Oil Palm Empty Fruit Bunches (EFB): Influence of Alkali and Acid Treatment on the Mechanical Properties of Medium Density Fibreboard (MDF)

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ARTICLE INFO	ABSTRACT
	Empty Fruit Bunches (EFB) are abundance residues from palm oil plantation was recognised as a potential material for manufacture such as particleboard and medium density fibreboard (MDF). The objective of this study is to identify and assess the potential effects of chemical treatment and concentration on the mechanical and bonding properties of MDF from EFB oil palm fibres. The EFB fibres were treated at 0.2, 0.4, 0.6 and 0.8% of sodium hydroxide (NaOH) and acetic acid (CH_3COOH), and the fibres were used in the MDF production. Mechanical test included modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB) were conducted. Between the chemical treatment and the concentration, the chemical types used had a more prominent effect compared to the concentration. This study also shows that both treatments resulted in different MDF performance, where acid acetic fibre produced better bending and bonding properties. The optimum condition was 0.4% for NaOH and 0.6% for CH_3COOH to produced panels with good strength and better dimensional stability. Such sample for 0.4% NaOH had 17.9 MPa, 1297 MPa and 0.48 MPa, and for 0.6% CH_3COOH had 22.1 MPa, 1641 MPa and 0.56 MPa, for MOR, MOE and IB, respectively.

1. Introduction

Medium density fiberboard (MDF) is one of the largest wood-based industry in Malaysia. Normally, the MDF industry uses rubberwood, acacia wood and mixed hardwood to produce MDF.

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Due to the lack of wood and the tendency to use lignocellulosic sources as fiber materials, natural fibers have been identified as a potential source for the MDF industry. Natural fibre uses in the composite industry are widely used because of their availability and strength properties. An empty fruit bunch (EFB), as being one of the world's largest producers of palm oil, could be utilised by the composite sector as a fibrous raw material. Oil palm empty fruit bunch (EFB) are the most used for composite materials among others oil palm fibers include the trunk and frond. Several studies showed that EFB fibers can be utilized such as in the production of polymer composite, particleboard, fiberboard, and fuels [1-5].

However, the EFB fibers have several disadvantages include high oil content, contains waxy layer on the fiber surface and have alkaline pH. Poor physical and mechanical properties would be caused by the EFB 's properties. Thus, the treatment of EFB fibers is essential to overcome the unfavorable properties of producing fibers with matrix-compatible properties, especially in composite production. Numerous research and studies have been conducted in relation to the treatment of EFB fibers [6-11]. In addition, several types of chemicals, such as silane, succinic anhydride, acrylic acid, formic acid, and phosphoric acid, are also used for the treatment of EFB fibers [9-13].

The modification of EFB fibers treated with acetic and propionic anhydride has been shown to increase the bonding strength of MDF. Such research was carried out by Khalil *et al.,* [14] and they found that the cause for the increase was due to better compatibility between the matrix resin surfaces. The modification enhanced the internal bonding (IB) as the hydrophobic properties of the fiber surface were increased. Pan *et al.,* [15] on the other hand, found that CH_3COOH treatment lowers the pH value for straw fiber and has a significant impact on the straw fiberboards' bonding strength, with no clear improvement in bending strength of four treated types of straw fiberboards. They also concluded that treat with CH_3COOH is optimum treatment for producing good quality of straw fiberboard. The chemical changes also contributed to the low bending and bonding strength of MDF. Li *et al.,* [16] examined the chemical changes in the cell wall resulting from the hemicelluloses extraction and found make that acid treated fiber and UF resin less compatible. At the same time, acid treatment can lower the pH value of the treated fibers and lead to the self-condensation of UF resin and contribute to the reduction of IB strength.

It has been noted that the better adhesion properties can be improved by pre-treatment using varieties of chemicals such as sodium hydroxide (NaOH), acid acetic (CH_3COOH), sodium perchlorate (NaClO4), and others. It is believed that the correct selection of the chemical during pre-treatment will produce fiber with acceptance properties. Since the adhesion between the EFB fiber and the resin is an important element in the realization of the mechanical properties of the MDF, studies have focused on pre-treatment fibers to improve the properties of the panel. Thus, the main objective of this was to identify and assess the effects of NaOH and CH_3COOH as treatment material at different concentrations in medium density fiberboard (MDF) manufacture. Mechanical properties of the MDF panel as a function of treatment and concentration were also evaluated.

2. Methodology
2.1 Materials

Oil palm EFB was obtained from the Sri Langat Oil Palm Mill located at Dengkil, Selangor, Malaysia with initial moisture content of 110%. The EFB was then transported to Malaysian Palm Oil Board (MPOB) Pilot Plant and chipped using a drum chipper.

2.2 Pre-treatments and Refining of EFB

The EFB then were treated with two types of chemicals: one being sodium hydroxide (NaOH), while the other was acetic acid (CH₃COOH) – tested at four different concentrations: 0.2, 0.4, 0.6 and 0.8%. The parameters selected for this study was based on findings and observations made in the previous study [3,9,10]. The EFB was soaked in a chemical solution at room temperature for a whole day, 24 hours. The treated EFB was initially washed with water to remove any impurities and air was then dried for 24 hours prior to the refining process.

2.3 Refining of the EFB

The treated EFB fiber were heated at 0.6 MPa of refining pressure for 5 minutes followed by refined at refiner temperature of 150 to 170°C through the usage of a Sprout-Bauer (ANDRITZ) refiner, at an MDF pilot plant that was located at the MPOB/UKM Research Station. The moisture content was below 5% after the fibres were dried.

2.4 Fibreboard Production

Refined EFB fibers with MC of 3-4% were blending with Urea-Formaldehyde (UF) resin with solid content of 64%. The UF resin was sprayed onto the fibers using a rotary drum blender and the resinated fibers were formed into mats. The mats had to first go through a process of cold pressing with a pressure of 50 kg/cm² for an estimation of 3 minutes, which is subsequently followed by being hot pressed at pressure of 160 kg/cm² at 175 °C for 5 minutes. The target density of the board was 0.70 g/cm³ with board dimensions of 300 x 300 x 12 mm. The MDF panels were kept conditioning at 20±2 °C and 65% relative humidity in the conditioning room until reached balance moisture content.

2.5 Mechanical Properties Evaluation

The bending strength of the panel were conducted according to EN 622-5, 2006 using Zwick Universal Testing Machine with load of 10 kN, using crosshead speed of 3 mm/min and span length of 240 mm. Samples were glued to the metal blocks of a surface area of 50x 50 mm with a hot melt resin to be tested for IB. There is another heated metal block containing heated glue on the other side of the IB sample. The samples were then kept in the conditioning room 24 hours before the test. The crosshead speeds for all specimens were 3 mm/min. The data were statistically analyzed using Statistical Analysis System (SAS) software. Analysis of Variance (ANOVA) was used to examine the effects of treatment on the panel properties. Least Significant Difference (LSD) method was used for mean separation to further evaluate the effects of different treatments and concentrations, and the interaction between both parameters.

3. Results

The summary of ANOVA on the panel properties is summarized in Table 1. The effect of different treatments and concentrations on the MDF mechanical properties including modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB) were determined.

Table 1
Analysis of variance (ANOVA) on the MDF mechanical properties for differences EFB treatment effects

Variables	d.f	p-value		
		MOR	MOE	IB
Treatment (T)	2	***	***	***
Concentration (C)	4	n.s	*	n.s
Interaction (T x C)	8	***	***	***

Note: ***: Significantly different at p≤ 0.01, *: Significantly different at p≤ 0.10, ns: not significant

It was found that, high significant interactions (p≤0.01) between treatment and concentration were observed for all panel mechanical properties. Between the two variables, treatment has more dominant effect (p≤0.01) on all mechanical properties of MDF. Compared to the concentration which has no effect on all mechanical properties.

3.1 Bending Strength

The MOR measures the maximum load that the composite panel can support and demonstrates the amount of stress required to cause failure, and the MOE measures the stiffness of the composite panel and demonstrates the ability to resist deflection. Figure 1 and Figure 2 show the value of the MOR and MOE of the EFB treated fiber panel. Treatment at 0.2 and 0.4% of NaOH, the MOR and MOE of the panels were 15.5 and 1101 MPa, and 17.9 and 1297 MPa respectively. Both properties were, however reduced to the range of 11.2 to 13.6 MPa of MOR and 810 to 927 MPa of MOE when the concentration has increased to 0.6 and 0.8%. The MOR and MOE values decreased significantly when the NaOH concentration was increased from 0.4 to 0.6 and 0.8%. Nevertheless, the panels produced from CH_3COOH treated fibres had relatively higher bending strength values in both MOR and MOE than panels manufactured from NaOH treated fibres. The value of MOR increased from a range of 18.4 to 20.8 MPa, and the value of MOE increased from a range of 1291 to 1638 MPa with increased concentration of CH_3COOH from 0.2 to 0.8%.
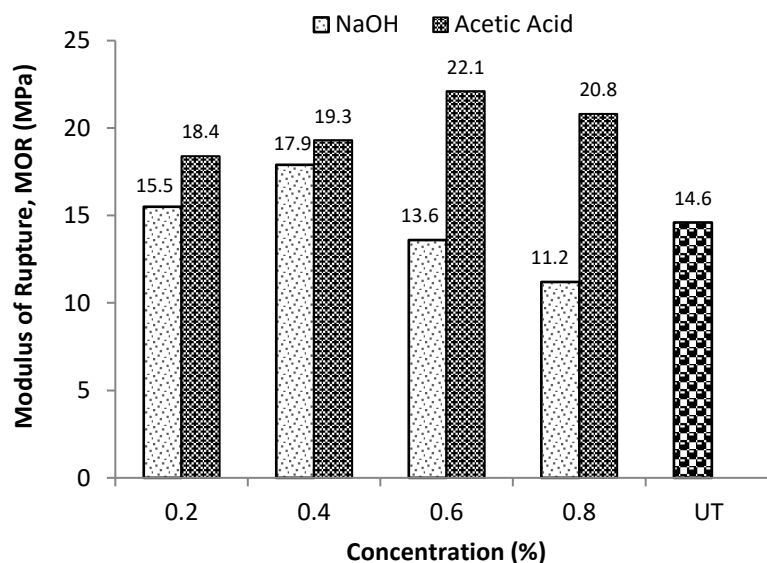
Fig. 1. The mean values of Modulus of Rupture (MOR) of MDF from treated empty fruit bunch (EFB) fibres

Based on the approach of the Least Significant Difference (LSD) method, means figures that were subsequently followed by the same letters do not differ significantly, at p≤0.05. UT = Untreated fibres.
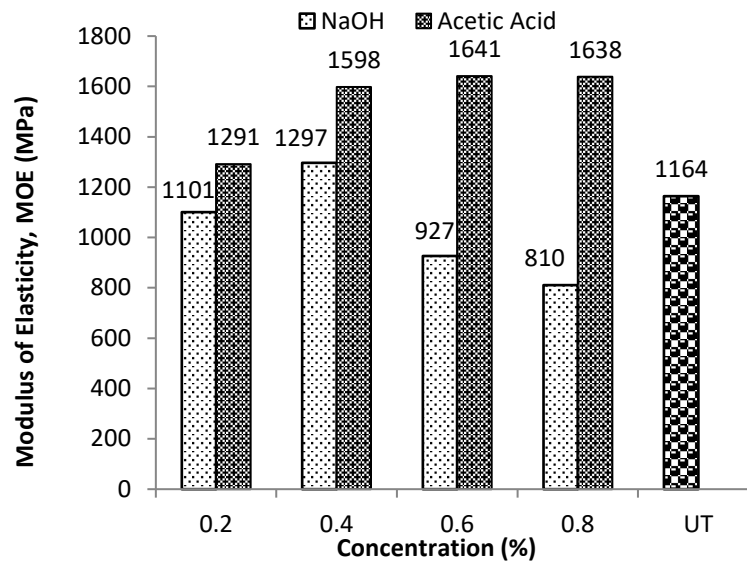
Fig. 2. The mean values of Modulus of Elasticity (MOE) of MDF from treated empty fruit bunch (EFB) fibres

Based on the approach of the Least Significant Difference (LSD) method, means figures that were subsequently followed by the same letters do not differ significantly, at p≤0.05. UT = Untreated fibers.

The results show that the MOR and MOE increased to 17.9 and 1297 MPa, respectively as the EFB fibers were treated with NaOH concentration from 0.2 to 0.4%. Increased values are likely due to the removal of impurities, including residual oil and wax, on fiber producing EFB fibers with a more accessible surface. The removal of impurities can attribute to better wetting of the EFB treated fibers and UF. Nevertheless, as NaOH concentration was increased to 0.6% and 0.8%, the results show a substantial decrease in the values of MOR and MOE. The reduction in bending strength is related to the formation of a fiber lump. Fiber lump formed as the result of excessive NaOH amount during the treatment, thereby increase OH concentration [12]. Consequently, the availability of cellulose makes the fiber much more flexible, thus results in the formation of fiber lumps [3,17]. During hot pressing, the lumps would be pressed into dense areas relative to the remainder of the MDF. This inhomogeneity generates excess internal stress, mostly in bending, and therefore lowers the MOR and MOE values of the panel. According to Ibrahim *et al.,* [7], excessive treatments can degrade the molecular chains of lignocellulosic materials, specifically the cellulose. The drop in MOR and MOE is in agreement with earlier observation by Ndazi *et al.,* [18] who obtained that at high concentration of NaOH, the size of rice husks was reduced and non-uniform interface bonding in the panels occurred due to the increasing the surface area. Moreover, at high concentration of NaOH, the physical and chemical degradation of EFB fibers occurred and caused deterioration of the fiber stiffness and strength.

Conversely, panels made from CH_3COOH treated fibers, both MOR and MOE values increased as the concentration increased from 0.2 to 0.6%. Mechanical properties of the panels were found to be influenced by the tensile strength of the fiber. Treatment at higher concentration of CH_3COOH produced fibers with higher tensile strength due to the lots of cellulose content. Aji *et al.,* [19] stated that the tensile strength of fiber increases with the increasing of cellulose content. This supported by

Nishino *et al.,* [20] who indicated that the crystal region gives the maximum value for the specimen modulus of panel. However, as the concentration of CH_3COOH increases to 0.8%, the MOR and MOE value decreases. This phenomenon could be related to the strong acid characteristic that can destroy the cellular fiber and reduce the tensile strength of the treated fiber. The higher level of hydrolysis at high levels of CH_3COOH appears to have caused a decrease in the strength of the fiber, thus, leading to a reduction in the values of MOR.

3.2 Internal Bonding (IB) Strength

IB strength indicates the homogeneity of the fiber to the fiber adhesion and was related to the dimensional stability of the panels. Figure 3 provides the IB values of panels from NaOH and CH_3COOH EFB treated fibres. As can be seen from the figure, the IB value of the samples increased with an increase in concentration in both treatments. However, following the treatment using NaOH it was observed that 0.6 and 0.8% concentration of NaOH had detrimental effect on the bonding properties of the fibres. The value of IB strength for the panel produced from NaOH treated fibre increased from 0.43 to 0.48 MPa significantly when the concentration increased from 0.2 to 0.4% and the IB value decreased in the further concentration, whereas the panel produced from CH_3COOH treated fibre showed an improvement in IB strength from 0.47 to 0.53 MPa with the increment of CH_3COOH concentration.
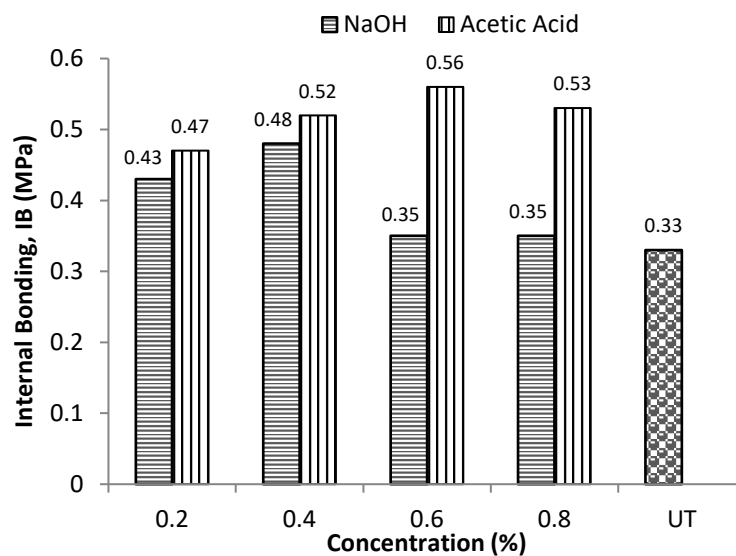
Fig. 3. The mean values of Internal Bonding (IB) of MDF from treated empty fruit bunch (EFB) fibres

Based on the approach of the Least Significant Difference (LSD) method, means figures that were subsequently followed by the same letters do not differ significantly, at $p \leq 0.05$. UT = Untreated fibres.

The IB strength increase when NaOH concentration was increased from 0.2 to 0.4%. The improvement of bonding is mainly due to the partial removal of oil and waxes on the surface of the fiber by NaOH treatment, thus providing better wetting properties of the surface and thus increasing the absorption of UF by the surface of the fiber. This surface may disrupt the efficiency of UF as a coupling agent by preventing penetration, thereby affecting the contact between the fibers and the adhesive resulting in a low IB value. This effect may ultimately increase the properties of the fiber adhesion, resulting in improved bonding properties for the panel. Previous researchers [3,21,22]

reported that NaOH treatment removed some oil, waxes, and other impurities from EFB fibers, and allows the resin to spread easily on the fiber surfaces.

However, the values of IB of the NaOH treated panels dropped significantly as the concentration of NaOH increased to 0.6 and 0.8%. Decreasing IB strength may be associated with increasing pH values that will affect the rate of UF reaction during panel production [23]. The UF resin reaction is then triggered and propagated in an acidic condition that originates from the fibers themselves or from the addition of acid chemicals. Increased pH values have a profound effect on the formation of alkaline fibers that will negatively affect UF, an acid-based resin that requires the acidic environment to be cured. Fibers with different pH values will yield different resin curing times during hot pressing [23]. In general, UF resin mixed with fibers with high pH value will require more time to cure. The decreasing IB indicated that the hot-pressing time (5 minutes) used in this study accompanied with high pH fibers is insufficient to cure the resin, thus resulting in poor bond strength.

The results also indicated that treated fiber with NaOH at high concentration surface shows deposition of impurities and the existence of fiber lump. The presence of these substances on the surface of the fiber inhibits the ability of the UF resin to spread on the surface of the fiber and thus negatively affects the contact between the fiber and the resin and produces panels with poor bonding properties. Gillah *et al.,* [24] stated that the resulting low IB values for panels were caused by the refined fibers with resin being poorly mixed, with samples failure occurring at the interface of fiber lumps.

3.3 Acid Treatment

Treatment of EFB fibers with CH_3COOH prior to panel manufacturing has been found to significantly improve the properties of IB. From the finding, CH_3COOH treated fibres with enough CH_3COOH yield in relatively higher IB strength. The significant changes in the bonding effects of the panels in the treatment of CH_3COOH can be attributed to the removal of residual oil in fibers. The presence of oil inhibits the rate of adhesive spread because this oil layer prevents the adhesive from distributing well on the surface of the fibre, creating poor adhesion between the fibre and the adhesive. Lee and Wang [25] reported that wax, oil and pectin on the fiber surface influenced the bonding properties of boards, due to the reason that these elements may form a thin film on the fiber surfaces, thus interrupt the fiber-UF bonding.

The oil content in the CH_3COOH treated fibres is the main factor that contributed to the bonding properties. Higher IB values have been found to be associated with better fibre contact resulting from properly treated fibres, which may lead to the removal of certain materials, including wax, globular protrusions, oil and impurities from the surface of the fibres. The elimination of these substances leads to a more consistent distribution of the glue on the smooth surface of the fiber, resulting in further fiber-to-fiber contact being overlapped, thereby allowing stronger interlocking of the fiber adhesive and thus improving the bonding properties of the plate. A previous study by Nemli *et al.,* [26] supported similar results by stating that if more contact is made within the fibre, this will increase the bonding of the fibres with improved resin efficiency during hot pressing.

The pH value of the fibre can be correlated with the good bonding ability of the generated panels. This result can be explained by the fact that good compatibility between fibre and UF resin can lead to the good distribution and curing of the UF matrix with the EFB fibres. Xing *et al.,* [27] argued that the change in pH could affect UF curing and influence UF bonding. Because the UF resin is an acid-based adhesive, it needs an acidic environment to wet the fibres in order to achieve adequate bonding and to develop bonds. Xing *et al.,* [28] concluded that the strength of the IB could be affected by a change in the pH values of the fibers which would result in different resin curing

times during hot pressure. Fiber acidity has played a vital role in the gel time and curing behavior of the resins and affects the performance of the MDF panels. There are number of studies that have assessed the influence of the fibre acidity on the performance of MDF panels [3,23,24,29]. Slight reduction of properties at 0.8% of CH_3COOH can be attributed to pre-curing of UF. More highly acidic fibers are expected to result in higher degrees of pre-curing during panel processing and to create poor fiber-to-fiber bonding. One study by Li *et al.,* [16] also reported similar finding where the IB strength of acid treated MDF reduced because low pH lead to self-condensation of UF resin.

4. Conclusions

The study found that the state of treatment, which requires various types of chemical and concentrations, influences the properties of the plate. Both pre-treatments are said to have a much more pronounced effect on the properties of the panel compared to the concentration of the material. Treated fiber with CH_3COOH produced panel with better strength and bonding properties compared to alkaline treatment. Alkaline treatment at concentration higher than 0.6% produced samples with relatively lower strength and IB properties. The MOR, MOE and IB properties of the panels treated with NaOH were found to markedly reduce at more severe treatment. The findings of the current study also suggest that the condition of treatment was crucial to the production of superior MDF properties. The optimum properties of MOR, MOR and IB were 22.1 MPa, 1641 MPa and 0.56 %, respectively at 0.6 % of CH_3COOH resulted in acceptable mechanical properties according to the EN 622-5 (Requirement for Ultra-light MDF board).

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