

## Disruption of Oil Palm Trunks and Fronds by Microwave-Alkali Pretreatment

Long-Wee Lai <sup>a,c</sup> and Ani Idris <sup>a,b,\*</sup>

In this study, lignocellulosic biomass from oil palm trunk (OPT) and oil palm frond (OPF) of oil palm tree, *Elaeis guineensis*, were treated using the microwave-alkali (Mw-A) method, and their chemical constituents, namely cellulose, hemicellulose, and lignin, were analyzed. A number of instruments, *i.e.* FESEM, FT-IR, and XRD, were employed to analyze the morphology and structural changes of biomass. After the Mw-A pretreatment, it was revealed that the amount of cellulose released was up to 41.55% for OPT and 64.42% for OPF. There was also a huge degree of reduction in hemicellulose, up to 64%, but lignin removal saw a fair reduction with only 15.33% for OPT and 17.97% for OPF. The results revealed that the Mw-A pretreatment is capable of disrupting the OPT and OPF.

*Keywords:* Lignocellulosic biomass; Microwave-alkali pretreatment; Oil palm frond; Oil palm trunk; Cellulose; FESEM; XRD; FT-IR

*Contact information:* a: Department of Bioprocess Engineering, Faculty of Chemical Engineering, Universiti Teknologi Malaysia, Skudai 81310, Malaysia; b: Institute of Bioproducts Development, Universiti Teknologi Malaysia, Skudai 81310, Malaysia; c: Faculty of Science and Biotechnology, Universiti Selangor, Bestari Jaya 45600, Malaysia.

\* Corresponding author: ani@cheme.utm.my

### INTRODUCTION

Malaysia, the world's second largest palm oil producer, possesses huge plantations with areas up to 5.038 million hectares (MPOB 2012). Sulaiman *et al.* (2012) claimed that oil palm tree (*Elaeis guineensis*) has recently become one of the most pivotal and valuable commercial crops in Malaysia. Each year, there is an increase in the amounts of oil palm lignocellulosic biomass generated from palm oil sources. Chen and Danapal (2012) reported that the total amount of solid oil palm biomass wastes, which contributed to a total of 59 million tons annually (dry weight), includes palm kernel shells (PKS) and oil palm empty fruit bunches (OPEFB) obtained from the mills, oil palm fronds (OPF) obtained during routine pruning, and oil palm trunks (OPT) derived from the fields when replanting is required.

As reported by Zhang and Zhao (2010), the chemical constituents of lignocellulosic biomass are polymeric carbohydrates, which consist of cellulose, hemicellulose, and lignin. Cellulose is the source that can be converted into glucose as a cheaper fermentation feedstock.

However, the digestibility of cellulose present in lignocellulosic biomass, such as oil palm biomass, is impeded by various physiochemical, structural, and compositional factors (Binod *et al.* 2012). The existence of hemicellulose and lignin in ligno-biomass is aimed to impart strength to plant cell walls and protect cellulose from enzymatic

degradation. Hence, the efficiency of lignocellulosic pretreatment must first be addressed to maximize the cellulose released from ligno-biomass and eventually convert it into fermentable sugars. In line with this, laborious pretreatment protocols are inevitable so as to provide a more accessible form of substrate for enzyme digestion. Generally, most published protocols can be categorized into three main groups: physical, chemical, and biological. Some examples include the use of hot compressed water on oil palm biomass for ethanol production (Goh *et al.* 2010), sequential acid-alkali pretreatment on oil palm empty fruit bunches biomass (Kim *et al.* 2012), acid in ionic liquid in treating lignocellulosic materials (Li *et al.* 2008), ionic liquid on oil palm frond pretreatment (Tan *et al.* 2011), and ionic liquid technology for cellulosic ethanol production (Wang *et al.* 2011).

Nowadays, pretreatment methods involving the aid of microwave technology have gained more attention and have been scrutinized by many researchers. For instance, microwave irradiation can create volumetric heating in a relatively short time by producing efficient internal heating *via* direct coupling of microwave energy with molecules that are present in the reaction mixture (Kappe 2009), thus providing a rapid energy-efficient heating on biomass pretreatment. Consequently, the temperature increment in the whole volume is simultaneously increased, whereas in a conventional heated vessel the reaction mixture in contact with vessel wall is heated *via* conduction and convection. This would lead to a slow heating process and the development of a temperature gradient within the sample, resulting in local overheating that might significantly decompose the product, substrate, or reagent. Yin (2012) has mentioned that conventional thermal heating is a relatively slow and inefficient heating method if compared to the microwave-irradiation.

Studies have shown that pretreatment systems using microwave irradiation exhibit improvement of organic reaction efficiency (Zhang and Zhao 2010) and can alter the ultrastructure of cellulose, degrade lignin and hemicellulose in lignocellulosic materials, and thus increase the susceptibility of lignocellulosic materials (Binod *et al.* 2012). Hamzah *et al.* (2009) demonstrated treating oil palm empty fruit bunch (EFB) biomass using microwave-alkali treatment, whereas Chen *et al.* (2011) conducted dilute acid pretreatment on sugarcane bagasse under microwave conditions. Khamtib *et al.* (2011) performed microwave-assisted sulfuric acid pretreatment on oil palm trunk for hydrogen production.

To the best of our knowledge, pretreatment using microwave-alkali (Mw-A) has been performed mostly on EFB biomass only (Hamzah *et al.* 2009), not on the other parts of oil palm biomass such as OPT and OPF. Therefore, the novelty in the present study is to provide complementary data to OPEFB and serve as perceptive information for subsequent or future research database.

In addition to that, under the predetermine microwaves' conditions (700 W, 80 °C, and 60 min), the change in treated biomass structure and morphology was evaluated in depth. The field emission scanning electron microscope (FESEM) images, Fourier transform infrared (FT-IR) pictograms, and X-ray diffraction (XRD) patterns of raw and pretreated OPT and OPF biomass were examined and analyzed so as to reveal the influence of microwave irradiation on the structure and chemical constituents of the OPF and OPT.

## EXPERIMENTAL

### Raw Materials

The oil palm biomass samples OPT and OPF were collected from FELDA palm oil plantation, Tenggaraoh, Johor, Malaysia. Both samples were washed to remove unwanted dirt, soil, dust, and insects prior to air-drying under sunlight. The dried samples were further ground using a disk mill (model FFC-15, China). The ground sample was fractionated by sieving to obtain a size less than 1.0 mm (mesh size: < No.18) using a Restuch sieve shaker (AS 200 basis, Germany). The powdered samples were kept at 60 °C overnight prior to compositional analysis.

### Chemical Reagents

The chemical, acid solutions, and solvents used in the experiments such as sodium hydroxide (NaOH), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), acetic acid (CH<sub>3</sub>COOH), ethanol (C<sub>2</sub>H<sub>5</sub>OH), and toluene (C<sub>7</sub>H<sub>8</sub>) were purchased from Merck (M) Sdn. Bhd. The sodium chlorite (NaClO<sub>2</sub>) was bought from Sigma-Aldrich (M) Sdn. Bhd. All chemicals were of analytical grade with highest purity. These chemicals (H<sub>2</sub>SO<sub>4</sub>, CH<sub>3</sub>COOH, C<sub>2</sub>H<sub>5</sub>OH, C<sub>7</sub>H<sub>8</sub>, and NaClO<sub>2</sub>) were used in the compositional analysis experiment as described by TAPPI test methods.

### Methods

#### *Microwave-Alkali (Mw-A) pretreatment*

A total of 20.0 g biomass sample was drenched in 200 mL of 2.5 M sodium hydroxide solution for 2.0 h prior to microwave treatment. The 2.5 M sodium hydroxide concentration has been used to treat OPEFB biomass (Hamzah *et al.* 2009). Since OPT and OPF are parts of the oil palm tree, the selection of 2.5 M NaOH was thought to be appropriate in the investigation. The Sineo microwave (MAS-II, Shang Hai, China) was used to perform microwave-alkali experiment under normal pressure. It possesses basic controller functions such as temperature, time, and power control that enable users to adjust to desired setting points. In this study, the Mw-A conditions were preset at the following values of microwave power, temperature, and duration *viz.* 700 W, 80 °C, and 60 min, respectively. Upon completion, the slurry was first cooled to room temperature and subsequently filtered through Whatman filter paper, no 3. The filter cake was washed under running tap water, followed by distilled water. The washing process was completed when the pH of filtered solution showed a neutral pH value. The treated residues were then dried and kept for subsequent compositional analysis. The raw biomass was treated in exactly the same manner as the pretreated one but did not undergo microwave treatment.

### Compositional Analysis

The compositional analysis for raw and microwave-alkali treated OPT and OPF were recounted as follows. All verification procedures were performed in duplicates. In order to ensure that attained results were reproducible, the relative percentage difference (RPD) was set to no more than 5%, revealing the reliability of the fiber analysis (Van Soest *et al.* 1991). A slightly modified protocol, as described by Mantanus *et al.* (2009), was used as the reference method in determining the moisture content of the OPT and OPF samples. The Soxhlet extraction process was conducted as described by a TAPPI test method (1997) to determine the percentage of extractive in the biomass samples. The

holocellulose content was measured as the sodium chlorite delignified residue (Teramoto *et al.* 2009). A slightly modified version of the corresponding TAPPI test method (2009) was used to determine the  $\alpha$ -cellulose content, and the hemicellulose was calculated as the difference between the holocellulose and cellulose contents. The acid insoluble lignin or Klason's Lignin content was quantified using a slightly modified version of the corresponding TAPPI test method (2002).

### Morphology Analysis

The field emission scanning electron microscopy images were captured using a Supra Zeiss instrument (Germany) for surface analysis. The FT-IR spectra profile was recorded in the range of 4000 to 370  $\text{cm}^{-1}$  using a Pelkin Elmer spectrometer (Model 2000), USA. The Siemens X-ray Diffractometer (D 5000), USA, was used to test and measure the crystalline index of the biomass samples. The crystallinity of cellulose was calculated according to the empirical method proposed by Segal *et al.* (1959),

$$\text{CrI (\%)} = [(I_{002} - I_{18^\circ}) / I_{002}] \times 100 \% \quad (1)$$

where CrI is the crystalline index,  $I_{002}$  is the maximum intensity of the (002) lattice diffraction, and  $I_{18^\circ}$  is the intensity diffraction at  $18^\circ$ ,  $2\theta$  degree.

## RESULTS AND DISCUSSION

### Chemical Compositional Analysis

The dry weight of some basic constituents including cellulose, hemicellulose, lignin, and extractives components for raw and microwave-alkali (Mw-A) treated oil palm trunk and oil palm frond are tabulated in Table 1.

**Table 1.** Chemical Composition Analysis of Raw and Mw-A Treated OPT and OPF (g/100g biomass)

Pretreatment Type	(g/100g biomass)			
	Cellulose	Hemicellulose	Lignin	Extractives
Raw OPT	50.78	30.36	17.87	0.99
Mw-A treated OPT	71.88	11.77	15.13	1.22
Raw OPF	41.88	33.61	20.65	3.86
Mw-A treated OPF	68.86	11.84	16.94	2.36
All measurements were within the relative percentage difference (RPD) below 5% (Van Soest <i>et al.</i> 1991)				

Table 1 provides an insight into the variation of the constituents affected by microwave-alkali (Mw-A) pretreatment. By comparing to the raw biomass samples, the cellulose content in both Mw-A treated samples increased up to 41.55% and 64.42% for treated OPT and OPF, respectively. The increment may be due to the disruption of the ultrastructure of lignocellulosic oil palm biomass under microwave irradiation conditions. For instance, Hamzah *et al.* (2009) performed Mw-A heating on oil palm empty fruit bunch (OPEFB) samples, which also showed a significant increase of cellulose content in the treated sample.

In line with this, it was strongly asserted that the combination of the 2.5 M NaOH aqueous solution with microwave heating conditions was able to break down the recalcitrant biomass complex by interfering with the inter- and intra-molecular hydrogen bonds embedded within the matrix of lignin and hemicellulose in the biomass, thus resulting in more cellulose being released.

Other researchers also observed the same phenomenon when treating lignocellulosic biomass using the microwave heating approach (Zhu *et al.* 2005; Binod *et al.* 2012), where microwave-assisted pretreatment showed a higher content of cellulose than the untreated ones. On the other hand, the lower value of hemicellulose was attained in Mw-A treated OPT and OPF, *i.e.* 11.77 and 11.84 (g/100g biomass), respectively. In general, hemicellulose is slightly crosslinked, relatively amorphous, and more easily hydrolyzed than cellulose. Therefore substantial loss of hemicellulose is common in many pretreatment techniques.

The significant reduction may be attributed to the ability of Mw-A pretreatment to depolymerize the hetero-polysaccharides sugar building blocks into oligosaccharides. The microwave heating transfers and induces heat to the direct interaction between the applied electromagnetic field to the lignobiomass, such as OPT and OPF, and eventually disrupts the structure of the hetero-polysaccharides sugar building blocks of hemicellulose (Ebringerova 2006).

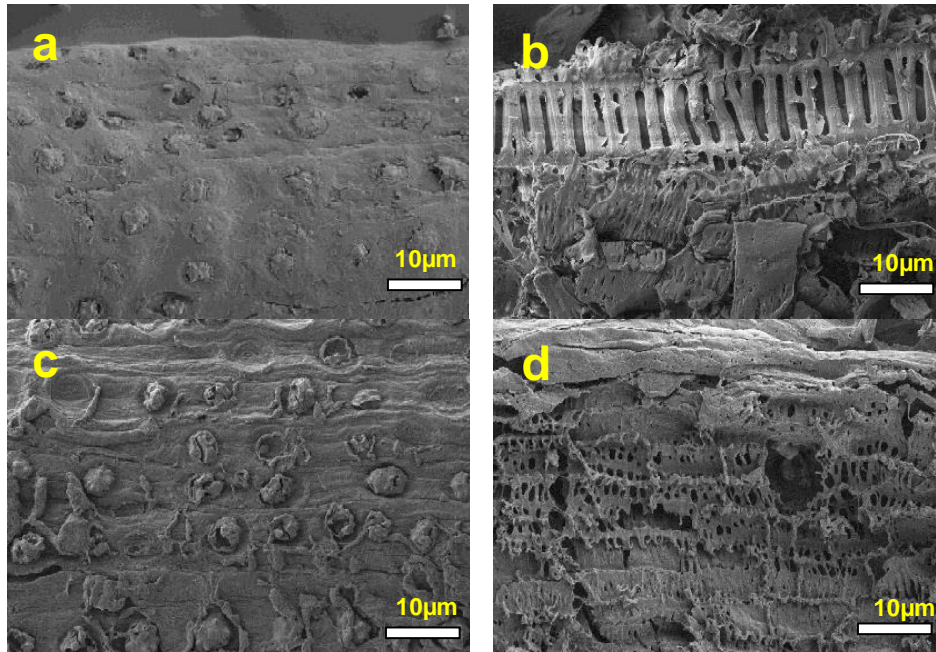
The efficiency of lignin removal by the Mw-A technique was also examined by lignin estimation after pretreatment. There was only up to 15.33%-OPT and 17.97%-OPF of reduction. Kim *et al.* (2012) also reported that the lignin content in dilute acid treated OPEFB was fairly pronounced, approximately 19.79% for 1% (v/v) sulfuric acid concentration. The higher amount of lignin in OPT and OPF is a disadvantage, as it limits the biodegradability of lignocellulosic biomass in the subsequent enzymatic hydrolysis process.

According to the review by Hendriks and Zeeman (2009), lignin removal was sufficient in low heating pretreatment processes for low-lignin biomass compounds, but not for high-lignin contained biomass. Since OPT and OPF biomass were the trunks and fronds of oil palm trees, the encrusted lignocellulosic polymer contained high-lignin and thus might require higher temperatures (>160 °C) in order to achieve higher lignin removal (Chen *et al.* 2011). In summary, the Mw-A technique is more effective in extracting the cellulose and hemicellulose fractions than lignin in both pretreated OPT and OPF samples.

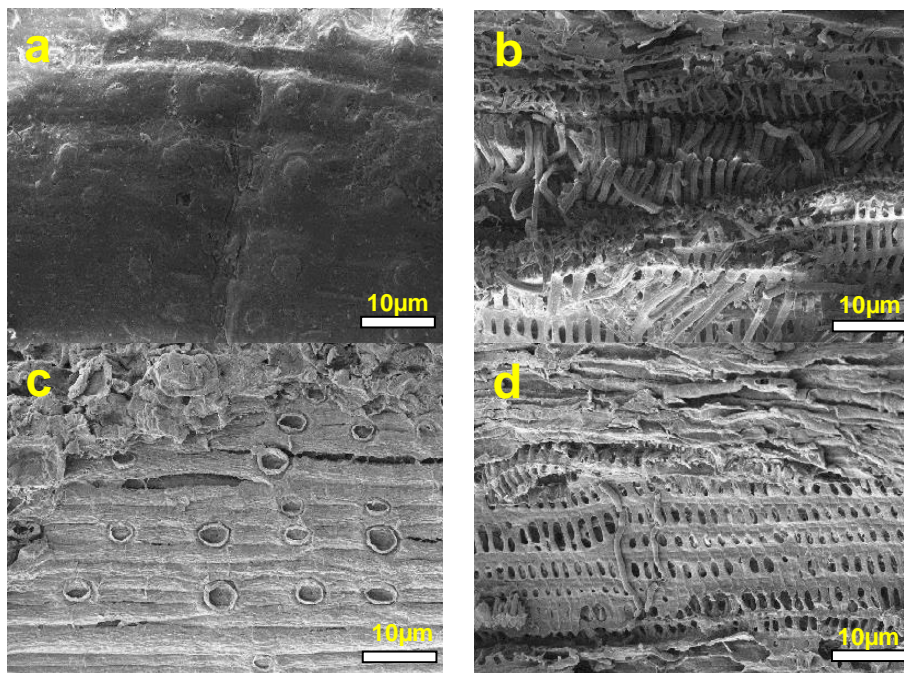
### FESEM Analysis

The field emission scanning electron microscope (FESEM) images of raw and Mw-A treated OPT are depicted in Fig. 1 (a-d) and OPF in Fig. 2 (a-d). For the raw biomass, a completely smooth with rigid, well-shaped fibril can be seen at the surface of the samples (Figs. 1a and 2a).

The neat order and compact lignocellulosic internal structure was clearly observed as well (Figs. 1b and 2b). After undergoing the Mw-A pretreatment process, cracks and tiny cavities were observed on the surfaces of both OPT and OPF (Figs. 1c and 2c). The irregular and disorder of lignocellulosic internal structure can also be detected as illustrated in Figs. 1d and 2d.



**Fig. 1.** Field emission scanning electron microscopic (FESEM) images at 500X magnification, 5.0 kV, and 10  $\mu\text{m}$  of raw and microwave-alkali (Mw-A) treated oil palm trunk (OPT): (a) raw surface, (b) raw internal structure, (c) Mw-A treated surface, and (d) Mw-A treated internal structure



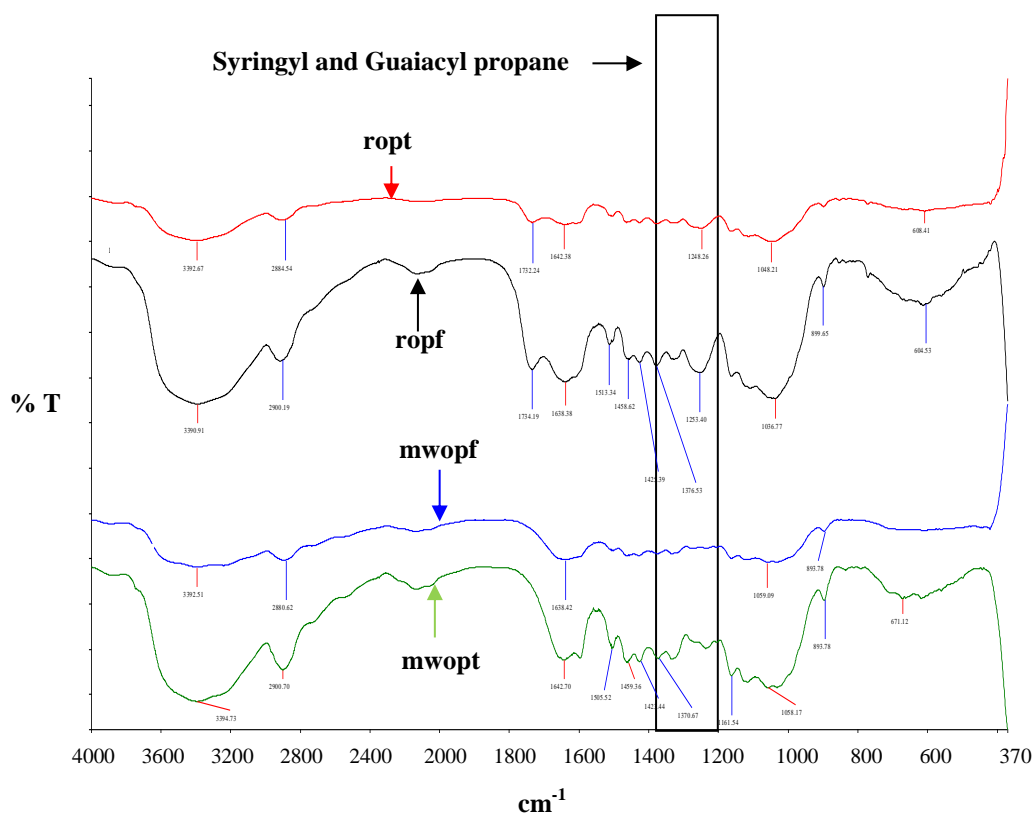
**Fig. 2.** Field emission scanning electron microscopic (FESEM) images at 500X magnification, 5.0 kV, and 10  $\mu\text{m}$  of raw and microwave-alkali (Mw-A) treated oil palm frond (OPF): (a) raw surface, (b) raw internal structure, (c) Mw-A treated surface, and (d) Mw-A treated internal structure

The results apparently indicate that microwave irradiation is able to induce physical changes to the OPT and OPF biomass. The damage to the structure allows more

cellulose to be exposed and released, and this assumption is also supported by the increase in cellulose content after Mw-A pretreatment (see Table 1). The amount of hemicellulose and lignin were, however, decreased as well, as shown in Table 1. Similar structural changes were earlier reported for OPEFB with Mw-A heating (Fazlena *et al.* 2009), rice straw microwave-alkali pretreatment (Zhu *et al.* 2005), microwave-NaOH pretreatment liquor distiller's grain (vinasse) for lactic acid production (Liu *et al.* 2012), microwave-assisted dilute ammonia treated sorghum (Chen *et al.* 2012), and microwave-assisted alkali switchgrass pretreatment (Hu and Wen 2008).

### FT-IR Analysis

FT-IR spectroscopy was used to investigate the chemical structure changes of raw and Mw-A treated biomass, as shown in Fig. 3. The broad absorption at 3394 - 3390  $\text{cm}^{-1}$  is related to the stretching of H-bonded OH groups, whereas the band at 2900-2800  $\text{cm}^{-1}$  is related to the C-H stretching (Wang *et al.* 2007).





swelling and thus enzyme accessibility more so than syringyl lignin. Thus, the disappearance in the absorbance at  $1245\text{ cm}^{-1}$  and reduction at  $1375\text{ cm}^{-1}$  represents the destruction of the guaiacyl units and reduction in the syringyl propane units, respectively, in the lignin structure of the Mw-A treated samples. Reduction occurring at absorbencies of  $1509$  to  $1422\text{ cm}^{-1}$  indicates that lignin was reduced in the Mw-A biomass samples. The disrupted crystalline cellulose region was observed at  $1059$ - $1058\text{ cm}^{-1}$  for both Mw-A pretreatment biomass samples. These bands indicate the breaking of hydrogen-bonds in the Mw-A treated samples (Sun *et al.* 2009).

Under microwave heating conditions, the NaOH was able to penetrate the lignocellulosic structure more efficiently, thus causing disruptions in such bonds. During the pretreatment, sodium hydroxide mainly serves as a microwave absorber. The raw biomass is not a good microwave absorber. It is mainly the quick moisture release induced by the efficient in-core volumetric and uniform microwave heating that changes the biomass composition and physical properties. The sharp bands ranging between  $899$  and  $893\text{ cm}^{-1}$  have been attributed to  $\beta$ -glycosidic linkage between the sugar units in cellulose (Sekkal *et al.* 1995). All three spectra profiles are clearly exhibited, except for the raw-OPT. As reported by Chen *et al.* (2011), the subtle band in raw-OPT is probably due to coverage of cellulose by hemicellulose and lignin. Table 2 illustrates typical infrared band frequencies and FT-IR spectra of wood components in units of wavenumber,  $\text{cm}^{-1}$ .

**Table 2.** Typical Infrared Band Frequencies and FT-IR Spectra of Wood Component

Infrared Band <sup>a</sup> $\text{cm}^{-1}$	Common Functional Groups Assignment	Infrared Band <sup>b</sup> $\text{cm}^{-1}$	Wood Component
			Assignment
3500-3200	O-H (H-bonded)		
2840-2690	C-H (aldehyde C-H)		
1740-1720	C=O (saturated aldehyde)	1735	C=O in xylans (Hemicelluloses)
1695-1630	C=O, C=C	1647	Absorbed OH ad conjugate C=O
1500-1450	C=C (in ring) (2 bands)	1505	Aromatic skeletal vibration in lignin
		1421	CH deformation in lignin and carbohydrate
1300-1000	C-O	1235	Syringyl ring and CO stretch in lignin and xylan.
		1371	CH deformation in cellulose and hemicellulose
		1319	CH vibration in cellulose, CO vibration in syringyl derivative
		1155	C-O-C vibration in cellulose and hemicellulose
		1030	C-O vibration in cellulose and hemicellulose
900	C-H	897	CH deformation in cellulose

<sup>a</sup> Michigan State University: Typical Infrared Absorption Frequencies

<sup>b</sup> Pandey and Pitman (2003)

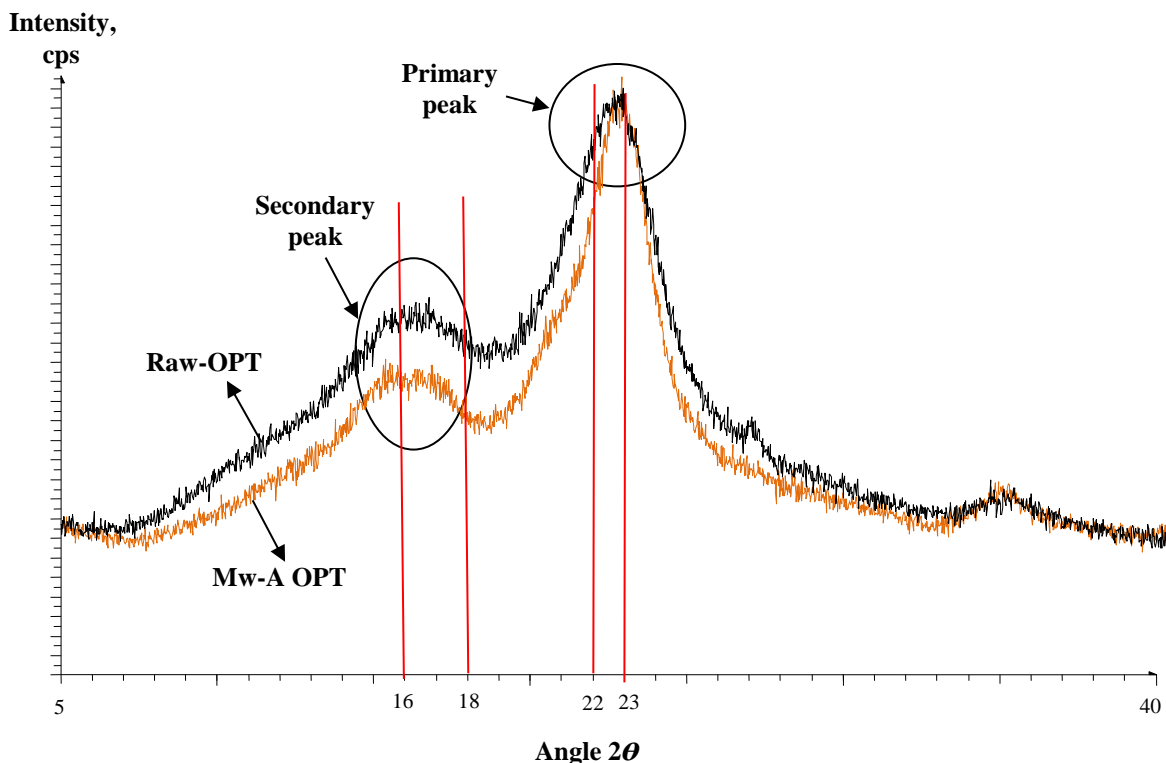
### XRD Analysis

The XRD patterns of the raw and Mw-A pretreated biomass are illustrated in Figs. 4 and 5. Chen *et al.* (2010) reported that the major diffraction peak for cellulose crystallography can be identified for  $2\theta$  ranging between  $22^\circ$  and  $23^\circ$  as a primary peak, whereas a secondary peak is in the range of  $16^\circ$  to  $18^\circ$ . From the XRD distribution



pattern, tangible peaks can be observed within the mentioned range for both the raw and Mw-A treated oil palm biomass, which confirms the presence of crystalline and amorphous structure of cellulose constituent.

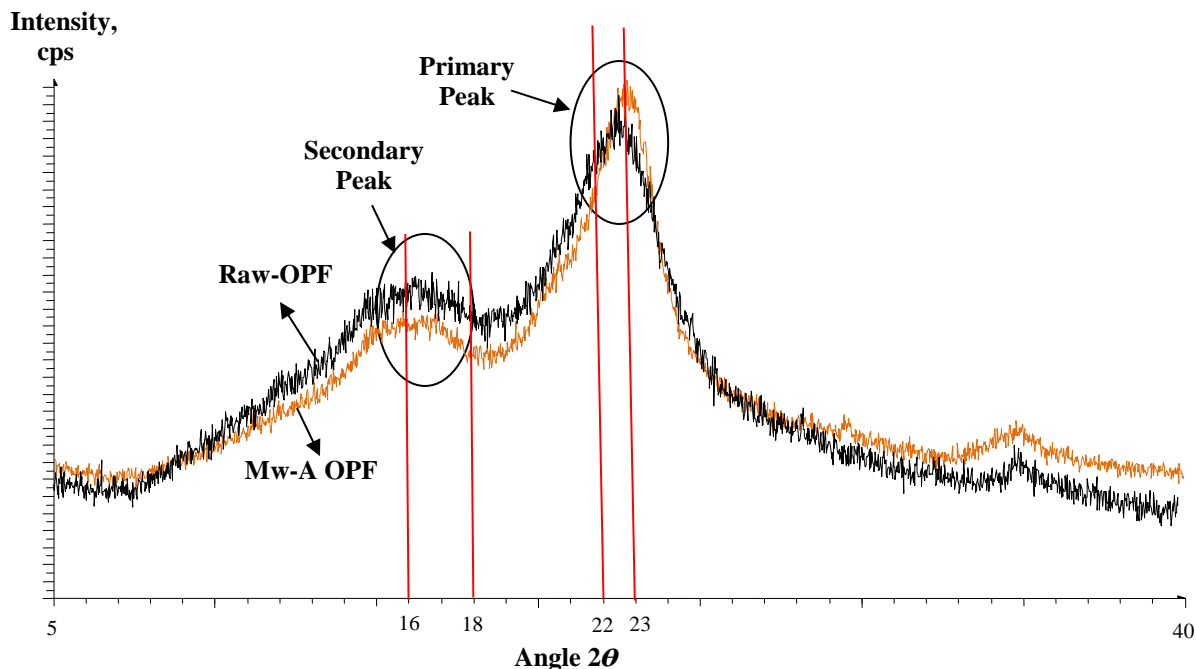
As reported by Liu *et al.* (2012), the  $I_{002}$  peak intensity (the maximum intensity of the 002 lattice diffraction) represents the primary peak and is classified as the diffraction intensity of crystalline regions, whereas the secondary peak represents the diffraction intensity of the amorphous zone. The primary peak of Mw-A treated OPT and OPF samples displayed a certain degree of narrowing, indicating a reduction in the crystallinity of the structure. The variation in the width of crystallization peak reflects intensity transformation in the cellulose molecular hydrogen bonding. In other words, the disruption of hydrogen bond in Mw-A pretreated samples occurs due to the rapid heating caused by microwave irradiation that can effectively increase the splitting effect of NaOH on the crystalline cellulose chain and maximize the expansion of the amorphous state (Liu *et al.* 2012). In addition, the NaOH solution behaves as an intracrystalline swelling agent that is capable of penetrating and swelling both the accessible amorphous and crystalline regions, as previously reported by Shujun *et al.* (2007). At the same time, the destruction of the cellulose crystalline structure occurred and the highly ordered fibrils in cellulose were distorted. As a consequence, the microfibrils are separated from the connected structure and become fully exposed, increasing the external surface and porosity of the cellulose.



**Fig. 4.** X-ray diffractometer (XRD) pattern within  $2\theta$  scale ranging from 5 to  $40^\circ$  for raw OPT and Mw-A treated OPT

The crystallinity index of cellulose has been used to interpret changes in cellulose structure after pretreatment. A higher crystallinity index reflects a more amorphous state

compared to a low crystallinity index. Table 3 shows the crystallinity index for raw OPT and OPF which are 42.07% and 37.21%, respectively.



**Fig. 5.** X-ray diffractometer (XRD) pattern within  $2\theta$  scale ranging from 5 to  $40^\circ$  for raw OPF and Mw-A treated OPF

Upon Mw-A pretreatment of the OPT and OPF samples, crystallinity index values of 56.08% and 44.20% were obtained. The increase in the percentage of crystallinity index denotes that the microwave heating plus NaOH solution disrupts the crystalline cellulose structure, thus exposing the cellulose structure. The disruption of the crystalline structure into a more amorphous state eventually facilitates the enzyme accessibility. Thus, the Mw-A pretreated samples are now readily available for enzymes, where enzymes can rapidly digest the “easy amorphous” material rather than the “difficult crystalline” cellulose.

Binod *et al.* (2012) postulated that the increase of crystallinity index might be attributed to the lignin removal under microwave-assisted reaction. This phenomenon is supported by the decrease in amount of lignin after Mw-A, as exhibited in Table 1 (15.33% for OPT and 17.97% for OPF).

**Table 3.** Intensity and Crystallinity Index (%) of Raw and Mw-A Treated OPT and OPF

Material	Intensity, cps		Crystallinity Index (%)
	Primary peak, $I_{002}$	Secondary peak, $I_{18}$	
Raw OPT	3107	1800	42.07
Mw-A OPT	2960	1300	56.08
Raw-OPF	2150	1350	37.21
Mw-A OPF	2631	1468	44.20

## CONCLUSIONS

1. Mw-A pretreatment was successful in reducing hemicellulose and lignin, whereas it increased the proportion of cellulose.
2. The FESEM images revealed that Mw-A pretreated OPT and OPF possess rough surfaces and disordered internal structures, indicating disruption of hemicellulose and lignin bonds thus releasing the cellulose.
3. The FT-IR spectra showed the stretching of hydrogen bonds of pretreated biomass, which illustrates the structural changes caused by microwave pretreatment.
4. The XRD profile showed that the raw biomass had a low crystallinity index when compared to treated biomass.
5. The Mw-A pretreatment is an effective and rapid treatment method for OPT and OPF.
6. This investigation might provide insight to further develop a microwave-alkali technology, particularly in treating recalcitrant lignocellulosic materials.

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