during blended			
	film production			
Hot Plate Stirrer	It is used to mix and heat up the PVA/CCP/glycerol			
	solution			
Measuring Cylinder	It is used to measure the amount of chemicals			
Weight Balance	It is used to measure the amount of the CCP and glycerol			
Magnetic Bar	It is used to stir the mixture			
Tray	It is used to place the CCP for drying purpose			
Water Bath	It is used to heat up the PVA/CCP/glycerol solution			
Drying Oven	It is used to dry the CCP before film production			
Dumbbell Cutter	It is used to cut the PVA/CCP/glycerol blended film into			
	dumbbell shape for tensile test			
Tensile Tester	It is used to determine the tensile strength of			
	PVA/CCP/glycerol blended film			
Attenuated Total Reflectance	It is used to determine the functional group of CCP and			
(ATR) Spectroscopy	PVA/CCP/glycerol blended film			
Scanning Electron Microscopy	It is used to obtain the information of the surface			
(SEM)	topography and composition			

3.4 Collection and Preparation of Raw Material

Corn Cob Powder (CCP), Polyvinyl alcohol (PVA), and glycerol were the main raw materials that would be used in the research. The prepared CCP was placed on a tray and put it into a drying oven to dry for one day at a temperature of 60°C. The purpose of drying the CCP was to remove the moisture. After dried the CCP, it was kept in a dry place for further usage. PVA and glycerol were obtained from Sigma Aldrich where the molecular weight for PVA and glycerol were 44.05 g/mol and 92.09 g/mol.

3.5 Synthesis of PVA/CCP/Glycerol Blended Film by using Solution Casting Method

Initially, 8g of PVA powder was added into 100mL of distilled water in a 250mL beaker. The solution was stirred for 30 minutes with a constant speed (20 rpm) and constant temperature (90°C) on a hot plate stirrer by using magnetic stirrer bar. The solution was required to stir until the PVA was completely dissolved. At the meantime, 2g of CCP was added into 40mL of distilled water in a 100mL beaker. The solution was also required to stir with a constant speed (20 rpm) and constant temperature (90°C) for 30 minutes. After that, 1mL of glycerol was added into CCP solution and stirred it continuously. Then, the CCP solution was mixed together with the PVA solution and placed it into the water bath for 20 minutes in order to remove all the bubbles and foams. After removing all the bubbles and foams, the PVA/CCP/glycerol solution was poured onto a clean glass plate and leave it to dry at room temperature for 24 hours. The dried PVA/CCP/glycerol solution would then turn into blended film, and it was further dry in oven with a temperature of 30°C for 30 minutes. After that, the PVA/CCP/glycerol blended film was completely synthesize and ready for further analysis. The steps were repeated for another 12 sets of parameters in term of different composition of CCP and glycerol. *Table 3.4* showed the experimental design of different formulation of PVA/CCP/glycerol blended film.

3.6 Optimization using Response Surface Methodology (RSM)

The optimization was conducted by using Response Surface Methodology (RSM) through Central Composite Design (CCD). Composition of CCP and glycerol were the two parameters that would be varied throughout the experiment. *Table 3.3* showed the variables and levels used for CCD for optimization of media composition. The results were analyzed by analysis of variance (ANOVA) using Design Expert 6.0.6 software. The 3D plots and their respective contour plots were obtained based on the effect of levels of the two factors. Based on the 3D plots, the simultaneous interactions of the two factors on the responses were studied. The optimum region was then determined based on the main parameters in the overlay plot. *Table 3.4* showed the experimental design for optimization of mechanical properties of the PVA/CCP/glycerol blended film. The responses that had been quantified in the optimization were tensile strength (unit: MPa), elongation at break (unit: %), and elastic modulus (unit: MPa).

Table 3.3: Variables and Levels used for CCD for Optimization of Media Composition

Independent Variable	Symbol (Coded)	Low Level	High Level
ССР	А	2.00	6.00
Glycerol	В	1.00	3.00

Table 3.4: Experimental Design for Opt	timization of N	Mechanical	Properties (of the
PVA/CCP/Glyc	erol Blended I	Film		

Blend	PVA (g)	CCP (g)	Glycerol (mL)
1	8	2	1
2	8	6	1
3	8	2	3
4	8	6	3
5	8	1.17	2
6	8	6.83	2
7	8	4	0.59
8	8	4	3.41
9	8	4	2
10	8	4	2
11	8	4	2
12	8	4	2
13	8	4	2

3.6.1 Verification of Data

The optimum condition for optimization study was obtained from RSM coupled with CCD. Each of the experiment was repeated five times and each of the result was compared with the predicted values so that the validity of the model was determined.

3.7 Data Analysis

Attenuated Total Reflectance (ATR) Spectroscopy was used to analyze the composition of the CCP sample and PVA/CCP/glycerol blended film while Scanning Electron Microscopy (SEM) was used to analyze the surface of cross section morphology of the PVA/CCP/glycerol blended film. Apart from that, mechanical properties of PVA/CCP/glycerol blended film was analyzed by using tensile tester while biodegradability of PVA/CCP/glycerol blended film was analyzed by using soil burial test.

3.7.1 Tensile Testing Machine

Tensile testing machine was an electromechanical test system that involved application of tensile force onto the material (Roell, 2022). Tinius Olsen H10KS-0784 tensile testing machine was used to determine the tensile strength, elongation at break, and elastic modulus of PVA/CCP/glycerol blended film. Before the PVA/CCP/glycerol blended film went for tensile tester, it was required to cut into five pieces of dumbbell shaped specimens with an average thickness of 0.10 mm, length of 65 mm, and width of 3 mm by using the dumbbell cutter. After that, the dumbbell shaped specimen was ready for the test. It was tested at crosshead speed of 50 mm/min in the tensile tester where the speed was standardized based on the ASTM D638. The results of tensile strength, elongation at break, and elastic modulus of PVA/CCP/glycerol blended film were showed in a stress-strain curves. It was then repeated for another four dumbbell shaped specimens in order to obtain average result. *Figure 3.2* showed the Tinius Olsen H10KS-0784 tensile testing machine.



Figure 3.2: Tinius Olsen H10KS-0784 Tensile Testing Machine

3.7.2 Attenuated Total Reflectance (ATR) Spectroscopy

Attenuated Total Reflectance (ATR) Spectroscopy was a contact sampling method that involved crystal with high refractive index and good IR transmitting properties whereby the light beam would be emitted onto the sample through the crystal and some of the light would be reflected. When the intensity of the reflected light reduced, attenuated total reflectance was occurred and thereby, some of the IR radiation would penetrate beyond the crystal and absorbed by the PVA/CCP/glycerol blended film. Hence, the absorbance was translated into IR spectrum in order to obtain the structural and compositional information of the PVA/CCP/glycerol blended film (Subramanian, 2009). ATR was non-destructive on the sample, shorter time needed, and no sample preparation was required. Meanwhile, Perkin Elmer Spectrum Two was used for ATR analysis and it was showed in *Figure 3.3*. Before the ATR analysis commenced, isopropanol alcohol was used to wipe the diamond cell and a suitable amount of prepared CCP was then placed on the it. After that, the pressure town was being screw down and the sample was pushed firmly against the crystal until the pressure gauge on the software program displayed optimum level of pressure, which was approximate 70% to 80%. In fact, the ATR spectra analysis was measured within a wavelength range of $400cm^{-1}$ to $4000cm^{-1}$ with a resolution of $4cm^{-1}$ for 32 scans. All the steps were repeated by replacing the sample with different formulation of PVA/CCP/glycerol blended film.



Figure 3.3: Perkin Elmer Spectrum Two

3.7.3 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) was a microscopy that used a focused beam of electrons to scan on the surface of the samples provided and generate a high resolution image. The image that produced by SEM able to illustrate information on the surface composition and topography of the particular material (TWI, 2022). Therefore, a PVA/CCP/glycerol blended film sample was prepared and placed it on the SEM machine for analysis. A focused beam of electrons would emitted and scanned on the surface of the PVA/CCP/glycerol blended film. The information of PVA/CCP/glycerol surface composition and topography would be obtained from the high resolution image of SEM. *Figure 3.4* showed the SEM machine.



Figure 3.4: SEM Machine

3.7.4 Biodegradability Test

Biodegradability test was conducted through soil burial test whereby the purpose was to identify the biodegradability of PVA/CCP/glycerol blended film. Initially, PVA/CCP/glycerol blended film were cut into dumbbell shape and the weight of the films were measured before they were buried into the soil. After that, 200g of soil was measured and then stored into a container. The dumbbell shaped PVA/CCP/glycerol blended film was then buried into the soil for 1 month under natural weather. The weight of the blended film was being measured and recorded every 7 days. The percentage of degradation of PVA/CCP/glycerol blended film was calculated by using *Equation 3.1* (Marichelvam, 2019).

Percentage of degradation,
$$\% = \frac{W_o - W}{W_o} \times 100\%$$
 (Equation 3.1)

Where:

 $W_o = Weight of the film before test$ W = Weight of the film after test

CHAPTER 4

RESULT AND DISCUSSIONS

4.1 Characterization of PVA/CCP/Glycerol Blend Films

4.1.1 Attenuated Total Reflectance (ATR) Spectroscopy

Attenuated total reflectance spectroscopy (ATR) was used to determine the functional group of CCP and PVA/CCP/glycerol blended film. However, the functional group of CCP and PVA/CCP/glycerol blended film were identified only in the region 1500 cm^{-1} and above. It was because the region below 1500 cm^{-1} was known as fingerprint region, whereby this region was difficult to identify individual peaks due to large number of peaks were contain in the region. *Figure 4.1* showed the spectra of ATR for CCP. According to the IR spectra from *Figure 4.1*, it showed that CCP had a broad peak at 3329 cm^{-1} , which was corresponding to O-H stretching bond due to the intramolecular hydrogen bond of cellulose. There was also a narrow peak in CCP at 1638 cm^{-1} , whereby this narrow peak attributed to the C=C stretching bond.

According to the study from Liu (2022), it showed that pure PVA blended film was at a peak of $3286 \ cm^{-1}$, which indicated the presence of free hydroxyl groups due to strong intermolecular bond and intramolecular bond. Since glycerol and PVA were hydrophilic, the interaction between the hydroxyl groups from PVA and glycerol could cause a slight changed in the O-H tensile strength when the PVA was blended with the glycerol.

Furthermore, *Figure 4.2* showed the spectra of ATR for PVA/CCP/glycerol blended film with different composition of glycerol and CCP loading. According to the IR spectra of set 1, which was PVA/CCP/glycerol blended film with 2g of CCP and 1mL of glycerol, it showed a

board peak at $3271 \ cm^{-1}$. This peak indicated the blended film had functional group of O-H stretching bond. Besides that, it also had another two narrow peak at 2937 cm^{-1} and 1651 cm^{-1} . The peak at 2937 cm^{-1} attributed to the C-H stretching bond while the peak at 1651 cm^{-1} was corresponding to the C=C stretching bond. The presence of the C-H stretching bond indicated that there were cellulose and hemicellulose in the blended film. Moreover, the other PVA/CCP/glycerol blended film also have nearly like IR spectra as set 1 even though they had different composition of glycerol and CCP loading. Meanwhile, *Table 4.1* showed the infrared absorption peaks of CCP and each of the PVA/CCP/glycerol blended films.



Figure 4.1: Spectra of ATR for CCP









Figure 4.2: Spectra of ATR for PVA/CCP/glycerol Blended Film with Different Composition of Glycerol and CCP Loading (Set 1: Blended film with 2g of CCP & 1mL of glycerol, Set 2: Blended film with 6g of CCP & 1mL of glycerol, Set 3: Blended film with 2g of CCP & 3mL of glycerol, Set 4: Blended film with 6g of CCP & 3mL of glycerol, Set 5: Blended film with 1.17g of CCP & 2mL of glycerol, Set 6: Blended film with 6.83g of CCP & 2mL of glycerol, Set 7: Blended film with 4g of CCP & 0.59mL of glycerol, Set 8: Blended film with 4g of CCP & 3.41mL of glycerol, Set 9: Blended film with 4g of CCP & 2mL of glycerol)

Wavelength, cm^{-1}										
ССР	Blended film with 2g of CCP & 1mL glycerol	Blended film with 6g of CCP & 1mL glycerol	Blended film with 2g of CCP & 3mL glycerol	Blended film with 6g of CCP & 3mL glycerol	Blended film with 1.17g of CCP & 2mL glycerol	Blended film with 6.83g of CCP & 2mL glycerol	Blended film with 4g of CCP & 0.59mL glycerol	Blended film with 4g of CCP & 3.41mL glycerol	Blended film with 4g of CCP & 2mL glycerol	Functional Group
3329	3271	3272	3278	3277	3275	3276	3271	3276	3276	O-H Stretching
-	2938	2938	2937	2937	2937	2936	2937	2938	2937	C-H Stretching
1638	1653	1652	1653	1651	1656	1649	1655	1652	1653	C=C Stretching (isolated)

Table 4.1: Infrared Absorption Peaks of CCP & PVA/CCP/Glycerol Blended Film

4.2 Optimization Study on Tensile Strength, Elongation at break & Elastic Modulus of PVA/CCP/Glycerol with Glycerol as Plasticizer

Response Surface Methodology (RSM) was used to carry out optimization study on the tensile strength, elongation at break, and elastic modulus of PVA/CCP/glycerol blended film where glycerol act as plasticizer. The tensile strength, elongation at break, and elastic modulus of PVA/CCP/glycerol blended film were evaluated based on the loading of CCP and composition of glycerol. It was revealed that blended film that consist of 6.83g of CCP and 2mL of glycerol had the highest tensile strength and elastic modulus but lowest in elongation at break. The higher tensile strength indicated that the strengthen effect of filler incorporated into the blended film had worked. It was because the presence of CCP act as filler and filler was usually added into polymer material to make it stronger by enhancing its rigidity. Meanwhile, higher elastic modulus indicated that the PVA/CCP/glycerol blended film had higher stiffness. It was because the incorporated filler, which was CCP had enhanced the stiffness of the blended film. This might be due to the equal distribution of filler in the PVA/CCP matrix and subsequently hinders the chain movement effectively during deformation (Onuoha, 2017). However, lower elongation at break indicated that the blended film had improved its stiffness but decreased in ductility. Hence, the higher the CCP loading, the lower the elongation at break as the ductility of the blended film decreased. Besides that, the presence of

glycerol can also affected the elongation at break of the blended film. It was due to the intermolecular bond between the PVA molecule was replaced by the intermolecular bond between PVA and glycerol and thus, the blended film can be easily break due to the strength of the bond had decreased (Dianursanti, 2018).

Based on theory of Central Composite Design (CCD), the central point for the optimization process should be 6.83g for CCP loading and 2mL for composition of glycerol. However, the central point for the optimization point in this case was 4g for CCP loading and 2mL for composition of glycerol due to the initial range of CCP loading and composition of glycerol had set to 2 - 6g and 1 - 3mL respectively. *Table 4.2* showed the design layout of CCD with the response.

	Para	meter	Response			
Standard Run	Loading of CCP (g)	Composition of Glycerol (mL)	Tensile Strength (MPa)	Elongation at Break (%)	Elastic Modulus (MPa)	
1	2.00	1.00	7.74 ± 0.4	29.12 ± 4	116.5 ± 10	
2	6.00	1.00	8.73 ± 0.4	23.18 ± 4	118.6 ± 10	
3	2.00	3.00	7.90 ± 0.4	81.7 ± 4	66.8 ± 10	
4	6.00	3.00	5.80 ± 0.4	23.19 ± 4	97.9 ± 10	
5	1.17	2.00	10.97 ± 0.4	74.3 ± 4	100.4 ± 10	
6	6.83	2.00	11.46 ± 0.4	11.78 ± 4	209.9 ± 10	
7	4.00	0.59	8.73 ± 0.4	55.9 ± 4	149.3 ± 10	
8	4.00	3.41	4.689 ± 0.4	110.8 ± 4	51.5 ± 10	
9	4.00	2.00	9.26 ± 0.4	46.14 ± 4	87.4 ± 10	
10	4.00	2.00	8.93 ± 0.4	51.1 ± 4	91.2 ± 10	
11	4.00	2.00	7.01 ± 0.4	38.33 ± 4	92.6 ± 10	
12	4.00	2.00	7.71 ± 0.4	54.9 ± 4	91.3 ± 10	
13	4.00	2.00	7.40 ± 0.4	93.2 ± 4	61.6 ± 10	

Table 4.2: Central Composition Design (CCD) Matrix with Response

4.2.1 Analysis of Variance (ANOVA)

The analysis of variance (ANOVA) was a type of analysis that used to analyze optimization in CCD. There were several statistical models available in ANOVA which are linear, quadratic, cubic, and two factor interaction (2FI). Based on the lowest p-value, the statistical model would be chosen so that the important terms in certain model can be identify.

Table 4.3 showed the ANOVA for response surface quadratic model of tensile strength. According to result of fit summary from design expert software, it showed that all data for quadratic source were the lowest among other sources. Therefore, the analysis of ANOVA of the experimental data should be graphically quadratic as the description statistics were used as a secondary checkpoint for usefulness of the model. Besides that, the F-value for quadratic source was pointed as a references for the suggested analysis by ANOVA. Therefore, the model to be used would be in quadratic.

Furthermore, the model F-value that showed in Table 4.3 was 4.75, which implied that the model was significant and there was only 3.26% of chances that the model F-value of this large could occur due to noise. Since p-values for the data was less than 0.0500, thus it proved that the model terms were significant. The significant model terms in this case were B and A^2 where A indicated CCP, and B indicated glycerol. However, whenever the p-value was greater than 0.1000, it indicated that the model terms were not significant. Therefore, the model reduction might occur to enhance the model. Nevertheless, lack of fit were analyzed. It is known as the significance of data that fits to the full model. When the model provided results as significant as the lack of fit, it would not be selected for the predictions. It would compared the pure error with the residual error from replicated design points. According to Table 4.3, it showed that the lack of fit of F-value was 1.91, which implied that it was not significant relative to pure error. There was 26.96% of chance that "Lack of Fit F-value" of this large could occurred due to noise. Non-significant of lack of fit was good for the model as the model needed to be fit. Adequate precision was used to measure the signal to noise ratio, and it stated that a ratio greater than 4 was desirable for the model. Since the ratio of current model obtained was 7.730, which indicated that it was an adequate signal, thus this quadratic model can be used to navigate the design space. CV% was used to judge the capability of a process in several industries. In this model, the CV% was 14.15%. However, the lower the CV%, the better the model.

Final Equation in Terms of Coded Factors:

Tensile Strength = +8.06 - 0.052 (A) - 1.06 (B) + 1.22 (A²) - 1.03 (B²) - 0.77 (A*B)

The equation in terms of coded factors were used for prediction of the response at given levels of each factor. The codes were +1 and -1 by default, which indicated high level and low level of the factors respectively. Besides, the usage of the coded equation able to identify the relative impact of the factors by comparing the factor coefficients.

Final Equation in Terms of Actual Factors:

Tensile Strength =
$$+7.95897 - 1.69669$$
 (CCP) + 4.60915 (Glycerol) + 0.30539 (*CCP*²) - 1.03119
(*Glycerol*²) - 0.38625 (CCP*Glycerol)

The equation in terms of actual factors were used for prediction of the response at the given levels of each factor. The levels were specified in the original units for each factor in the equation. However, the equation was not used to determine the relative impact of each factor due to the coefficients were scaled to accommodate the units of each factor and the interception was not at the center of the design space as well.

The comparison between the Predicted R-squared and Adjusted R-squared with the adequate value was illustrated with the diagnostic part of the optimization. *Figure 4.3* and *Figure 4.4* showed the normal probability plot for tensile strength and predicted against actual graph for tensile strength respectively. The normal probability plot was used to analyze whether that the residuals able to follow the normal distribution. Generally, the residuals would followed the straight line of the graph, and it was expected to have some scatters within the normal data. In order to analyze a more definite patterns, a slight "S-Shaped" curve was showed in the graph, and it attributed that a transformation of response might provide a better data analysis. Besides that, the predicted against actual graph was basically a comparison between the predicted value and actual value. The comparison was analyzed to detect for a value or a group of values that were not easily to be predicted by the model.

Source	Sum of Squares	DF	Mean Square	F-value	p-value	
Model	31.82	5	6.36	4.75	0.0326	Significant
А	0.022	1	0.022	0.016	0.9022	
В	9.00	1	9.00	6.72	0.0359	
A^2	10.38	1	10.38	7.75	0.0272	
B ²	7.40	1	7.40	5.52	0.0511	
AB	2.39	1	2.39	1.78	0.2237	
Residual	9.38	7	1.34			
Lack of Fit	5.52	3	1.84	1.91	0.2696	Not Significant
Pure Error	3.86	4	0.96			
Cor Total	41.20	12				
Standard Deviation	1.16	R ²	0.7723			
Mean	8.18	Adjusted R ²	0.6097			
C.V. %	14.15	Adequate Precision	7.730			

Table 4.3: ANOVA for Response Surface Quadratic Model (Tensile Strength)



Figure 4.3: Normal Probability Plot for Tensile Strength



Figure 4.4: Predicted against Actual Graph for Tensile Strength

Table 4.4 showed the ANOVA for response surface linear model of elongation at break. According to result of fit summary from design expert software, it showed that all data for linear source were the lowest among other sources. Therefore, the analysis of ANOVA of the experimental data should be graphically linear as the description statistics were used as a secondary checkpoint for usefulness of the model. Besides that, the F-value for linear source were pointed as a references for the suggested analysis by ANOVA. Therefore. The model to be used would be in linear.

Moreover, the model F-value that showed in *Table 4.4* was 4.59, which implied that the model was significant and there was only 3.26% of chances that the model F-value of this large could occur due to noise. Since p-values for the data was less than 0.0500, thus it proved that the model terms were significant. The significant model term in this case was A where A indicated CCP. However, whenever the p-value was greater than 0.1000, it indicated that the model terms were not significant. Therefore, the model reduction might occur in order to enhance the model. Meanwhile, lack of fit were also analyzed. According to *Table 4.4*, it showed that the lack of fit

of F-value was 1.04, which implied that it was not significant relative to pure error. There was 49.93% of chance that "Lack of Fit F-value" of this large could occurred due to noise. Non-significant of lack of fit was good for the model as the model needed to be fit. Furthermore, adequate precision was used to measure the signal to noise ratio, and it stated that a ratio greater than 4 was desirable for the model. Since the ratio of current model obtained was 6.497, which indicated that it was an adequate signal and thus, this quadratic model can be used to navigate the design space. CV% was used to judge the capability of a process in several industries. In this model, the CV% was 40.36%. However, the lower the CV%, the better the model.

Final Equation in Terms of Coded Factors:

Elongation at Break = +61.84 - 19.11 (A) + 16.28 (B) - 13.78 (A^2)

Final Equation in Terms of Actual Factors:

Elongation at Break = +12.38299 + 18.00171 (CCP) + 16.27879 (Glycerol) - 3.44448 (CCP²)

The comparison between the Predicted R-squared and Adjusted R-squared with the adequate value was illustrated with the diagnostic part of the optimization. *Figure 4.5* and *Figure 4.6* showed the normal probability plot for elongation at break and predicted against actual graph for elongation at break respectively. The normal probability plot was used to analyze whether that the residuals able to follow the normal distribution. Generally, the residuals would followed the straight line of the graph, and it was expected to have some scatters within the normal data. In order to analyze a more definite patterns, a slight "S-Shaped" curve was showed in the graph, and it attributed that a transformation of response might provide a better data analysis. Besides that, the predicted against actual graph was basically a comparison between the predicted value and actual value. The comparison was analyzed to detect for a value or a group of values that were not easily to be predicted by the model.

Source	Sum of Squares	DF	Mean Square	F-value	p-value	
Model	6384.44	3	2128.15	4.59	0.0326	Significant
А	2921.03	1	2921.03	6.30	0.0333	
В	2119.99	1	2119.99	4.57	0.0612	
A ²	1343.42	1	1343.42	2.90	0.1229	
Residual	4173.27	9	463.70			
Lack of Fit	2357.46	5	471.49	1.04	0.4993	Not Significant
Pure Error	1815.81	4	453.95			
Cor Total	10557.71	12				
Standard Deviation	21.53	<i>R</i> ²	0.6047			
Mean	53.36	Adjusted R ²	0.4730			
C.V. %	40.36	Adequate Precision	6.497			

Table 4.4: ANOVA for Response Surface Linear Model (Elongation at Break)



Figure 4.5: Normal Probability Plot for Elongation at Break



Figure 4.6: Predicted against Actual Graph for Elongation at Break

Table 4.5 showed the ANOVA for response surface quadratic model of elastic modulus. According to result of fit summary from design expert software, it showed that all data for quadratic source were the lowest among other sources. Therefore, the analysis of ANOVA of the experimental data should be graphically quadratic as the description statistics were used as a secondary checkpoint for usefulness of the model. Besides that, the F-value for quadratic source was pointed as a references for the suggested analysis by ANOVA. Therefore. The model to be used would be in quadratic.

Table 4.5 showed model F-value was 9.50, which implied that the model was significant and there was only 0.38% of chances that the model F-value of this large could occur due to noise. Since p-values for the data was less than 0.0500, thus it proved that the model terms were significant. The significant model terms in this case were A,B, and A^2 where A indicated CCP, and B indicated glycerol. However, whenever the p-value was greater than 0.1000, it indicated that the model terms were not significant. Therefore, the model reduction might occur in order to enhance the model. Nevertheless, lack of fit were analyzed. According to *Table 4.5*, it showed that the lack of fit of F-value was 4.86, which implied that it was not significant relative to pure error. There was 7.53% of chance that "Lack of Fit F-value" of this large could occurred due to noise.

Moreover, adequate precision was used to measure the signal to noise ratio, and it stated that a ratio greater than 4 was desirable for the model. Since the ratio of current model obtained was 9.782, which indicated that it was an adequate signal, thus this quadratic model can be used to navigate the design space. CV% was used to judge the capability of a process in several industries. In this model, the CV% was 22.67%. However, the lower the CV%, the better the model.

Final Equation in Terms of Coded Factors:

Elastic Modulus = +85.40 + 23.51 (A) - 26.09 (B) + 28.10 (A^2)

Final Equation in Terms of Actual Factors:

Elastic Modulus = +202.96343 - 44.44648 (CCP) - 26.08876 (Glycerol) + 7.02500 (CCP²)

The comparison between the Predicted R-squared and Adjusted R-squared with the adequate value was illustrated with the diagnostic part of the optimization. *Figure 4.7* and *Figure 4.8* showed the normal probability plot for tensile strength and predicted against actual graph for tensile strength respectively. The normal probability plot was used to analyze whether that the residuals able to follow the normal distribution. Generally, the residuals would followed the straight line of the graph, and it was expected to have some scatters within the normal data. In order to analyze a more definite patterns, a slight "S-Shaped" curve was showed in the graph, and it attributed that a transformation of response might provide a better data analysis. Besides that, the predicted against actual graph was basically a comparison between the predicted value and actual value. The comparison was analyzed to detect for a value or a group of values that were not easily to be predicted by the model.

Source	Sum of Squares	DF	Mean Square	F-value	p-value	
Model	15453.65	3	5151.22	9.50	0.0038	Significant
А	4420.65	1	4420.65	8.15	0.0189	
В	5444.99	1	5444.99	10.04	0.0114	
A ²	5588.99	1	5588.01	10.31	0.0106	
Residual	5588.01	9	542.12			
Lack of Fit	4879.10	5	838.01	4.86	0.0753	Not Significant
Pure Error	689.05	4	172.26			
Cor Total	20332.75	12				
Standard Deviation	23.28	R ²	0.7600			
Mean	102.69	Adjusted R ²	0.6800			
C.V. %	22.67	Adequate Precision	9.782			

Table 4.5: ANOVA for Response Surface Quadratic Model (Elastic Modulus)



Figure 4.7: Normal Probability Plot for Elastic Modulus



Figure 4.8: Predicted against Actual Graph for Elastic Modulus

4.2.2 Three Dimensions (3D) Surface Plot

3-Dimensions surface plot graph provided the image of optimum point of data. *Figure 4.9* showed the 3D surface graph on tensile strength for interaction between loading of CCP and composition of glycerol. According to the 3D graph, it did not showed a proper dome shape due to several reasons where one of them was the tensile strength data itself. Besides that, the composition of glycerol was yet to reach its optimum composition for obtaining an optimum formulation for the blended film. The 3D graph also showed that the tensile strength of PVA/CCP/glycerol blended film would decreased if the composition of glycerol reach its optimum condition. It was because glycerol was one of the plasticizer and plasticizer able to enhance the workability and flexibility of the blended film by increasing the intermolecular separation of the blended film molecules. Hence, it had explained and proved that the reduced of tensile strength in present study was correct.



Figure 4.9: 3-Dimension Surface Graph on Tensile Strength for Interaction between Two Factors

Moreover, *Figure 4.10* showed the 3D surface graph on elongation at break for interaction between loading of CCP and composition of glycerol. According to the 3D graph, it also did not showed a proper dome shape due to several reasons where one of them was the elongation at break data itself. Meanwhile, the loading of CCP was considered reached its optimum condition as the trend of the graph showed that the loading of CCP was going towards the center point. The optimum result of elongation at break was 110.8% with optimum composition of glycerol and loading of CCP. According to Novianti (2020), it stated that any of the elongation at break of blended film had exceeded 10%, it considered good, and even considered well if the elongation at break was break exceeded 50%. Hence, it had explained and proved that the optimum result of elongation at break in present study was correct.



Figure 4.10: 3-Dimension Surface Graph on Elongation at Break for Interaction between Two Factors

Nevertheless, *Figure 4.11* showed the 3D surface graph on elastic modulus for interaction between loading of CCP and composition of glycerol. According to the 3D graph, it also did not showed a proper dome shape due to several reasons where one of them was the elastic modulus data itself. Furthermore, the composition of glycerol was yet to reach its optimum composition for obtaining an optimum formulation for the blended film. It can also be seen from the 3D graph that the elastic modulus would decreased when the composition of glycerol reach its optimum condition. The reason that the elastic modulus of PVA/CCP/glycerol blended film would decreased was due to the elastic deformation of the grain boundaries, which indicated that the blended film would became more elastic and less stiff. Therefore, the result fulfilled the theory that the blended film would have lower elastic modulus when the composition of glycerol reach its optimum condition. Hence, it had explained and proved that the reduced of elastic modulus in present study was correct.



Figure 4.11: 3-Dimension Surface Graph on Elastic Modulus for Interaction between Two Factors

4.2.3 Model Verification

The design expert plot illustrated the interaction between the loading of CCP and composition of glycerol. The optimum condition to obtain the optimum tensile strength, elongation at break, and elastic modulus were 3g of CCP and 1mL of glycerol. In this study, the predicted result for tensile strength, elongation at break, and elastic modulus were 10.0333MPa, 12.6707%, and 163.096MPa respectively.

In order to verify the model adequacy, 5 sets of experiments were repeated at optimum condition. The percentage error of tensile strength, elongation at break, and elastic modulus between the experimental result and predicted result were shown in *Table 4.6*. It showed that all of the percentage error were in the range of 7.28% to 9.23%. Since the percentage error were less than 10%, thus the validity had been proved.
Run	Experimental Result			Predicted Result			Percentage Error (%)		
	Tensile	Elongation	Elastic	Tensile	Elongation	Elastic	Tensile	Elongation	Elastic
	Strength	at Break	Modulus	Strength	at Break	Modulus	Strength	at Break	Modulus
	(MPa)	(%)	(MPa)	(MPa)	(%)	(MPa)	(MPa)	(%)	(MPa)
1	9.164	11.72	148.6	10.0333	12.6707	163.096	8.66	7.50	8.89
2	9.191	11.55	151.4	10.0333	12.6707	163.096	8.4	8.84	7.17
3	9.303	11.58	148.7	10.0333	12.6707	163.096	7.28	8.63	8.83
4	9.118	11.50	147.5	10.0333	12.6707	163.096	9.12	9.23	9.56
5	9.149	11.64	149.8	10.0333	12.6707	163.096	8.81	7.35	8.14

Table 4.6: Validation of Data & Model Constructed

4.3 Scanning Electron Microscopy (SEM)

In this case, SEM with magnification of 500 was used to determine the cross section morphology of PVA/CCP/glycerol blended film. Figure 4.12 showed the cross section morphology of three types of PVA/CCP/glycerol blended film, which were blended film that had lowest tensile strength (A), blended film that had highest tensile strength (B), and blended film that had moderate tensile strength (C). Based on the images provided, it was observed that several starch fibers especially for the blended film that have the lowest tensile strength, were pulled out from the polymer matrix and formed large cavity (shown in red arrow) during the fracture process. This scenario might occurred due to the used of unevenly distribution of treated fibers. It was because the unevenly distribution of treated fibers might contributed to poor interfacial adhesion between the starch fibers and polymer matrix. Therefore, it caused the stress transfer between starch fibers and polymer matrix become ineffective (Scanning electron microscopy (SEM), 2022). Hence, the blended film would break easily during tensile testing as the starch fibers were pulled out and formed cavity easily. Besides that, it can also be observed that there were several breakages of starch fibers around the blended film during fracture process, especially for the blended film that had highest tensile strength. However, the fibers surface of the breakages had showed many traces of polymer matrix adhering to it, which indicated that the blended film had better wetting of starch fibers due to close contact between starch fiber and polymer matrix. A closer contact between starch fibers and polymer matrix also attributed to good fiber-matrix adhesion. Hence, it indicated that blended film that have lower tensile strength required better wetting of starch fibers and good fiber-matrix adhesion.



Figure 4.12: SEM image of Fracture Surface of PVA/CCP/glycerol Blended Film at 500x Magnification (A) Lowest Tensile Strength (B) Highest Tensile Strength (C) Moderate Tensile Strength

4.4 Biodegradable Properties

All the PVA/CCP/glycerol blended film had conducted the biodegradability test for a month and the weight of each of the blended film had been measured and recorded every week. The result of biodegradability test for all the PVA/CCP/glycerol blended film were showed in Figure 4.13. According to the graph from Figure 4.13, it showed that the trend of the graph for all the blended film were increased gradually, which indicated that all the blended film had degraded every week. Based on the result showed, PVA/CCP/glycerol blended film with 2g of CCP and 1mL of glycerol had the highest percentage of degradation while the blended film with 4g of CCP and 3.41mL of glycerol had the lowest percentage degradation after 1 week of biodegradability test, which were 17.69% of degradation rate and 11.24% of degradation rate respectively. After the following week of biodegradability test, the highest percentage of degradation was no longer the blended film with 2g of CCP and 1mL of glycerol, but it was the blended film with 6g of CCP and 3mL of glycerol. It had 24.57% of degradation rate in the second week. Meanwhile, the blended film with 6.83g of CCP and 2mL of glycerol had the lowest percentage of degradation, which was 16.85% only. When the biodegradability test came to the third week, the highest percentage degradation was the blended film with 4g of CCP and 2mL of glycerol, which had a percentage of degradation of 32.14% while the lowest degradation rate was belong to the blended film with 4g of CCP and 0.59mL of glycerol, which was 20.95%. However, after 1 month of biodegradability test, the highest percentage of degradation was belonged to the blended film with 2g of CCP and 1mL of glycerol again. It had a percentage degradation rate of 43.24%. Meanwhile, the blended film with 6.83g of CCP and 2mL of glycerol had the lowest percentage of degradation, which was 25.30%. According to the result, it showed that the loading of CCP and composition of glycerol do affected the biodegradability of the blended film, but the premise was fabricated the blended film with optimum loading of CCP and composition of glycerol instead of fabricating blended film with higher loading of CCP even though higher content of CCP might helped to enhance its biodegradability. However, the content of CCP and glycerol in the blended film have to be optimum so that the biodegradability of blended film can be achieved. The composition of glycerol in the blended film might affected the degradation rate because glycerol could leached out from the blended film as glycerol had strong affinity towards the water and absorption of glycerol by the soil. It was due to water act as a strong driving force to leach the



glycerol out from the blended film to soil moisture. In addition, glycerol of the blended film would be absorbed by the soil to increase the bioavailability of soil water.

Figure 4.13: Result of Biodegradability Test for each PVA/CCP/glycerol Blended Film

Furthermore, the physical appearances for each of the blended film were shown in *Figure* 4.14. According to the result that showed in *Figure 4.14*, all of the PVA/CCP/glycerol blended film were shrunk after 1 month of biodegradability test. It was because annealing effect happened as the blended film was shrunk and swelled easily when it exposed to sunlight. Besides, the weather was extreme during the biodegradability test. Therefore, it might also be one of the factor that caused the blended film shrunk and swelled. According to Suki (2014), the study stated that blended film was considered as a hydrophilic polymer and it able to absorb water during raining period, which then allowed the blended film occurred annealing effect easily. Moreover, the blended film might had cracking on the surface when the scenario of shrunk and swelled occurred.

There were several factors that might affected the result of biodegradability test. Firstly, the soil might stick onto the blended film and affected the accuracy of the weight of blended film. Next, there might be some pieces of blended film lose in the soil and the microorganism colonies

would formed on the blended film, which was very difficult to clean it. However, these can avoid when the procedure was done completely (Ostadi., 2020). Lastly, the dispersion of glycerol in the blended film might affected its biodegradability rate as well.



Figure 4.14: Physical Appearances for PVA/CCP/Glycerol Blended Film with Different Composition of Glycerol and CCP Loading (Set 1: Blended film with 2g of CCP & 1mL of glycerol, Set 2: Blended film with 6g of CCP & 1mL of glycerol, Set 3: Blended film with 2g of CCP & 3mL of glycerol, Set 4: Blended film with 6g of CCP & 3mL of glycerol, Set 5: Blended film with 1.17g of CCP & 2mL of glycerol, Set 6: Blended film with 6.83g of CCP & 2mL of glycerol, Set 7: Blended film with 4g of CCP & 0.59mL of glycerol, Set 8: Blended film with 4g of CCP & 3.41mL of glycerol, Set 9: Blended film with 4g of CCP & 2mL of glycerol)

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Summary of Study

In this study, the PVA/CCP/glycerol blended film were successfully fabricated by using solution casting method. Besides that, the effect of glycerol composition and CCP loading on the mechanical properties of PVA/CCP/glycerol blended film by using optimization tool were accomplished successfully as well.

Nevertheless, the optimization of tensile strength, elongation at break, and elastic modulus of PVA/CCP/glycerol blended film were analyzed by using RSM method. However, all of them did not showed a dome shape 3D surface graph as the composition of glycerol and loading of CCP were yet to reach its optimum. Instead, the software calculation had recommended the best result from the data obtained to construct the 3D surface graph. Meanwhile, the calculated data from ANOVA had showed significant result where each of the R-squared value of tensile strength, elongation at break, and elastic modulus were 0.7723, 0.6047, and 0.7600 respectively, which is nearly to 1.000. In a nutshell, the results obtained considered a good result even though the optimization point was yet to obtain in this study.

5.2 Recommendation for Future Study

Throughout the results and discussions of this research project, there were several aspects that can be modified or enhanced the hand skill in order to improve the results and the quality of this research project. There were several recommendations listed down below:

- i. During preparation of PVA/CCP/glycerol blended film, CCP can blended finer in order to ensure the blended film able to mix uniformly with the PVA as well as the glycerol.
- ii. When the PVA/CCP/glycerol solution was poured into the glass plate, the solution was ensured to be poured uniformly throughout the glass plate so that the thickness of the PVA/CCP/glycerol blended film was uniform.
- iii. Further characterizations of the CCP and PVA/CCP/glycerol blended films can be performed by using Energy Dispersive X-Ray (EDX), Particle Size Analysis (PSA), water permeability test and more characterization.
- iv. Different types of plasticizers, additives and crosslinking agents can be studied to determine the mechanical properties of PVA/CCP/glycerol blended films.
- v. For the biodegradability test, the duration for degradation of the PVA/CCP/glycerol blended film can be extended in order to allow the blended film to have enough time to degrade it in the soil.
- vi. Further analysis on the biodegradability test can be done such as undergoes ATR to determine the functional group after degradation, FESEM can be done by observe the type of microorganisms that grow on the surface and mechanical properties of the PVA/CCP/glycerol blend films.

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APPENDICES

APPENDIX A: Mechanical Properties



A. Tensile Strength for PVA/CCP/Glycerol Blended Film in different loading of CCP & Composition of Glycerol



B. Elongation at Break for PVA/CCP/Glycerol Blended Film in different loading of CCP & Composition of Glycerol



C. Elastic Modulus for PVA/CCP/Glycerol Blended Film in different loading of CCP & Composition of Glycerol

APPENDIX B: Degradation Rate for Biodegradability Test

٨	Waisht of Dash	DVA/CCD/Class	anal Dlandad Eilm	during Call Duriel Test
А.	weight of Each	PVA/CCP/GIYC	erol Blended Film	1 during Son Burial Test

Number of Days	7	14	21	28
Blended film with 2g of CCP & 1mL of glycerol	17.68519	18.51852	29.25926	43.24074
Blended film with 6g of CCP & 1mL of glycerol	14.0989	18.79853	25.58235	26.604
Blended film with 2g of CCP & 3mL of glycerol	14.54813	23.07127	31.52094	40.19104
Blended film with 6g of CCP & 3mL of glycerol	13.43658	24.57095	25.11511	29.55211
Blended film with 1.17g of CCP & 2mL of				
glycerol	11.91904	20.83958	23.46327	31.4093
Blended film with 6.83g of CCP & 2mL of				
glycerol	13.02113	16.84752	23.75785	25.29983
Blended film with 4g of CCP & 0.59mL of				
glycerol	11.85455	20.58182	20.94545	33.01818
Blended film with 4g of CCP & 3.41mL of				
glycerol	11.24145	19.20821	24.78006	27.56598
Blended film with 4g of CCP & 2mL of glycerol	14.84794	24.44842	32.14073	39.05784

• Highlighted cells represent the highest weight of the blended film among all the PVA/CCP/glycerol blended film for the particular week