

DEVELOPMENT OF CHEMICALLY TREATED SUGAR PALM FIBRE REINFORCED PLA COMPOSITE THROUGH FUSED DEPOSITION MODELLING 3D PRINTING.



BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY (PROCESS) WITH HONOURS

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Faculty of Mechanical and Manufacturing Engineering Technology



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Bachelor Of Manufacturing Engineering Technology (Process) With Honours

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2021

DECLARATION

I declare that this Choose an item. entitled "Development of chemically treated sugar palm fibre reinforced PLA composite through Fused Deposition Modelling 3D Printing." is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours.



DEDICATION

In the name of Allah, the Most Gracious, the Most Merciful and all praises to the Prophet, Muhammad S.A.W. Alhamdulillah, praise to Allah for His mercy, I have successfully completed this project in a timely manner.

I would like to take this opportunity to extend my utmost gratitude and sincere appreciations, to my mother, Nor Fazila Binti Zabri and my father Abdul Razak Bin Mat Noh for their support and sacrifice to confront with all problems and difficulties along this journey, mentally and physically.

May Allah rewards all of you with goodness and prosperity, here and hereafter

تىكنىكا ملىسىا ملاك

ABSTRACT

Nowadays, 3d printers is an advanced technologies that use to tangible objects that occupy space. Like a normal paper printer, 3d printer also need an ink or filler to perform printing process but what make it difference is, ink for 3d printer is in solid state and it is called filament. Polylactic acid (PLA) and Acrylonitrile Butafiene Styrene (ABS) is a common material use to create filament. Printing using filament that contain 100% PLA can only withstand small load or force which mostly use to create product that can be use as a prototype or merchandise. Other than that, PLA can be recycle but due to its low melting point, recycling PLA is not main consideration. In this research, the main focus is to create biocomposite material to use as a material to create a filament that will has stronger and higher thermal deflection properties. The idea is sugar palm fibre as natural fibre will reinforced by PLA as matrix. Creating this biocomposite material will need several process from degradation of SPF, fibre treatment, mixing, extrusion process until the analysis phase which is thermogravimetric (TGA), Differential scanning calorymetri, and rhological studies. Creating hypothesis of this research required 4 specimen to undergoes these 3 analysis which is untreated SPF, and treated SPF that divided in three treatment which is NAoH solution, silane solution, and NAoH + Silane treatment. It is believed that biocomposite material will have higher melting point, stronger bond, and higher thermal stability which make it will be usable as a daily product and better at recycling process.

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ABSTRAK

Hari ini, pencetak 3D ialah teknologi canggih untuk objek ketara yang menggunakan ruang. Seperti pencetak kertas biasa, pencetak 3d juga memerlukan dakwat atau pengisi untuk melakukan proses pencetakan, tetapi perbezaannya ialah dakwat pencetak 3d adalah pepejal, yang dipanggil filamen. Asid polilaktik (PLA) dan akrilonitril butadiena stirena (ABS) adalah bahan biasa untuk membuat filamen. Pencetakan dengan filamen vang mengandungi 100% PLA hanva boleh menahan beban atau dava vang kecil dan digunakan terutamanya untuk mencipta produk yang boleh digunakan sebagai prototaip atau produk komersial. Di samping itu, PLA boleh dikitar semula, tetapi mengitar semula PLA bukanlah pertimbangan utama kerana takat leburnya yang rendah. Dalam penvelidikan ini, tumpuan utama adalah untuk mencipta biokomposit untuk digunakan sebagai bahan untuk membuat filamen dengan sifat ubah bentuk haba yang lebih kuat dan lebih tinggi. Ideanya ialah gentian kelapa sawit sebagai gentian semula jadi akan diperkukuh oleh PLA sebagai matriks. Pembuatan biokomposit ini memerlukan beberapa proses bermula daripada degradasi SPF, pengendalian gentian, pencampuran, proses penyemperitan hingga ke peringkat analisis termogravimetrik (TGA), kalorimetri pengimbasan pembezaan dan kajian reologi. Membina hipotesis untuk kajian ini memerlukan 4 sampel untuk 3 analisis ini, SPF tidak dirawat dan SPF dirawat, dibahagikan kepada tiga rawatan, larutan NAoH, larutan silane, dan rawatan NAoH + silane. Adalah dipercayai bahawa biokomposit akan mempunyai takat lebur yang lebih tinggi, ikatan yang lebih kuat dan kestabilan haba yang lebih tinggi, membolehkan ia digunakan sebagai barangan harian dan dikitar semula dengan lebih baik.

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No hundo.

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CHAPTER 1

INTRODUCTION

1.0 Introduction

In the first chapter, project introduction was explained which contain the project background, problem statement, objective and work scope

1.1 Background

In this decade, the expanding rate of technology is very high. This scenario has conducted inventory of many products that use to create others product for example is the existence of 3d printer. The first 3D printers were introduced to the market in the mid-1990s. In this day, A variety of technologies are used in 3D printers. Fused deposition modelling (FDM), also known as fused filament fabrication, (FFF) is the most well-known technologies used . It involves melting and depositing layers of acrylamide butane styrene (ABS), polylactic acid (PLA), or another thermoplastic filament through a heated extrusion nozzle.



Figure 1 Fused Desposition Modelling 3D-printers (Fused Deposition Modeling FDM 3D Printing Technology | How It Works, n.d.)

Fig.1.1 Fused Desposition Modelling 3D-printers (Fused Deposition Modeling FDM 3D Printing Technology | How It Works, n.d.)

Commonly, the usage of 3d printer is only limited from presenting level such as demonstration, prototype and model, to moderate or soft duty product. It is due to the properties of the filament is not strong enough to be use in bigger or stronger field..

Creating better environment for 3d printer by using bio-composite filament for example by reinforcing PLA with natural fibre, such as sugar palm. Bio-composites have less environmental footprints, hence, they are safer for humans and other living habitats and they are recyclable and reusable. The mechanical performance of fiber-reinforced composites can be affected by many factors including the volume or weight fraction of the reinforcement, the orientation of the fibers, the fiber aspect ratio, fiber-matrix adhesion, fiber alignment, distribution, use of additives, and chemical treatment of fibers. It is important to add that the moisture absorption of the composites also affects the mechanical behavior of the composites which leads to the poor interfacial bonding between fiber and hydrophobic matrix polymer (Thyavihalli Girijappa et al., 2019).

The degraded fibre will undergoes chemical treatment that has the main purpose which is to enhance the properties of the fiber itself by modifying their microstructure along with improvement in wettability, surface morphology, chemical groups and tensile strength of the fibers (Siakeng et al., 2019)

Mixing polylactic acid(PLA) with natural fibre (sugar palm fibre) is to create sturdy, higher melting point and etc filament for fusion filament fabrication or fused deposition modelling (FDM) . new biocomposite material will went through Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) to characterize materials used in various environmental, food, pharmaceutical, and petrochemical applications and

measure the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic and exothermic processes respectively. Thermogravimetric analysis (TGA) is performed on an instrument called a thermogravimetric analyzer. As the temperature of the sample changes over time, the thermogravimetric analyzer continuously measures the mass. Mass, temperature, and time are considered as baseline measurements in thermogravimetric analysis, and many other measurements can be derived from these three baseline measurements (Bottom, 2008).

Differential Scanning Calorimetry (DSC) is a thermal analysis technique in which heat entering or leaving the sample is measured as a function of temperature or time while the sample is exposed to a controlled temperature program. It is a very powerful technique that can be used to evaluate material properties such as glass transition temperature, melting, crystallization, specific heat capacity, curing process, purity, oxidation behavior, and thermal stability. DSC analysis can provide test data for various materials including polymers, plastics, composites, organic materials, rubber, petroleum, chemicals, explosives, biological samples (*Differential Scanning Calorimetry (DSC) Analysis*, n.d.).

UNIVERSITI TEKNIKAL MALAYSIA MELAKA 1.2 Problem Statement

3D printing, also known as additive manufacturing, is becoming popular with manufacturers. The demand is growing due to some of the revolutionary benefits that it can provide. Like almost all technologies it has its own drawbacks that need considering.

3D printing process can create items from a variety of plastics, the available raw materials are not exhaustive such as PLA . PLA material can be recycle but due to its low melting point, recycling PLA is not main consideration and not giving to much benefit.

Due to a tremendous rise in demand and a concurrent supply deficit, the price of PLA filament has been continuously rising. Because this is one of the most often used 3D printing materials, the price hikes have many customers in a frenzy.

There are various ways for 3D printing, but the most common is a technique known as Fused Deposition Modeling (FDM). FDM printers use a thermoplastic filament, which is heated to its melting point and then extruded, layer by layer, to create a three dimensional object. Under this circumstances, due to properties that is low in strength and low heat deflection temperature FDM is only limited in modelling or prototype rather than functional parts.

Creating biocomposite filament that have good in strength, the data of its thermal properties need to be obtain so that the temperature of the nozzle of the 3d printer can be sync with the filament thermal properties. The deformation of biocomposite filament when flow at different speed during melt state need to be observe to see whether the filament suit the nozzle of the 3d printer.

1.3 Research Objective | TEKNIKAL MALAYSIA MELAKA

The objective of this project are :

- • To develop sugar palm fibre reinforce PLA composite through extrusion mixed with different treatment fibre and untreated fibre.
- To Characterize thermal and reological properties of the composite

1.4 Scope of Research

- I. Sugar palm fibre as natural fibre.
- II. Matrix used is polyactic acid (PLA).

- III. Sugar palm fibre (SPF), will go through degradation process until the SPF is in powder form (125 μ m).
- IV. Different treatment SPF which is alkali solution, silane solution treatment, alkalisilane solution and untreated SPF will mix with PLA .
- V. Filament deposite through twin-screw extruder machine.
- VI. Thermal properties using TGA and DSC analysis and rheological study finding.



CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

Chapter 2 will be some discussion about the research background related to the project. This chapter also discusses the journals as references and examples from other sources linked to the project.

2.1 Natural Fibre

Natural fibre, any hair-like raw material obtained directly from an animal, vegetable, or mineral source and converted into nonwoven textiles such as felt or paper, or woven cloth after spinning into yarns.



Figure 2 Sugar Palm Fibre

A natural fibre is further described as an aggregation of cells with a diameter that is minimal in contrast to the length. Aside from economic concerns, the commercial utility of a fibre is decided by qualities such as length, strength, pliability, elasticity, abrasion resistance, absorbency, and other surface qualities. The majority of textile fibres are thin, flexible, and somewhat robust.. They are elastic in that they stretch when put under tension and then partially or completely return to their original length when the tension is remove (Kamalnath et al., 2018).

2.2.1 Classification of Narutal fibre

Natural fibres are classed based on whether they are derived from plants, animals, or minerals . Natural plant fibres, on the other hand, are the most often utilised reinforcing material in biocomposites. Plant fibres are classified according to the kind of plant or portions of the plant from which the fibres were collected. Figure below depicts the major plant fibre classifications, which include bast, leaf, seed, and fruit, stalk, and grass fibers. All these mentioned plant fibers are term as non-wood fibers. Of late, many researches focus on the usage of non-wood fibers (Sanyang et al., 2016).



Fibres



Figure 3 Chart of classification of Natural fibre (Kamalnath et al., 2018)

2.2.2 Sugar palm fibre (Arenga Pinnata)

Sugar palm is a massive and tall palm with a single unbranched stem that may reach 20 metres in height and 65 centimetres in diameter. The trunk is covered in long black fibres and the roots of broken leaves. The trunk can also be used to store starch. The starch is generally converted into sugars at the commencement of flowering for the production of seeds or tapped palm juice.



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The palmae family includes the sugar palm tree (Arenga pinnata). This plant is commonly found in the hot, humid parts of the Asian tropics, and it has a wide range of uses, making it a useful palm species. Sugar palm fibre, as a relatively new natural fibre in comparison to other natural fibres, can be classified in a variety of ways. The reason for this is that the fibre of the sugar palm tree might come from a cluster, a frond, or the trunk (Essaadani et al., 1991).

Malaysia, as a tropical country, has an abundance of natural fibre resources. Sugar palm fibre (locally known as ijuk fibre) is one of the plentiful natural fibres found in Malaysia,

however it has not been widely employed as reinforcement in the fabrication of polymer composites.

2.2 Polylactic acid (PLA)

Polylactic acid polymers, also known as polylactides, are lactic acid polyesters that have recently been commercialised for goods that need biodegradability. Polylactic acid is a versatile polymer derived from sustainable agricultural source resources that have been fermented to produce lactic acid. The lactic acid is subsequently polymerised to the desired length using a cyclic dilactone, lactide, ring opening. The lactic acid is subsequently polymerised to the desired polylactic acid by a cyclic dilactone, lactide, ring opening. Certain modifications are made to the polymer, which improves its temperature stability and reduces its residual monomer content (Oksman et al., 2003).



Figure 5 Polylactic Acid (PLA)

PLA can be treated in a manner similar to polypropylene, flax reinforced composites via extrusion should be viable. Tensile testing was used to investigate the mechanical characteristics of the composites. (Oksman et al., 2003)

2.3 Biocomposite

Biocomposites are biopolymers reinforced by natural fibers. Researchers have been developing these materials as alternatives to traditional materials, which may be non-renewable, rebellious, or manufactured using polluting emission processes. Although the industrial-scale production of biocomposites is increasingly feasible, the durability of these natural materials limits their application in many settings. This chapter defines biocomposite materials and provides examples of specific biocomposite materials and their material properties. The definition of biocomposites varies widely throughout the literature, some of which include composites composed of synthetic fibers or polymers (Mohanty et al., 2005a; Wool and Sun, 2005).

2.4.1 Properties of sugar palm composite

Sugar palm is a new type of natural fiber source with ecological and economic advantages. These low-density biodegradable materials can be used with polymer materials to produce partially biodegradable composite materials. The properties of sugar palm are in the range of natural fibers, which qualifies it as a reinforcing material for natural fiber composites. According to the available data, the diameter of the sugar palm fiber is 81 to 500 pm, the cellulose content is 37.3 to 66.5%, the hemicellulose content is 4.7-20.6% and the lignin content is 17.9 to 46.4% (Mukhtar et al., 2016).

2.4.1.1 Thermal properties of sugar palm fibre

The sugar palm fibre that has been bleached with 4ml of acetric acid and 8 g of sodium chloride each our for 7 hours (SPBF07) and alkali-treated sugar palm fibre (SPC07) possessed certain advantages such as a better thermal stability (Ilyas et al., 2017).

Sample	Water evaporation		1 st Thermal		2 nd Thermal				
				Degradation		I	Degradatio	on	
	TOnset	T_{Max}	W_L	T _{Onset}	T_{Max}	WL	Tonset	T_{Max}	W_L
	(°C)	(°C)	(%)	(°C)	(°C)	(%)	(°C)	(°C)	(%)
SPBF07	42.4	103.7	9.9	195.7	271.6	15.2	288.4	324.4	52.4
SPC07	43.5	101.2	8.6	207.9	345.1	73.7		-	-

Table 1 TGA finding

As the fiber is heated, the weight of the initial material will decrease due to the loss of combined water and volatile extracts, and the less volatile extracts tend to migrate to the surface of the fiber. The migration of volatile extracts is due to the evaporation of water on the surface of the fiber, and the water moves from the inside of the fiber to the surface of the fiber (due to the migration from the low-potential water on the fiber surface to the high-potential water). Therefore, the volatile extract is collected with water and remains on the surface of the fiber (Ishak et al., 2012).

The fiber loading was found to increase the thermal stability of the biocomposite. The first mass loss, in the range of 31 to 100 oC, is due to the evaporation of the water. Thus, the loss of mass of the SPF / SPS biocomposites between 150 oC and 380 oC is due to the decomposition of the three main components of natural fibers; hemicellulose, cellulose and lignin. Generally, the thermal decomposition of these fibers consists of four stages. The

first stage is the decomposition of the hemicellulose, then the cellulose, the lignin and finally its ash (Sahari et al., 2013).

2.4.2 Properties of polylactic acid (PLA) composite

Biocomposite with different ratio of PLA and thermoplastic starch (TPS) create different properties values . The high values of the TPS and low PLA content have a partial aggregation, suggesting insufficient interfacial adherence between PIA and TPS. This resulted in a greater peel of tensile strength. This confirms that the TPS content had an increase to PLA, and confirmed that tensile strength decreases and additional elongation to rest due to the breeze of TPS. Even so, as the addition of TPS increased, it seemed to improve of young's modulus (Nazrin et al., 2020).

2.4.2.1 Thermal properties of PLA

The glass transition (Tg) is known to be a complex phenomenon that depends on several factors, including intermolecular interactions, steric effects, chain flexibility, molecular weight, branch and crosslink density (Krishnamachari et al., 2009).

Due to the low cold crystallization temperature, additional exothermic crystallization peaks that have not completed PLA crystallization are displayed. This peak is due to the melting / recrystallization mechanism, which can be explained by the increase in the thickness of the crystalline lamella formed during cold crystallization (Frone et al., 2013).

With the addition of cellulose nano-fibers, the cold crystallization peak becomes broader and is shifted to lower temperatures as compared to the cold crystallization of neat PLA. The lower cold-crystallization temperature observed in the heating run can be an indication of faster crystallization induced by cellulose nanofillers which act as nucleating agents for PLA (Kang et al., 2008).

Sample	T _g (°C)	T _{cc} (°C)	T _c ^b (°C)	T _{mc} (°C)
PLA	56.9	87.3	151.5	169.1
PLA/CNF	56.3	85.2	151.5	168.0
PLA/CNFS	57.2	89.8	151.4	167.9

Table 2 DSC data correst	ponding to	the first	heating-coo	ling scan	(Frone et al	2013)

DSC thermograms obtained during the second heating scan for neat PLA and nanocomposites. The thermogram of the composite material only shows the melting event. The glass transition temperature is hardly observed, and low-temperature crystallization is not observed. This shows that the material is highly crystallized after cooling to 2 °C/min. Instead, it should be noted that the shoulder melting peak appeared just before the main melting peak (Frone et al., 2013).

	Air			
Sample	T _g (°C)	$T_{cc}(^{o}C)$	$T_{c}^{b}(^{o}C)$	T _{mc} (°C)
51		6.6	5 A	
PLA	61.1	87.3	S 151.5	169.1
		10 10		
PLA/CNF	VER 59.9 TEK	85.2 ALA	YSIA51.5LAK	(A 168.0
PLA/CNFS	60.3	89.8	151.4	167.9

 Table 3 DSC data corresponding to the second heating-cooling scan (Frone et al., 2013)

2.4.3 Thermal properties of different treatment of fibre

The combination treatment of alkali and silane has shown properties that are good for thermal properties. However, it is believed that the weight loss gained by hybrid composite materials after being subjected to a low temperature was due to the higher moisture absorption caused by the hemicelluloses. Based on a work reported, the lignocellulosic fibres were not good at bonding with polymers. (Puglia et al., 2013)

Hybrid designation	Initial degradation	Final degradation	Final residue (%)
	temperature, IDT °C	temperature, FDT	
		(°C)	
UTSP	156	435	28.48
TNSP	146	480	30.69
TSSP	132	481	29.35
TNSSP	138	507	32.02

 Table 4 TGA data for hybrid composite with fibre treatment(Atiqah, Jawaid, et al., 2019)

 2.4.4 Rheology properties of composite

2.4.4.1 Rheological properties of composite polymers and fibre

A collection of rheological parameters known as material functions can characterise the deformation behaviour or fluid dynamics of polymeric melts. These functions describe how the fluid reaction varies as a result of the flow conditions placed on the melt. (Advani et al., 1997)

According to other finding, fiber content, fibre length, fibre orientation, fiber-tomatrix bonding, fibre arrangement, and filler all have an impact on the rheological behaviour of hybrid nanocomposite polymers. The rheological behavior did not much affect when low constant shear stress (0.1%) is applied. The rheological characteristics of hybrid nanocomposites increased in direct proportion to the percentage of charge applied. (Hsissou et al., 2020) In other work on determine the rheological properties of composite, there are finding that show stable rheological properties with change less than 2% in time-sweep experiments over 15 min. (Bagheriasl et al., 2016)

2.4 Processing technologies for natural fibre reinforced thermoplastic (biocomposite)

The choice of the production methods depends directly on the materials and the desired properties of the final product. The subtopic below discusses the principle and implementation of conventional compression, extrusion, injection moulding and Long fiber thermosplastic-direct (LFT-D) method

2.5.1 Compressing method

Due to its high reproducibility and low cycle time, the pressure method is very popular in the manufacture of natural fiber composite materials. The two methods used are compression molding and flow compression molding. These processes differ in the type of semi-finished products used and their cutting. In the compression molding process, semifinished flat products or mixed wool that are larger than the shape or accurately cut to the required part size are usually used (Oksman et al., 2003).

The compression moulding method in biocomposite product fabrication offers a fastest forming process compared to solution casting technique. Compression moudling method is one of method that has low in cost compared to other process. On the other hand, compression molding technology is suitable for high-pressure manufacturing processes, and it is also suitable for the molding of complex biocomposites with natural fiber-reinforced polymers. (Shamsuri, 2015).

2.5.2 Extrusion

The extrusion process is used by the plastics industry for the continuous production of pellets and semi-finished products or parts. For this process, single-screw or twin-screw extruders can be used to work in joint or counter-rotation. When the mixing effect does not need to be very high, a single screw extruder is used. Due to the excellent mixing effect of the twin-screw extruder, the natural fiber material can be evenly distributed and wetted in the thermoplastic melt (Faruk et al., 2012).



Figure 6 Extrusion process (*What Is Plastic Extrusion - Plastic Tubing and Profile - PBS Plastics*, n.d.)

The common mixing process is carried out by a twin screw extruder , where the thermoplastic polymer is heated from the outside or mechanically sheared by the internal screw . This processing is limited to short fiber compositions. Most long continuous fibers are not used. However, in recent research, this has aroused great interest and started to be carried out on a laboratory scale. The heating temperature should be careful not to exceed a certain limit of (about 190-200 degrees), so as not to cause rapid decomposition of the fiber. This will result in failures and / or unsatisfactory compounding performance during the compounding process. Generally, thermoplastic biocomposites can be extruded

directly through a die (in this case, they must be installed in a machine) into the final product; alternatively, they can be extruded, granulated and packaged for reprocessing procedures (Dahy, 2019).

2.5.3 Injection moulding

Injection molding is a manufacturing process for the mass production of parts. Most typically, is used in mass production processes, where the same part is continuously manufactured thousands or even millions of times. Injection cuts a significant proportion of the original plastic block or sheet, resulting in a lower scrap rate compared to traditional manufacturing processes such as CNC machining. However, this has the potential to be negative compared to additive manufacturing processes such as 3D printing, but the scrap



Figure 7 Injection moulding process(Xie et al., 2011)

Complex geometric parts with functional elements can be produced quickly and in large batches by injection molding. Compared with compression molding, it has many advantages (economy of scale, minimum warpage and shrinkage, high functional integration, use of recycled materials) and almost does not need finishing (Faruk et al., 2012).

2.5.4 Long fibre thermoplastic - direct (LFT-D)

The LFT-D basic mechanism is to directly bond natural fiber mats and polymer melts to press tools using a combination of extrusion and press methods. With the help of an adjustable extruder, the molten polymer film is placed in the press and the natural fiber fleece is pressed together in layers in addition to the molten mass. Another possibility for processing natural fibers with the LFT-D method is to feed the fiber yarn or silver color directly into a two-screw extruder or injection molding machine. According to investigations, it has been shown that, depending on the optimal configuration of the screw, long-fiber structures can be maintained. However, handling of silver is quite problematic for industrial use (high throughput extrusion) (Faruk et al., 2012).



Figure 8 Long fibre thermoplastic - direct (LFT-D)

2.5 3D printing of natural fibre composite

2.6.1 Fused Deposition Modelling (FDM) process

The working principle of FDM technology is to use a metal wire or plastic filament unrolled from a spool and supply the material to an extrusion nozzle that can open and close the flow. During this process, the nozzle is heated to melt the material and can be moved horizontally and vertically by a numerical control mechanism, which is directly controlled by computer-aided manufacturing (CAM) software. After extruding from the die, the object is produced by extruding the molten material to form a layer. If necessary, the software accompanying the technology will automatically generate the support structure. The machine uses two materials, one for the model and the other to form a disposable support structure (*FDM 3D Printer Fused Deposition Modeling Technology for 3D Printing | Stratasys*, n.d.)

2.6.2 Composite filament

Composite filaments have been developed, which incorporate a range of different synthetic fibres. The addition of short glass fibre in an acrylamide–butane–styrene (ABS) matrix for use in FDM has shown an improvement (Zhong et al., 2001). It was found that adding short carbon fiber to ABS polymer can increase the tensile strength and Young's modulus by 115% and 700%, respectively (Tekinalp et al., 2014). 3D printing with continuous carbon fibre in a PLA matrix has also been conducted using a modified FDM printer, giving strengths of 185 and 57 MPa, respectively (Aruan Efendy & Pickering, 2014).

2.6.3 Properties of biocomposite filament

2.6.3.1 Natural fibre(Harakeke and Hemp) filament properties comapre to glass fibre

It was found that the strength of harakeke fiber in its untreated state was reduced by 25%, and more stringent treatment was required to cause fiber separation, which is believed to be due to the higher lignin content in its composition. However, its strength is 782 MPa, which is generally expected to provide significant reinforcement in the more common polymer matrix. It was found that treatment with NaOH and Na2SO3 can improve separation and remove surface components from harakeke and hemp fibers. Due to the removal of components, the fiber diameter, the maximum tensile load and the tensile strength of are reduced in most cases (Aruan Efendy & Pickering, 2014).

1				
Sample	Tensile 🔁	Net increase	Young's	Net increase
Storen TEL	strength (MPa)	(%)	modulus (MPa)	(%)
Plain pp	كل ميمي	کنیک	ىيونى ⁸⁹² يىنى ت	9
10 wt% Glass UNIVE	29 RSITI TEK	33 NIKAL MALA	" 1995 YSIA MELAK	124 (A
20 wt% Glass	32	39	2016	126
30 wt% Glass	36	62	3386	280
10 wt% Harakeke	28	25	1612	81
20 wt% Harakeke	36	62	2336	162
30 wt% Harakeke	39	74	2767	210
10 wt% Hemp	28	27	1683	89
20 wt% Hemp	34	55	2261	153
30 wt% Hemp	38	71	2681	201

Table 5 Mechanical properties of natural fibre compared to glass fibre reinforced 3dprinting filament (Stoof & Pickering, 2017)
Wood fiber %	T _g	Tc	T _m	
	(glass transition)	(crystallization)	(melting)	
0 (pure PLA)	62.1	102.5	167.6	
1	62.7	103.6	168.8	
3	62.1	103.5	168.3	
5	62.1	103.0	167.9	
7	62.6	104.3	168.3	
9	61.7	105.4	168.7	

2.6.3.2 Thermal stability of poplar fibre on the properties of printed biocomposite

Table 6 The thermal performance of 3D-printed biocomposite (Yang et al., 2021)



CHAPTER 3

METHODOLOGY

3.0 Introduction

In this chapter, the methodology of this research will be discussed, which explain the purpose of literature review and the objective. This chapter will specify the method in producing bio-composite material from Sugar palm fibre and PLA and the test used to analyze the properties of this bio-composite material.

3.1 Project flow chart

Flow chart below shows the flow of the research methodology. This research was carried out in proper step from start to end and many process were involve. Purpose of illustrating step using flow chart is to show and clarify the position of first objective and second objective so it can be achieved properly.

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Table 7 Project flow chart

3.2 Degradation of SPF size

First and for most, sugar palm fibre were purchase from Hafiz Adha Enterprise in form of long and continuous fibre. In this fabrication, sugar palm fibre (SPF), is needed in form of powder with maximum size of 125 µm. Process of clean, cut, crush and sieve was done to get the desired type of fibre. The SPF then went through chemical fibre modification to removes impurities to improve the surface of the fibres (Atiqah et al., 2019) .SPF and PLA will be mix and extrude by using twin screw extruder machine and crush again to get into a pallet form. The new bio-composite material then will be characterized to study the thermal and rheological properties using TGA, DSC and RC which is Thermogravimetric analysis (TGA) ,Differential scanning Calometry (DSC) and Rheological study (RC).



Figure 9 Strip form Sugar Palm Fibre (SPF)

The new bio-composite material is the mixture of PLA and Sugar palm fibre (SPF). SPF must be in powdered form or dust (125 μ m) so that it can mix with PLA and create a better bond. There are steps by steps to change from strip of fibre into powdered form fibre.

When the Sugar palm fibre (SPF) was extracted from the plant in the strip form, a lot of foreign and unwanted matter comes with it. Clean and wash process was done to remove these scraps. This process only use tap water and bear hand to remove the scraps. These need to be done so that it will not affect the properties of the final result (bio-composite). The washed and cleaned fibre then dried in the room temperature



Figure 10 Process of Cleaning SPF



Figure 11 Dried SPF

Next, the dried fibre need to be downgrade the size step by step until 125 μ m. Before using crusher to crush the fibre, the SPF was cut into smaller size which is 1 mm to 5 mm to avoid the SPF stuck in the blade of the crusher . The smaller size of fibre will smooth the crushing process and create more dust form (125 μ m).



Figure 12 Preparation of cutting SPF



Figure 13 Smaller size SPF (1mm to 5mm)

Furthermore, crusher is used to crush the smaller size fibre. Bit by bit of the fibre are being inserted into the crusher and not in huge amount to make sure it crushed smoothly. Since the crusher is easily overheat, the crushing process need to be stop for 5 minutes for each 10 minutes of usage . Crusher takes 1 hour to produce roughly 90 g of 125 μ m fibre dust. Atleast 75 g SPF powder required for test (TGA and DSC) in this research. Crushed SPF was packed in zip bag to avoid from water and ready to sieve.





Figure 15 Crushing Process

In crush process ,because of limitation of the crusher machine, the amount of SPF powder (125 μ m) produced is not the same as the amount of SPF inserted into the crusher. A few amount of bigger size of SPF still need to be separated and sieve to get 125 μ m SPF powder. Sieve shaker machine were used to sieve crushed SPF and the mesh size used is 125 μ m. This machine takes about 10 minutes to produce 20 g of 125 μ m of SPF. Sieved SPF that did not pass through the 125 μ m mesh sieve, was gathered and insert again the crusher to crush again.



Figure 16 Process of sieving



Figure 17 SPF powder (125 µm)

Sieve and crush process was repeated until required amount of SPF powder is fulfilled. SPF powder is packed and ready for treatment.

3.4 Fibre treatmentERSITI TEKNIKAL MALAYSIA MELAKA

Chemical treatment

Sodium Hydroxide (NoAH)

The alkaline treatment is a chemical treatment where SPF will be immersed in sodium hydroxide (NoAH)solution for a period of time. Purpose of this alkali treatment is to modifies the surface of fibres by removing the certain amount of lignin, hemicellulose, wax and oils that covers the external surface of natural fibres. During the alkaline treatment, the fibers are separated from one another, resulting in an increase in the effective surface area available for wetting by the resin. In addition, the alkaline treatment modifies the crystallinity, the unit cell structure, and fiber orientation

STEP	PROCESS	FIGURE
1	Preparation NoAH :	
	NaOH (6%) + H2O(94%) =	
	Volume of mixture (100%)	A THE AND A THE
	$=\frac{6}{100} \times 1000 g$	
	= 60 g of NaOH	00 ml — /AKI (14:2) //EKI (14:2
	ALAYSI.	- 600
	AL ALL ALL	- 400
	1) TEKIII,	
2	SPF powder was soaked into the	
	NaOH solution. All SPF was make sured to immersed	اونيور ميتي تيك
	completely and soaking time was	AL MALAYSIA MELAKA
	2 hours at room temperature	
3.	After 2 hours, SPF powder was	
	rinsed throughly with tap water	



 Table 8 Process of fibre treatment using NaOH solution

Silane

The SPF will go through Silane treatement .Silane treatment improves the adhesion and moisture resistance properties of SPF. The treatment of SPF in silane, preceded by mercerization, provides improved wettability, mechanical properties, and water resistance of SPF + PLA composites. This treatment improves wettability of the fibers because of the increase in density of the composite. The additional sites of mechanical interlocking as a result of treatment lead to improvement of interfacial bonding and promote resin/fiber interpenetration at the interface.

The high hydrophobic resin pick-up also accounts for the reduction in water absorption and hence improved mechanical properties under wet conditions. Although treatment of sisal fibers in silane preceded by mercerization produces very little change in the mechanical properties of dry composites, mechanical performance under wet conditions, and hence water resistance, can be improved. The treatment in 100% silane produces fibers that are almost hydrophobic. Water molecules at the interface tend to replace the resin– fiber covalent bond by weaker hydrogen bonds, hence silane plays an important role in reducing water absorption in cellulosic–fiber-reinforced composites.

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STEP	PROCESS	FIGURE		
1	Preparation of Silane : Silane (2%) + Methanol 90% + H2O (10%) = 500 ml			
	Silane Methanol H2O $\frac{2}{100}$ $\frac{90}{100}$ $\frac{10}{100}$ $\times 500 ml$ $\times 490 ml$ $\times 490 ml$			
	= 10 ml $= 441 ml$ $= 49 ml$	- 600 - 100		
	WALAYSIA MA			
2	The Silane solution then adjusted its PH to 3.5 using Acetic Acid and stir continuously for 10 minutes	TeM		
3.	SPF will be fully immersed into the solution for 3 hour.	ALAYSIA MELAKA		

4.	Rinsed SPF usng distilled water
5.	Dried the SPF in the oven at 60°C for 72 hours
6	The fibre with silane treatment is ready

 Table 9 Process of fibre treatment using Silane solution

3.5 Extrusion

Extrusion is the process to deposit inserted material in continuous and long thread form. This method is suitable to create filament. All different treatment of SPF sample with load of fibre loading which is 2.5 wt% will be insert into the twin-screw extruder and mix with PLA to deposit bicomposite (SPF/PLA) filament . Hot melt extrusion (HME) is the process of applying heat and pressure to melt a polymer and forcing it through an orifice in a continuous process.



Figure 18 Extrusion process (*What Is Plastic Extrusion - Plastic Tubing and Profile - PBS Plastics*, n.d.)

3.5.1 Process of extruding sample

STEP	PROCESS	FIGURE
NO		
1.	Turn on the machine and start configure the	
	machine to get suitable setting.	
2	After the temperature reached need value,	
	insert the fibre and PLA into the hopper	
	تيكنيكل مليسيا ملاك UNIVERSITI TEKNIKAL MALAY	SIA MELAKA
3	Twin screw inside the extruder will mix the SPF and the PLA which will create a	
	SIT and the TETT which will breake a	
	composite material. Then, the composite will	
	come out as filament through the machine.	



3.6 Characterization composite properties

SPF/PLA biocomposite filament will went through Thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC) and Rheological (RS) study to characterize SPF/PLA biocomposite material.

There are 4 different sample that will undergoes characterizing process which is untreated SPF and treated SPF with NAoH solution, silane solution and NAoH+silane solution that mix with PLA with 2.5% wt of each sample of fibre loading .

Thermal analysis for this sample was done at Kolej Kemahiran Tinggi Malaysia (KKTM) Masjid Tanah.

3.6.1 Thermogravimetric analysis (TGA)

In thermogravimetric analysis (TGA), the sample is weighed continuously while heating, because inert gas will pass through it. Many solids undergo reactions to produce gaseous by-products. In TGA, these gaseous by-products are removed and changes in the remaining mass of the sample are recorded. This is ideal for studying substances that are known to decompose in various ways at different temperatures and to better characterize how they decompose.

TGA (Universal v4.5A TA instrument) procedures :

STEP	PROCESS
1	Place an empty crucible in the sample platform's groove and tare. The crucible will be loaded and unloaded by the device
2	It's possible that the crucible is out of alignment if it won't load. A second effort will be made by the instrument to load it.
3	Please do not disrupt the instrument throughout this operation and avoid bumping the table
4	The pan will be returned to the sample platform after the tare is finished
5	Fill the pan with your sample after removing it from the platform. Please ensure that the sample fits into the pan comfortably
6	As polymers heat up, some of them expand. If this occurs and the substance is close enough to the thermocouple, the thermocouple will "glue" itself to the pan
7	If the programme is already open and the parameters are set, hit the run button, and the instrument will load and weigh the sample before starting the



Table 11 TGA procedure

3.6.2 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry analysis perform to measure the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic and exothermic processes. Differential scanning calorimetry (DSC) measures the energy transferred to or from a sample undergoing physical or chemical changes. Cellulose, hemicellulose, and lignin, which are components of lignocellulosic biomass, have been analyzed by DTA and DSC to determine their thermal response. WALAYS !!

DSC procedure:

C proced	
STEP	PROCESS
1	Tare the scale by placing an empty T-Zero pan on it.
2	Fill the pan with sample. It is necessary that the sample establish excellent contact with the pan's surface.
3	The weight of the sample $(15 - 20 \text{ mg})$
4	Place the top die on top of the pan, then the lower die on top of that.
5	Place the assembly in the press with the cover on the pan.
6	Bring the press lever down slowly.
7	Move the lever position up and back to its original position
8	Remove the pan and place in a position in the auto chamber
9	Repeat for each sample

Table 12DSC procedures

3.6.3 Rheological study

While, Rheological study is to perfom while the SPF/PLA biocomposite in melt phase or while it has been dissolved in a solvent for intrinsic viscosity and relative viscosity. To measure the rheological properties of a material, rheometers are used. Rheometers help those involved in industries such as sciences, geophysics, human biology, pharmaceuticals and food science measure how substances respond to particular forces or stressers.



Rheology analysis procedure:

STEP NO	PROCESS	FIGURE
1	The process start with switch on the main switch and rehometer machine switch.	
TEKU	AN MALAYSIA AREAN	
2.	Switch on the pc and open the	
U	ceast view software. NIKAL N	ALAYSIA MELAKA







 Table 13 Rheology analysis procedure

CHAPTER 4

RESULT AND DISCUSSION

4.0 Characterization composite properties

In this chapter, the result of thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC) and Rheological (RC)) study

4.1 Thermogravimetric analysis (TGA) result

TGA is used to measure the mass change, thermal decomposition and thermal stability of the materials in the temperature range over which the materials can be used to the point of noticeable degradation. The analysis of thermal stability for all samples of untreated SPF and treated SPF mix with PLA biocomposite had been analyzed using TGA. The TGA test was conducted with temperature ranging between 30°C to 800°C. The weight loss (TG) and its derivative (DTG) curves of all samples for different treatment had been shown in table .

Properties	Тетре			
Sample	Onset (°C)	Peak (°C)	FR,Wt%	
Untreated SPF	98.35	310.76	6.36	
	(92.6% wt)	(67.19% wt)	(790°C)	
SPF+NAoH	149	331.7	21.11	

4.1.1 Sugar palm fibre TGA data

	(91.875% wt)	(41.7% wt)	(720°C)
SPF+NAoH+Silane	105.45 (93.99%	315.02	26.75
	wt)	(73.54 % wt)	(790°C)

Table 14 Sugar palm fibre TGA data



Figure 20 Sugar Palm fibre TGA curve

Properties	Tempe		
Sample	Onset (°C)	Peak (°C)	FR,Wt%
Untreated	114.98 (99.35%	379.70	0.33
SPF+PLA	wt)	(1.37% wt)	(540 °C)
SPF+PLA+NAoH	36.49 (99.24%)	355.45	0
	wt)	(2.68% wt)	(789°C)
SPF+PLA+	36.42 (99.32%	375.24	0.38
NAoH+Silane	wt)	(1.75% wt)	(789.54°C)
SPF+PLA+Silane	38.81 (99.48% wt)	377.84 (1.79% wt)	0.28 (530.68°C)
1. Ide		- · ·	

4.1.2 Composite filament of sugar palm fibre and poly-lactic acid (PLA) TGA data.

Table 15 Composite filament of sugar palm fibre and poly-lactic acid (PLA) TGA data





Figure 21 Composite filament of sugar palm fibre and poly-lactic acid (PLA) TGA curve



Figure 22 TGA data combined graph of composite filament

TGA and derivative (DTG) curves of filament with untreated and treated fibre are shown in figure 21. Activation energy of thermal degradation for the composites was investigated with 2.5% fiber volume fraction for each composite sample .It can be observed that after sample that undergoes treatment with the silane solution, it reach 0% wt at lower temperature which indicate that, the thermal stability of the composite filament decreased slightly, while the alkali treatment improved the thermal stability of composite filament significantly by reaching 0 weight at higher temperature . It is seem the thermal stability at best when the same fibre undergoes those two treatment, it takes the highest temperature compare to other sample which is 789.54°C to decompose the filament, this is because it need more energy to completly decompose it. Decomposition of the filament with untreated SPF shows start at higher temperature, indicating the presence of different components that decompose at higher temperature. The delay on weight losses can be attributed to the evaporation of moisture on the surfaces of these foreign materials. The filament with treated fibers have shown a reduced weight loss earlier, indicating that alkali treatment removes a part of the absorbed molecules like fats and waxes .

4.2 DSC result

SPF/PLA composite's material properties such as glass transition temperature, melting, and crystallization will be determined.

4.2.1	Composition of	f sugar pa	lm fibre and	poly-lactic	acid (P	'LA) DSC	data.
				The second se	3 S (1 S S S S S S S S S S S S S S S S S	La la part e se part	

Properties Sample	Tg(°C)	T _c (°C)	T _m (°C)
Untreated	53.98	154.97	387.15
SPF+PLA			
SPF/PLA+NAoH	60.87	155.13	358.36

SPF/PLA+	58.87	154.25	378.93
NAoH+Silane			
SPF/PLA+Silane	54.89	153.98	383.38

Table 16	Composition	of sugar paln	n fibre and	poly-lactic acid	(PLA) DSC data.
----------	-------------	---------------	-------------	------------------	-----------------





Figure 23 Composition of sugar palm fibre and poly-lactic acid (PLA) DSC data

As shown in Figure and Table, DSC tests were used to characterize the thermal properties of sugar palm fibre mixed with PLA filaments with different type of treatment. The glass transition temperature (Tg) and melting temperature (Tm) of sugar palm fibre mixed with PLA filaments with different type of treatment are provided in Table. It can be seen from Table that the filament of untreated spf mix with PLA has the lowest glass transition temperature ,Tg. The filament with fibre that has been treated with NAoH has shown to have the highest Tg. The Tg of filament with fibre that has been treated with NAoH + silane show lower value of Tg compare with filament with fibre that has been treated with NAoH only. Filament with fibre that has been treated with silane only has the lowest Tg. Since silane is use to reduce moisture which can conlcude that silane can reduce the value of Tg of the filament which explain the reduction of Tg value of filament with fibre that has been treated with NAoH+silane. Different treatment between the four sample did not much affected the value crystallization temperature Tc. This indicates that, all these four sample seem to be in the crystallization phase nearly at the same temperature. Even so, the melting point temperature, Tm, of the four filaments sample show slightly different. From the table, when compare with all the samples, as expected from TGA result, it can be seen that the filament of untreated spf mix with PLA has the highest Tm and this is due to foreign matters or dust that melt at high temperature . In other hand, among the treated fibre, fibre with treatment of NAoH+silane has highest melting point. It is show that this filament has strongest intermolecular foreces which basically show it is stronger than the other filament samples. This was expected since the treatment was done for filament to improve their thermal properties.

4.3 Rheology analysis result

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The rheology testing is the study of how the stress in a material or force applied is related to deformation and flow of the material. The sample for this testing is the same as the thermal testing. The machine that use for this testing is rheometer (Instron SR20).

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 Table 18 Data of rheology analysis


🔳 100 PLA 📕 Untreated SPF/PLA 🖩 SPF/PLA + NAoH + Silane 📒 SPF/PLA+NAoH 📕 SPF/PLA+Silane

Figure 25 Viscosity data

Rheology properties for this four composite filament sample has the same pattern of viscosity graph pattern. Based on the sample result, it is shows wheather untreated or treatment of the fibre does effect much the viscosity of the samples. This can be say, when the graph is compare to pure PLA graph that show huge value of difference. The viscosity changes with each shear rate. When the shear rate increase, all four samples viscocity will decrease . This is called a "shear-thinning" fluid.

All pattern of graph show shear thinning fluid. Shear thinning is the non-Newtonian behavior of fluids whose viscosity decreases under shear strain. It is sometimes considered synonymous for pseudoplastic behaviour, where the composite fluid increase viscosity as force is applied and is usually defined as excluding time-dependent effects. Shear stress arises within the fluid because of the gradient of flow rates from this boundary to the centre of the rheometer chamber.

On the shear stress data, among the treated sample, treatment with both solution (NAoH + Silane) has the highest max shear rate volume which is 0.9312 kPa. This show that sample of SPF/PLA + NAoH + silane having highest shear modulus of elasticity that need more force to deform. Compare to pure PLA sample, treated fibre with NAoH + silane does increase its properties in term of strength. Eventhough the untreated sample show better result , it cannot be compare to treated sample because untreated fibre contain foreign matter that that make the result seem better.

4.4 Discussion

There are a few things that can be discussed through this journey of this study. During extrusion process, the first time extruding the filament, it become so brittle that it could easily break. It happen because the filament was submerged into the water right after filament being extrude. It makes the temperature drop drastically lead to less mobility that can cause brittle behavior.



Figure 26 Brittle filament

In rheological testing , the amount insert into the chamber should be observe carefully. It will affect the testing data. The sample will come out differently if the amount did not insert the right amount,



Figure 27 Differences between low amount sample (left) and proper amount sample (right)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

At the end of this study of "DEVELOPMENT OF CHEMICALLY TREATED SUGAR PALM FIBRE REINFORCED PLA COMPOSITE THROUGH FUSED DEPOSITION MODELLING 3D PRINTING", there are a few conclusion that can be made. The treatment of fibre surely show different in many aspect compared to untreated fibre. The samples show variant result is because of the different bonding condition between the PLA. Based on the finding of thermal analysis which is thermogravimetrix analysis (TGA) and Differential Scanning Calorimetry (DSC), it is seem the thermal stability at best when the same fibre undergoes those two treatment, it takes the highest temperature to degrade all the sample which is 789.54°C compare to other sample. Decomposition of the filament with untreated SPF shows start at higher temperature 114.98°C, indicating the presence of different components that decompose at higher temperature. In other hand, among the treated fibre, fibre with treatment of NAoH+silane has highest melting point 378.93°C. It is show that this filament has strongest intermolecular forces which basically show it is stronger than the other filament samples. This was expected since the treatment was done for filament to improve their thermal properties. Composite filament with untreated an treated fibre does give impact and effect on rheology behavior when compare to pure PLA. Fibre with both treatment show best rheological behavior with highest shear rate value (0.9312 kPa) and highest max viscosity (450 Pa*s). All of this properties finding can help in reducing of PLA which can reduce the cost . Instead of using 100% PLA in fused

deposition modeling 3d printing, using this composite material (NAoH + Silane) can produce the better properties in terms of termal stability and rheology behavior.

5.2 Recommendation

Further work that can be extended in this study is to study the composite other properties like mechanical properties to define its young modulus, in-plane shear modulus, major Poisson's ratio and ultimate tensile strength. Other aspect that can e bestudy on this composite is, changing the type of fiber, fiber length and types of thermoplastics. The fiber composition should be optimized and the maximum fiber composition should be determined in the chemical treatment, the optimum alkaline solution and silane solution should also be measured. This can improve the mechanical properties of the composite and more mechanical test should be carried out to determine the mechanical properties of the composite. The definition of composite density should be define to so that its ability can be explore. The specimen should be checked with the Scanning Electron Microscope(SEM) machine for detailed observation of the specimen.

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APPENDIX

APPENDIX 1 : Gantt chart PSM 1

APPENDIX 2 : Gantt chart PSM 2

	Alua				Weeks										
Project Activity	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
	11/10 -	18/10 -	25/10 -	1/11 -	8/11 -	15/11 -	22/11 -	29/11 -	6/12 -	13/12 -	20/12 -	27/12 -	3/1- 7/1	10/1-	17/1-
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Compile all chapter			2				×		-				5		
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TAJUK: Development of Chemically Treated Sugar Palm Fibre Reinforced Pla Composite Through Fused Deposition Modelling 3d Printing

SESI PENGAJIAN: 2021/22 Semester 1

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	Figure number (chapter. No of figure) before Figure Title, just separate the title					
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