

**DEVELOPMENT AND CHARACTERIZATION
OF NANO OIL PALM ASH AS FILLER
IN PHENOLIC RESIN FOR BONDING
OIL PALM VENEER AND HYBRID PLYWOODS**

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UNIVERSITI SAINS MALAYSIA

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IN PHENOLIC RESIN FOR BONDING
OIL PALM VENEER AND HYBRID PLYWOODS**

by

CHE KU ABDULLAH BIN CHE KU ALAM

**Thesis submitted in fulfilment of the requirements
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الرَّحِيمِ الرَّحْمَنِ اللَّهُ بِسْمِ

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BS	British Standard
BSI	British Standard Institute
ISO	International Organization for Standardization
NIOSH	National Institute of Occupational Safety and Health
SCCP	Scientific Committee on Consumer Products
MPOB	Malaysia Palm Oil Board
OPA	Oil Palm Ash
POFA	Palm Oil Fuel Ash
OPT	Oil Palm Trunk
EFB	Empty Fruit Bunch
OPF	Oil Palm Frond
PKS	Palm Kernel Shell
OPKS	Oil Palm Kernel Shell
MF	Mesocarp Fiber
MDF	Medium Density Fibreboard
LVL	Laminated Veneer Lumber
PSL	Parallel Strand Lumber
OSB	Oriented Strand Board
POME	Palm Oil Mill Effluent
PF	Phenol Formaldehyde
FOB	Free On Board / Freight On Board
RPM	Rotation Per minute

MOR	Modulus of Rupture
MOE	Modulus of Elasticity
W/B	Weight -to-Binder
S/N	Signal-to-Noise
L9	Level 9
DOF	Degree of Freedom
DOE	Design of Experiment
D-Value	Diameter of Sphere
ANOVA	Analysis of Variance
SS	Sum of Square
MS	Means of Square
MOF	Metal Organic Framework
HEBM	High Energy Ball Milling
FT-IR	Fourier Transform Infrared Spectroscopy
KBr	Potassium Bromide
TGA	Thermogravimetric Analyzer
DTG	Derivative Thermogravimetry
IDT	Initial Degradation Temperature
FDT	Final Temperature Degradation
FESEM	Field Emission Scanning Electron Microscope
TEM	Transmission Electron Microscope
XRD	X-ray Diffraction
PWV	Plywood Veneer
PWH	Plywood Hybrid
PWC	Plywood Commercial

LIST OF SYMBOLS

%	Percentage
g	Gram
g/cm ³	Density (Gram centimetre cubic)
Kg/m ³	Density (Kilogram meter cubic)
g/m ²	Gram Meter Square
MPa	Megapascal
GPa	Gigapascal
°C	Degree Celsius
m ³	Meter cubic
m ²	Meter square
mm	Milimeter
µm	Micrometer
cm	Centimeter
nm	Nanometer
mg	Miligram
Å	Atom / Molecule
cp	Viscosity
Wt.	Weight
psi	Pound force per square inch
Δ	Delta
cm ⁻¹	Wavelength per unit distance in centimeters
a.u	Arbitrary Unit
J	Joule

J/m	Joule Per meter
m	Mass
V	Volt
X	Magnification
θ	Theta
λ	Lambda

LIST OF APPENDICES

APPENDIX A SIGNAL TO NOISE (S/N) AVERAGE CALCULATION

**PEMBANGUNAN DAN PENCIRIAN NANO ABU KELAPA SAWIT
SEBAGAI PENGISI DALAM RESIN FENOLIK UNTUK PEREKATAN
PAPAN LAPIS VENIR KELAPA SAWIT DAN HIBRID**

ABSTRAK

Fokus dan minat dalam penggunaan abu kelapa sawit (OPA) adalah tinggi, terutamanya disebabkan oleh sumber yang boleh diperbaharui, peluang untuk meningkatkan sifat dan kemungkinan untuk digunakan dalam pelbagai aplikasi. Tesis ini membincangkan pengoptimuman proses menghasilkan nanopartikel OPA dan untuk mengkaji prestasi nanopartikel OPA dalam komposit papan lapis. Analisis statistik berdasarkan kaedah Taguchi telah digunakan untuk mencari parameter optimum untuk penyediaan nanopartikel OPA dengan menggunakan teknik pengisaran bola. Tatasusunan ortogonal L_9 dan nisbah 'signal-to-noise' (S/N) digunakan untuk mengkaji ciri-ciri prestasi parameter yang dipilih iaitu masa pengisaran (jam), kelajuan pengisaran (rpm), dan saiz bebola besi tahan karat (mm). Taburan saiz nanopartikel OPA telah diperiksa oleh '*Particle Size Analyzer*' dan analisis TEM. Dalam kajian ini, analisis statistik dan ujian pengesahan menunjukkan bahawa saiz bola (20 mm) adalah parameter yang paling berpengaruh untuk menghasilkan nanopartikel OPA diikuti dengan masa pengisaran (24 jam) dan kelajuan pengisaran (90 rpm). Bagi meneroka potensi nanopartikel OPA dalam aplikasi resin, resin fenol formaldehid (PF) diisi dengan muatan nanopartikel OPA yang berbeza. Pengkajian kualitatif nanokomposit daripada resin PF diisi nanopartikel OPA diselidiki oleh FT-IR, TGA, XRD dan FESEM. Ia menunjukkan bahawa nanopartikel OPA menambahbaik kestabilan haba dan kristalografi nanokomposit

resin PF. Taburan nanopartikel OPA juga menunjukkan perbezaan dari segi kadar muatan disebabkan oleh aglomerasi nanopartikel OPA dalam nanokomposit. Penyiataan mengenai prestasi komposisi papan lapis yang digunakan dengan resin PF diisi dengan pemuatan berbeza nanopartikel OPA dianggap sangat penting kerana sifat venir yang lemah. Keputusan yang diperolehi dalam kajian ini menunjukkan bahawa kehadiran nanopartikel OPA sangat ketara dalam mempengaruhi sifat fizikal, mekanik dan termal panel papan lapis. Penambahbaikan yang ketara dalam kestabilan dimensi dari penyerapan air dan perubahan ketebalan diperolehi untuk panel papan lapis dengan pemuatan nanopartikel OPA tertinggi dalam resin PF. Sifat mekanikal menunjukkan bahawa komposit papan lapis menunjukkan peningkatan dalam sifat lenturan, ricih dan kekuatan impak dengan kadar pemuatan partikel nano abu kelapa sawit (OPA) yang optimum. Morfologi permukaan patah juga menunjukkan keberkesanan nanopartikel OPA dalam pengurangan keretakan lapisan akibat daya dan pengagihan tekanan. Prestasi kestabilan haba menunjukkan bahawa PF diisi nanopartikel OPA menyumbang kepada kestabilan haba dalam panel papan lapis. Oleh itu, hasil yang diperolehi dalam kajian ini menunjukkan bahawa nanopartikel OPA pastinya akan meningkatkan ciri-ciri kayu lapis venir dan papan lapis hybrid.

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ABSTRACT

The focus and interest in the utilization of oil palm ash (OPA) is high, mainly due to their renewable material, opportunity to enhance the properties and possibility to use in a wide range of applications. This thesis discusses the optimization of OPA nanoparticles preparation and to study the performance of OPA nanoparticles in the plywood composite. A statistical analysis based on the Taguchi method has been used to find the optimal parameters for the preparation of OPA nanoparticles by using ball milling technique. L₉ orthogonal array and signal-to-noise (S/N) ratio are applied to study performance characteristics of the selected parameters which are milling time (hours), milling speed (rpm), and size of stainless-steel balls. The size distribution of the OPA nanoparticles was examined by the particle size analyzer and TEM analysis. In this study, a statistical analysis and confirmation test showed that the ball's size (20 mm) was the most influential parameter to produce OPA nanoparticles followed by milling time (24 hours) and milling speed (90 rpm). In order to explore the potential of the OPA nanoparticles in resin application, the phenol formaldehyde (PF) resin filled different loading of OPA nanoparticles were prepared. The qualitative characterizations of PF resin nanocomposite filled OPA nanoparticles were investigated by FT-IR, TGA, XRD and FESEM. It was indicated that OPA nanoparticles remarkably improved the thermal stability and crystallinity of PF resin nanocomposites. The distribution of OPA nanoparticles also showed differences in

loading rates due to the agglomeration of OPA nanoparticles in nanocomposites. Investigation on the performance of plywood composite applied with PF resin filled different loading of OPA nanoparticles is considered very important due to the poor properties of OPT veneer. The results obtained in this study showed that the presence of OPA nanoparticle significantly affected the physical, mechanical and thermal properties of the plywood panels. Significant improvements in dimensional stability from water absorption and thickness swelling experiments were obtained for the plywood panels with the highest OPA nanoparticles loading in PF resin. The mechanical properties indicated that plywood composites showed improvement in flexural, shear and impact properties until certain loading of OPA nanoparticles in PF resin. Fracture surface morphology also showed the effectiveness of OPA nanoparticles in reduction of layer breakage due to force and stress distribution. The thermal stability performance showed that PF filled OPA nanoparticles contribute to the thermal stability of the plywood panels. Therefore, the results obtained in this study showed that OPA nanoparticles certainly improve the characteristic of the plywood veneer and plywood hybrid respectively.

CHAPTER 1

INTRODUCTION

1.1 Introduction and Background

Over the last few decades, Malaysia was the world's largest producer of palm oil where the oil palm planted area amounted to 5.90 million hectares but was overtaken by Indonesia in the early years of the 21st century. As the world's second largest producer and exporter of palm oil, it was projected that its production would increase to 72 million tons by 2019 (Al-mulali et al., 2015, Jackson et al., 2019, Senawi et al., 2019). However, in any industry in the world, no exception for the palm oil industry, there are secondary products that are produced alongside the main product. During oil palm fruit harvesting or replanting of oil palm tree, approximately million tons dry weight of oil palm frond (OPF) and oil palm trunk (OPT) was produced annually. On the replanting season, as much as 15 tons and 75 tons per hectare of dried OPF and OPT, respectively, were chipped and left to rot at the plantation as mulch. In a yearly basis, OPF and OPT can amount up to 4 and 18 million tons (dry weight) respectively. It is estimated that the total potential oil palm biomass from 4.69 million hectares of oil palm planted area in Malaysia is 77.24 million tons per year and these figures are based on the estimation of 5% oil palm plantation that due for replantation (Abdul Khalil et al., 2013b, Awalludin et al., 2015, Chew et al., 2020). Even though several types of research are investigating the ways of utilizing these wastes, there is still a large fraction of the residues abundantly unattended (Idris et al., 2010).

Extensive research on the conversion of OPT, EFB, and fronds into value-added products such as particleboard, medium density fiberboard (MDF), cement-bonded particleboard, fiber-reinforced plastics, and plywood has been initiated with

great commercial potentials (Bhat et al., 2011). Plywood panels are among wood composite materials that are made from wood veneers as renewable bio-resources. Plywood has been successfully produced from oil palm trunk by using hardwood as the back and face of the veneers. The laminated veneer lumber has been manufactured from oil palm trunk using dried veneer by the conventional plywood manufacturing process, except that the arrangement of the layers is parallel to the grains as stated by Abdul Khalil et al. (2010a).

Meanwhile, the palm oil mill industries are no exception. Palm oil mills produce biomass alongside palm oil, and they are a shell, empty fruit bunch, fiber and palm kernel. This biomass traditionally used as solid fuels for steam boilers. The steam generated is used to run turbines for electricity production. However, the burning of these solid fuels creates another environmental problem in which the emissions of dark smoke and the carryover of partially carbonized fibrous particulates is due to incomplete combustion of the fuels. The carbonized fibrous particulates are known as oil palm ash (OPA). Approximately, 5 % of OPA by weight of waste materials is produced after fuel combustion (Tay and Show, 1995a, Aprianti et al., 2015, Yusoff, 2006).

Various research work has been done to utilize OPA such as a cement replacement material (Tangchirapat et al., 2007), as a natural low-cost adsorbent (Chu and Hashim, 2002), flue gas desulphurization (Zainudin et al., 2005), transesterification of waste cooking oil into biodiesel (Boey et al., 2011, Chin et al., 2009) and recently as a filler material in a polymer (Abdul Khalil et al., 2013b, Ooi et al., 2014). However, most of the ash is still disposed of in a landfill that requires a lot of land area which contributed to the rapid increase in the cost of ash disposal services (either in landfill or transportation) and could lead to environmental problems for the industry and the public (Ooi et al., 2014, Hawa et al., 2013). Hence, creating,

manipulating and exploring OPA in nano-composite technology will create an advanced bio-agricultural waste material.

The utilization of OPA in advance or conventional composites to reduce the cost, appearance and properties of final products, has gained the attention of many researchers. Therefore, the utilization of OPA becomes an essential and vital topic to be further investigated (Zainudin et al., 2005, Chu and Hashim, 2002). In order to obtain high-quality OPA with fine particle size, homogenous size distribution and special morphology, various preparation techniques have been used to synthesize ultrafine OPA including ball milling and grinding technique (Abdul Khalil et al., 2012c, Abdul Khalil et al., 2011c).

In the past decade, nanocomposite materials have become a rapidly growing field with potential applications ranging from the advanced application to a daily used product. Nanocomposites are a type of composite in which the scale of the dispersed phase is less than 100nm in at least one dimension. Due to the nanoscale dispersion and the high aspect ratios of filler, nanocomposite exhibit lightweight, dimensional stability, heat resistance, high stiffness, barrier properties, and improved toughness and strength with far less reinforcement loading than conventional composite counterparts (Ku et al., 2012b, Zhou et al., 2008).

Phenolic resins were the first thermoset resins to be synthesized commercially in 1907. These resins are not only low-cost and easy to produce but also exhibit excellent fire performance, good dimensional stability, superior thermal insulation properties, good chemical and corrosion resistance. These features enable phenolics to be used in myriad applications, such as household appliances, business equipment, wiring devices, and electrical systems (Zhou et al., 2008, Eesaee and Shojaei, 2014).

For further improvement of the properties and performance of PF resin, nanomaterial-reinforced composite has been intensively investigated in recent years (Candan and Akbulut, 2014a, Cai et al., 2008, Natali et al., 2011). It was concluded that the addition of small percentages of nanofiller increase the performance of nanocomposite such as permeability resistance, improved thermal stability, enhanced mechanical properties and it also decreased the formaldehyde emission values (Zhang et al., 2011b, Lei et al., 2008a, Zhang and Smith, 2010, Srikanth et al., 2013, Candan and Akbulut, 2013a).

Surprisingly, reports by other researchers in the utilization of OPA nanoparticles as reinforcement material in the polymer matrix are still low due to difficulty to prepare OPA nanoparticles. Moreover, the usage of other nanoparticles such as carbon-based nanoparticles, metal nanoparticles, ceramic nanoparticles and polymeric nanoparticles to produce nanocomposite causing the usage of the OPA to not be fully utilized. Besides, the usages of the nanoparticle particularly focus on manufacturing nanocomposite which is more on high-end performance products. However, findings by Candan and Akbulut (2014b) in reporting the usage of nanomaterials in the wood-based product show that the implementation of nanomaterial in plywood enhances the durability performance and significantly improves the dimensional stability. Therefore, this research is aimed to explore the potential utilization of OPA incorporated into phenolics resin, so that the concerns about the application problems of these carbonized materials can be solved. At the same time, by using the nano-engineering aspect, preparation of plywood composites applied with PF resin filled OPA nanoparticles at proper loading level is expected to produce a plywood composite having improved properties and substantial performance.

1.2 Problem Statement

The generation of oil palm ash (OPA)/boiler ash (BA) was estimated to be over 4 million tons/year in Malaysia. The generated secondary waste could lead to health-related issues, environmental problems and also financial loss (Boey et al., 2011, Khankhaje et al., 2016a, UI Islam et al., 2016). Although there are some studies on the utilization of OPA such as a partial replacement of cement in concrete (Sehaqui, 2011), as an adsorbent for the removal of heavy metal from aqueous solution (Chu and Hashim, 2002), flue gas desulphurization (Fu et al., 2018) and recently as a nano-structured material in a polymer (Abdul Khalil et al., 2013b), most of the ash is still disposed of in a landfill that requires a lot of land area.

The interest in the utilization of oil palm ash is high, mainly due to their renewable material, opportunity to enhance the properties and possibility to use in a wide range of applications. Therefore, the utilization of OPA becomes an essential topic to be further investigated. There have been several investigations to simulate the dynamics of the ball milling technique process as well as analyze the parameters to produce nano-structured materials and to create the process more efficient. Furthermore, to transform the OPA particle into nano-scale particles by ball milling technique, there is no conclusive method on the best parameter have been developed. Thus, the design optimization of the ball milling process has to be developed to study the parameter combination and the most influential parameter to prepare OPA nanoparticles.

The phenol formaldehyde resin is well known as the oldest synthesized and one of the lowest cost adhesives for structural purposes. For their excellent properties such as easy to apply, heat resistance, flame retardancy, good electrical insulation and so on, the PF resin has been widely used in many industrial fields. Some researchers introduced organic and inorganic materials into phenolic resin to improve their

thermal stability which could prevent the propagation of the combustion. Therefore, to enhance the PF properties, incorporating OPA nanoparticles into the phenolic resin is one of the investigations in this study. The presence of the OPA nanoparticles is expected to enhance the physical and the thermal properties of PF resin due to interfacial interaction by the active groups of OPA nanoparticle and PF resin.

Oil palm trunk (OPT) and empty fruit bunch (EFB) are the renewable biomass derived from oil palm plantation. Over the years, numerous research and development efforts have been carried out to optimize the utilization of OPT and EFB. Despite all the extensive research work mostly on a wood-based product, pulp and paper making, high quality organic fertilizer and so on, the development of high-quality value-added products still low and in small-scale production. Based on the literature study, extensive research on the utilization of hybrid plywood and plywood veneer showed improvement in properties. However, due to the properties of the PF resin and OPT veneer, some other issues or problems related to physical, mechanical and thermal properties compared to commercial plywood composite still could not be solved. Thus, with the great potential of properties enhancement in PF resin filled OPA nanoparticles, it could be assumed that the PF resin matrix characteristic improvement simultaneously helps to enhance the performance of hybrid plywood and plywood veneer respectively. In line with the need to minimize the biomass waste, maximize the biomass utilization and develop a wood-based sustainable platform, this study will take the initiative to prepare and explore OPA nanoparticles as promising material, corporate with PF resin to develop high quality plywood composite in term of durability and reliability performance.

1.3 Objectives of the Study

The objectives of this present research work are:

1. To optimize the parameters of nanoparticle production from oil palm ash (OPA) using a ball milling technique.
2. To study the effect of OPA nanoparticles loading (1-5%) into phenol formaldehyde resin on the physical, thermal and morphological properties.
3. To study the effect of phenol formaldehyde (PF) resin filled OPA nanofiller on the physical, mechanical, thermal properties and fracture morphology of hybrid plywood and plywood veneer.

1.4 Organization of Thesis

- Chapter 1 - Introduction focused on introducing and background, major challenges/gaps, the scope of study and objectives of the study.
- Chapter 2 - Focused on the literature review of various aspects of natural fibers, matrix, EFB and OPT veneer and its composites. It also covered detail scientific information about hybrid composites.
- Chapter 3 - Explains about materials and methodology of development and characterization of OPA nanoparticles, PF nanocomposites and plywood panels.
- Chapter 4 - Deals with results and discussion of mechanical, physical, thermal properties and morphology of OPA nanoparticles, PF nanocomposites and plywood panels.
- Chapter 5 - Summarizes the overall conclusions and recommendations for future research proposals of this study.

CHAPTER 2

LITERATURE REVIEW

2.1 Oil Palm in Malaysia

Over the last few decades, the Malaysian palm oil industry has grown to become a very important agriculture-based industry, where total exports of palm oil and palm oil products in 2019 reached 27.9 million tons contributing the RM64.8 billion total revenue to the country (Tan and Lim, 2019, MPOB, 2019). The percentage of oil palm production by country is shown in Figure 2.1. Today, Malaysia is the second world's largest producer and exporter of palm oil. Over the years, Malaysian palm oil production increased from about 4 million tons in 1985 to 6 million tons in 1990, 10.85 million tons in 2000 and to 19.8 million tons in 2019. On the other hand, oil palm production in Malaysia is expected to grow with the increasing global demand for edible oil, biodiesel and oleo-chemicals derived from palm oil (Kong et al., 2014, Awalludin et al., 2015, Tan and Lim, 2019).

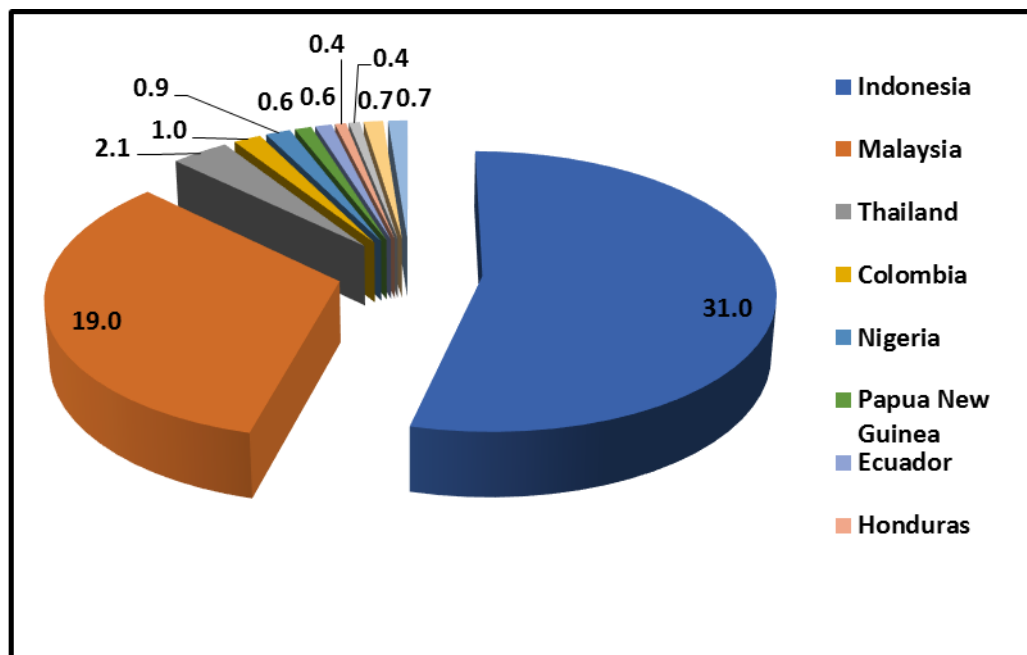


Figure 2.1: Percentage of oil palm production by country (Murphy, 2014).

2.2 Oil Palm Biomass

Biomass is defined as organic matter available on a renewable basis, and these include forest and mill residues, wood wastes, agricultural waste, domestic garbage, as well as livestock wastes. In the palm oil industry, the oil extraction rate is only about 10% from palm oil production with the majority 90% left as biomass (Abdullah and Sulaiman, 2013). At a conservative estimate, for each kg of palm oil extracted, there is approximately another 4 kg of dry biomass generated such as empty fruit bunch (EFB), palm kernel shell (PKS) and mesocarp fiber which derived from fresh fruit bunch (FFB). While oil palm trunk (OPT) and oil palm frond (OPF) is generated after the oil palm fruits harvesting or during oil palm tree replantation inside the plantation estates (Umar et al., 2013, Awalludin et al., 2015). The oil palm biomass generated per annum from the palm oil industry and oil palm plantation is shown in Figure 2.2, while Figure 2.3 illustrates a simplified flow of oil palm biomass derived from oil palm plantation. These biomasses have a high potential of turning into value-added products and renewable energy.

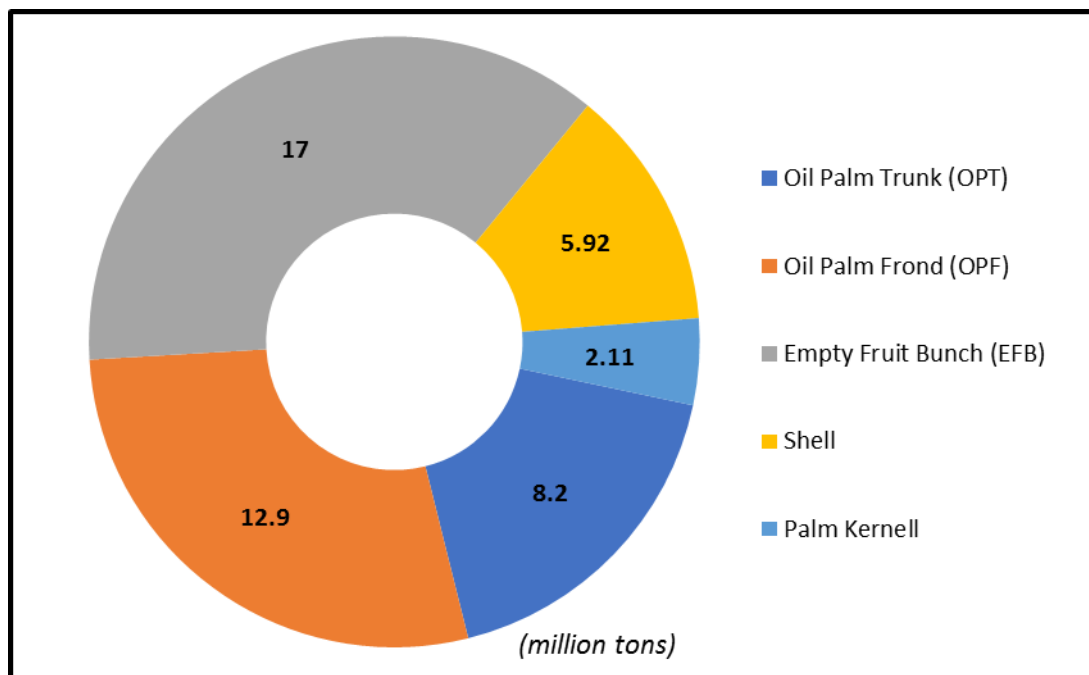


Figure 2.2: Oil palm biomass (Onoja et al., 2018)

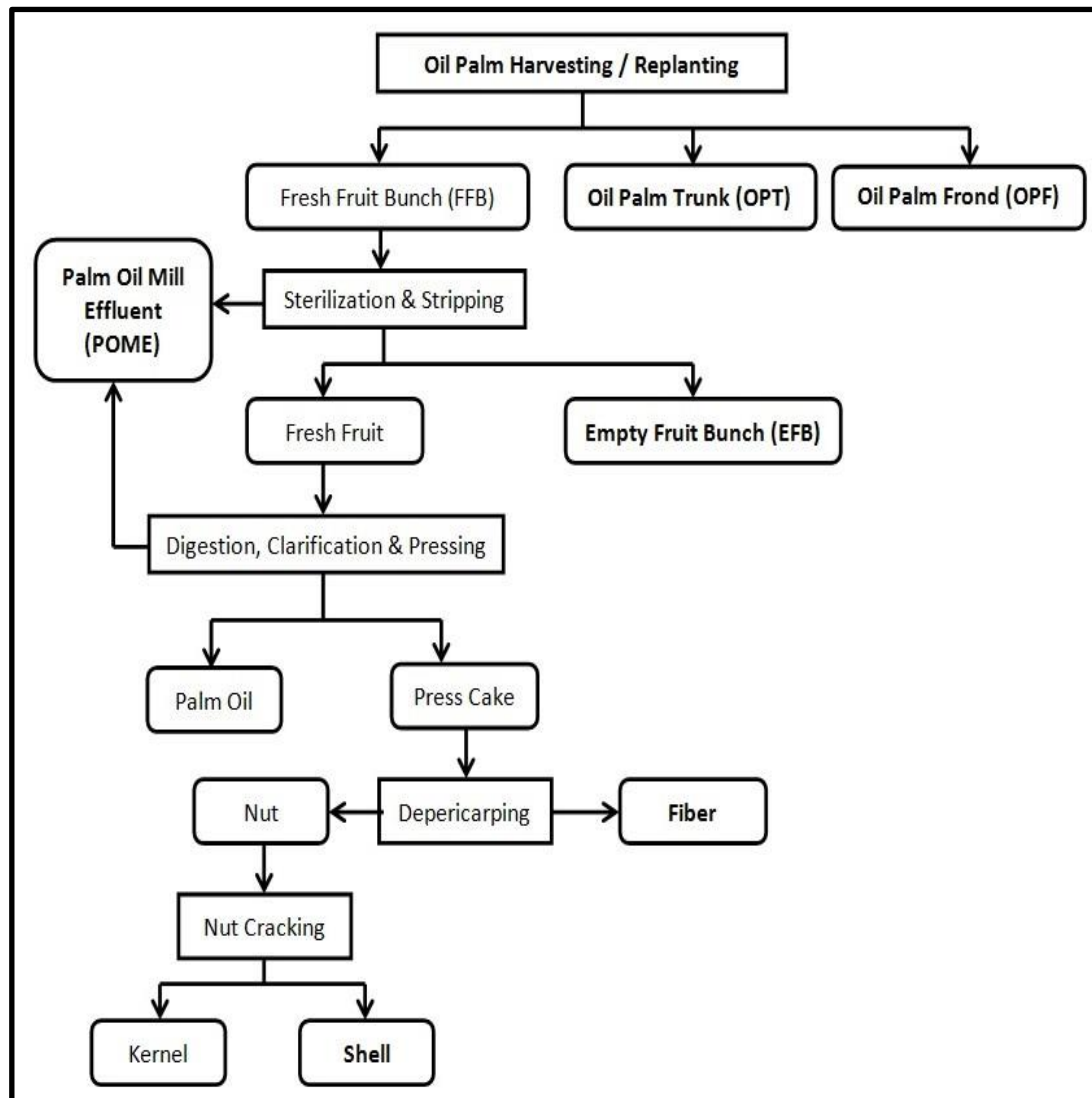


Figure 2.3: Simplified process flow diagram of an oil palm biomass is generated (Sulaiman et al., 2011, Mahlia et al., 2001, Latif Ahmad et al., 2003).

2.2.1 Oil Palm Empty Fruit Bunch (EFB)

Every year in Malaysia, a vast quantity of palm biomass is generated from the palm oil industry. Approximately one ton of empty fruit bunch (EFB) is generated for every ton of palm oil produced from fresh fruit bunch (Chang, 2014b). Oil palm EFB fiber is lignocellulosic biomass and produced in abundance as a residue product after the palm oil extraction process in the palm oil mill. About 20% of a solid residue of the fresh fruit weight is an empty fruit bunch (EFB) after oil extraction at palm oil mills. Sreekala et al. (1997) reported about 400g of EFB fiber produced

from a fresh fruit bunch. Formerly, EFB often used as fuel to generate steam at the mills. However, this practice produces gases with particulates such as tar and dust (Igwe and Onyegbado, 2007) and creates environmental pollution problems with additional methane emission into the atmosphere (Amal et al., 2008). In the past decades, most of the oil palm empty fruit bunch is utilized as fuel, fertilizer and mulching material. Recently, some studies process EFB fiber into various dimensional grades to suit specific applications such as mattress manufacturers and stabilize material for erosion control (Hasibuan and Wan Daud, 2007, Mahjoub et al., 2013). EFB offers the best prospect for commercial exploitation since it is readily available at the palm oil mill which can minimize transportation and procurement costs. Considered as waste in oil palm mill, EFB fiber can be a potential reinforcing agent and has emerged as a competitor with other lignocellulosic fiber. Besides, the possibilities of using EFB fiber as reinforcement materials for conventional building materials would be cost-effective and value-added substitutes in the building industry. EFB also has become a potential raw material for utilization in bioconversion technology to produce bio-based products (Tajuddin et al., 2019). An empty fruit bunch (EFB) and EFB fiber mat can be seen in Figure 2.4.



(a)

(b)

Figure 2.4: (a) Empty fruit bunch and (b) empty fruit bunch fiber mat

2.2.1(a) Chemical Composition of Empty Fruit Bunch

Oil palm empty fruit bunch (EFB) is a potential natural fiber resource due to highly consist of cellulose and hemicellulose. The chemical composition of EFB is an important chemical property that determines its utilization performance in several applications. Similar to other natural fibers, the soft and amorphous matrix of hemicellulose and lignin are surrounded by crystalline cellulose microfibrils which lead to rigid structure (Chang, 2014b). The composition and microstructure of EFB have attracted the industry players to utilize EFB as raw material for compost, mulch for landscaping, organic fertilizer and fuel to generate electricity (Palamae et al., 2017). The average composition of empty fruit bunch (EFB) fiber is shown in Table 2.1, with cellulose (up to 65%), followed by lignin (up to 19%), hemicellulose (up to 12.79%) and extractive (up to 3.2%). The EFB also contains small quantities of pectin, protein, extractives (nonstructural sugars, nitrogenous material, chlorophyll and waxes) and ash (Coral Medina et al., 2016). The major composition of EFB viz. cellulose, hemicellulose and lignin might differ due to plant age and growth conditions, soil conditions, weather effect, and testing methods used. Based on the literature review, these differences in composition can affect the EFB utilization in terms of material conversion and product yield (Hassan et al., 2010).

Table 2.1: Average composition of empty fruit bunch (EFB) fiber from different researchers

Composition	(Sreekala et al., 1997)	(Abdul et al., 2002)	(Kelly-Yong et al., 2007)	(Abdul Khalil et al., 2008)	(Piarpuzán et al., 2011)
Cellulose (%)	65	15	38.3	50.49	13.75
Hemicellulose (%)	-	11.73	35.3	-	12.79
Lignin (%)	19	7.14	22.1	17.84	7.79
Extractive (%)	-	-	2.7	3.21	-
Ash Content (%)	2	0.67	1.6	3.4	0.63

2.2.1(b) Physical & Mechanical properties of Empty Fruit Bunch

The physical and mechanical properties of the EFB fiber are determined by the amount of cellulose and non-cellulosic constituents in a fiber. Properties such as density, electrical resistivity, tensile strength, modulus, moisture regain and crystallinity are related to the composition and internal structure of the fibers (Reddy and Yang, 2005). Sreekala et al. (1997) also found that properties of lignocellulosic fibers depend mainly on the cellulose content and microfibril angle which give hard and tough characteristics to EFB fibers. Mahjoub et al. (2013) have compared the character of the EFB fiber in terms of tensile strength, young's modulus, elongation at break and so forth with other researchers and hence found out that there was a variation of strength value between researchers. These findings have been reported by Virk et al. (2010) that, the wide range of value was due to the various type of EFB fiber and irregular sectional area. Besides, due to lighter weight compared to synthetic fibers, its mechanical properties also unfavourable to be utilized (Afzaluddin et al., 2019). Therefore, due to the variation of performance, only a small portion of the total volume of EFB fiber has been utilized as a reinforcement agent in composites. Table 2.2 showed the data for physical and mechanical properties of EFB fiber and synthetic fibers. The table also shows that the mechanical and physical properties of the fibers are different for each fiber based on the fiber types that have been tested.

Table 2.2: Physical and mechanical properties of EFB fiber and synthetic fibers (Bledzki and Gassan, 1999, Sreekala et al., 1997).

Properties	EFB Fiber	Glass fiber
Density (g/cm ³)	0.7 – 1.55	2.56
Strength (MPa)	248	3530
Stiffness (GPa)	2.0	72
Strain (%)	14	4.8

2.2.1(c) Utilization of Empty Fruit Bunch (EFB)

The utilization of EFB has rapidly increased in the last two decades. Long-time ago, EFB biomass has limited uses. Most of EFB biomass was used as a fuel to generate steam by incineration at the mills. However, the incineration process was replaced with other fuel due to high moisture content (>60% of total EFB weight), which released a large amount of white smoke and had a significant impact on the surrounding environment. Some EFB biomass is used as a substrate for mushroom production or left to rot and returned to the fields as supplementary fertilizer (Chang, 2014a). Nowadays, there is still abundantly of EFB available that can be used for other purposes. As reported by many researchers, EFB fiber has portrayed great potential as reinforcement material in polymers. Further, with the specific properties that showed previously, reinforcement into polymer may develop superior bio-composite materials. The high content of lignocellulosic composition in EFB is a potential source of making biofuels such as ethanol and other products. The utilization of oil palm EFB fiber showed in Table 2.3.

Table 2.3: Utilization of oil palm EFB fiber

Material	Utilization	Researcher
Oil palm EFB fiber	Plywood/hybrid plywood	(Abdul Khalil et al., 2010c)
	Bioethanol/Bio-oil production	(Chiesa and Gnansounou, 2014)
	Organic mulch plantation	(Mohammad et al., 2012)
	Bio-char	(Mubarak et al., 2014)

Besides, the promising properties of EFB fibers also to be an ideal material for the making of mattresses, seats and insulation products (Kelly-Yong et al., 2007). On the other hand, the possession of EFB fiber such as low density, non-corrosive and comparable specific properties to synthetic fibers has increased interest in using EFB fiber with various polymeric materials. For example, some parts in automotive industries are replaced with natural fiber such as interior and insulation panels

(Hassan et al., 2010). Besides, the transformation of EFB fiber into nanofiller as a reinforcement agent also showed a great performance in the structural stability of nanocomposite (Saba et al., 2017). A recent study by Nasir et al. (2018) reported that EFB fiber has great potential as adsorbent material specifically in the removal of pollutants from wastewater.

2.2.2 Oil Palm Trunk (OPT)

In general, the productivity of oil palm trees becomes lower after 20-25 years. Due to the lower production of palm oil fruits, it is necessary to cut down the oil palm trees after 25-30 years and replant new seedlings at the plantation site (Amouzgar et al., 2010). During the replanting process as shown in Figure 2.5, it is estimated that seven million tons of oil palm trunk wastage available per year could be produced from 4.69 million hectares plantation (Chin et al., 2011). Furthermore, the OPT cannot easily be burned in the field due to high moisture content. The process to dispose of oil palm trunk by burning is now considered unacceptable, as it creates air pollution due to white smoke and affects the environment (Abdullah et al., 2012). Research by Abdullah et al. (2012) found that a minimum of five years to decompose the stem on the field after cut down.



(a)

(b)

Figure 2.5: (a) Felled Oil Palm Trunk and (b) Oil palm trunk log

Based on the literature, the decomposition period of oil palm trunk will be encouraging insect attacks such as ants, termites and tree worms. Moreover, *Ganoderma Boninense*, a soil fungus pathogen will attack basal stem root in OPT that can weaken the stem stiffness and reduce the strength of OPT structure (Rosli et al., 2016, Najmie et al., 2011). For a decade, the utilization of OPT as an alternative material for wood-based industries has been carried out and showed the potential to reduce the use of wood consumption. Moreover, a worldwide shortage of solid wood will be a good reason for OPT to be utilized as an alternative source for future wood-based panel industries.

2.2.2(a) Chemical Composition of Oil Palm Trunk

The natural characteristic and chemical composition of OPT are the main challenges in making it be a manageable raw material for producing various products. As reported by Komariah et al. (2019) the OPT is composed mostly of vascular bundles that provide mechanical support, whereas 70% of the inner part is composed of parenchyma that is soft and highly hygroscopic. The poor water resistance that clarified by Lamaming et al. (2013) was due to the chemical components of OPT. The parenchymatous tissues are soft and contain mainly short-chain polysaccharides and starch. Table 2.4 showed the chemical composition of oil palm trunk (OPT) fiber.

Table 2.4: Chemical composition of oil palm trunk (OPT) fiber
(Abdul Khalil et al., 2010a).

Chemical Constituents	Composition (%)
Cellulose	29.2
Lignin	18.8
Hemicellulose	16.5
Pentosan	18.8
Ash	2.0

Generally, OPT fiber showed the highest percentage of lignin composition compared to empty fruit bunch (EFB) fiber (Abdul Khalil et al., 2008). This was due to matured tissues at the base (trunk) accumulate higher amounts of metabolic products than the younger parts at the top (frond and branches) (Ververis et al., 2004). Research by Rowell et al. (2000) found that chemical composition varies from plant to plant and within plants from different parts of the same plant. It also varies within plants from different geographic locations, ages, climate, and soil conditions.

2.2.2(b) Physical and Mechanical Properties of Oil Palm Trunk

Being a monocotyledon plant, OPT does not have any vascular cambium, secondary growth, growth rings, ray cells, sapwood and hardwood, branches and knots. Moreover, the number of parenchyma tissues and vascular bundle contribute into trunk density widely between 200 and 700 kgm⁻³ and moisture content ranging from 100% - 500% (Mokhtar et al., 2011a). The morphological of dried oil palm trunk structures, a particularly vascular bundle was presented in Figure 2.6. The micrograph showed the existence of parenchymatous ground tissue, fibers, and vessels was easily recognized and identified.

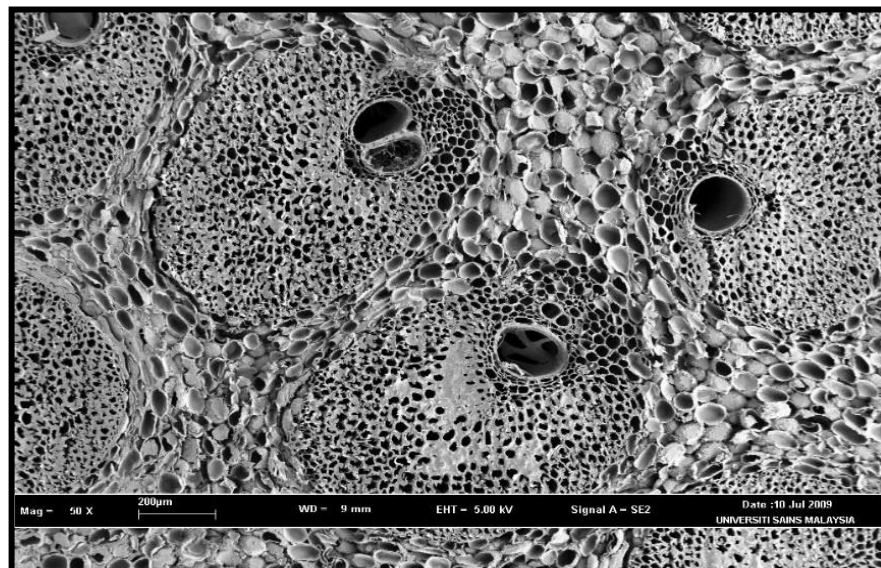


Figure 2.6: Scanning electron micrograph (SEM) of dried oil palm trunk (50x Mag)

The mechanical properties of OPT do not comparable with wood. This was due to its character which very hygroscopic in nature, shrinking and swelling at a higher rate than wood. In the meantime, the density variation observed in the trunk both in radial as well as in the vertical direction also affects the mechanical properties of OPT. Lim and Gan (2005) also found that the modulus of elasticity (Moezzi pour et al.), modulus of rupture (MOR) and compressive strength along the grain are found to be linearly correlated to the density. The OPT can be classified under weak timber such as Geronggang, Sesendok, Pulai and Terentang. Table 2.5 illustrated the variables of OPT strength properties correlated to OPT density.

Table 2.5: Oil palm trunk basic strength properties (Lim and Gan, 2005)

Density (kgm⁻³)	Modulus of Elasticity (MPa)	Modulus of Rupture (MPa)	Compressive Strength (MPa)
>500	6744	68.7	38.1
350 – 500	5094	52.5	31.4
<350	1712	18.1	13.0

2.2.2(c) Utilization of Oil Palm Trunk

Malaysia wood-based industries have concerned about the future supply of raw material especially in plywood production. This was due to forests that are gazetted to be a reserved forest. In the meantime, the collaboration between researcher and industry partners are trying to find a good alternative raw material to replace or support to wood-based industries. Currently, oil palm biomass has seemed like the most viable alternative source (Hamid et al., 2014). There were about millions of tons OPT have generated annually and this lignocellulosic material provides a continuous supply for the biomass industry. Many efforts have been made to use OPT into substitute materials such as in plywood, Laminated Veneer Lumber (LVL), Parallel Strand Lumber (PSL), Oriented Strand Board (OSB) and Medium Density Fiberboard (MDF) (Edi et al., 2006). Recently, with the revolution of

nanotechnology, the OPT becomes a preferred material to produce cellulose nanofiber (CNF) due to the high composition of holocellulose in the range of 72 to 78%. In line with sustainable green materials, the CNF can be utilized in a wide range of applications such as biomedicine purposes, cosmetics, electronics, packaging and so forth (Abdul Khalil et al., 2012a). Table 2.6 showed the utilization of OPT until nowadays.

Table 2.6: Utilization of oil palm trunk (OPT)

Material	Utilization	Researcher
Oil palm trunk veneer	Oil Palm plywood	(Mokhtar et al., 2011a)
Oil palm trunk panel	Sound Absorber	(Kerdongmee et al., 2016)
Oil palm trunk	Ethanol Extraction	(Jung et al., 2011)
Oil palm trunk	Oil palm trunk lumber	(Abdullah, 2010)
Oil palm trunk	Cellulose Nanofiber	(Lamaming et al., 2015)

2.3 Palm Oil Industry Waste

The large production of palm oil in Malaysia also abundantly produced waste from the oil palm industry which caused criticism and complaint. Report by MPOB in 2008, waste disposal from oil palm mills in Malaysia is one of the environmental challenges for industries after meeting the demand for oil at domestic and global levels (Abdul Khalil et al., 2012c). A large number of solid wastes were obtained such as palm fibers, nutshells, palm kernel and empty fruit bunches due to palm oil processing for oil extraction. Singh et al. (2010) and Yacob et al. (2005) reported that about 26.7 million tons of solid biomass were generated from 381 palm oil mills in Malaysia in 2004. Furthermore, due to the processing of oil palm fruit, the factory generated secondary waste such as palm oil mill effluent (POME) which possesses enormous high organic content and linked to water pollution. At the same, it also generates a huge amount of oil palm ash with a high content of particulate matters and generally linked to air pollution problems (Alhaji et al., 2016).

Moreover, these wastes were incinerated at temperature 800-1000°C as fuels to generate electricity in palm oil mills. From this process, two types of palm ashes were produced which is boiler ash and palm oil fuel ash (POFA). The utilization of both wastes respectively is still underutilized due to the unique properties and wide range of element compositions. The utilization of palm oil industry waste by-product from the power plant by some researchers to be value-added products is shown in Table 2.7 and Table 2.8.

Table 2.7: Utilization of the main palm oil industry waste

Palm Oil Industry Waste	Utilization	Researcher
Oil palm kernel shell (OPKS)	As coarse aggregate in pervious cement	Khankhaje et al. (2016b)
Empty fruit bunch (EFB)	Hybrid plywood Fiber reinforced hybrid composite	Abdul Khalil et al. (2010c) Jawaid et al. (2010)
Palm oil mill effluent (POME)	Extraction process to produce chemicals.	Md. Din et al. (2014)

Table 2.8: Utilization of by-products from a power plant to generate electricity and boilers operation

Boiler and power plant residue	Utilization	Researcher
Palm oil fuel ash (POFA)	Cement replacement materials	Ul Islam et al. (2016)
Oil palm ash (OPA) / Boiler ash	Absorbent materials	Mohamed et al. (2005)

Boiler ash was obtained from burning the palm fiber and kernel shells in the boiler where it consists of clinkers and ash. Meanwhile, by-products that consist of residue from palm fiber, shell and empty fruit bunch from a power plant that generates electricity is called POFA (Kroehong et al., 2011, Subramaniam et al., 2008, Zarina et al., 2013b). As reported by Khankhaje et al. (2016a) and Ul Islam et al. (2016), as much as 10 million tons of POFA was produced in Malaysia alone and

this could lead to health-related issues, environmental problems and also financial loss. However, despite waste produced from oil palm mills, researchers also explore the utilization of waste to value-added products from time to time. The investigation of by-products from the power plant and boilers by other researchers is shown in Table 2.8.

2.3.1 Oil Palm Ash (OPA)

The large production of palm oil by oil extraction in palm oil mill has abundantly produce secondary waste which causes criticism and complaint. The solid waste such as palm kernel, palm fiber and palm kernel shell are the most waste obtain from palm oil extraction. There are two types of palm ashes which know as boiler ash and palm oil fuel ash (POFA). Palm oil mill produces a large amount of boiler ash which use palm fibers and kernel shell as fuel. The boiler ash consists of clinkers and ash. Meanwhile, POFA is produced from the power plant that uses palm fiber, kernel shell and EFB as fuel and burns at 800 – 1000°C to generate electricity (Zarina et al., 2013a). This by-product is estimated to be over 4 million tons/year and escalates huge criticisms and complaints, mainly attributed to its persistent, carcinogenic and bio-accumulative effects (Vijaya et al., 2008). On the other hand, the increase in cost to dispose of the ash either in landfills or ash pond has opened up opportunities to be utilized efficiently and effectively to other value-added products (Boey et al., 2011). Although various applications of oil palm ash have been explored, it is still not widely used in industry-scale processes (Lau et al., 2019). Four types of OPA can be collected from four different stages of water tube boiler such as furnace ash, pit ash, blower ash and hopper ash (Abdul Khalil et al., 2012c). The raw images of oil palm ash obtained at different stages were shown in Figure 2.7 (a-d).

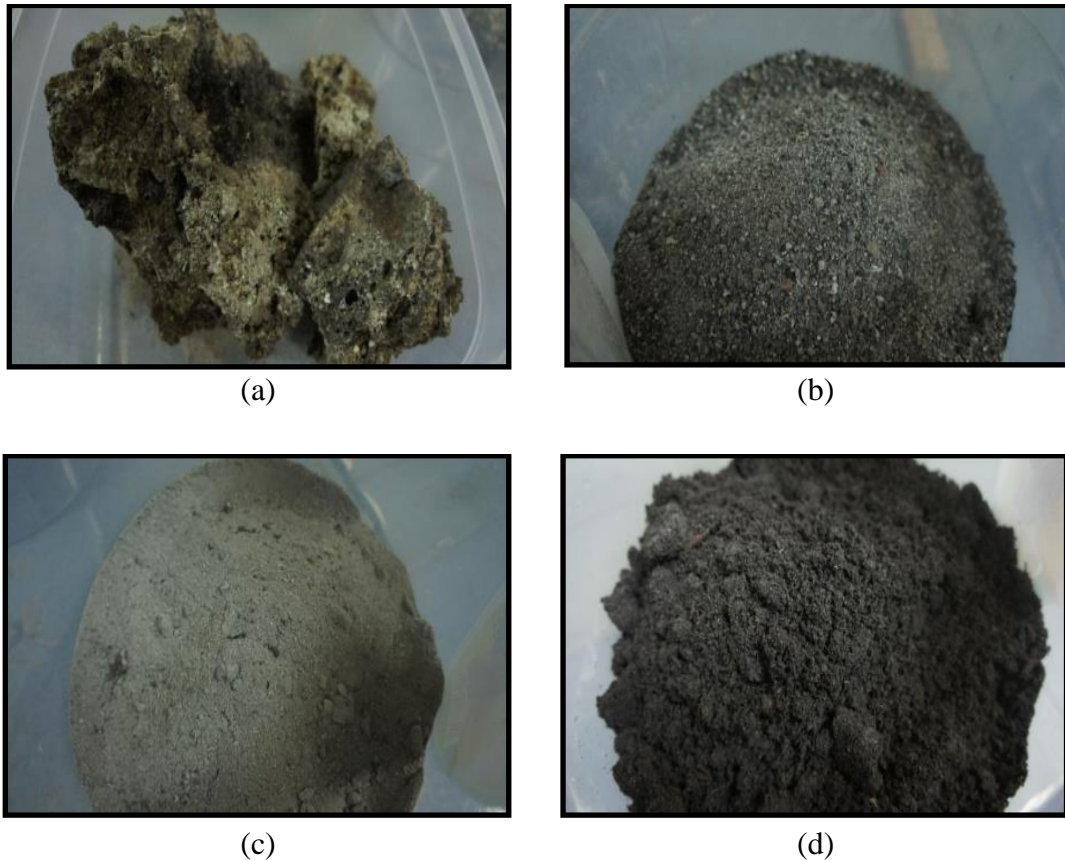


Figure 2.7: (a) Furnace ash, (b) Pit ash, (c) Blower ash and (d) Hopper ash

2.3.2 Properties of Oil Palm Ash

The raw OPA consists of irregularly shaped particles and a porous structure with a median size of about 20 μm . After the ball milling process on certain parameters, the OPA particles became smaller and individually noted containing irregular and crushed shapes (Abdul Khalil et al., 2011c, Jaturapitakkul et al., 2011, Chindaprasirt et al., 2008). Since the OPA is from the incineration process, the fresh OPA is greyish in colour and further will darken due to the increasing proportion of unburnt carbon. Its compositions indicate a high amount of silica which is considered to have high potential as replacement materials in cement and porcelain industries (Jamo et al., 2013, Altwair et al., 2011). The SEM and TEM images of OPA with different morphology were presented in Figure 2.8.

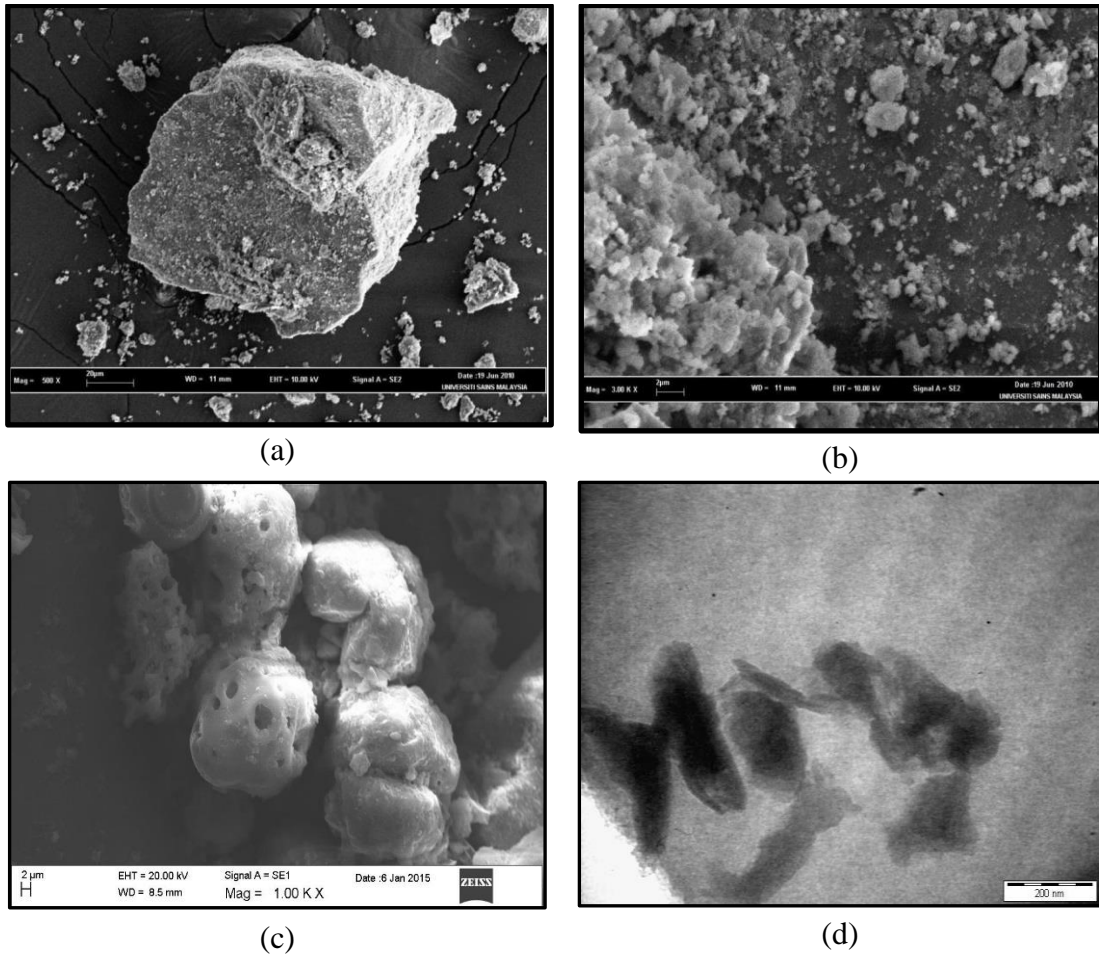


Figure 2.8: SEM Micrographs of (a) Raw OPA, (b) Ground OPA, (c) Spherical OPA and TEM micrograph of (d) OPA nanoparticles (Abdul Khalil et al., 2011c, Abdul Khalil et al., 2012b).

Tables 2.9 exhibit the composition analysis of the OPA. The chemical elements of most OPA obtained from palm oil factories are found to be silicon dioxide, aluminum oxide, iron oxide, calcium oxide, magnesium oxide, sodium oxide, potassium oxide and sulfur trioxide. The high silica (SiO_2) content in OPA could be the potential to be used in various engineering applications (Ginting et al., 2020). The mineral composition of ashes tends to be very complex and fluctuating upon the varieties of a proportion of irrigated area, geographical conditions, fertilizers used, climatic variation, soil chemistry, timeliness of production and agronomic practices in the oil palm growth process (Foo and Hameed, 2009a).

Table 2.9: The composition analysis of the oil palm ash (OPA) chemical composition (%)

Compositions	Chindapasirt et al. (2008)	Tangchirapat et al. (2009)	Jaturapitakkul et al. (2007)	Awal and Hussin (1997)	Zarina et al. (2013b)
Silicon dioxide	63.6	57.7	65.3	43.6	51.18
Aluminium oxide	1.6	4.5	2.5	11.4	4.61
Iron oxide	1.4	3.3	1.9	8.4	3.42
Calcium oxide	7.6	6.5	6.4	4.8	6.93
Magnesium oxide	3.9	4.2	3	0.4	4.02
Sodium oxide	0.1	0.5	0.3	4.7	5.52
Potassium oxide	6.9	8.2	5.7	3.5	5.52
Sulphur trioxide	0.2	0.2	0.4	2.8	0.36
Loss of ignition	9.6	10.5	10	18	21.6

2.3.3 Utilization of oil palm ash

Typically, the morphology of oil palm ash (OPA) is characterized by a spongy and porous structure in nature which suitable to be converted to the adsorbent. Research by Zainudin et al. (2005) has utilized OPA as one of the material to be adsorbent for flue gas desulfurization using the hydration process. Meanwhile, an investigation by Ahmad et al. (2007) found that palm ash could be a low-cost adsorbent material due to its availability and high adsorption to remove direct blue dye from an aqueous solution. Within years, many researchers have studied the usage of OPA as constituents in concrete. Their outcomes have revealed that oil palm ash contained a high amount of silica in amorphous form and could be utilized as a pozzolanic material. A pozzolanic material was defined as a material that contains siliceous or siliceous and aluminous material by composition (Tangchirapat et al., 2009, Bamaga et al., 2013). In most studies, the utilization of OPA as a supplementary cementitious was reported remarkably depresses the water permeability, water-to-binder ratio (W/B) and drying shrinkage, while enhances the modulus elasticity, expansion, compressive and tensile strength, thus facilitating the