

THE FUSED DEPOSITION MODELING OF POFA REINFORCED COMPOSITE
POLYMER

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A thesis submitted in
fulfillment of the requirement for the award of the
Degree of Master of Mechanical Engineering with Honours



Faculty of Mechanical and Manufacturing Engineering
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AUGUST 2021

For my beloved parents, my family and my best friends, thank you for everything especially for their support and everlasting love. To my supervisor and research mates, thank you for any help and cooperation during my toughest moment and being such an understanding person.



ACKNOWLEDGEMENT

In the name of Allah S.W.T., the most Gracious, the most Graceful. Alhamdulillah, with His permission and blessing, finally my project report has been accomplished perfectly in the time frame given.

Firstly, I would like to express my sincere appreciation to my kind supervisor, Ts. Dr. Omar Mohd Faizan bin Marwah for the support, guidance and advice given throughout the duration of this project. To my co-supervisor, Dr. Nasuha bin Sa'ude and Dr. Mohd Yussni bin Hashim, a big appreciation for them because give a lot of information and guidance for me to improve my research on making the filament composite success.

Next, from the deepest of my heart, I would like to give special thanks to my lovely mother Jamila binti Hj Mohd Taha, my father Darsani bin Kadir and all my family for the full support that they gave to me along the time to complete this project. Then, also a lovely thank for my special person Jalal Zaiki and my best friends (Nur Fatihah, Muhammad Amirulhakim, Nur Nadia, Nor Fariza) for being there whenever I want them to be. I am very grateful and happy to have them in my life I just want them to know that I love them so much.

Lastly, thanks to everyone that is involved directly or indirectly in the compilation of this project report and may all the deeds will be blessed by Allah S.W.T.

ABSTRACT

Palm oil fuel ash (POFA) is one of the waste products in the palm oil industry, whereas the abundance of that waste largely contributed to environmental effects. A strong interest in the development of renewable products to reduce the environmental effects has been focusing on the use of waste source as reinforcements in polymer matrix composites. In 3D Printing Fused Deposition Modeling (FDM), composite filament material has started to be used in the pattern development application. However, in FDM pattern development, the composite filament has a common issue especially in reference to the nozzle clogging problem that relates to the critical process in composition mixing and filament preparation. Therefore, this research aims to investigate the capability of different percentage composition in the range of 20vol% - 60vol% of POFA with polypropylene (PP) materials formed into solid composite filament for FDM machine. The composite materials were mixed homogeneously using the Brabender mixer machine and the addition of 5vol% and 8vol% maleic anhydride grafted polypropylene (MAPP) as a compatibilizer to improve the bonding ability of the PP matrix and POFA as well as to improve flowability during the next process. The Melt Flow Index (MFI) test was conducted on POFA+PP+MAPP compositions to investigate the data of viscosity which is important for the filament extrusion process. The single screw extrusion machine was used in the fabrication of composite filaments with a diameter and length range of $1.70 \pm 0.02\text{mm}$ and 60 to 70cm respectively. Then, the filaments were run for deposition test on the FDM machine by printing the ISO527-1 (dog bone sample). Finally, the properties in terms of mechanical and surface observation were investigated for the successful printed sample. The investigation results of the POFA filament deposition test on FDM shows that 40vol% POFA, 55vol% PP, and 5vol% MAPP gave the highest tensile strength which is 27.40MPa compared to other composition and virgin PP, 20% net increment of elongation at break compared to 20%vol POFA, lower warping defect and high initial degradation temperature with 318°C.

ABSTRAK

Abu bahan bakar minyak sawit (POFA) merupakan salah satu produk sisa dalam industri kelapa sawit, di mana lambakan sisa tersebut memberi kesan besar terhadap alam sekitar. Dalam Permodelan Pengendapan Lakur 3D (FDM), penggunaan filamen berunsurkan bahan komposit mula digunakan dalam aplikasi pembinaan produk. Walau bagaimanapun, dalam pembuatan produk menggunakan FDM, filamen komposit mempunyai masalah umum terutamanya mengenai masalah penyumbatan pada muncung pengeluaran, di mana faktor utama yang berkaitan dengan permasalahan itu adalah proses kritikal semasa pencampuran komposisi dan penyediaan filamen. Oleh itu, penyelidikan ini bertujuan untuk mengkaji keupayaan komposisi peratusan yang berbeza dalam lingkungan 20vol% - 60vol% POFA dengan bahan polipropilena (PP) telah dibentuk menjadi filamen komposit padat bagi digunakan di mesin FDM. Proses campuran bahan komposit itu diadun sehati dengan menggunakan mesin pengadun Brabender dan ditambahkan dengan 5vol% dan 8vol% maleic anhydride polypropylene (MAPP) sebagai alat penyesuaian untuk meningkatkan keupayaan ikatan matriks PP dan POFA serta memudahkan proses pengaliran semasa proses seterusnya. Ujian Melt Flow Index (MFI) dilakukan ke atas semua komposisi POFA+PP+MAPP untuk menyiasat data kelikatan yang penting untuk proses penyemperitan filamen. Mesin penyemperitan skru tunggal digunakan untuk membentuk filamen komposit dengan diameter dan panjang masing-masing iaitu 1.70 ± 0.02 mm dan 60 hingga 70cm. Kemudian, filamen dijalankan untuk ujian pemendapan pada mesin FDM dengan mencetak ISO527-1 (sampel tulang anjing). Akhirnya, sifat dari segi pemerhatian mekanikal dan permukaan disiasat untuk sampel bercetak yang berjaya. Hasil penyiasatan ujian pemendapan filamen POFA pada FDM menunjukkan bahawa 40vol% POFA, 55vol% PP, dan 5vol% MAPP memberikan kekuatan tegangan tertinggi iaitu 27.40MPa berbanding komposisi lain dan PP dara, kenaikan bersih pemanjangan 20% pada waktu rehat berbanding dengan POFA 20% vol, kecacatan melengkung yang lebih rendah dan suhu penurunan awal yang tinggi iaitu dengan 318°C.

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LIST OF ABBREVIATIONS

AM	-	Additive Manufacturing
FDM	-	Fused Deposition Modeling
MAPP	-	Maleic Anhydride Polypropylene
MFI	-	Melt Flow Index
POFA	-	Palm Oil Fly Ash
PP	-	Polypropylene
RP	-	Rapid Prototyping
SEM	-	Scanning Electron Microscope
TGA	-	Thermogravimetric Analysis



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CHAPTER 1

INTRODUCTION

This research is generally about the composite filament for Fused Deposition Modeling (FDM) application. The composite filament consists of the main matrix, filler and compatibilizer agent which acted as an additive material to improve the bonding between them. In this chapter, the introduction of this research was explained generally. Firstly, the explanation was given on the background of the research. Then, the issues and objectives of the research were critically highlighted in this chapter. Lastly, the scope of the study was defined to outline the research limitations and the expected results were discussed as predicted outcomes for this research.

1.1 Research Background

Natural fibre composites (NFCs) are alternatively referred as the bio-composites which gained much awareness due to their numerous advantages in a variety of applications and appliances (Khan *et al.*, 2012). Composite materials consist of two or more elements of matrix materials and reinforcement materials. In NFCs, they are usually made from a combination of plastics and fibres, whereas plastics as a matrix and natural fibres as the reinforcement. However, before the applications of NFCs are being commercialised, it is necessary to utilise the true advantage of material properties and gain a general understanding of the challenges involved in implementing them, particularly on the processability (Pandey *et al.*, 2015).

Malaysia is actively involved in the oil palm plantation industry which is also known as the world's largest palm oil producer and exporter due to millions hectares of land was dedicated in oil palm cultivation (Ng *et al.*, 2019). Moreover, a statistic reported by Malaysian Palm Oil Board (MPOB) in 2018 as shown in Table 1.1

proves that the total area of matured and immatured oil palm trees planted in Malaysia was 5849330 hectares (ha) in 2017. This country is capable to provide a million tonnes of palm oil and palm kernel oil. Palm oil is derived from the flesh of the fruit, while palm kernel oil is derived from the seeds or kernels (Sobuz *et al.*, 2019).

Table 1.1: Statistical area (ha) of Oil Palm Planted 2017 (MPOB, 2018)

STATE	MATURED	%	IMMATURED	%	TOTAL	%
JOHOR	680,562	91.0	67,000	9.0	747,562	12.8
KEDAH	82,287	91.1	8,007	8.9	90,294	1.5
KELANTAN	121,085	77.9	34,287	22.1	155,372	2.7
MELAKA	51,237	90.2	5,574	9.8	56,811	1.0
NEGERI SEMBILAN	167,026	89.1	20,425	10.9	187,451	3.2
PAHANG	653,535	86.4	102,614	13.6	756,149	12.9
PERAK	364,090	88.1	49,221	11.9	413,311	7.1
PERLIS	641	94.1	40	5.9	681	0.0
PULAU PINANG	14,042	95.5	660	4.5	14,702	0.3
SELANGOR	123,139	90.3	13,222	9.7	136,361	2.3
TERENGGANU	149,519	88.5	19,395	11.5	168,914	2.9
PENINSULAR MALAYSIA	2,407,163	88.3	320,445	11.7	2,727,608	46.6
SABAH	1,378,655	89.0	170,590	11.0	1,549,245	26.5
SARAWAK	1,403,526	89.3	168,951	10.7	1,572,477	26.9
SABAH & SARAWAK	2,782,181	89.1	339,541	10.9	3,121,722	53.4
MALAYSIA	5,189,344	88.7	659,986	11.3	5,849,330	100.0

However, from the production, a huge amount of wastes over 100 million tonnes of biomass were generated from the milling process in the forms of fibres, nutshells, and empty fruit bunches (Fahmi *et al.*, 2015). Scientific Malaysian Magazine (2017) also reported that without introducing modern methods of planting, harvesting, processing, and land management into mainstream operations, the palm oil mills in Malaysia contribute 80,000 metric tonnes of dry biomass annually and it keep rising in the coming years as the increasing of projected palm oil plantations and mills. Figure 1.1 shows the process involved in the palm oil industry and how the biomass is reused productively.

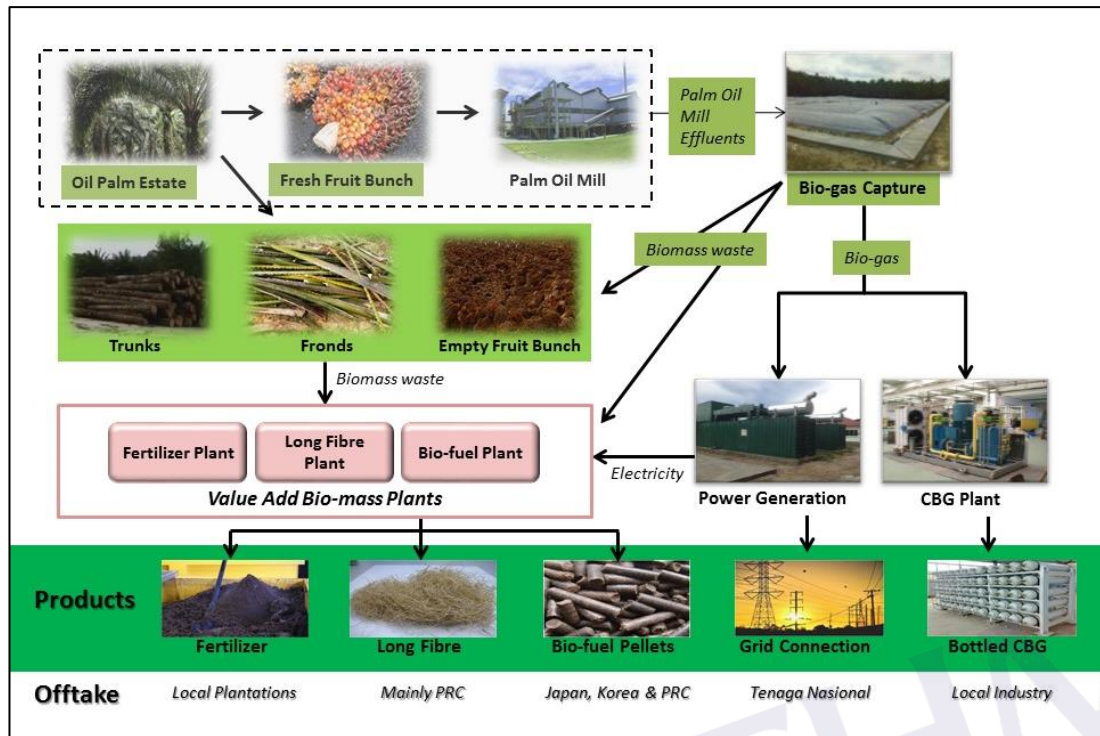


Figure 1.1: The process of oil palm industry (Scientific Malaysian Magazine 2017)

Palm oil fuel ash (POFA) is a source of silicate which the end products of oil palm bunches and kernels burning process at 800 to 1000°C that used as a fuel to generate electricity (Cheng *et al.*, 2019). The production process of POFA from palm oil processing is shown in Figure 1.2. Combustion in a steam boiler produces approximately 5% of POFA, whereas Malaysia is estimated to produce 3.14 million tonnes of POFA annually. It is also expected to grow annually in line with the increasing production of palm oil (Garba *et al.*, 2019). The major disposal problem occurs due to the presence of these oil palm waste that badly affecting the environment. These wastes are typically disposed of in landfills. Nevertheless, they may cause environmental and health risks because as the wind passes through the landfill, they could be harmful to breathe (Wei *et al.*, 2019). However, due to its major potential as supplementary cementing materials (SCMs) in the building industry, previous researchers attempted to use POFA as the secondary cementitious or pozzolanic materials to minimise the unhygienic disposal of POFA (Garba *et al.*, 2019).

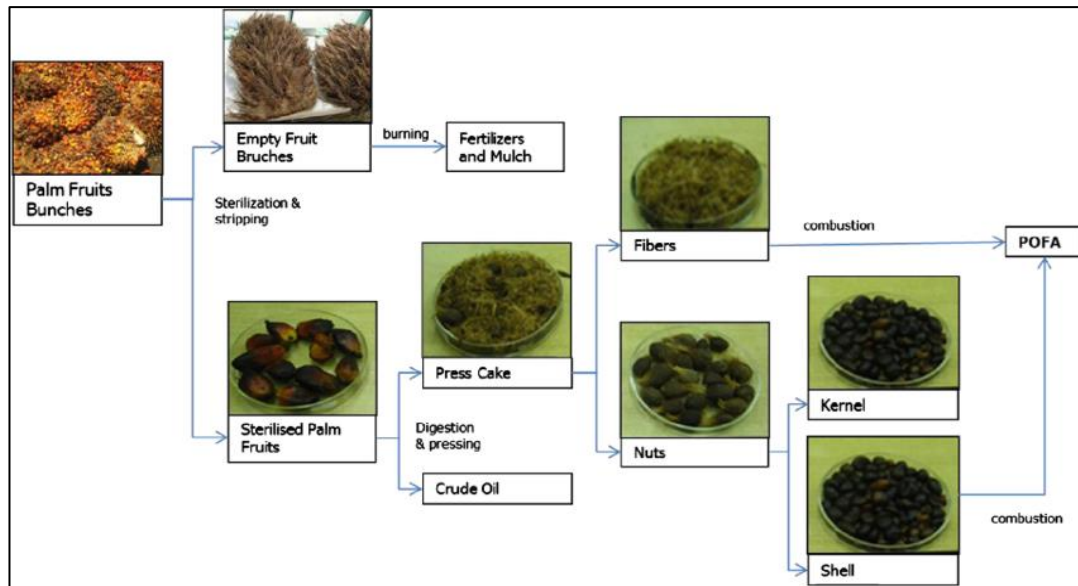


Figure 1.2: The schematic diagram for POFA production (Nagaratnam *et al.*, 2016)

Lately in Malaysia, biomass source has been announced as an important issue that gives profit for the country. According to environmental concerns, the natural source becomes one of the interesting and fascinating materials in the industrial application that should be explored by engineers and researchers (Reza *et al.*, 2013). Khalid *et al.* (2019) reported that based on consideration of the environmental issue and sustainable development in the modern production of polymeric materials, the modification of waste materials has also become an interesting research topic. This is a good opportunity for the industry to enhance the technologies from biomaterial as a new innovative product (Palm *et al.*, 2015). Therefore, the reuse of POFA from landfill sites in the composite material application is a successful way to minimise environmental and health impacts and at the same time to support the efficiency of using composite materials.

Currently, in FDM application many researchers addressed the potential of polypropylene (PP) as a candidate for filaments in FDM process due to their widely available and interesting characteristics for industrialists such as reasonable price, nontoxic polymer with good recyclability, lightness, resistance to chemicals and good electrical insulation (Carneiro *et al.*, 2015). In addition, the PP material was suitable to be used in some industrials such as food packaging, automotive, textile, construction, piping and medical facilities (Sodeifian *et al.*, 2019). However, due to the crystalline nature and tendency toward warping PP not always suitable or

remains quite rare in the 3D printing market (Spoerk *et al.*, 2018). Therefore, Silva *et al.* (2017) was evaluated the potential solution to prevent printed parts of PP material from shrinking and warping by incorporate fillers such as short glass fibres into the polymer. In addition, in other research also, to improve the mechanical and thermal performance of PP material as the requirements demanded by engineering applications, PP was usually modified with additives and particles as a reinforced material (Costantino *et al.*, 2019). Then, the fibre-reinforced thermoplastic matrix composites showed the commercial success in many application where various fibres are widely used as reinforcement polypropylene (PP) matrix to prepare the composites (Shubra *et al.*, 2011).

Pandey (2015) revealed that the mixture of natural fibre with various polymers as a polymer composite filament obtained from the extrusion process has shown the potential of 3D printing to develop new products. Milosevic *et al.* (2017) reported that both strength and stiffness increase with natural fiber content when harakeke and hemp fiber filled in PP as composite filament. Then, Hui *et al.* (2011) has been successfully produced the FDM filament by mixing 30% 100-mesh wood flour, 60% HDPE, 6% maleic anhydride polypropylene (MAPP), and 3% stearic lubricant. The mechanical properties of composite materials made from maleic anhydride-grafted polyhydroxyalkanoate (PHA-g-MA) and coupling agent treated palm fibre (TPF) showed an improvement in tensile strength and interfacial adhesion, hence successfully fabricated as FDM filaments (Wu *et al.*, 2017). Besides, the study by Lek (2016) showed the capability of 10% 0.1mm Oil Palm Frond (OPF) fibre mixed with HDPE to be used as composite filament in 3D printing. However, some improvements in the interaction between fibre and polymer are required due to poor interfacial derived from the hydrophobic nature of the fibre.

As stated in previous reports, there were numerous attempts on blending the biomass materials with a polymer to perform a composite material. However, there is a lack of research in making composite filament for FDM 3D printing using oil palm biomass.

The purpose of this study is to produce the composite filament with POFA filled by a single extrusion machine in PP polymer and deposited by FDM machines via layered production processes. The mixing ratio of the constituent material for POFA+PP+MAPP is investigated for optimal composition. Then, an effective compatibilizing agent material has been introduced to be used in the process of

mixing the composite filament as a medium to improve the melt flowability in wire filament form. Therefore, to achieve a homogeneous mixing, wire filament manufacturing must adopt the compounding technique, methods, mixing ratio and time to ensure a constant diameter of approximately $1.75\text{mm} \pm 0.2\text{mm}$ in standard filament sizing for this FDM unit.

1.2 Problem statement

Green environmental awareness has attracted researchers and industries to use biomass as an alternative primary filler for polymer bio-composites which can decrease the percentage of synthetic thermoplastics usage in the fabrication of products and help in reduced the environmental impact due to the overloading biomass waste in the land (Mazzanti *et al.*, 2019).

Malaysia has held the title as one of the most productive palm oil producers in the world. POFA is one of the abundant waste materials and it was estimated that 3 million tons per annum of fly ash were produced in (Razi *et al.*, 2016). Most of the abundance of fly ash especially in Malaysia is underutilized whereas this POFA have higher potential to be used in developing a polymer composite as a filler material (Elammaran *et al.*, 2020).

Lately, bio-polymer is one of the hot topics among researchers as a sustainable material in the industry based on its mission to achieve sustainable development goals. Among the emerging technologies of Industry 4.0, FDM has been acknowledged as the technology in additive manufacturing (AM) machines which commonly used thermoplastic materials as feedstock filaments. Most of the research proved the benefits and improvement of the product from the application of composite material due to several factor such the lightweight character, partially recyclable nature, biodegradability and high specific strength as their advantage in industrial applications (Kumre *et al.*, 2017). Therefore, the innovation material of composite filament for FDM becomes an interesting issue to be explored among the researchers.

However, there are three critical problems that should be addressed for composite material filaments, namely (1) mixing or compounding, (2) preparation of feedstock, and (3) manufacturing filament through extrusion machine (Sa'ude, 2016). First, the mixing and compounding phase is one of the most important parameters in

material compounding. The requirement of the melting temperature of each material for the main matrix, filler and binder, is the core issue in composite materials. The compounding of various materials may involve certain mixing methods, where when the temperature selection over the melting temperature and some of the material weight is decreased and vaporised, the potential of material degradation is high. Furthermore, the mixing and filament extrusion phase temperature must be lower than the degradation temperature of the binder (Sa'ude *et al.*, 2014).

Then, the volume ratio of various materials such as matrix material, a conductive filler, and binder would provide a continuous linkage and efficient concentration for conductive composite material in a continuous network material for feedstock preparation (Sancaktar *et al.*, 2011). For volume ratio material preparation, the best formula and calculation provided a good interconnection between matrix and filler material to maintain the conductive portion with good flexibility, stiffness, and viscosity in filament manufacturing.

Next, homogeneous mixing and bonding material are some critical factors in the manufacture of filament. It can influence the conductive portion with constant viscosity in the manufacturing filament. Besides, the constant distribution of fibre-filled polymer matrix was important in mixing process. The filament in wire form was difficult in the manufacturing process when the matrix material is filled with high fibre material and produced as a brittle composite material. Therefore, some of the binder was added in a matrix material to minimise the brittle characteristic of that material and to improve the flow during the fabrication of filament (Sa'ude *et al.*, 2016, Mostaffa *et al.*, 2011).

The POFA has the potential as an effective reinforcement in thermoplastics materials. It was found on previous reports that there were numerous attempts on blending the biomass materials with a polymer to perform a composite material. However, by comparing to previous research, less research was conducted on making composite material that specifically focuses on the composite filament for FDM using the POFA reinforced with a polymer.

1.3 Objective

The primary goal of this research was to investigate the natural composite filament material for FDM use. These research objectives are as follows:

- i. To investigate the best composition percentages of the POFA+PP+MAPP materials to be formed as a favourable result of filaments.
- ii. To investigate the feasibility of POFA+PP+MAPP composite filament deposition using FDM machine.
- iii. To evaluate the mechanical and observation defects of the deposited POFA+PP+MAPP samples.

1.4 Scope of the Study

These are the scope of the study:

- i. 20%, 30%, 40%, 50%, and 60% volume percentage of POFA was used in the mixture process of POFA+PP+MAPP composite material.
- ii. 5% and 8% volume percentage of MAPP was added in the mixture of POFA+PP+MAPP as compatibilizing agent to improve the bonding between fibre and polymer.
- iii. Material preparation and compounding by the Brabender plastograph mixer of POFA+PP+MAPP filaments with sufficient melting temperatures and mixing times.
- iv. The development of a new POFA+PP+MAPP filament wire (diameter: $1.75\text{mm} \pm 0.02$) with melting temperature at 170 to 190°C by the custom-made single extruder machine.
- v. The development of sample (ISO527-1) for POFA+PP+MAPP filament on the FDM machine.

1.5 Significant of Study

The purpose of this research is to provide contributions in the manufacturing of FDM filament material specifically for composite filament material in terms of the bio-waste product by oil palm, which is POFA reinforced with polymer-based. The composite filament fabrication process parameter was studied in this research by gaining enough information in fabricating a new wire filament form that can be applied uniformly in FDM deposition.

At the end of this research, the expected outcomes of this study is the development of bio-waste filament material using POFA reinforced with PP as a new alternative composite filament material in Additive Manufacturing (AM) technology. In other words, a small effort in reduced the oil palm waste is believed to create a significant benefit to the environment and contribute to a more sustainable green technology.



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CHAPTER 2

LITERATURE REVIEW

In this chapter, a literature review was briefly covered all the important things related to the study from the previous studies and journal papers. On the other hand, it was highlighted the issue of material selection such as the main matrix, filler, and additive material in the composite material area. Then, the process involved in composite filament production and their application in FDM technology was critically reviewed as a guideline in the methodology process. Therefore, the combination of information and data collected in this chapter shows the research gap as a reference for the research process in this study.

2.1 Palm Oil in Malaysia

The species *Elaeis guineensis* (oil palm tree) is mainly planted in oil palm plantations in Malaysia (Keshvadi *et al.*, 2011). Malaysia has reached 4.98 million hectares of land devoted to the production of palm oil, recognised as one of the world's leading palm oil producers and exporters. The United States Department of Agriculture reported that by 2017, the global palm oil output was about 64.5 million tonnes and about 80% of the world palm oil production is from Malaysia and Indonesia (Garba *et al.*, 2019).

2.2 Oil Palm Issues

By successfully integrating the beneficiaries of the palm oil industry, Malaysia is one of the countries that have a responsibility for the global transformation of the oil palm industry through substantial contributions and persistent dedication (Awalludin

et al., 2015). Undoubtedly, palm oil has become Malaysia's main agricultural crop and one of the biggest contributors to the country's economic growth (Jamilah *et al.*, 2019). While the industry is considered as financially secure and gives a considerable income to the economy, it also contributes a huge amount of palm oil biomass waste at the same time (Awalludin *et al.*, 2015).

It has been estimated that approximately 7 tonnes of fibre, 25 tonnes of empty fruit bunches, and 20 tonnes of palm kernel shells are produced for every 100 tonnes of raw fruit bunches harvested (Iskandar *et al.*, 2018). Besides, Ng *et al.* (2019) reported that the Malaysian Palm Oil Board (MPOB) has announced that Malaysia is capable of producing millions tonnes of palm oil per year, which has added a substantial amount of waste due to this massive production. In this industry, solid biomass waste is produced in two ways, which are from oil palm plantations and palm oil extraction plants.

As seen in Figure 2.1, waste in the form of harvested trunks (OPT) is available during replanting phase, while pruned fronds (OPF) are available during the fruit harvesting process. Meanwhile, in Figure 2.2, waste from palm oil extraction mills is derived from empty fruit bunches (EFB), palm kernel shell (PKS), and mesocarp fibre (MF). The EFB is produced after oil extraction, where the fresh fruit bunches are pressurised, cooked, and extracted from the bunch. The kernel shells are collected after the process of removing the nut from its kernel, while the mesocarp fibre is produced during the palm oil extraction process. However, the existence of such oil palm waste poses a significant waste disposal problem (Abdullah and Sulaiman, 2013).



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