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EFFECT OF TREATMENT ON THE OIL CONTENT AND SURFACE MORPHOLOGY OF OIL PALM (*ELAEIS GUINEENSIS*) EMPTY FRUIT BUNCHES (EFB) FIBRES

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ABSTRACT

The effect of sodium hydroxide (NaOH) and acetic acid treatment at different concentration levels on the morphological structure of oil palm empty fruit bunches (EFB) fibres were investigated. The effects of the treatments on the surfaces of EFB fibres subjected to thermo-mechanical pulping (TMP) were observed using scanning electron microscopy (SEM). The residual oil content of the EFB refined fibres was also determined. The results indicated that both treatments had successfully removed the residual oil, with NaOH treatment removing more oil than acetic acid treatment. However, both treatments resulted in different surface characteristics. The acid treatment resulted in rougher fibre surface and more removal of silica bodies. Results showed that 0.4 of NaOH and 0.6 % of acetic acid were good enough to remove the oil and impurities. The acetic acid treatment led to better surfaces than NaOH.

KEYWORDS: Empty fruit bunches (EFB), sodium hydroxide (NaOH) treatment, acetic acid treatment, silica bodies, fibre surface characterization.

INTRODUCTION

Oil palm is one of the main agricultural crops in Malaysia with oil as the main product. In 2013, the oil palm plantation covered about 5.23 hectare (Anon 2013) and these huge area generated abundant amount of biomass per year in the form of trunk, frond, pressed fruit fibre (PFF), palm oil mill effluent (POME) and empty fruit bunches (EFB) (Salleh et al. 2007). Abdul Khalil et al. (2010) found that, the oil palm industry in Malaysia produced over 11.9 million tons with 100 million tons of biomass. From the total of oil palm biomass generated, EFB contributed about 30.5 % from the oil palm biomass (Shuit et al. 2009).

EFB is the fibrous mass left behind after separating the fruit from fresh fruit bunch (FFB), generated from oil processing. The EFB consist of a bunch of fibres left after the oil extraction. The EFB fibre is hard, tough, and show similarity to coir fibre in term of cellular structure (Sreekala et al. 1997). In addition, Law et al. (2007) found large quantities of silica bodies attached to the surface of EFB fibres with the remaining oil, and this may affect the properties of the composite materials.

The EFB fibres contain about 4.5 % of residual oil (Abu Bakar et al. 2006) and this oil forms a layer on the outer surface of the EFB fibres (Rozman et al. 2001). Many researchers (Ridzuan et al. 2002; Paridah and Zaidon 2000a; Nor Yuziah et al. 1997; Kobayashi et al. 1985) claimed that the residual oil interrupts the penetration of binding agent and affect the properties of the final EFB-products.

Many studies were carried out on the utilization of the EFB fibres in particleboard, medium density fibreboard (MDF) and composites (Rozman et al. 2005; Ridzuan et al. 2002; Koh and Yeo 2000; Paridah et al. 2000b). Kobayashi et al. (1985) found that residual oil deteriorate the product properties. Thus, removing the residual oil is necessary in order to improve the final product properties. Sreekala et al. (2002) reported that EFB fibre cannot withstand heavy load thus lead to the composite failure. On the other hand, EFB fibre is hydrophilic due to the large numbers of hydroxyl groups from the cellulose and hemicelluloses in the cell wall. Besides, the present of residual oil and silica in the EFB fibre affected the fibre properties and eventually the composites.

In order to remove the residual oil in EFB, fibre pre-treatment is necessary to clean the fibre surface, remove impurities, remove certain amount of lignin, wax and oils covering the external surface, depolymerise the native cellulose structure, improve the bonding, lower the moisture absorption and modify the fibre. The treatment can be physical, thermal, mechanical or chemical, and it can be done before (pre-treatment), during or after (post-treatment) fibre processing.

There are various methods of treatments that can be used for the removal of silica bodies from the surface of EFB fibres; heat treatment, chemical treatment and mechanical treatment. Yunus et al. (2010) treated the EFB fibres by using a combination of heat and chemical treatment; acid hydrolysis at 100°C and ultrasonic pre-treatment. On the other hand, Rosman et al. (2013) treated the EFB using alkali followed by silane treatment and found that the surface treatment of EFB fibres increases the compatibility with the matrix thus producing superior mechanical properties of the reinforced polymer composite. The modifications of EFB fibres treated by sodium hydroxide and succinic acid increase the availability of functional groups through chemical modification, and interact strongly with the matrix polymer to get better interfacial bonding between fibres and matrix (Bhat et al. 2011).

This study focused on treating EFB fibres with different types of chemicals at different concentration levels. The EFB fibres were treated with sodium hydroxide (NaOH) and acetic acid to study the properties of the fibre surface.

MATERIAL AND METHODS

Oil palm EFB were obtained from Malaysian Palm Oil Board (MPOB). The EFB were shredded to strand sizes of 10 to 20 cm via chipping.

Fibre treatment and production

The strands were soaked in different concentration levels of NaOH and acetic acid (0.2, 0.4, 0.6 and 0.8 %) for 24-hours. The NaOH and acetic acid were supplied by a local chemical supplier. The strands were later washed off any impurities and subjected to thermo-mechanical pulping (TMP) using Sprout-Bauer (ANDRITZ) refiner at an MDF pilot plant located in MPOB/Universiti Kebangsa no space an Malaysia (UKM) Research Station. The refined fibres were air dried for a few hours followed by oven-drying for 24-hours.

Residual oil determination

The oil content in the EFB after treatment and refining were determined by soxhlet extraction method. For each types of treatment, about 5g of dried EFB fibres were weighed and placed in the extraction thimble in the soxhlet extractor. The extraction was carried out at a temperature of 140°C. The chemical reagent used to extract the oil residues was hexane. The extraction process was done for about 4 hours. Then, the flask containing residual oil together with hexane and extracted fibres were placed in an oven set at 103 ± 2°C for 1 hour to allow for complete evaporation of the hexane followed by cooling stage where the fibres were placed in a desiccator over silica gel. The collected extract consisted of concentrated extract which contained the remaining oil.

SEM *examination*

The surface properties of EFB fibres were examined using variable pressure scanning electron microscope (SEM). Hitachi 3400 SEM was used to study the effect of the treatments on the fibre surface. All samples were sputter-coated with gold with the acceleration voltage set at 15 kV. The surface morphology of the fibre was observed.

RESULTS AND DISCUSSION

Oil content

The average values of oil content of treated fibres are presented in Fig. 1. In general, the oil content was reduced with increased concentration of chemical used in both treatments. Treatment using NaOH resulted in more reduction of oil content in the fibres after refining process compared the treatment using acetic acid. Untreated EFB fibres contain about 0.50 % of residual oil after refining.

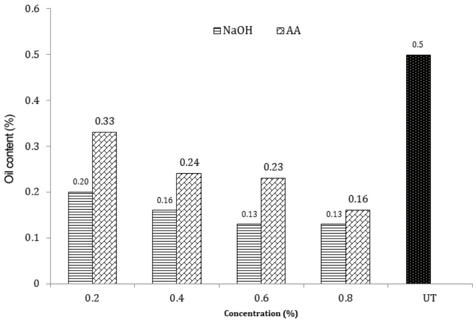


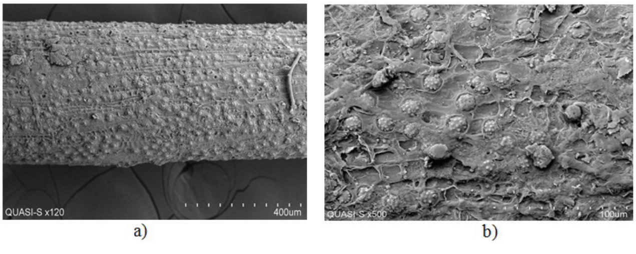
Fig. 1: The oil content of treated empty fruit bunch (EFB) fibres.

The fibres treated with NaOH had distinctly lower oil content after going through the fibre treatment with the amount of oil content lesser at higher concentration of NaOH compared to lower concentration. The introduction of NaOH during soaking induce swelling of the fibres through the activation of -OH groups on the cellulose structure, and revealing chemically reactive functional groups (Sreekala and Thomas 2003). The hydrogen bonding between cellulose molecules were broken and replaced by Sodium ion and formed water as by-products along the impurities. Being a swelling agent, NaOH is able to penetrate the crystalline region, thus more hydrogen bonding were broken, resulted in more impurities including oil that were removed. Mwaikambo et al. (2007) suggested that treatment using NaOH, certain portion of hemicellulose, lignin, pectin, wax and oil that covered the fibres were partially removed together with hydroxyl groups. This observation was more severe at higher concentration of NaOH as indicated by the low oil content.

Results also indicated that treatment using acetic acid removed the oil off the fibres; oil content decreased with concentration significantly at 0.6 and 0.8 %. The dissimilar reaction with NaOH occurred in acetic acid treatment, where the cell wall hydroxyl groups were substituted with acetyl groups, rendering the surface more hydrophobic. The hydroxyl groups react with lignin, hemicellulose and other substances including wax and oil (Rowell et al. 1996). The presence of oil and waxy substances cover the reactive functional groups, thus acetic acid used in the treatment removed these substances and exposed more reactive groups (Dash et al. 2000).

Scanning electron microscope (SEM) analysis

Scanning electron microscope (SEM) images of untreated EFB fibres is shown in Fig. 2. It can be clearly seen that silica bodies were found embedded on the fibre surface. Substances which



a) b)

Fig. 2: Scanning electron microscope (SEM) view of untreated empty fruit bunch (EFB) fibre: at a) 120 magnification and b) 500 magnification.

may include oil and other impurities covered the surface of untreated fibre. A similar result was obtained in a studies conducted by Bahrin et al. (2012) and Baharuddin et al. (2011) who found that the silica bodies are found in the untreated EFB structures.

For fibres treated with NaOH, the SEM images show that alkali treatment made the fibre surface rougher with less amount of silica bodies. Most of the lignin and small amount of silica bodies were removed resulting in a rough surface. The SEM images of fibre treated with 0.2 and 0.4 % of NaOH (Figs. 3 a-d) show that the silica bodies were still attached to the fibre surface indicating that at higher concentration of NaOH is required to effectively remove the silica bodies and other substances.

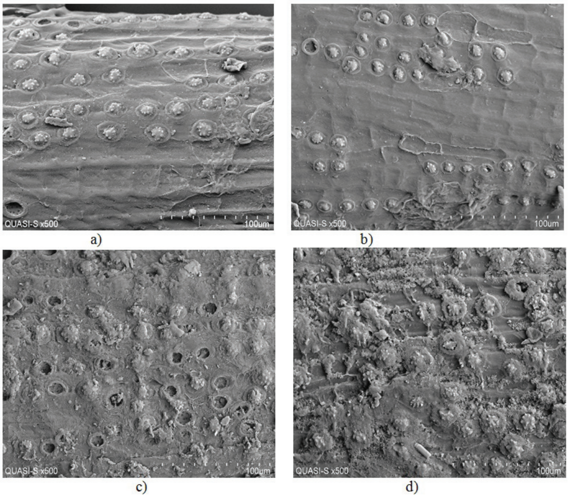


Fig. 3: Scanning electron microscope (SEM) view at 500 magnification of empty fruit bunch (EFB) fibre : a) 0.2 % NaOH treated fibre, b) 0.4 % NaOH treated fibre, c) 0.6 % NaOH treated fibre and d) 0.8 % NaOH treated fibre.

For the treated EFB fibre at 0.6 % of NaOH (Fig. 3c), some of the silica bodies had been removed from the EFB structure and some pores were exposed on the fibre surface. The pores became more prominent on the surface of the fibre after alkali treatment as obtained by Sreekala et al. (2007), who found that some silica were removed after being treated with NaOH, and the pores were found on the fibre surface with average diameter of 0.15μm. They also claimed that the porous surface morphology is useful for better mechanical interlocking with the matrix. However, further addition of the NaOH concentration to 0.8 % (Fig. 3d) resulted in the surface of the fibre to become coarse and uneven with some deposition of several substances, believed to be the residue from the fibre degradation due to the higher NaOH concentration. Fig. 4 showed some silica bodies still embedded but some of that was removed, thus leaving pores on the surface of the fibres. If the NaOH concentration was higher than the optimum condition, excess delignification of fibre takes place, resulting in damage of the fibre (Wang et al. 2007).

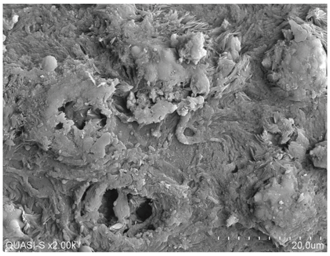


Fig. 4: Scanning electron microscope (SEM) view of empty fruit bunch (EFB) fibre at 0.8 % NaOH treated fibre at 2000 magnification.

The surface morphology of EFB fibres treated with acetic acid is given in Figs. 5 a-d. After being treated with acetic acid, the surface becomes rougher and more silica bodies were removed compared to the NaOH treated fibre surface. More voids resulted from the removal of silica bodies could be noticed on the surface of fibre treated at 0.6 % acetic acid (Fig. 5c). The removal of silica bodies revealed that the bottom of the silica crater is perforated and it would enhance the chemical penetration in pulping (Law et al. 2007). Nascimento et al. (2012) stated that the silica bodies contributed to the strength and rigidity of EFB fibre. However, removal of this silica bodies is believed to open up the siliceous pathway and expose more amorphous region of the fibres (Omar et al. 2014).

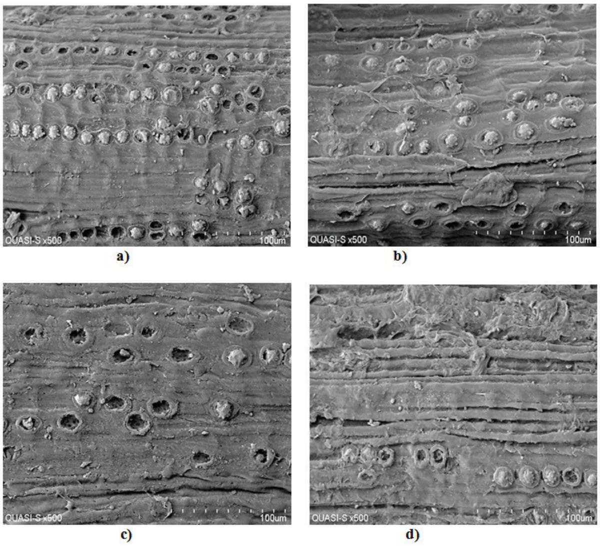


Fig. 5: Scanning electron microscope (SEM) view at 500 magnification of empty fruit bunch (EFB) fibre: a) 0.2 % acetic acid treated fibre, b) 0.4 % acetic acid treated fibre, c) 0.6 % acetic acid treated fibre and d) 0.8 % acetic acid treated fibre.

Further additional concentration of acetic acid at 0.6 (Fig. 5c) and at 0.8 % (Fig. 5d), produced a rougher surface with some cleavage line on the surface. The condition of fibre surface treated with acetic acid at 0.8 % concentration, the cleavage became more prominent and severe delamination of fibres take place resulting in a rougher surface. Many studies claimed that increasing surface roughness results in better mechanical interlocking that improved adhesion ability of fibre with the matrix (Bahrin et al. 2012; Karthikeyan and Balamurugan 2012; Sreekala et al. 1997). In addition, surface cracks (Fig. 6) were visible at EFB fibres treated with acetic acid at 0.8 % concentration suggesting that the fibre surface experienced severe treatment, thus exposing more surface area and increase porosity.

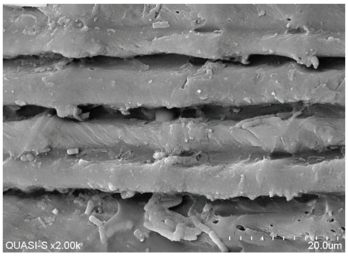


Fig. 6: Scanning electron microscope (SEM) view of empty fruit bunch (EFB) fibre at 0.8 % acetic acid treated fibre at 2000 magnification.

CONCLUSION

Based on the results from this study, it can be concluded that:

1. The chemicals and the concentration levels have a great effect on the residual oil content and the surface morphology of treated refined fibres. Treatment of the EFB fibres with NaOH and acetic acid resulted in different properties of both fibres.
2. Treatment using NaOH successfully removed more residual oil from the EFB surface compared to those treatments using acetic acid.
3. Acetic acid treated fibre surface were rougher than NaOH fibre surface that resulted from more removal of silica bodies, thus revealing more pores.
4. At higher concentration, NaOH fibre surface becomes uneven and contains some substances deposited on the surface, while the acetic acid surface have clear cleavage lines and cracks formation were also observed.

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