## PHYSICOCHEMICAL PROPERTY CHANGES OF OIL PALM MESOCARP FIBERS TREATED WITH HIGH-PRESSURE STEAM

Noor Seribainun Hidayah Md Yunos,<sup>a</sup> Azhari Samsu Baharuddin,<sup>a,\*</sup> Khairul Faezah Md Yunos,<sup>a</sup> M. Nazli Naim,<sup>a</sup> and Haruo Nishida<sup>b</sup>

> High-pressure steam treatment (HPST) is a potential alternative method for the modification of lignocellulosic materials. The effect of HPST on oil palm mesocarp fibers (OPMF) was successfully investigated with treatment conditions of 170 °C/ 0.82 MPa, 190 °C/ 1.32 MPa, 210 °C/ 2.03 MPa, and 230 °C/ 3.00 MPa for 2 min. treatment time. Significant changes in the colour, smell, and mechanical properties of the samples were observed after the treatment. Scanning electron microscope (SEM) images revealed changes in the surface morphology of the OPMF after the pretreatment. The degradation of hemicelluloses and changes in the functional groups of the lignocellulosic components were identified using Fourier Transform Infrared (FTIR) and Thermogravimetric (TG) analysis. These results suggest that HPST is a promising method for the pretreatment of OPMF.

Keywords: High-pressure steam; Lignocellulosic materials; Oil palm mesocarp fiber; Physicochemical properties

Contact information: a:Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM, Serdang Selangor, Malaysia; b: Department of Biological Functions and Engineering, Graduate School of Life Science and System Engineering, Kyushu Institute of Technology, 2-4 Hibikino, Wakamatsu-ku, Kitakyushu, Fukuoka 808-0916, JAPAN; \*Corresponding author: azharis@eng.upm.edu.my

## INTRODUCTION

Malaysia experiences favorable weather conditions throughout the year, which is advantageous for palm oil cultivation (Yusoff 2006). According to Yoshizaki *et al.* (2012), 80% of waste material from palm cultivation, such as empty fruit bunch (EFB), mesocarp fiber, and palm kernel shell (PKS), are utilized for plantation nutrient recycling or are burnt inefficiently in the mills. Out of this biomass, it is expected that the future generation of oil palm mesocarp fiber (OPMF) will grow, beyond what can be burned by the limited boiler capacity of the mills (Hock *et al.* 2009). Generally, oil palm mesocarp fiber (OPMF) is a waste material after the oil extraction; this waste material creates a significant environmental problem. Currently, OPMF is used as a mulching medium, a boiler fuel source, and as a fiber source for composites used in furniture and mattress manufacturing. Accordingly, an alternative disposal method or new technological procedures are necessary to overcome the large quantity of biomass waste. Through observations at palm oil mills, it appears the abundant oil palm mesocarp fiber is a potential substrate for the bioconversion process.

OPMF is a lignocellulosic material that can be converted into a valuable feedstock for the production of biosugar, biocompost, biochemicals, and bioethanol. However, the conversion of lignocellulosic material requires an effective pretreatment to enhance sugar production. Shamsudin *et al.* (2012) and Baharuddin *et al.* (2012) indicated that OPMF

pretreatment is a crucial step for the efficient saccharification of the biomass through enzymatic hydrolysis. Several methods such as mechanical, biological, chemical, and thermal (*i.e.*, steam) pretreatments have been proposed to improve the digestibility of the lignocellulosic material. Mechanical combination utilizes chipping, grinding, and milling processes to reduce the cellulose crystallinity and size. However, there are limitations in this pretreatment, because the power consumption is usually higher than inherent biomass energy (Kumar *et al.* 2009). Alternatively, biological pretreatment utilizes microorganisms to treat the lignocelluloses to enhance enzymatic hydrolysis. The applied microorganisms usually degrade the lignin and the hemicellulose while leaving the cellulose intact, since it has more resistance to the biological attack. However, the rate is very low in most biological pretreatment processes (Taherzadeh and Karimi 2008). Additionally, chemical pretreatment such as ozonolysis, utilize ozone to reduce the lignin content of lignocellulosic wastes. Unlike another chemical treatment, it does not produce toxic residues. This technique degrades most of the lignin and some of the hemicelluloses, while leaving the cellulose intact; however, it requires a large amount of ozone, which is expensive (Kumar et al. 2009).

Steam pretreatment is a new suggested pretreatment method that is available at palm oil mills. There are two types of steam treatment – superheated steam treatment and high-pressure steam treatment. Superheated steam treatment is a form of unsaturated steam produced by the addition of heat to saturated steam. In this treatment, the heat helps the saturated steam temperature to exceed the boiling point of the liquid at a certain pressure value (Bahrin et al. 2012). However, superheated steam treatment requires longer treatment times of 20 to 80 min. and only produces one type of byproduct, which is the pretreated solid. Meanwhile, high-pressure steam treatment (HPST) utilizes highpressure steam that is available at palm oil mills (Baharuddin et al. 2012). Interestingly, high-pressure inputs can be supplied by the excess steam generated at the palm oil mill. In addition, the high-pressure steam treatment has advantages of less treatment time (2 to 8 min.), as well as the production of both pretreated OPMF liquid and solid, in contrast to superheated steam treatment. The HPST loosens the lignocellulosic structure of the OPMF, which increases its accessibility towards enzymatic saccharification. Therefore, the objective of the present work is to investigate the effects of high-pressure steam treatment on the physicochemical changes of oil palm mesocarp fibers (OPMF).

## EXPERIMENTAL

#### Materials

Oil palm mesocarp fibers (OPMF) were obtained from Seri Ulu Langat Palm Oil Mill (Dengkil Selangor, Malaysia). About 10 g of OPMF was dried at 60 °C for 24 hours prior to the treatment.

## **High-Pressure Steam Pretreatment**

The high-pressure steam treatment (HPST) of OPMF was conducted in a 500 mL high-pressure autoclave (START 500, Nito Kuatsu, Co. Ltd, Japan) equipped with temperature and pressure control systems. The autoclave has the ability to reach temperatures up to 250 °C and pressures up to 9.4 MPa. The treatment temperature and pressure were 170 °C / 0.82 MPa, 190 °C / 1.32 MPa, 210 °C / 2.03 MPa, and 230 °C / 3.00 MPa, respectively, and the treatment time was for 2 minutes. At the end of each

treatment, one exhaust valve of the autoclave was opened to release the steam. Afterwards, the treated OPMF samples were oven dried at 105 °C for 24h. Changes in surface texture and color of the treated OPMF were examined.

## Scanning Electron Microscope (SEM)

Surface textures of the raw and the pretreated OPMF samples were observed utilizing a scanning electron microscope (SEM) (S-3400N, Hitachi, Japan) at an accelerating voltage of 10 kV. The samples were air dried and coated with gold-palladium in a sputter coater (E-1010, Hitachi, Japan).

## Fourier Transform Infrared (FTIR)

The Fourier transform infrared (FTIR) spectra were recorded on a Perkin Elmer GX2000R infrared spectrophotometer in a range of 500 to 4000 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>. Reflection spectra of the OPMF samples were measured on a Golden Gate Diamond attenuated total reflectance (ATR) (10500) module with a germanium crystal, by the single-reflection ATR method.

## Thermogravimetric and Differential Thermal Gravimetric Analysis (TG/DTG)

A thermogravimetric analyzer (TG/DTG 6200, SII NanoTechnology Inc, Japan) was used to investigate the mass loss of the samples. The treated samples were ground into powdered form and weighed (5 to 7 mg) for each analysis in order to avoid heat and mass transfer limitations. During TG/DTG analysis, the samples were heated from 30 to 550 °C at a heating rate of 10 K/min. Purified nitrogen was flushed at a flow rate of 100 ml/min. to provide an inert atmosphere for thermal decompositions.

## **RESULTS AND DISCUSSION**

## Physicochemical Properties of Treated OPMF

Figure 1 shows the physical appearance of the untreated and the treated OPMF at different treatment conditions of 170 °C / 0.82 MPa, 190 °C / 1.32 MPa, 210 °C / 2.03 MPa and 230 °C / 3.00 MPa. It can be observed that the initial colour of the untreated OPMF was slightly brown. However, when the samples were exposed to the high-pressure steam treatment at 170 °C (Fig. 1b), the samples turned brown and became darker. In addition, the colour of the samples remained unchanged until the treatment temperature of 210 °C (Fig. 1d).

As the treatment temperature increased from 210 °C to 230 °C (Fig. 1e), the sample colour became darker and smelled of "burnt sugar." In addition, the treated samples also became brittle and partially broken when compressed in the hand. This is similar to previous findings by Bahrin *et al.* (2012), who examined the superheated steam treatment of oil palm empty fruit bunch (OPEFB). According to Bahrin *et al.* (2012), increasing the superheated steam temperature (>210 °C) reduced the mechanical strength of OPEFB material. Moreover, it has been suggested that colour changes were probably related to the chemical breakdown of lignin and wood extractives at high temperatures (Negro *et al.* 2003).

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**(e)** 

**Fig. 1.** Physical appearance of high-pressure steam treatment (HPST) of raw OPMF (a) and treated OPMF with various treatment conditions for 170 °C/ 0.82 MPa (b), 190 °C/ 1.32 MPa (c), 210 °C/ 2.03 MPa (d), and 230 °C/ 3.00 MPa (e) for 2 min. treatment time.

The significant colour changes that occurred during the pretreatment were due to the effect of high pressure and temperature. Sun *et al.* (2005) noted that after steam treatment of wheat straw, brownish products resulted from the degradation of carbohydrates caused by the high steam temperatures. Negro *et al.* (2003) and Sun *et al.* (2005) also proposed that the saturation component of colours increased after pretreatment, suggesting changes in chromophore groups and the appearance of brownish products in treated samples. This may be related to "pseudo-melanoidins" formation from the products of sugar degradation that are produced at high pretreatment temperatures. Meanwhile, another factor that contributes to the significant colour changes of samples is the partial solubilization of lignin. Excoffier *et al.* (1991) stated that lignin slowly softens and depolymerizes under heat and solubilizes a small part of the original lignin. Furthermore, Sampedro *et al.* (2011) reported that the darkening of the treated chips after steam explosion treatment suggested that some kind of reaction had occurred.

## Scanning Electron Microscopy (SEM)





**(a)** 

**(b)** 



(c)

(**d**)



**(e)** 

**Fig. 2.** SEM micrograph of raw OPMF (a), treated with various treatment conditions of HPST at 170  $^{\circ}$ C / 0.82 MPa (b), 190  $^{\circ}$ C / 1.32 MPa (c) , 210  $^{\circ}$ C / 2.03 MPa (d), and 230  $^{\circ}$ C / 3.00 MPa (e) for 2 min. Object in the box indicates the present of silica bodies.

The morphological characteristics of oil palm mesocarp fiber (OPMF) at different temperature conditions are shown in Fig. 2. There were obvious changes between the surface morphology of the untreated and the treated fibers. Initially the surface of the untreated fibers appeared rough and rigid. In addition, the fibers of the untreated samples were found to be arranged in the highly ordered fibrils. Meanwhile, silica bodies were embedded along the inner structure of OPMF samples. According to Law *et al.* (2007), the silica bodies at about 10 to 15  $\mu$ m in diameter were spread uniformly over the strand surface. This finding was similar with the previous study in which silica bodies were observed on the surface of oil palm empty fruit bunch (OPEFB) (Bahrin *et al.* 2012 and Baharuddin *et al.* 2012).

Moreover, for sample fibers treated at 170 °C (Fig. 2b), no significant changes were observed. The outer layer of the sample fibers was not disrupted and silica bodies remained attached. Nevertheless, in Fig. 2c, there were a few changes on the OPMF surface where some of silica bodies were removed – the surface was visibly separated and more defined when compared to Fig. 2b. This indicates that high pressure and temperature had altered the structure of the OPMF. When the temperature increases above 150 to 180 °C, parts of the lignocellulosic biomass, firstly the hemicelluloses and shortly after that the lignin, will start to solubilize (Garrote *et al.* 1999).

In addition, as treatment temperature was raised from 210 °C to 230 °C (Fig. 2d and 2e), the surface of the OPMF appeared more uniform and smoother. At high treatment temperature (> 120 °C), the surfaces of fibers had quite similar features in which most of the outer layers were almost completely disrupted and no silica bodies were observed. This indicated that sufficient energy was achieved to modify the lignocellulosic components and remove the silica bodies. As the silica bodies are removed from the OPMF surface, the accessibility of the enzyme through the internal cellulose layers in the lignocellulosic structure will increase (Dietrich *et al.* 2003). It was concluded that the high-pressure steam treatment (HPST) had successfully altered the lignocellulosic structure and solubilized the hemicelluloses at treatment temperatures above 210 °C.

## **FTIR Spectral Analysis**

Infrared spectroscopy is frequently used for investigating the functional groups and the chemical changes of lignocellulosic materials during delignification (Zhao *et al.* 2010). Figure 3 presents the FTIR spectra comparing the raw and the treated OPMF with high-pressure steam treatment (HPST) for 2 min. treatment time. The results of FTIR spectroscopy showed the most significant changes at wave numbers of 1730 cm<sup>-1</sup>, 1645 cm<sup>-1</sup>, 1516 cm<sup>-1</sup>, 1505 cm<sup>-1</sup>, 1246 cm<sup>-1</sup>, 1232 cm<sup>-1</sup>, and 1000 to 1200 cm<sup>-1</sup> range.

From Figure 3, the intense peak of carbonyl band (C=O) at 1730 cm<sup>-1</sup> in the OPMF represented aldehyde, ketone, or carboxylic acids in the hemicelluloses, which became weaker and disappeared as the treatment temperature increased. In our hypothesis, unstable compounds containing double bonds, such as aldehyde, ketone, and carboxylic acid, will decompose into stable single bonds. These factors were caused by the high temperature and pressure conditions of the treatment. In addition, absorption at 1232 cm<sup>-1</sup>, which was attributed to the hemicellulose acetyl groups, disappeared as the treatment temperature increased. Hemicellulose within the biomass is attached to the lignin and the cellulose by covalent bonds, with a few hydrogen bonds; these bonds are easily broken down compared to the bonds associated with crystalline cellulose (Jacobsen and Wyman, 2000). These results suggested the removal of a large portion of

hemicelluloses as the HPST temperature increased from 170 °C to 230 °C. According to Hideno *et al.* (2009), the solubilization of the hemicellulose increased as the mean pore size of the substrate increased, resulting in higher accessibility to the cellulose and in higher xylose yields.



Fig. 3. Peak wavenumbers of bands for raw and treated OPMF with HPST for 2 min. of treatment time

The band at 1645 cm<sup>-1</sup> was assigned to the bending mode of the adsorbed water associated with cellulosic fiber. The 1516 cm<sup>-1</sup> absorption was assigned to the aromatic skeletal vibration. The bands at 1505 cm<sup>-1</sup>, which referred to the aromatic ring of lignin, increased significantly with the treatment temperature, particularly at 230 °C. Pandey (1999) suggested that the range 1590 to 1600 cm<sup>-1</sup> wave numbers corresponded to the characteristic bands of an aromatic skeleton, such as that of lignin. The increase of lignin and cellulose content contributed to the disappearance of hemicelluloses, where lignin and cellulose were released and re-deposited on the surface of the lignocellulosic material (Kristensen et al. 2008). The absorption at 1420 cm<sup>-1</sup> increased when utilizing higher steam temperature. According to Bahrin et al. (2012), important bands used to identify the cellulose component are 1420 and 1430 cm<sup>-1</sup>, which correspond to the amorphous/ crystallize cellulose structure. Meanwhile, the absorption at 1246 cm<sup>-1</sup> corresponded to the characteristic of polysaccharides such as cellulose, which were present in the samples. The increase in the intensity of this peak showed an increase in cellulose content after the pretreatment. In Fig. 3, the peak around 1000 to 1200 cm<sup>-1</sup> has been ascribed to the C-O, C-O-C stretching, and C-OH linkages in cellulose and lignin. It becomes visible with increasing temperature treatment and most significantly at 230 °C. In conclusion, the changes of components in the OPMF samples after the steam treatment were expected to be an advantageous approach for further bioconversion processes.

Wavenumbers location (cm <sup>-1</sup> )	Vibration	Functional group	Component	References
1730	C=O	Aldehyde, ketone, carboxylic acids	Hemicellulose	Kristensen <i>et al</i> . 2008; Tandy <i>et al</i> . 2010
1648	O-H	Absorbed water molecules associated with cellulosic fiber		Xiao <i>et al</i> . 2001; Himmelsbach et <i>al.</i> 2002
1590-1600	C=C	Aromatic skeleton	Lignin, Cellulose	Pandey 1999; Zhao <i>et</i> <i>al</i> . 2010
1516		Aromatic skeletal vibration		Bahrin <i>et al.</i> 2012; Wang <i>et al</i> . 2009
1505		Aromatic ring	Lignin	Xiao <i>et al</i> . 2011
1430			Cellulose	Bahrin <i>et al</i> . 2012
1246	C-O-C,	Polysaccharides	Cellulose	Wetzel <i>et al</i> . 2001
1235	COOH	Carboxyl group, Acetic acid esters	Hemicellulose	Smidt <i>et al</i> . 2005
1000-1200	С-О-С, С-О, С-ОН	Polysaccharides	Cellulose, Lignin	Yang <i>et al</i> . 2007

#### Thermogravimetric and Differential Thermal Gravimetric Analysis (TG/DTG)

The TG and DTG curves of the untreated OPMF and the treated OPMF at various treatment conditions of 170 °C/ 0.82 MPa, 190 °C / 1.32 MPa, 210 °C / 2.03 MPa and 230 °C / 3.00 MPa are shown in Fig. 4. From the analysis of thermogravimetric curves (Fig. 4a) it can be observed that the biomass degradation took place in two steps: the first step was weight loss, which occurred in the temperature range of 0 to 280 °C. This is due to the vaporization of moisture content and unstable functional groups in the samples that contribute to about 5% weight loss. Then the second step occurred at temperatures above 290 °C; the changes were attributed to the removal of heat-instable factors by the steam treatment, such as hemicelluloses and cellulose. The TG curve of weight loss for all the samples became significant with the increase in temperature. The curves at this stage were drastically reduced, showing about 40 to 60% of weight loss. This implied that the treated samples had higher thermal stability compared to the untreated samples. Meanwhile, untreated samples contained unstable compounds that were volatile with the increasing of temperature. Thermal degradability was influenced by the chemical composition of the material, because different components of a lignocellulosic biomass have different thermal behaviors (Hideno et al. 2007). According to Wang et al. (2009), hemicelluloses were the easiest components to be removed from the main stem and were easily volatile at low temperature due to their amorphous structure, which is rich in branches and consists of various saccharides that appear in a random organization.



**Fig. 4.** TG and DTG data comparison of raw and treated OPMF with various treatment conditions for 2 min. of treatment time

The DTG profiles (Fig. 4b) for the rate of weight loss of OPMF samples show three distinct peaks. The degradation of components in the samples took place in three stages. The initial mass loss at 0 to 100 °C was due to the moisture content present in the sample. It was observed that the untreated samples contained the largest amount of moisture compared to the treated samples. Second stage occurred in the temperature range 200 to 315 °C, where the decomposition of hemicelluloses was identified. Lastly,

the third stage occurred in the temperature range 315 to 400 °C, where cellulose and lignin degradation took place. It was observed that the peaks of DTG distribution were likely to move rightward to a higher temperature after the treatment. These profiles indicate that the thermal decomposition of cellulose and lignin shifted to a higher temperature. However, lignin was the most difficult to decompose with a maximum decomposition rate at 456 °C (Xiao *et al.* 2011). According to Yang *et al.* 2007, the easiest component to be removed was hemicellulose, with degradation occurring at 220 to 315 °C, followed by cellulose at range 315 to 400 °C, and lastly, lignin at maximum range 160 to 900 °C.

These findings were also supported by SEM and FTIR analysis results that suggested the degradation of hemicellulose and removal of some silica bodies were due to the effect of high-pressure steam treatment on the lignocellulosic materials of the samples. Therefore, results of this study suggested that high-pressure steam treatment is efficient treatment for the alteration of lignocellulosic materials with optimum conditions of 230 °C and 3.00 MPa.

## CONCLUSIONS

- 1. The physicochemical, surface chemistry and thermal degradation characteristics of raw and treated oil palm mesocarp fibers (OPMF) with high-pressure steam treatment were studied.
- 2. The raw and treated samples were analyzed using SEM, FTIR, and TG/DTG to investigate the changes that occurred in the lignocellulosic materials of OPMF. Results from this study show that after high-pressure steam treatment, changes in compositions, properties, and behavior of lignocellulosic materials occurred in the OPMF samples.
- 3. Based on the results obtained, high-pressure steam treatment at 230 °C / 3.00 MPa were the optimal conditions for the efficient modification of lignocellulosic materials in the OPMF. Therefore, it can be concluded that the HPST is an attractive approach for the treatment of lignocellulosic materials because it can be directly implemented at palm oil mills and can successfully alter lignocellulosic materials in the OPMF.

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