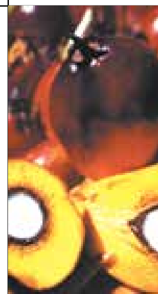

Physico-chemical Treatment of Oil Palm Biomass into Applicable Feedstock

Fatiha Ismail*; Noorshamsiana Abdul Wahab*; Nur Eliyanti Ali Othman* and Astimar Abdul Aziz*

INTRODUCTION

The depletion of fossil fuels and natural raw materials has encouraged the search for new resource materials for the production of bio-based materials (Alekhina *et al*., 2014). Oil palm biomass (OPB) is classified as lignocellulosic residues comprised mainly of cellulose, hemicellulose, and lignin in their cell walls (Raveendran *et al*., 1995). This lignocellulosic material can be converted into valuable feedstock for the production of biosugar, biocompost, biochemical and bioethanol. Due to the lignocellulosic nature of OPB, countless research and development activities were undertaken by various agencies in order to improve the transformation of OPB into more valuable substrate for producing a variety of chemicals that will have huge potential in food, chemical and pharmaceutical industries. The chemical constituents in OPB varied considerably due to their diverse origins and types (Chew and Bhatia, 2008). The chemical composition of different OPB is shown in *Table 1*.

Cellulose forms a major constituent of OPB. Only cellulose and hemicellulose can be converted into fermentable sugars. These lignocellulosic chemicals are reinforced in a lignin matrix similar to that of other natural fibres. Recovery of these components from the OPB requires some kind of pre-treatment. The pathway of different pre-treatments on OPB to extract lignocellulosic chemicals and production of fermentable sugars is shown in *Figure 1*. A pre-treatment method needed to remove lignin, reduce crystallinity of the cellulose and increase the porosity of lignocellulosic materials in order to make cellulose and hemicellulose more amenable to hydrolysis and fermentation processes that are used to convert these lignocellulosic chemicals to fermentable sugars in higher yield (Moiser *et al*., 2005; Balat, 2011). Various pre-treatment methods have been developed to extract these lignocellulosic chemicals.

Oil palm biomass were subjected to physico-chemical treatment in order to cleave the cellulosic matrix into individual component for effective chemical or biological process. The increased in surface

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TABLE 1: CHEMICAL CONTENT IN COMMON OIL PALM BIOMASS FEEDSTOCKS

Composition	Oil palm biomass chemical composition (wt %)				
	EFB	OPF	OPT	OPMF	Kernel shell
Cellulose	38-70	40-50	22-44	39-42	13-28
Hemicellulose	10-35	23-38	12-41	9-24	21-22
Holocellulose	68-86	70-83	42-73	49-64	42-47
Lignin	13-37	18-32	18-36	21-33	44-52
Xylose	29-63	26-52	15-55	40-49	63-64
Glucose	23-66	20-67	18-32	23-29	21-22
Ash	1-6	2-8	2-4	3-9	1-2

Note: EFB - empty fruit bunch. OPF - oil palm frond. OPT - oil palm trunk. OPMF - oil palm mesocarp fibre.

Source: Mohtar *et al*. (2015); Ching and Ng (2014); Rugayah *et al*. (2014); Mohd Basyaruddin *et al*. (2012); Shinoj *et al*. (2011); Bono *et al*. (2009); Chew and Bhatia (2008); Saka *et al*. (2008); Shibata *et al*. (2008); Abdul Khalil *et al*. (2006, 2008); Law *et al*. (2007); Wan Rosli *et al*. (2007); Abdul Khalil and Rozman (2004); Law and Jiang (2001); Sreekala *et al*. (2001); Kirkaldy and Susanto (1976).

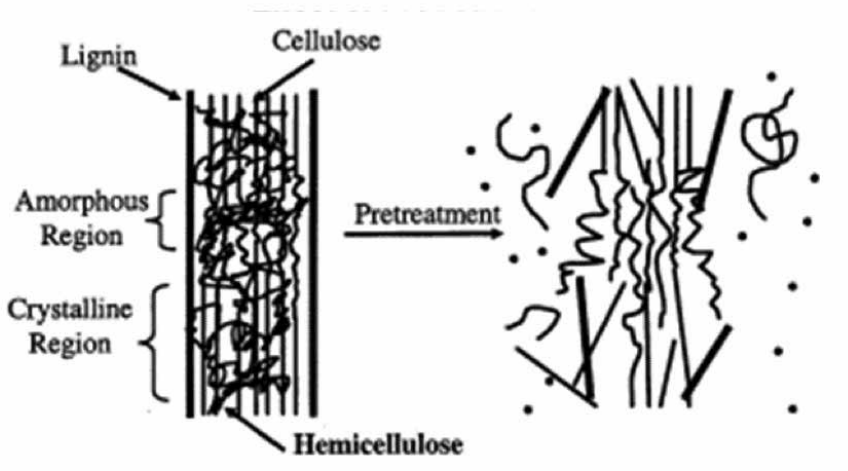

Figure 1. Cleavage of lignocellulosic matrix when subjected to physico-chemical treatment (Hsu et al. *1980).*

area of material would shorten the reaction time, hence the conversion of oil palm biomass into various higher value products is technically feasible and economically viable. *Figure 1* illustrates the cleavage of lignocellulosic matrix when subjected to physico-chemical treatment.

Types of Treatment

The physico-chemical processes are improvement of existing chemical processes, to reduce reaction time and improve the efficiency of the process. In order to enhance the removal of lignin and increase their efficiency, physical parameters such as pressure and temperature are added to the established chemical pre-treatment. In previous studies, various types of physico-chemical pre-treatments for oil palm lignocellulosic biomass were used. These include pre-treatments such as ultrasonic, microwave, ball milling, superheated and chemical thermo-mechanical. An insight review and description of various treatment processes, and discussion on their advantages and disadvantages are given below:

i) *Ultrasonication process*

Ultrasonic treatment is a relatively newer method in lignocellulosic biomass pre-treatment technology which is more

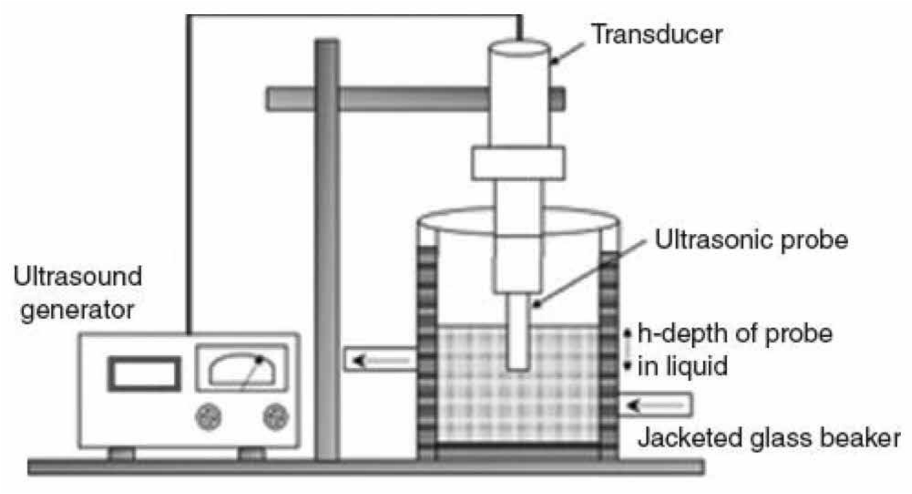

Figure 2. Set-up of ultrasonic treatment for oil palm biomass.

applicable at laboratory scale. The significant use of ultrasound on lignocellulosic biomass is to enhance the extractability of main component such as hemicelluloses, cellulose and lignin. Pre-treatment using ultrasound enhanced the hydrolysis yields from lignocellulosic biomass. Ultrasonic interactions with lignocellulose for degradation of lignin shows the key ultrasonic effects on lignocellulose. First, ultrasound increased the cleavage of bonds within lignin as well as the bonds between lignin and hemicellulose. The cleavage reactions were enhanced by radicals produced by ultrasound and the shear forces from ultrasonic mixing improved the degradation of polymer (Bussemaker and Zhang, 2013).

According to Asakura *et al.* (2008), ultrasound is also used to clean cellulosic fibre from used paper and to improve the susceptibility of lignocellulosic materials to biodegradation. In another study, Toma *et al.* (2007) found that by increasing the accessible surface area and influencing the crystallinity, it can generate a pre-treated substrate to be more easily hydrolysed. Previous research done by Robiah *et al.* (2010) have found that the ultrasonicated oil palm empty fruit bunch (OPEFB) fibre has achieved maximum xylose yield which occurred at low temperature. Thus, ultrasonication is an emerging and very effective mechanical pretreatment method by disrupting the physical, chemical and biological properties of the lignocellulosic biomass. *Figure 2* shows a set-up of ultrasonic treatment for oil palm biomass.

ii) ***Microwave process***

The treatment of biomass using microwaves has been in used since 1970s (Dominguez *et al.*, 2006). The microwave treatment of cellulose and wood has been optimised for the production of chemicals in the synthesis of natural products (Sarotti *et al.*, 2007). Microwave energy can cause a heating effect through ionic conduction where the electric field generates ionic motion causing rapid heating and reduce reaction time (Kappe, 2004). Factors such as starting material particle size, microwave power, irradiation time and source of biomass are all considered to have effect on the efficiency of microwave interactions.

Therefore, many studies have been carried out to investigate the appropriate operational parameters of the microwave pre-treatment in order to optimise the conditions for a further efficient hydrolysis of biomass. In general, the treatment of oil palm biomass commonly uses the microwave assisted-alkaline and microwave-assisted acid treatments. The combined microwave-chemical treatment of different feedstock resulted in higher sugar

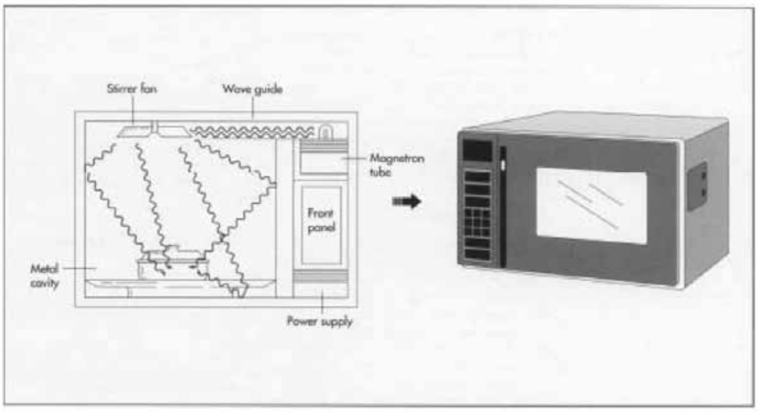

Figure 3. The working mechanism microwave heating process (Tyagi et al.*, 2013).*

recovery. Several chemicals were used in microwave/chemical pre-treatment such as microwave-assisted dilute ammonia (Chen *et al.*, 2012) and microwave-assisted $FeCl_3$ (Lu and Zhou, 2011). In previous research carried out by Komolwanich *et al.* (2014), pre-treatment on OPEFB was done using the combination of microwave and NaOH which removed lignin and hemicellulose, thus enhancing the cellulose accessibility during the pre-treatment. In 2005, Guanben and co-workers demonstrated that microwave drying with proper selection of power input, weight of drying material and drying time could increase the drying rate. When compared with the conventional drying methods, microwave drying could save up to 50% of energy and significantly decreases the volatile organic compound emissions. This view is supported by Ethaib *et al.* (2015) who concluded that although the energy consumption of microwave is relatively higher than that of oven drying, it could save more time and thus the total energy consumed is significantly lower than that of conventional methods. *Figure 3* illustrates the working mechanism of microwave process and a photo of actual machine available.

iii) *High-energy ball milling process*

Ball milling has been recognised as one of the proven treatment through its mechanical activation processes. Besides material synthesis, high-energy ball milling is a process to modify the conditions of chemical reactions by changing the reactivity of the solids (mechanical activation - increasing reaction rates, lowering reaction temperature of the ground powders) or by inducing chemical reactions during milling (mechanochemistry). Its function is mainly to increase the surface area of lignocellulosic biomass. During ball milling process, the particle sizes and crystallinity index values of the OPB were significantly reduced with extended ball mill processing time. This indicates that the particle size of treated OPB has been reduced after undergone the ball milling treatment. Zakaria *et al.* (2014) claims that the yield of glucose and xylose has increased when OPF fibre and OPEFB were pre-treated through ball mill. This makes ball milling a good and economical choice as a preliminary pre-treatment method for a wide variety of lignocellulosic feedstocks. *Figure 4* illustrates the rotation of the milling bowl and the beads during the ball milling reaction. Inside a ball mill, the balls move around in a circle movement as the mill turns, crushing the particle into powder form.

iv) *Superheated steam process*

Superheated steam (SHS) is a technology created to convert oil palm biomass into bio-composite material. Through this technology, it can effectively remove hemicellulose from oil palm biomass (OPB) fiber, which leads to surface modification of the fiber. This is because SHS treatment is conducted at high temperature and has a long retention time which causes the removal of cellulose. Previously, Warid *et*

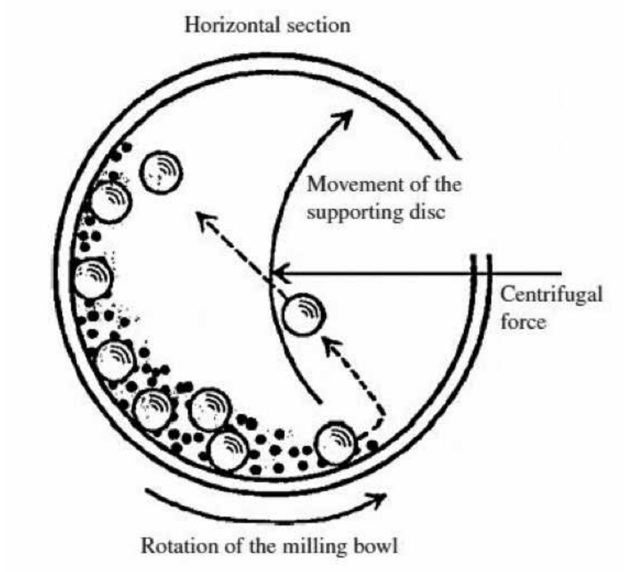

Figure 4. The illustration of the rotation of milling bowl and beads during the ball milling process.

al. (2016) conducted a study to optimise the SHS treatment temperature and retention time for three types of OPB fibers which are oil palm mesocarp fiber (OPMF), oil palm empty fruit bunch (OPEFB), and oil palm frond (OPF). The study successfully maintained the cellulose degradation below 5% while the removal of hemicellulose was in the average range of 65%.

SHS can be an advantageous treatment method for oil palm biomass as it is conducted at atmospheric pressure compared to steam explosion. The SHS treatment does not require hazardous chemicals for operation and it is a safe, non-hazardous, green technology. The entire SHS procedure is cost-savvy as it is operated at atmosphere pressure and prevents the abrasive wear and damage of the extruder screw. In addition, Bahrin *et al.* (2012) have claimed that their research on SHS treatment of OPEFB for fermentable sugars production is the first to be reported. In another major study, Ahamad Nordin *et al.* (2013) found that the utilisation of SHS for surface modification may contribute to the eco-friendly and sustainable treatment. *Figure 5* shows the photo of steam supply control system of the SHS oven.

Figure 5. Treatment of oil palm biomass by superheated steam (Warid et al.*, 2016).*

v) *Chemithermomechanical process*

The other alternative for pre-treatment of lignocellulosic biomass is via combination of chemical, thermal and mechanical treatments as one treatment process. There are various of treatment methods that can be used for removal of unwanted particles from the surface of OPEFB fibres; heat treatment, chemical treatment and mechanical treatment. The CTMP fractionation process would simultaneously remove lignin and disintegrate the microfibrils of the fibres, hence increasing the surface area of the fibres for the enzymatic saccharification. On the other hand, Rosman *et al.* (2013) treated the OPEFB using alkali followed by silane treatment and found that the surface treatment of OPEFB fibres increased the compactibility with the matrix, thus producing superior mechanical properties of the reinforced polymer composite.

The modifications of OPEFB fibres treated with sodium hydroxide and succinic acid increased the availability of functional groups through chemical modification and interacted strongly with the matrix polymer to get better interfacial bonding between fibres and

Figure 6. The soaking and the CTMP processes in MPOB Pilot Plant (Astimar et al.*, 2015).*

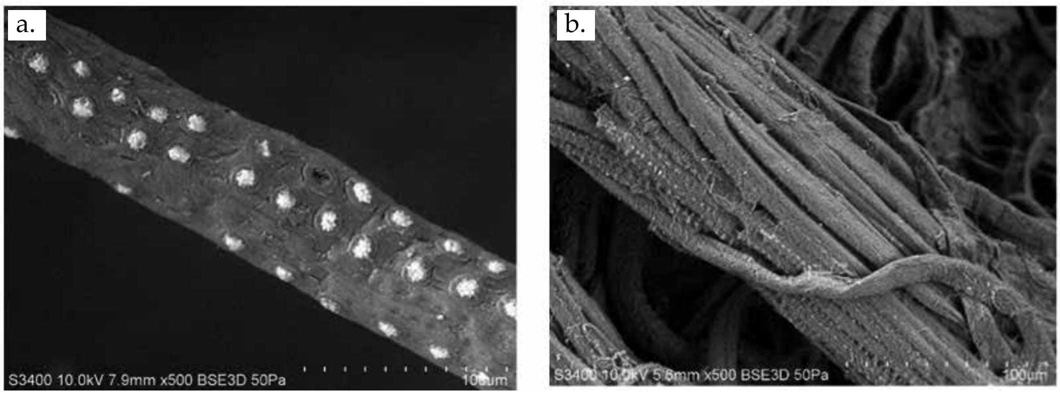

Figure 7. SEM micrographs of (a) untreated EFF and (b) treated EFB with 20% NaOH at 200 sec (160°C).

TABLE 2. ADVANTAGES AND DISADVANTAGES OF DIFFERENT TREATMENT METHODS ON OIL PALM BIOMASS

Treatment Types	Advantages	Disadvantages
Ultrasonic	• Green and sustainable treatment	Partial cleavage of the lignin-carbohydrate matrix
Microwave	• Less inhibitor formation • High reaction rate and yields	High energy consumption
Ball milling	• Green and sustainable treatment	Partial cleavage of the lignin-carbohydrate matrix
Superheated steam process	• A cost-effective operation • Transformation of lignin and-hemicelluloses • High yield of glucose and hemicellulose	• Partial disintegration of hemicelluloses • Acid catalyst needed to make process solubilisation efficient with high lignin content material • Toxic compound generation in two-step process
Chemithermomechanical	• Green and sustainable treatment	• Involve more than two steps for pre-treatment • Require more reaction time

matrix (Bhat *et al.*, 2011). Zawawi *et al.* (2015) reported that the effect of pre-treatments on the surfaces of EFB fibres are subjected to thermomechanical pulping (TMP) process. For fibres treated with NaOH, the SEM images showed that the alkali treatment made the fibre surface rougher with less amount of silica bodies (*Figure 7*). Most of the lignin and small amount of silica bodies were removed resulting in a rough surface. A study carried out by Ariffin *et al.* (2008) claimed that the combination of physical, chemical and thermal treatments have successfully altered the physical structure and chemical composition of the OPEFB, as well as reducing sugar production. OPEFB treated by chemical treatment followed by thermal is the best treatment for production of reducing sugars as compared to reversed treatment technique.

Summary of Oil Palm Biomass Treatments

The main goal of numerous treatment strategies that have been developed for OPB is to enhance the reactivity of cellulose and to increase the yield of fermentable sugars. The advantages and disadvantages of different treatment methods are listed in *Table 2*.

CONCLUSION

The important insights of this article is the objective of biomass treatment before biochemical conversion of lignocellulosic biomass into applicable feedstock, i.e. in biofuels and biorefinery applications. The treatment is depended on the choice of the optimum pretreatment process where it is a crucial step in order to enhance and activate the reactivity of cellulose. Therefore, the choice of a treatment method should also focus on important parameters such as economic viability and environmental impact, and not only on its potential yield.

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