Influence of Ultrasound on Alkaline Treatment of Empty Fruit Bunch Fibre: Preliminary Study

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Abstract. This research underlines the effect of ultrasound in NaOH surface modification of empty fruit bunch (EFB) fibre. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) were used to characterized the fibres. It can be seen that surface morphology treated by ultrasound assisted alkali treatment shows smooth surface with minor impurities. Additionally, the elimination of hemicellulose on EFB fibre surface can be proven by the disappearance of peak between 2900-1700 cm^{-1} (C-H). Furthermore, Ultrasound assisted alkali method demonstrate the admirable value (300 N/mm^2) in enhancing the tensile stress of EFB fibre and comparable with alkali soaking technique (292 N/mm^2). The findings indicate that ultrasound-assisted alkali treatment has the potential to be used as surface modification method in the industry.

1. Introduction

In Malaysia, there are more than 5,000,000 hectares of oil palm were cultivated and making the country world's largest exporter of Empty Fruit Bunch (EFB) fibre is one of the lignocellulose biomass waste from palm oil industry, conventionally it used as energy sources in boiler [1]. EFB fibres have the properties like light weight, nonabrasive and biodegradable which suitable to be used as reinforcing agent for plastic and composite application [2]. However, hydrophilic native property of EFB fibre leads to weak interface due to poor interfacial adhesion between EFB fibre surface and polymer matrix [3]. In order to overcome the drawbacks, surface treatment of EFB fibre is required such as physical and chemical treatments, to modify the surfaces by increasing surface roughness and remove impurities. Moreover, the surface modification treatment could enhance the number of hydroxyl groups active side or incorporate new functional groups, which enhance the bonding ability between fibre surface and matrix.

Ultrasound assisted method could further improvise the mechanism of surface modification, where ultrasound creates strong and irregular environmental chemistry in the solution through the generation of numerous tiny cavities that possible to expand and implode [4]. As a series of ultrasonic waves are produced to flow through the solution, this phenomenon creates a significant amount of heat. Ultrasound

technique can lead to fibre refining, regulated residual compressive stress and fibre size reduction, all of which are beneficial for improving mechanical strength of natural fibre reinforced materials. The influence of ultrasound involves both physical and chemical effects [5].

This study emphasized on morphology of different EFB fibre treatment through Scanning Electron Microscopy (SEM). Fourier Transform Infrared Spectroscopy (FTIR) technique was used to investigate the ultrasound influence in EFB fibre alkaline treatment. Furthermore, the fibre strength was measured by using universal testing machine and tensile strength was recorded.

2. Materials and method

2.1. Materials

EFB fibre were collected from Kilang Sawit Panching, Kuantan, Malaysia. Other notable chemicals which were used include analytical grade sodium hydroxide and acetic acid were purchased from Permula Chemical Sdn. Bhd.

2.2. EFB fibre sample preparation

The collected EFB fibre was washed with deionized water for several time and sun dried for 24 hours. EFB fibre treatment was performed by modification with ultrasound alkaline treatment. The fibres were weighed and placed in a beaker containing 3 wt% NaOH solution and maintain a fibre-to-water ratio of 1:20. The beaker and its contents were put inside the ultrasonic bath (GT-SONIC Ultrasound) and the method was carried out at room temperature 120 min. In order to extract excess alkali, the treated fibres were thoroughly washed. As washing continued, a few drops of very dilute acetic acid solutions were applied until signs of alkalinity were no longer shown. The treated EFB fibres were further oven dried for 2 hours at 60°C to obtain ultrasound treated EFB fibres (UEFB). For further research, the samples were placed in plastic bags. As for soaking treatment of EFB fibre, the raw sample of EFB fibre was soaked in 3wt% NaOH solution for 12 hours at room temperature. The soaked EFB fibre (SEFB) was then oven dried with the same temperature and time applied on ultrasound treatment EFB fibre.

2.3. Characterization of EFB fibre using SEM

Surface morphologies of non-treated EFB fibre (NEFB), SEFB and UEFB were analysed with scanning electron microscopy (ZEISS, EVO 50, Germany). Before observation under the SEM instrument, samples were coated with platinum using a sputter-coater.

2.4. FTIR analysis of EFB fibre

Bonding structure for NEFB, SEFB and UEFB was analysed through FTIR analysis (Model-Thermo scientific Nicolet 6700, Germany). The analysis was done using the standard KBr method and range of wavenumber was taken from 4000-700cm^{-1}.

2.5. Tensile test of EFB fibre

The tensile strength test for EFB fibre was conducted based on the ASTM standard (ASTM D3379) for single fibre. The preparation of test specimen was setup as per shown in Figure 1. The specimen was mounted on the testing jig and apply the load to obtain the breaking load henceforth calculate the tensile value. The Instron Universal Testing Machine with a 10 kN load-cell was employed in this test. The EFB fibre used in this test were selected based on hydration temperature category and surface morphological change. Three samples have been selected for tensile study (NEFB, SEFB and UEFB). The single fibre was glued to a cardboard frame shown in Figure 1. The process of sticking/glue EFB on cardboard conducted with carefully by placing a small amount of adhesive (epoxy) at the marks on the mounting cardboard that define the gauge length. The test specimens were gripped/clamped to the load train so that the test specimen is aligned axially along the line of action of the test machine.

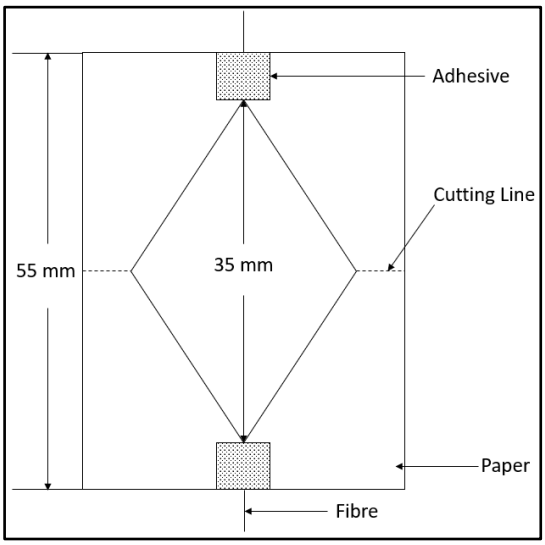

Figure 1. Tensile test experimental setup sketching diagram [6]

3. Results and Discussion

3.1. Characterization of EFB fibre using Scanning Electron Microscopy (SEM)

Figure 2. (a) SEM for NEFB, (b) SEM for SEFB and (c) SEM for UEFB.

Scanning electron microscopy is an effective method for studying fibre surface morphology and fibre composite surface fractures. Morphological changes that occurred before and after fibre treatment were investigated in this report. Figure 2a shows NEFB under SEM micrograph, one can see that it's contain wax, impurities, fatty substances and globular protrusions called ''tyloses''[7].

Compared to untreated ones, there were significant differences in fibre morphologies after NaOH soaking technique. Figure 2b visualise a significant change in the morphological structure of EFB fibres occurred in SEFB by soaking 3wt% NaOH for 12 hours. It can be seen that, the impurities on the surface such as wax, cuticle were washed away. As the binding materials are removed and some micropores emerge in the treated fibres, fibrillation has been found to occur. It was also confirmed that the removal of the surface layer was able to increase the contact area as the fibrils became more exposed. The fibre diameter is also reduced and the surface is rougher. For fibre-matrix adhesion, the removal of surface impurities on plant fibres is beneficial as it promotes mechanical interlocking and bonding reaction [8].

Besides that, UEFB surface morphology can be seen as irregular, coarse and with many exposed pore spaces on the surface, as shown in Figure 2c. This means that ultrasound treatment has been successful in extracting the cement material from the surface of the fibre. By eliminating the binding and sizing structures, cleaning of the fibre surface could have led to increased porosity and efficient fibre surface area by opening up the pore space. The opened spore shows that some structural changes occurred after ultrasound treatment on the fibre surface. This may be in the form of hydrogen bonding disturbances in the fibre structural framework. It can be seen that, morphology of UEFB have better quality compared to NEFB and SEFB.

3.2. FTIR Analysis

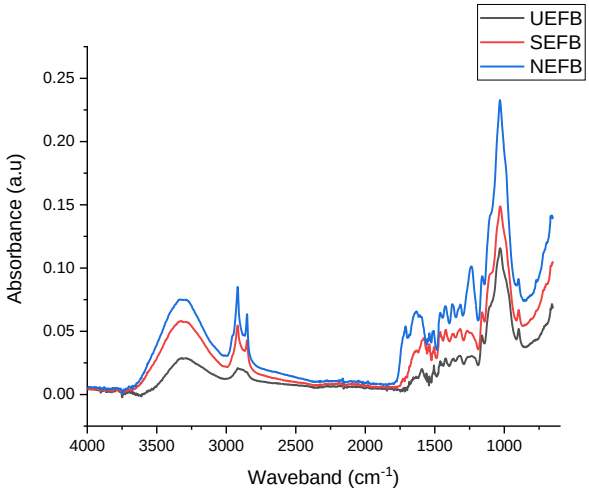

Figure 3. FTIR spectra on effect of treatment

The FTIR spectra of NEFB, SEFB and UEFB were illustrated in Figure 3. FTIR spectrum analysis determine that broad bands at 3500-3200 cm^{-1} and 2918-2916 cm^{-1}, correspond to the stretching vibration of hydrogen bonded O-H, N-H [9, 10] and C-H [11] respectively. The FTIR spectrum of NEFB shows four identical peaks appear at 2850, 1710, 1634 and 1374 cm^{-1}. The peak at 2850 cm^{-1} is due to C-H stretching in cellulose and hemicellulose. Band observe at 1710 cm^{-1} is from the absorption of carbonyl (C=O) stretching of fatty acids that exists in the fibres [12-14]. The peaks at 1634 cm^{-1} and 1374 cm^{-1} can correspond to the vibrations of the aromatic skeleton (=CH) and C-O stretch in lignin. [15]. The similar pattern has been reported by few other researchers [16, 17]. The bands ranging from 1323-1316 cm^{-1} may be assigned to bending of C-H and O-H [18]. The small bands detected at 900-896 cm^{-1} can be assigned from β-glucosidic linkages in hemicellulose and cellulose [19]. Both spectrum of SEFB and UEFB were identical in term of peak. It is noticed that, the peak at 2900-1700 cm^{-1} of SEFB and UEFB sample was disappeared which reveals the elimination of hemicellulose and fatty acid from EFB fibre after alkali technique [20, 21].

3.3. Tensile stress on EFB fibre

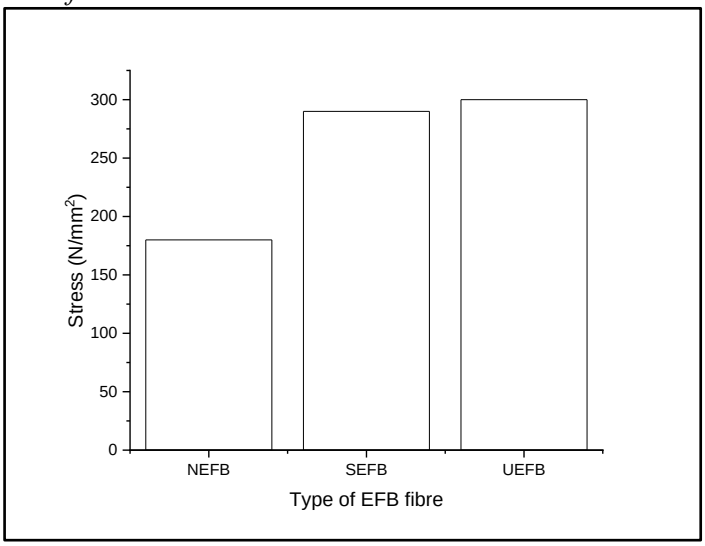

Figure 4. Effect of tensile stress on different treatment of EFB fibre

Effect of tensile stress on various treatment can be seen on Figure 4. The findings exhibit that the tensile strength of EFB fibres was substantially improved by alkali treatment. This might be due to the increased in crystallinity of fibres by alkali treatment. The findings are in agreement with Izani, Paridah [8], Li, Tabil [21], where reported that the natural fibre treated with NaOH exhibits higher tensile strength value than untreated fibre. Alkali treatment can depolymerize and excessively delignify the native cellulose fibre, which can adversely affect the strength of the fibre [22, 23]. It can be seen that, the tensile stress value of UEFB is comparable with SEFB. It is evidence from the morphology of the fiber, where UEFB is better than SEFB, less impurities were noticed. Similar finding was also reported by Islam, Beg [24], ultrasound method is better compared to soaking method in NaOH treatment. The treatment duration of UEFB is 6 times shorter compare to SEFB, but ultrasound treatment process is needed small amount of electricity for operation. It is indicating that, UEFB would be the potential candidate to be used as reinforcing agent in polymer matrix and apply in industrial scale.

4. Conclusion

The surface modification of EFB fibres was successfully prepared by different methods. The results of EFB morphology indicates that alkaline method possess cleaner surface compare to untreated fibre where most of the impurities were removed. FTIR spectra confirmed the removal of hemicellulose ranging from 2900-1700 cm^{-1} for samples of alkali treatment where the peak was depleted, while a significant peak was observed for untreated sample. Tensile stress indicate that alkaline treated fibre possesses higher tensile value compared to untreated fibre. Moreover, the tensile value of UEFB is slightly higher than SEFB, indicating ultrasound method is favourable. In view of the total preparation duration, ultrasound method possesses 6 times shorter in term of duration compared to soaking method, which operation cost effective.

5. References

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Acknowledgements
The author would like to acknowledge Minister of Higher Education Malaysia, for funding this work under FRGS grant (FRGS/1/2019/STG07/DHUAM/02/1).