Development of Binderless Fiberboards from Steam-exploded and Oxidized Oil Palm Wastes

Elizabeth Henao Mejía,[a] Germán C. Quintana,[a,*] and Babatunde O. Ogunsile [a,b]

Binderless fiberboards were made from oil palm (*Elaeis guineensis*) empty fruit bunches with two treatments: steam explosion and Fenton reagent oxidation. Fiberboards were prepared with a targeted density of 1.20 g/cm^3 and a thickness of 4 mm. A factorial experimental design 2^2 with two center repetitions and one repetition was applied for each treatment. The oil palm waste was oxidized with Fenton reagent using a H_2O_2/Fe^{2+} ratio of 2%/0.2% to 4%/0.4% and a pressing temperature of 170 to 190 °C. Steam explosion was carried out at a severity factor of 3.5 to 4.0 at the same pressing temperature. Both treatments were examined under two major response variables: mechanical properties (modulus of rupture, MOR, and modulus of elasticity, MOE) and physical properties (thickness swelling, TS, and water absorption, WA). Steam-exploded samples developed better physico-mechanical properties than those that underwent Fenton reagent oxidation. The best results were obtained from fiberboards treated with the highest steam explosion design conditions (severity 4 and pressing temperature 190 °C) to give optimum values of MOE 3100.09 MPa, MOR 28.49 MPa, TS 11.80%, and WA 22.74%. Binderless fiberboards made from steam explosion-treated pulp satisfied favorably well the Colombian Standard NTC 2261.

Keywords: Binderless fiberboard; Oil palm wastes; Steam explosion; Fenton reagent

*Contact information: a: Grupo Pulpa y Papel, Facultad de Ingeniería Química, Universidad Pontificia Bolivariana, Circular 1 N° 70-01, Medellín, Colombia; b: Department of Chemistry, University of Ibadan, Ibadan, Nigeria; *Corresponding author: german.quintana@upb.edu.co*

INTRODUCTION

In the preparation of fiberboards, synthetic resins are used as binders to make a good finish. Phenol-formaldehyde (PF) and urea-formaldehyde (UF) resins are approximately 60% of the total production cost of fiberboard manufacturing on a dry weight of only 8 to 10% (Abdul Khalil *et al*. 2010; Hashim *et al*. 2010). Furthermore, PF and UF resins cause environmental problems during fiberboard degradation (Rokiah *et al*. 2009). They constitute waste disposal problems because they are non-biodegradable and not recyclable. Many treatments have been studied to obtain binderless fiberboards that are cheaper and friendlier to the environment than the resins. The most common methods are the oxidative and the thermal treatments (Velásquez *et al*. 2002).

Fenton reagent was proposed as an oxidative treatment to modify fiber surface and degrade low molecular weight components by improving covalent bond formation between lignocellulosics polymers, so that during hot pressing, the reagent could penetrate porous cellular wall and react inside (Widsten *et al*. 2003a; Halvarsson*et al*. 2009). Fiberboards from wheat straw (Halvarsson *et al*. 2009), *Pinus radiata* (Riquelme-Valdés *et al*. 2008), fir fibers and beech (Widsten *et al*. 2003a), hardwood, and soft fibers (Widsten *et al*. 2003b) have been made with this method.

Steam explosion is a thermal method in which hemicelluloses are partially hydrolyzed to sugars by autohydrolysis, exposing cellulose and promoting lignin condensation (Laemsak and Okuma 2000; Quintana *et al.* 2009). In the process, lignin content is proportionally increased and redistributed onto the surface of cellulose, thereby increasing hydrophobicity (Negro *et al.* 2003). This method has been applied for the production of fiberboards from coconut husk (Van Dam*et al*. 2004), *Miscanthus sinensis* (Salvadó *et al.* 2003), softwood (Salomón *et al.* 2013), banana stalk (Quintana *et al.* 2009), and oil palm frond (Laemsak and Okuma 2000).

Oil palm is cultivated in 42 countries around the world (Salomón *et al.* 2013). Colombia, with 427.368 ha planted in 2011 (Martín-Sampedro *et al.* 2012), is the fourth biggest oil palm producer in the world, behind Malaysia, Indonesia, and Thailand. Oil palm mills generate between 260 to 480 kg of solid wastes per ton of fresh fruit bunches, of which empty fruit bunches (EFB) constitute around 60% (Salomón *et al.* 2013). EFB are normally burned at plantations (Chew and Bhatia 2008) or used as fertilizer (Salomón *et al.* 2013). EFB is hereby proposed as a raw material for the production of binderless fiberboards.

The present work is aimed at using steam explosion and Fenton reagent treatment methods to manufacture fiberboards without additives from EFB. Comparative studies of the mechanical and physical properties of the binderless fiberboards are made using a 2^2 experimental design matrix.

EXPERIMENTAL

Characterization of Raw Material

EFB was provided by CENIPALMA from Santander (Colombia). The sample was dried, ground, and chemically analyzed using standard methods for its content of moisture, ash, extractives, and Klason lignin, designated ASTM D-4442-07, ASTM D-1102-84, ASTM D-1105-96 and ASTM D-1106-96, respectively. The results were based on the oven-dried weight of EFB. The holocellulose content was estimated by the summative method, using the difference between the sample weight and the sum of the extractives and the Klason lignin.

Experimental Design

Each treatment was evaluated under a 2^2 factorial experimental design to evaluate two factors at two levels of operational variables. Accordingly, this work was aimed at producing fiberboards from EFB exploded with steam and pretreated with Fenton reagent. The effect of two operational variables was examined on each treatment method: severity (3.5 to 4) and pressing temperature (170 to 190 °C) for the steam exploded treatment (Table 1), reagent ratio ((2%/0.2% to 4%/0.4), and pressing temperature (170 to 190 °C) for the Fenton reagent treatment (Table 2). The Fenton reagent ratio was chosen from reported work in the literature (Halvarsson *et al*. 2009) and preliminary work in the laboratory. Also Halvarsson *et al*. (2009) reported that fiberboard properties were improved using hydrogen peroxide value from 2.5 to 4.0 %. Subsequently, preliminary evaluation of different ratios of hydrogen peroxide with Fe^{+2} was used to decolourise methylene blue dye, and the present ratio resulted in the highest rate of decolourisation. In the steam exploded treatment, two levels of severity factor (3.5 and 4) were combined with two levels of temperature (170 °C and 190 °C) to make a total of four distinct

conditions. A fifth condition was created by making use of the central point of each independent variable. A repetition of these total combinations is what we have in Table 1. Table 2 was similarly derived for the Fenton reagent treatment

Steam Treatment

A stainless steel 10-L cylindrical reactor was fed with 150 g of EFB chips. The sample was steam-treated with high pressure to the conditions desired and later depressurized into a 100-L collecting tank by a remote actuation pneumatic valve (Quintana *et al*. 2009). The treated sample was washed and dried at 40 °C for 48 h.

Fenton Reagent Treatment

EFB chips (100 g dry weight) and 450 mL of Fe^{2+} solution were mixed with 450 mL of H_2O_2 in a 5-L reactor at a constant temperature of 50 °C. Ten experimental ratios of H_2O_2/Fe^{2+} were used, as denoted in Table 2. The reaction time was 1 h under mechanical agitation. The treated sample was washed and dried for 48 h at 40 °C.

Table 1. Experimental Design Matrix for the Steam Explosion Treatments

Experiment	Severity (dimensionless)	Pressing temperature (°C)
1	4	170
2	3.5	170
3	4	190
4	3.75	180
5	3.5	190
6	3.5	170
7	3.75	180
8	3.5	190
9	4	190
10	4	170

Table 2. Experimental Design Matrix for the Fenton Reagent Treatments

Experiment	$H_2O_2\%/Fe^{2+}\%$	Pressing temperature (°C)
1	3/0.3	180
2	4/0.4	190
3	2/0.2	170
4	2/0.2	190
5	3/0.3	180
6	2/0.2	190
7	4/0.4	170
8	4/0.4	170
9	2/0.2	170
10	4/0.4	190

Fiberboard Preparation

The oven-dried treated samples were shaped by hand using a forming box (150 mm in length and 50 mm in width). Fiberboards of 4.0-mm thickness and a target density of 1200 kg/m^3 were prepared. After they were formed, the exploded EFB samples were pressed in a hydraulic press at 716 psi. The pressing temperature was varied from 170 to 190 °C, as higher temperatures cause thermal degradation of the material. Exploded EFB samples were pressed by a three-stage pressing method:

- Pre-pressing for 3 min at the desired pressure and temperature;
- 5 s breathing; and
- Pressing for 3 min at the desired pressure and temperature.

Physical and Mechanical Characterization

The modulus of rupture (MOR) and modulus of elasticity (MOE) were evaluated by the Colombian standard NTC 2261, while the internal bond strength (IB) was determined in accordance with ASTM E5651 standard, using a Hidromecánica universal assays device. Dimensional stability was maintained throughout the determination of thickness swelling (TS) and water absorption (WA) using the ASTM D-4442 standard.

RESULTS AND DISCUSSION

EFB Characterization

Data from the chemical analysis of the raw material are reported in Table 3. The result indicated that EFB contained 64.44% holocellulose and 20.87% lignin. These values are moderate and good for typical fiberboard making. They are comparable with values of 24.5% lignin and 67.0% holocellulose reported by Martín-Sampedro *et al.* (2012). The holocellulose contents include the cellulose, which acts as a support, and the simple sugars and polyoses that give union to the fibers (Shen 1986; Hashim *et al.* 2010). The lignin content acts like a binder between fibers in the adhesion process (Velásquez *et al.* 2002). Similar values were reported in the literature for some other agricultural wastes and raw material that resulted in the formation of good fiberboards without synthetic additives, such as *Miscanthus sinensis* (73.8% holocellulose and 19.9% klason lignin), the fibers and pith of coconut husk (36.3 and 21.0 wt.% α-cellulose and 31.9 and 24.1% klason lignin, respectively), and banana stalk (68.15 % holocellulose and 14.28 % klason lignin) (Salvadó *et al.* 2003; Van Dam *et al.* 2004; Quintana *et al.* 2009). The ash and extractive contents of EFB were moderate and relatively low compared to other lignocellulosic materials such as banana stalk that have approximately 17% extractives and 12% ash (Quintana *et al.* 2009).

Table 3. Chemical Characterization of EFB

Component	%
Acid-insoluble ash (%)	2.93
Organic extractives	8.86
Aqueous extractives	5.33
Klason lignin	20.87
Holocellulose	64.44

Fiberboard Mechanical Properties

Table 4 shows the results for the mechanical properties of untreated EFB fiberboards and the defined pressing conditions in experimental design. MOE and MOR values obtained were close to the minimum values accepted by the Colombian Standard NTC 2261 for 4-mm fiberboards (2400 MPa and 21 MPa, respectively).

Table 4. Mechanical Properties of Untreated EFB Fiberboard

Pressing temperature (°C)	MOR (MPa)	MOE (MPa)
170	19.94	2564.19
190	19.98	2561.27

Table 5 shows the mechanical properties of the Fenton reagent-treated fiberboards. The boards had values between 1656 and 2469 MPa for MOE and between 11.63 and 16.58 MPa for MOR. For the range of this investigation, the treatments caused a reduction in the values of MOE and MOR in comparison with the values obtained for the untreated EFB fiberboard (Table 4), due to the fact that Fenton reagent produces two effects: First, it generates radicals on the surface of the fiber that enhances hydrogen bonding. As the bonding and intermolecular forces increased, the fiber board becomes less flexible and rupture is increased (Hashim *et al.* 2010). Second, Fenton reagent can also degrade the macro molecular lignocellulosic components of the fiber that promotes adhesion, thus affecting the viability of the rupture (Yuhui Qian *et al*. 2004).

Table 5. Reagent-treated EFB Fiberboard

Experiment	MOR (MPa)	MOE (MPa)
1	15.32	2207.38
2	15.28	2469.33
3	12.78	1914.28
4	12.00	1656.27
5	12.60	1796.19
6	16.58	2460.35
7	12.28	2148.82
8	11.63	2432.37
9	11.86	1912.77
10	13.11	2093.05

Table 6 shows the mechanical properties of the steam explosion-treated fiberboard, which had values between 1249 and 3079 MPa for MOE and 12.26 and 31.75 MPa for MOR. The boards with low severity factors (Experiments 2, 5, 6, and 8 in Table 6) had lower values than those without fiber treatment (Table 4), suggesting that for these conditions, the treatments remove some hemicellulose; if the hemicellulose had remained in the fiber, then it would have aided fiber adhesion (Hashim *et al.* 2010).

Table 6. Explosion-treated EFB Fiberboard

Experiment	MOR (MPa)	MOE (MPa)
1	25.46	2787.75
2	17.44	1856.85
3	24.75	3079.47
4	19.63	2659.12
5	12.26	1590.67
6	12.72	1249.44
7	20.55	2385.63
8	15.20	1796.14
9	25.16	3100.12
10	31.75	2665.25

Figure 1 shows the Pareto chart for MOE at a 90% confidence level for both treatments. A Pareto chart gives a graphical representation of the factors considered in descending order by bars, showing the relative importance of factors and the effect (positive or negative) in the response variable.
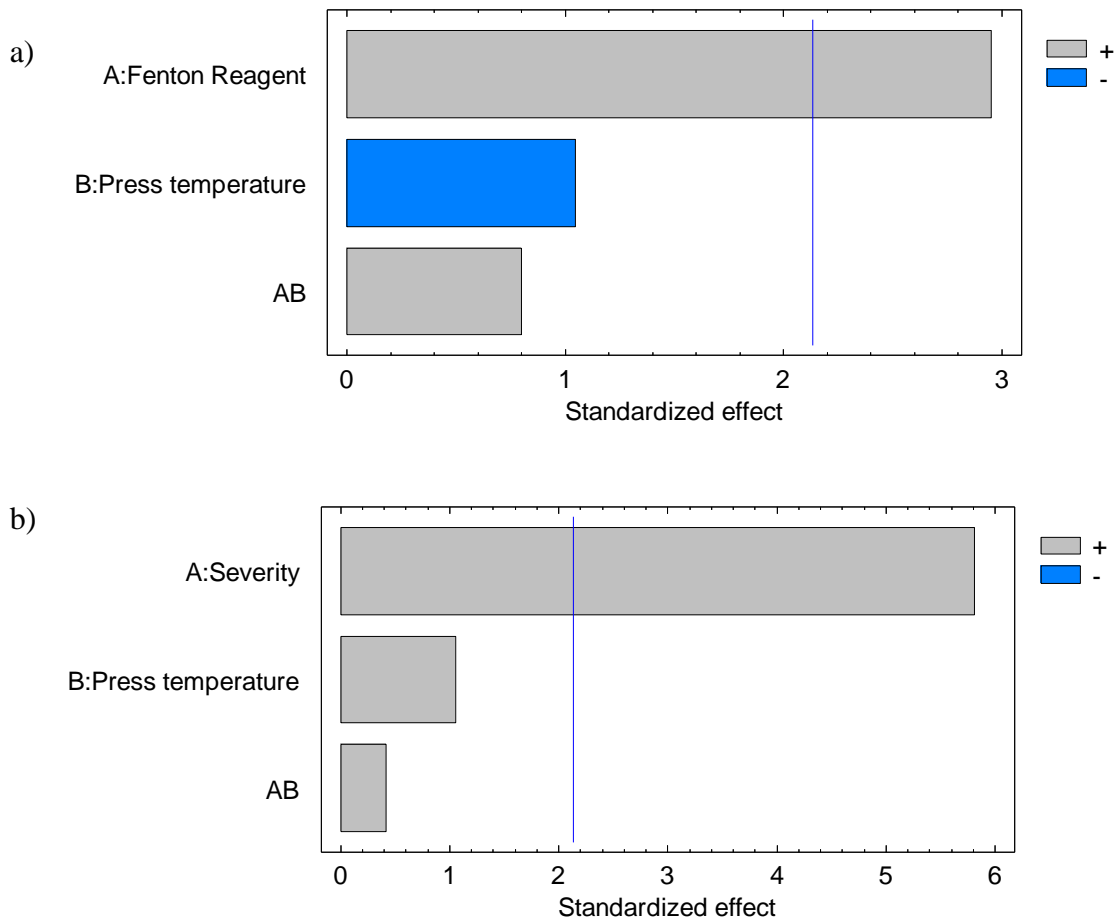
Fig. 1. Standardized Pareto chart for MOE: a) Fenton reagent treatment and b) steam explosion treatment

The purpose of the Pareto chart is to highlight the most important among a set of factors (Wilkinson 2006). Reagent concentration had a statistical influence on the Fenton reagent-treated fiberboard, while severity had a statistical influence on the steam explosion-treated board.

Colombian Standard NTC 2261 values were obtained with the fiberboard treated with steam explosion at severity factors of 3.75 and 4. Pressing temperature did not have an influence on MOE; the maximum pressing temperature was reduced due to the imperfections caused by the residual oil under high temperature.

The optimal MOE value predicted for the Fenton reagent treated fiberboards was 2332.05 MPa at a Fenton reagent ratio of $4\%H_2O_2/0.4\%Fe^{2+}$. The optimal MOE value for steam explosion treated fiberboards was predicted to be 3100.09 MPa with a severity factor of 4.0. Only the steam explosion treatment improved MOE values compared with untreated fiberboard. The MOE reported in the literature for steam explosion-treated fiberboard is 3063.13 MPa for banana stalk (Quintana *et al.* 2009) and between 1120 and 6070 MPa for *Miscanthus sinensis* (Velásquez *et al.* 2002). Norway spruce (*Picea abies*) fiberboard treated with Fenton reagent has a MOE ranging from 3200 to 5800 MPa, which increased with pressing temperature up to 202 °C (Martín-Sampedro *et al.* 2012).

Figure 2 shows the Pareto chart for MOR at a 90% confidence level for both Fenton reagent- and steam explosion-treated fiberboards.
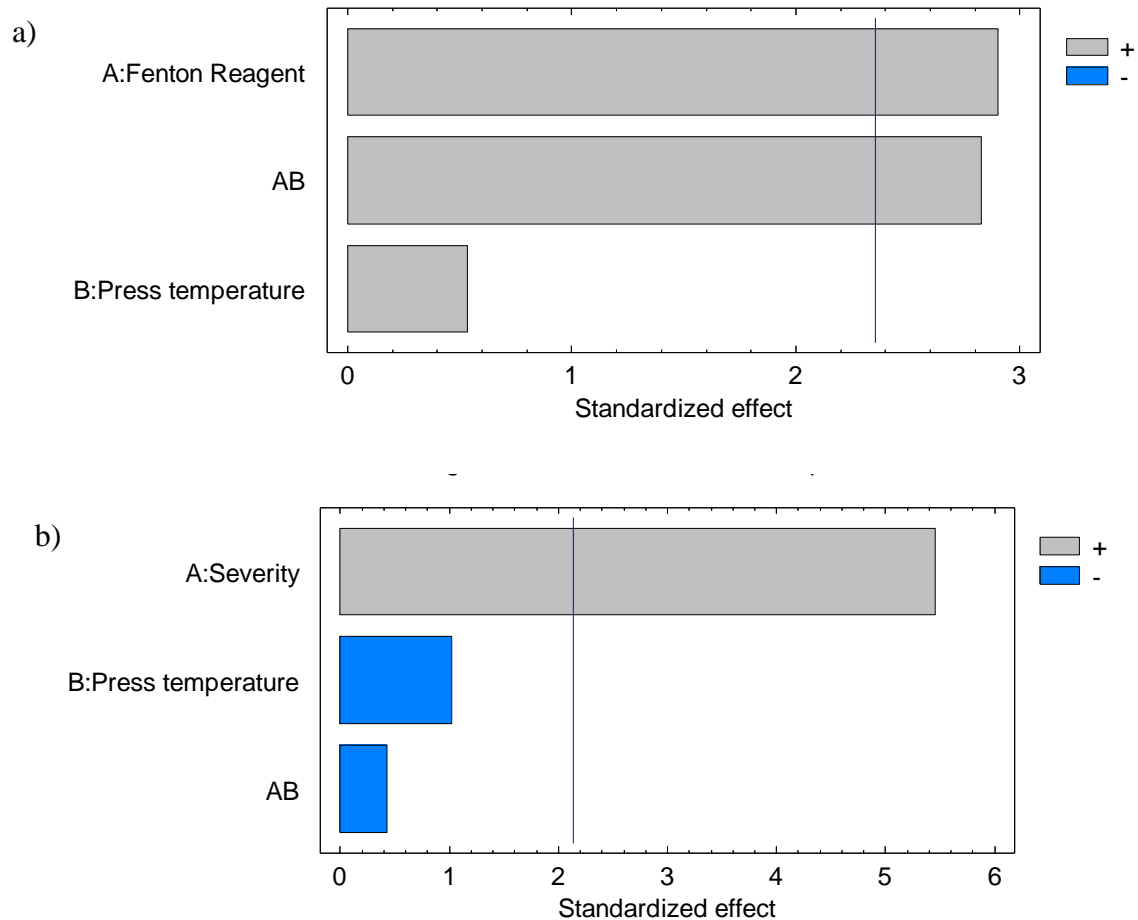

Fig. 2. Standardized Pareto chart for MOR: a) Fenton reagent treatment and b) steam explosion treatment

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The result of the MOR analysis showed that Fenton reagent ratio, and the interaction between pressing temperature and Fenton reagent ratio, had statistical influences on Fenton reagent-treated fiberboards (Fig. 2a), with temperature facilitating the covalent bond formation between activated lignocellulosic polymers (Widsten *et al.* 2003; Halvarsson *et al.* 2009). For steam explosion-treated fiberboards, severity was the only factor with statistical influence on MOR (Fig. 2b); higher severity factor increases the amount of lignin deposited on the fiber surface, which in turn gives support to the fiberboard formed (Van Dam *et al.* 2004).

The interaction plot in Fig. 3 confirmed the importance of pressing temperature and Fenton reagent ratio for the MOR of Fenton reagent fiberboards. High MOR values were obtained at high operating conditions. The influence of temperature was more pronounced, especially at low Fenton reagent ratios.

An optimal MOR value of 14.13 MPa was predicted for the Fenton reagent-treated fiberboards at a Fenton reagent ratio of $4\%H_2O_2/0.4\%Fe^{2+}$ and a pressing temperature of 190 °C; for steam explosion-treated pulp fiberboards at a severity factor of 4.0, the optimal MOR value was 28.49 MPa, computed at a statistical confidence level of 90%. Thus, the optimal value predicted for the steam explosion treatment in this study was twice that of the Fenton treatment. The MOR value was higher than the 22.36 MPa reported for steam-exploded fiberboard from palm frond (Laemsak and Okuma 2000), but close to the 30 MPa reported for softwood fiberboards (Widsten *et al.* 2003). However, the value was lower than the 48 MPa obtained for steam explosion-treated banana stalk fiberboards (Quintana *et al.* 2009) and the 47.2 MPa for *Miscanthus sinensis* (Salvadó *et al.* 2003).

The MOR values attained by the Fenton reagent-treated fiberboard were lower than the 21 MPa required by Colombian Standard NTC 2261. This may be due to the removal of sugars and low-molecular weight hemicelluloses coupled with the degradation of lignin, leading to a decrease in the chemical bonding properties of the fibers during pressing (Widsten *et al.* 2003a).
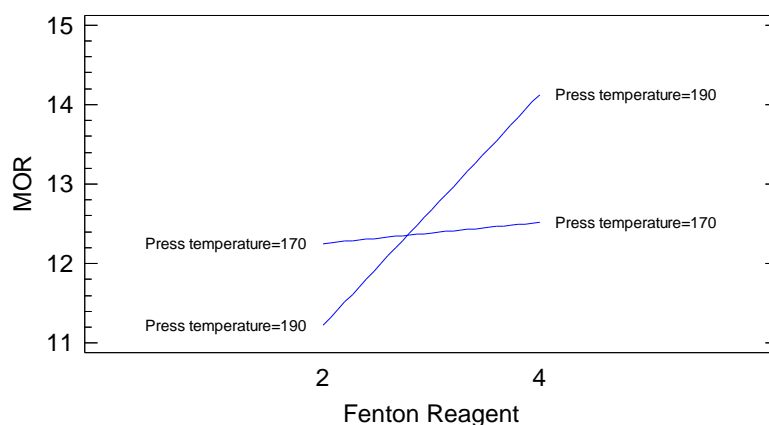

Fig. 3. Interaction plot for MOR of Fenton reagent treatment

Fiberboards made from untreated fibers and steam-exploded treatment at a severity factor of 4 has MOR values higher than Colombian Standard NTC 2261. In the steam-exploded sample, lignin plasticization and redistribution onto cellulose occurred,

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which increased the inter-fiber bonding and hence improved the MOR (Negro *et al.* 2003).

Physical Properties of Fiberboards

Water absorption (WA) and thickness swelling (TS) tests were carried by measuring the change in weight and dimensions of the fiberboards after soaking in distilled water at 20 °C for 24 h. The results of the physical properties of fiberboards made from untreated fibers, Fenton reagent-treated fibers, and steam-exploded fibers are presented in Tables 7, 8, and 9, respectively.

Fenton reagent pretreated fiberboards absorbed between 38.36 and 75.38% of water and returned TS values between 30.89 and 49.79%, showing an improvement over untreated EFB. Steam explosion treated fiberboards absorbed between 23.28 and 136.13% of water and returned TS values of between 13.47 and 57.88%, being lower than untreated fiberboards.

Table 7. Physical Properties of Untreated EFB Fiberboard

Pressing temperature (°C)	WA %	TS %
170	139.13	98.93
190	83.29	56.17

In the Fenton reagent-treated fiberboards, the ratio of Fenton reagent and the interaction between the reagent's concentration and pressing temperature had a negative statistical influence on the WA (Fig. 4a). This is because an oxidative reaction increases the formation of radicals, thus favoring binding capacity while decreasing the spaces for water penetration. The higher the pressing temperature, the greater is the bond formation of the fibers activated by the treatment (Widsten *et al.* 2003). The Pareto chart of the steam explosion-treated fiberboards (Fig. 4b) showed that severity factor had a negative statistical influence on water absorption. This may be attributed to the fact that high severity distributes the lignin onto the cellulose matrix, thus conferring hydrophobic properties to it (Salvadó *et al.* 2003; Van Dam *et al.* 2004), causing a possible reduction of exposed hydroxyl groups, which resulted in a decrease in the WA.

Table 8. Physical Properties of Fenton Reagent-treated Fiberboard

Experiment	WA (%)	TS (%)
1	60.57	49.09
2	47.99	34.97
3	63.43	49.79
4	59.61	30.89
5	68.17	53.16
6	75.38	46.25
7	48.83	41.25
8	57.87	47.5
9	50.58	32.53
10	38.36	30.89

Table 9. Physical Properties of Steam Explosion-treated Pulp Fiberboard

Experiment	WA (%)	TS (%)
1	38.50	19.94
2	89.91	57.88
3	24.33	13.66
4	37.45	20.54
5	62.52	54.23
6	136.13	100.30
7	44.59	31.85
8	57.28	53.11
9	24.87	13.47
10	23.28	13.35

Figure 5 illustrates the influence of interaction between the Fenton reagent ratio and pressing temperature in the water absorption capacity of the fiberboards. As shown, Fenton reagent treatment was the most important of the factors.
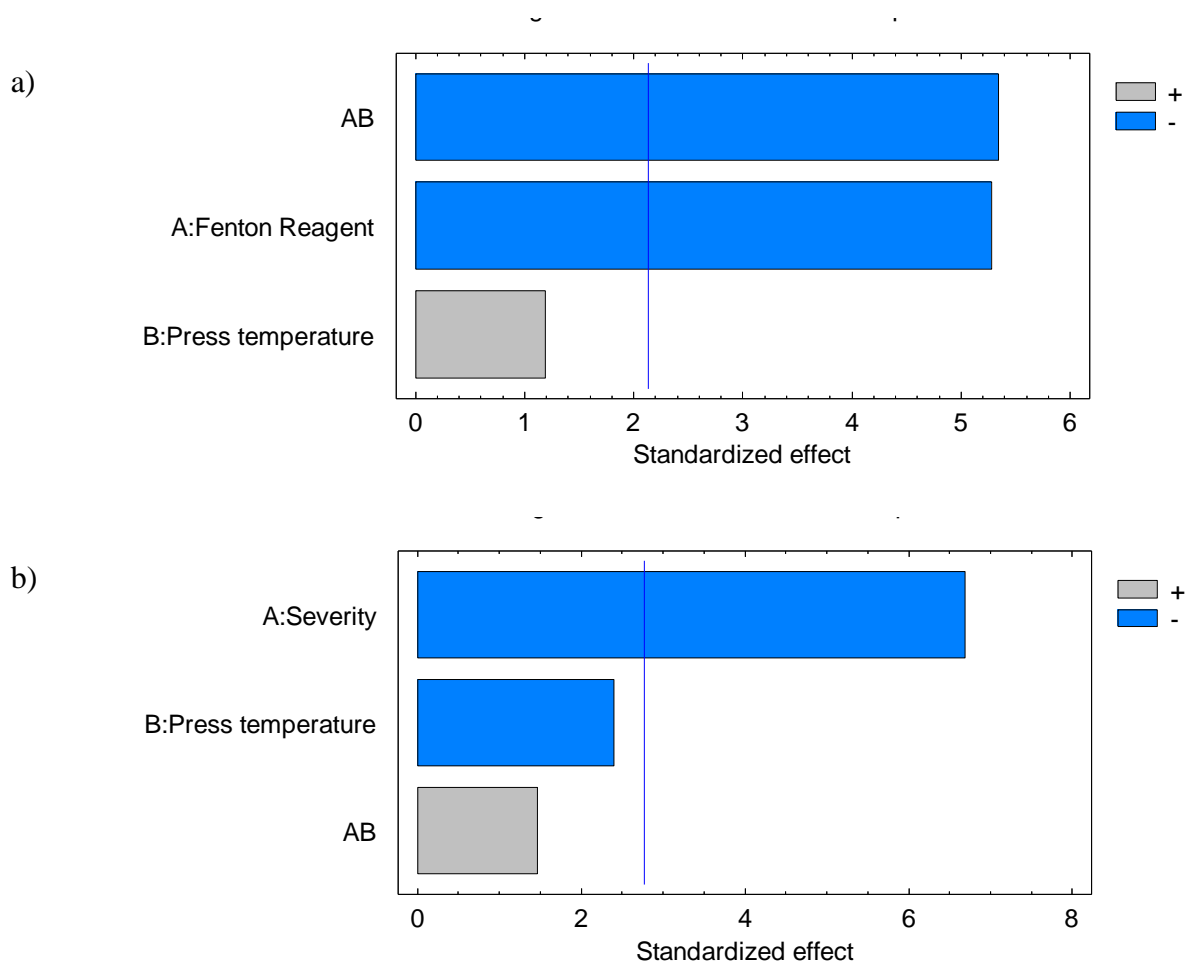
Fig. 4. Standardized Pareto chart for WA: a) Fenton reagent treatment and b) steam explosion treatment
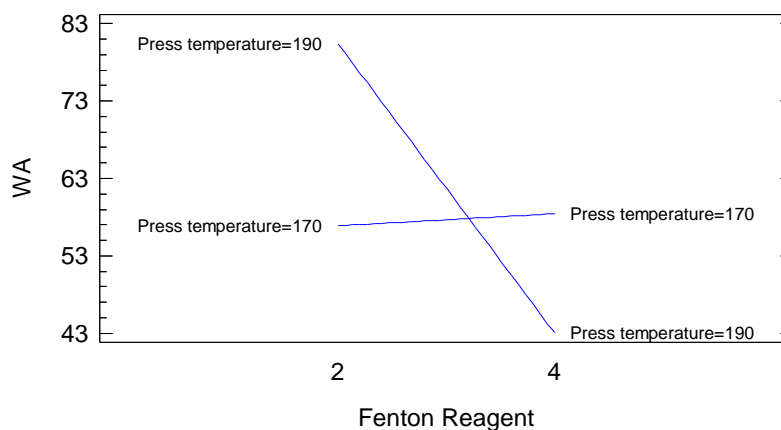
Fig. 5. Interaction plot for WA of Fenton reagent treatment

WA was low at a high Fenton reagent ratio. However, at high pressing temperature and low Fenton reagent ratio, a high WA was obtained because the low Fenton reagent ratio does not generate a sufficiently high concentration of free radicals necessary to activate the fibers, though pressing was done at high temperature. Apart from this, non-wood fibers such as EFB contain more hemicelluloses and less lignin content than their woody counterparts, and the hemicelluloses are more hygroscopic and less resistant to water than lignin. Hence, non-wood fibers in general possess higher WA properties than wood fibers (Halvarsson *et al*. 2009).

The optimum absorption by the Fenton reagent-treated fiberboards was 43.04%, at a Fenton reagent ratio of $4\%H_2O_2/0.4\%Fe^{2+}$ and a pressing temperature of 190 °C. The value for the steam explosion-treated fiberboards was 22.74% with severity 4.0, at a statistical confidence level of 90%. Because the minimum WA required for a high-density commercial board is a value less than 35%, the Fenton reagent-treated fiberboards did not meet this condition. Only the steam explosion-treated fiberboards with a severity factor of 4 reached this value, as a result of the hydrophobicity conferred on the fiber by the coated lignin (Negro *et al*. 2003).

ANOVA, represented by the Pareto diagram in Figs. 6a and 6b, showed that the interaction between Fenton reagent concentration and pressing temperature had a negative statistical effect on the TS of the Fenton reagent-treated fiberboards, while severity factor had a negative statistical effect on the TS of the steam explosion-treated fiberboards. TS behavior is associated with water absorption properties. The extracted lignin, distributed onto the cellulose structure, and the increment in the binding capacity caused by the increase in free radicals present, reduces the hydrogen bond formation to give rise to a more compact fiberboard (Salvadó *et al*. 2003; Widsten *et al*. 2003).
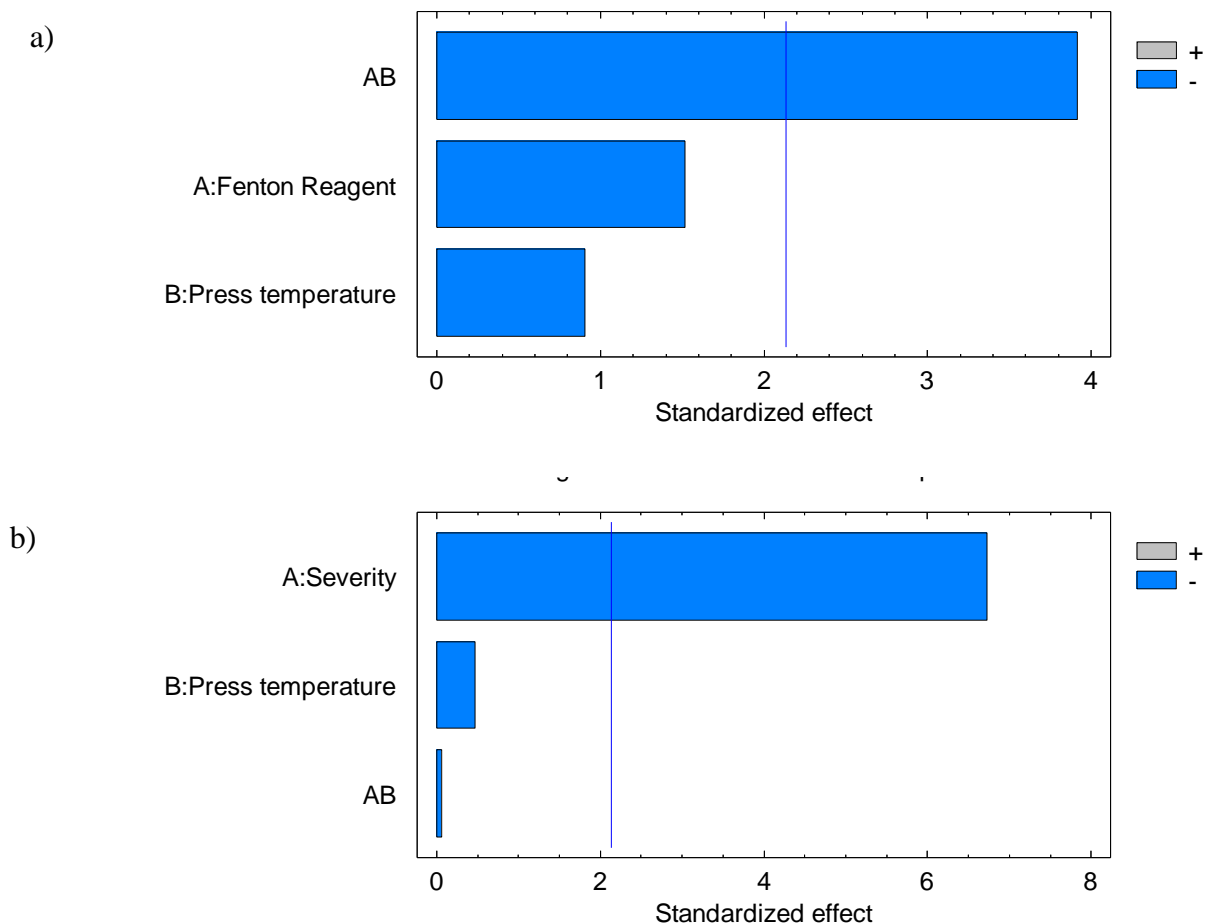
a)

b)

Fig. 6. Standardized Pareto chart for TS: a) Fenton reagent treatment and b) steam explosion treatment

The pattern of interaction for TS of the Fenton reagent treatment, as shown in Fig. 7, was similar to that observed in Fig. 5 for WA. Again, Fenton reagent ratio was the most important factor.

The Fenton reagent pretreated fiberboards had an optimal TS value of 33.13% at a Fenton reagent ratio of $4\%H_2O_2/0.4\%Fe^{2+}$ and a pressing temperature of 190 °C, while the steam explosion treated fiberboard had a value of 11.80% at a severity factor of 4. Better results were obtained for the steam explosion treatment. Steam explosion improved the properties of fiberboard due to lignin plasticization and bonding (Laemsak and Okuma 2000). The results indicated the formation of a stronger bond by the lignin within the fiber matrix during pressing than the bond formed by the increase in radicals. Although the Fenton reagent penetration of the lumen increased radical formation, causing intensification of reactions within the fibers and improving the resistance to swelling (Salomón *et al.* 2013), the effect was not as strong as the binding effect of the lignin (Laemsak and Okuma 2000). As Fenton reagent penetration of the lumen increases the formation of radicals, intensification of reactions within the fibers improves the resistance to swelling (Salomón *et al.* 2013).
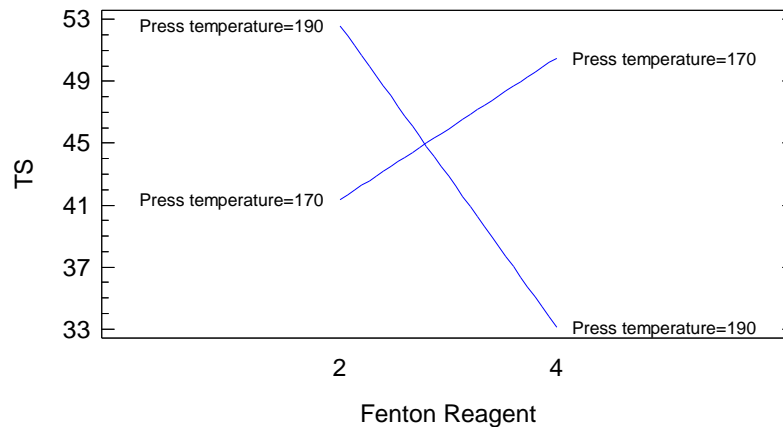
Fig. 7. Interaction plot for TS of Fenton reagent treatment

CONCLUSIONS

1. Steam-exploded oil palm wastes produced fiberboards with improved mechanical properties under the highest severity condition of treatment.

2. The Fenton reagent and steam explosion treated fibers produced fiberboards with better water absorption and thickness swelling properties than the untreated fibers.

3. The Pareto chart diagram revealed that severity factor and Fenton reagent ratio have the greatest significance influence on the fiberboard properties.

4. The physicomechanical properties of the boards were improved more significantly with increasing lignin content caused by the steam explosion treatments than with radical formation developed during oxidation with Fenton reagent.

ACKNOWLEDGMENTS

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