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Integrated Study of the Potential Application of Remediated CCA Treated Spruce Wood in MDF Production

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ABSTRACT: Public health awareness has increased in the past few years regarding the disposal of chromated copper arsenate (CCA) preservative-treated wood wastes. This study demonstrates the potential for using remediated CCA lumber and alternative fiber sources, such as sugar cane bagasse, to produce medium density fiberboard (MDF). The role of both remediated CCA loaded spruce wood and substitution of a part of it with sugar-cane bagasse fibers on the performance of MDF produced were evaluated. The remediation conditions were optimized from examining the FTIR-spectra and TGA analyses of the treated wood fibers resulted from changing the remediated pH (1.4−7.0) and temperature (20−80 °C) together with the efficiencies of removing the preservative metals. The results showed that CCA-remediated spruce fiber provided MDF with 59% and 75.5% reduction in water absorption and thickness swelling, respectively, and 93% increase in IB, compared to panels made from untreated wood. Blending both bagasse fibers with either untreated or remediated fibers had a positive impact on MDF properties. Compared to boards made with untreated control spruce fibers, boards made with up to 30% bagasse had up to 79% reduction in water absorption, 62% reduction in thickness swell, 38% increase in modulus of rupture (MOR), and 244% increase in internal bond (IB) strength. According to ANSI Standard for interior MDF, the remediated CCA-treated spruce was suitable for the manufacture of MDF, either individually or in blends with sugar cane bagasse.

INTRODUCTION

There was a rapid expansion of the use of chromated copper arsenate (CCA) wood preservative between 1975 and 1990, as a result of high consumer acceptance of the product for decks, fences, and other residential applications.[1] Until recently, these traditional biocidal CCA wood preservative treatments have been widely used to improve the durability of timber, because these preservatives are very cost-effective and possess qualities required for wood treatment. However, apart from human health and safety issues,[2,3] disposal of treated wood after the end of its service life is also a major concern.[4] Such concerns over the use of persistent toxic chemicals as wood preservatives have led to limitations and/or restrictions on their disposal by normal landfill, in many regions of the world.[5]

An environmentally sound and cost-effective method for recycling for CCA treated wood and other wastes will help alleviate the landfill and potential chromium and arsenic contamination problems. A number of alternative approaches have been evaluated:

- burning/incineration of spent CCA treated wood in incinerators[6,7] or cement kilns[8]
- reuse of spent CCA treated wood to make composites such as wood-cement, OSB boards, etc.[9−11]
- use of biological or biotechnological method to detoxify spent treated wood[12−15]
- chemical extraction of CCA from spent treated wood[16−18] prior to disposal or reuse

In our previous work a method for extracting nearly all preservative metals from CCA-treated lumber for use in the production of flake-boards was previously performed.[19,20] The extraction was carried out for 6-h at 80 °C using 1% oxalic acid and adjusted to pH 2.4 with sodium hydroxide. These conditions resulted in 100% removal of arsenic, 97% reduction in chromium, and a 91% removal of copper from preservative-treated flakes. In this present study, we are evaluating the structure changes by FTIR-spectra and the thermal stability of the chemical extracted CCA-spruce fibers treatment conditions, and applying the most promising remediated spruce fibers in production of MDF type composites. The role of both remediating the CCA loaded spruce wood and substituting a part of it by sugar cane bagasse on enhancing the performance of MDF produced was also evaluated. Whereas, the sugar cane bagasse regard the agro-fibers based MDF in Egyptian local mill. For bagasse-based MDF, recently Basta et al.[21] succeeded in producing high quality MDF from stored sugar cane bagasse by controlling the retention time of the steam digestion process.

The objective of the work reported in this paper was to identify if remediated CCA-treated wood fiber obtained using the methods of Sabo et al.[20] could be combined with sugar cane bagasse to manufacture a viable MDF panel suitable for commercial use.

EXPERIMENTAL SECTION

Chemicals. Oxalic acid dihydrate (VWR International) and sodium hydroxide (reagent grade, Sigma-Aldrich) were

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obtained in solid form and used for all treatments. Solutions of oxalic acid were prepared in deionized (DI) water unless otherwise explicitly stated as containing tap water. The oxalic acid weight concentrations were prepared on the basis of anhydrous oxalic acid. Huntsman Rubinate 1840 diphenylmethane diisocyanate (MDI) was used as a binder for MDF fabrication.

Fiber Preparation. Nominal 2- by 4-in. (38 mm × 89 mm) spruce lumber was treated with chromated copper arsenate (CCA) type C using a vacuum of 82 kPa followed by 0.8 MPa pressure for a total cycle time of about 3 h, resulting in about 8.3 kg/m^3 of preservative retention.[22] The lumber used for this study was naturally weathered for numerous years at the Forest Products Laboratory in Madison, WI. The treated spruce lumber selected for this study was MSR grade 2250f/1.9E and had 660, 7 mm-deep incisions per square foot. The lumber was chipped and screened to accept chips less than 3/4 in.

Preservative-treated chips were extracted by circulating 1% oxalic acid, pH adjusted to 1.4 −7.0 with sodium hydroxide, at 20−80 °C. The extracted chips were rinsed prior to refining. Wood chips were refined in a 12″ pressurized disc refiner following a 10 min pretreatment with saturated steam at 90 psi. The fibers were dried for approximately 8 h at 105 °C in a tray drier and left to cool, resulting in a moisture content of approximately 8%. The resulting wood fibers were mixed with bagasse fibers. The six blends used for making MDF panels are:

A. 100% untreated spruce
B. 90% untreated spruce +10% bagasse
C. 70% untreated spruce +30% bagasse
D. 100% remediated CCA-treated spruce
E. 90% remediated CCA-treated spruce, + 10% bagasse
F. 70% remediated CCA-treated spruce +30% bagasse

The blends were then passed through a hammer mill without screens to further disperse and blend the fibers.

Fiber Characterization. *FTIR Spectra Analysis.* Infrared spectra were recorded with a Jasco FT/IR, Nicolet, and model 670. The samples were mixed with KBr and pressed into a tablet. The bands were recorded in the region from 4000 to 400 cm^{-1} with a deuterated triglycine sulfate (DTGS) detector at the National Research Center. This test characterized the functional group in the un- and remediated CCA loaded spruce fibers. The technique of O'Connor et al.[23] was used to calculate the crystallinity index (Cr. I). The mean strength of hydrogen bonds (MHBS) was calculated according to Levdik et al.[24] The relative absorbance was also calculated as the absorbance of the band relative to the band corresponding to C−O of the pyranose ring, at ∼1050 cm^{-1}

Thermogravimetric Analysis (TGA). Thermogravimetric analyses (TG and DTG) of the examined samples were done using a PERKIN ELIMER thermogravimetric analyzer (TGA 7). The analyses were performed with a heating rate of 10 °C/min and a flow rate of 50 mL/min, under nonisothermal conditions and in the presence of nitrogen

TG-Curve Analysis. Kinetic studies, based on the weight loss data, were obtained by TG curve analysis. The activation energy has been evaluated by applying the Coats and Redfern method of analysis.[25] For pseudohomogeneous kinetics, the irreversible rate of conversion of the weight fraction of reactant was expressed by the following equation:

$$\frac{d\alpha}{dt} = k(1-\alpha)^n \tag{1}$$

where α is the fraction of material decomposed at time t, k is the specific rate constant, and n is the order of reaction. The temperature dependence of k is expressed by the Arrhenius equation:

$$k = Ae^{-E_a/RT} \tag{2}$$

where A is the frequency factor (s^{-1}) and T is the absolute temperature. For linear heating rate, a, (deg min^{-1})

$$a = \frac{dT}{dt} \tag{3}$$

For calculating the activation energy, E_a, of thermal decomposition when n = 1, eq 4 was used.

$$\log\left[-\log\frac{1-\alpha}{T^2}\right] = \log\frac{AR}{aE_a}\left[1-\frac{2RT}{E_a}\right] - \frac{E_a}{2.3RT} \tag{4}$$

When $n \neq 1$, eq 5 was used

$$\log\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \log\frac{AR}{aE_a}\left[1-\frac{2RT}{E_a}\right] - \frac{E_a}{2.3RT} \tag{5}$$

Plotting the left-hand-side value of the equation {i.e., log [$1-(1-\alpha)^{1-n}/T^2\,(1-n)$]} against $1/T$ using various values of "n" should give a straight line with the most appropriate value of "n".[26] The least-squares method was applied for the equation, using values of "n" ranging from 0.0 to 3.0 in increments of 0.5. The correlation coefficient (r) and the standard error (SE) were calculated for each value of "n". The "n" value, which corresponds to the maximum r and minimum SE, is the order of the degradation process. The activation energies and frequency factors were calculated from the slope and intercept, respectively, of the Coats−Redfern equation with the most appropriate value of "n".

MDF Production. Two MDF panels for each fiber blend were made using 5% MDI blended with fibers in a drum blender. For each board, 2500 g of fiber and resin were formed in a 20 in. × 20 in. forming box and then pressed at a platen temperature of 180 °C. The press cycle included a 90 s close time, followed by 90 s hold at 1/2 in, followed by a 60 s degass. The panels were left in a hot box overnight prior to trimming and cutting into test specimens.

Physical Testing. Thickness swelling, water absorption, internal bond (IB), and static bending (modulus of rupture and modulus of elasticity) tests were conducted according to ASTM D1037-06a.[27] The IB tests were performed at a crosshead speed of 6 mm per minute. Each value in the plots is the average values of 8 measurements. All error bars shown in subsequent Figures represent the standard error in the mean. The physical and mechanical property data were evaluated statistically using an analysis of variance and pairwise Tukey comparison (Larsen and Marx). A level of significance (alpla) of 0.05 was used for all statistical analyses.[28]

RESULTS AND DISCUSSION

Fiber Characterization. As we previously reported, oxalic acid is effective at removing As, Cr, and Cu from preservative treated wood.[19,20] For example, a 1% oxalic acid solution adjusted to pH 3 with sodium hydroxide was able to remove 100% of As, 97% of Cr, and 99% Cu from preservative-treated wood after 6 h at 80 °C.

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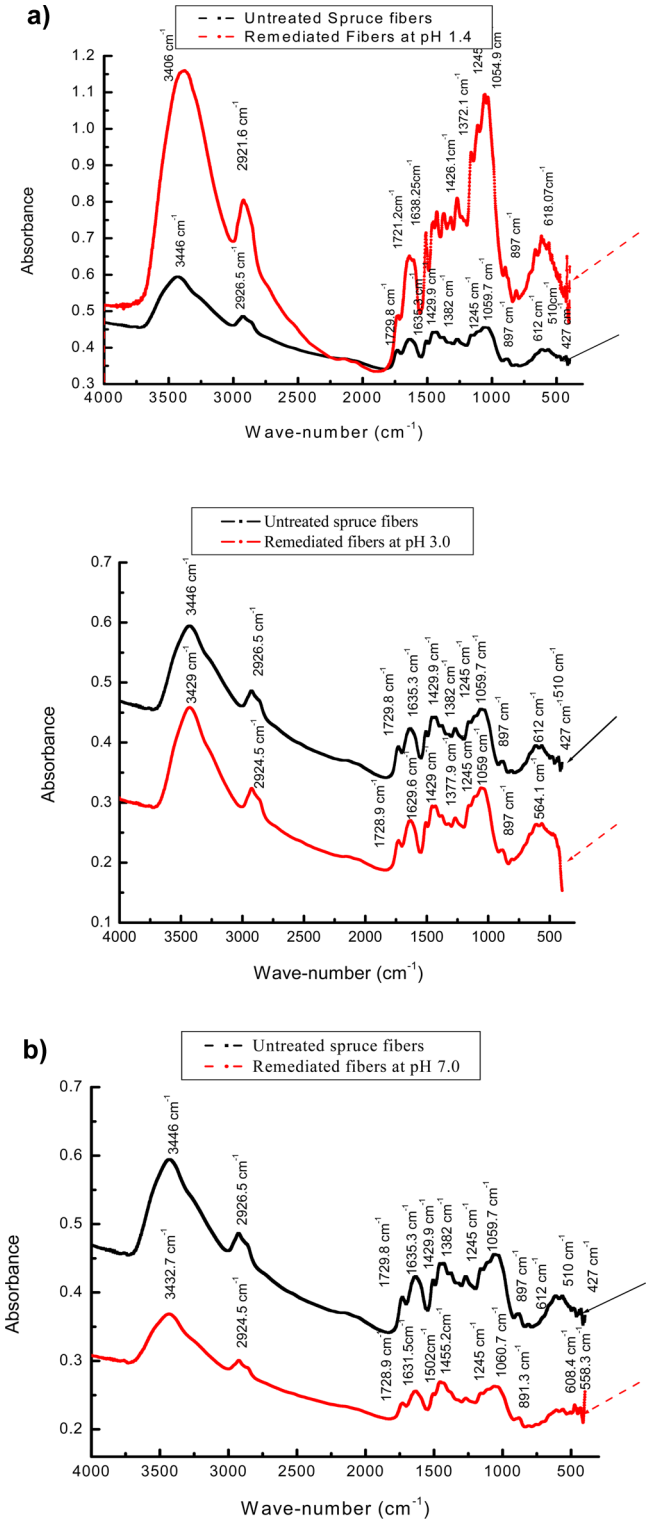

Figure 1. (a) FTIR-spectra of untreated and remediated spruce wood, at different pH's. (b) FTIR-spectra of untreated and remediated spruce wood, at different pH's.

IR Spectra. The practical uses of FTIR spectroscopy in different aspects of wood science was detailed by Faix and Beinhoff.[29] The Fourier transform infrared spectroscopic (FTIR) studies were conducted by Ostmeyer et al.[30] that help in differentiating the basic chemical nature of different species of wood and assist in distinguishing the changes during decay and deterioration. Because the oxalic acid extraction may lead to the deterioration of wood, IR spectroscopy is used in the present study to evaluate the changes in the chemical structure of major components like lignin, cellulose and hemicellulose.

Table 1. IR Measurements of CCA Preservative Spruce and Acid Extraction Treated Wood

sample	MHBS	Cr.I.
untreated fibers	1.22	1.20
treated at pH 1.4	1.44	1.25
treated at pH 3.0	1.42	1.33
treated at pH 7.0	1.23	1.24

The FTIR spectra of CCA preservatives containing spruce wood (untreated) and oxalic acid remediated CCA spruce wood at different pH's (1.4, 3, and 7.0) preservative treated panels are given in Figure 1. In the untreated spruce fiber sample prominent band was observed at ~3446 cm^{-1} was due to the presence of hydrogen bonded O−H stretch. The bands observed at position ~2927 cm^{-1} were due to the C−H stretching vibrations in lignin, cellulose or hemicellulose. In preservative extracted samples, especially at pH ~1.4 these peaks have shifted to lower wavenumber, at ~3406 and 2922 cm^{-1}. When compared to untreated sample, an increase in absorbance intensities of bands corresponds to OH and CH stretching vibrations was observed in the spectra of preservative extracted fibers at pH 1.4−3.0.This is related to the mean hydrogen bond strength in case of acid extraction fibers was higher than untreated and acid extraction at pH 7.0 (Figure 1).

It was observed that, by extracting the preservative, significant changes occur in carbonyl groups associated with lignin, cellulose, or hemicellulose. In unremediated fibers, a significant reduction was observed in band intensity at 1721 cm^{-1} characteristic to unconjugated C═O stretch of hemicellulose than extracted one, whereas the band position was shifted to higher wavenumber ~1729 cm^{-1}. This indicated that, acid extraction led to breakdown of bonds formed between preservative metals with cellulose fibers, and consequently promotes the stretching vibration of carbonyl content. When compared to that of untreated spruce fibers, the characteristic band of conjugated C−O stretch at 1638 cm^{-1} was lost in very acidic extraction treated fibers (pH 1.4). Due to the hydrolyzing effect of oxalic acid extraction at pH 1.4−3.0, the intensities of bands correspond to glycosidic linkage at ~890 cm^{-1} in preservative extracted fibers were lower than untreated ones. Interestingly, despite the extraction at pH 3.0 resulting in higher removal efficiencies of preservative metals, there was no significant reduction in the crystallinity index of cellulose. The ratio of absorbencies related to bands at ~1430 cm^{-1} to bands at ~900 cm^{-1} is around 1.2 (Table 1). This means that the hydrolysis mainly occurred in hemicellulose. This finding supports reduction in the relative intensities of band at 1245 cm^{-1}, from 0.91 to 0.69, which denotes the C−O stretch in lignin and xylan on acid extraction at lower pH. Also, the disappearance of the bands at 510 and 433 cm^{-1} for the spectra of remediated wood at pH 3.0 and 7.0, emphasized the view of removing the metal oxides preservative.

TGA Analysis. For thermal stability evaluation (Table 2 and Figure 2), it is observed that thermal decomposition of untreated CCA loaded spruce fibers is a four stage mechanism: moisture evolution, polymer degradation and volatilisation, and carbonization of the residue as well as decomposition of metal oxides of CCA preservative. Oxalic acid extraction of metals at

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Table 2. TGA and FTIR Measurements of CCA Preservative Spruce and Acid Extraction Treated Wood[a]

sample	stage	temp. range °C	DTG peak max, °C	*n*	E_a, kJ/mol	wt. remain, %
untreated fibers	1st	50−97				95.6
	2nd	201−307	268.6	1.0	120.0	28.3
	3rd	307−332	319.0	1.5	988.0	12.0
	4th	332−397	362.0	1.5	258.7	5.1
treated fibers at pH 1.4	1st	50−95				94.7
	2nd	190−318	276.7	1.0	121.2	28.0
	3rd	318−468	378.7	1.5	134.7	0.1
treated fibers at pH 3.0	1st	50−89				96.3
	2nd	193−313	279.2	1.0	119.2	29.1
	3rd	378−471	428.2	1.5	270.7	2.0
treated fibers at pH 7.0	1st	50−100				94.7
	2nd	186−284	275.4	1.0	129.5	38.9
	3rd	284−316	302.0	1.5	606.3	17.0
	4th	317−402	348.9	1.5	212.8	4.2

[a] *n* is the order of degradation reaction and E_a is the activation energy of degradation.

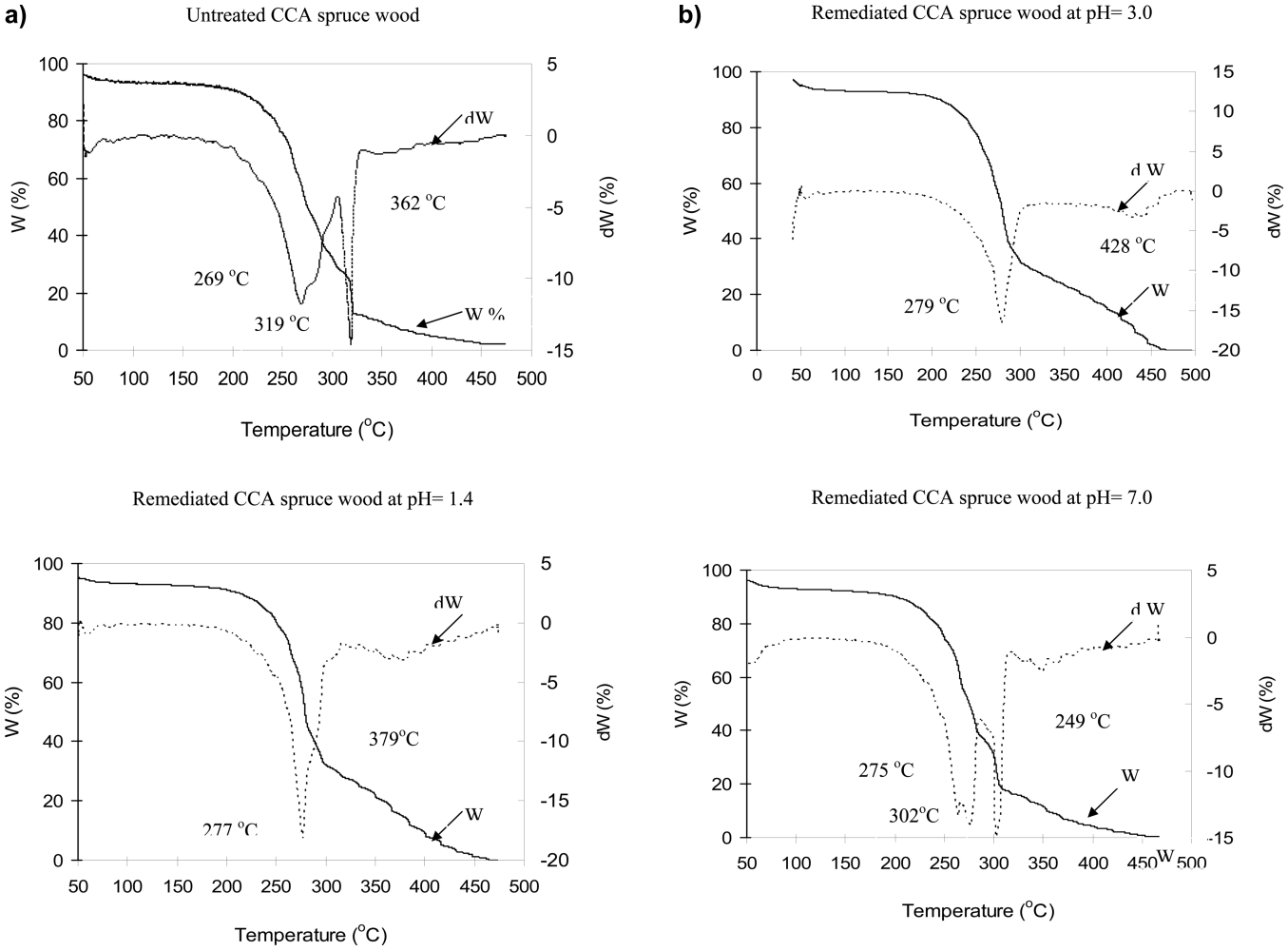

Figure 2. (a) TGA and DTGA curves of untreated and remediated CCA spruce wood. (b) TGA and DTGA curves of untreated and remediated CCA spruce wood.

acidic pH levels (1.4 and 3.0), the degradation stages are reduced to three stages, and this is ascribed to the removal of metals. Before extraction (untreated fibers), the peak maximum (DTG peak max.) and activation energy (E_a) of second degradation stages (~269 °C and 119 kJ/mol) are lower than extracted fibers (treated fibers), especially at acidic pH. Increase in the DTGA peak maximum and activation energy is noticed for treated fibers at pH 3.0 (279 °C and ~230 kJ/mol). This is ascribed to the relatively higher removing the metals which leads to promote the formation of hydrogen bond between the metals-free hydroxyl groups and carbonyl contents of spruce wood constituents. The activation energy of fibers treated at pH 3.0 was higher than at pH 1.4, despite the removal metal efficiencies being higher at pH 3.0. This emphasizes the

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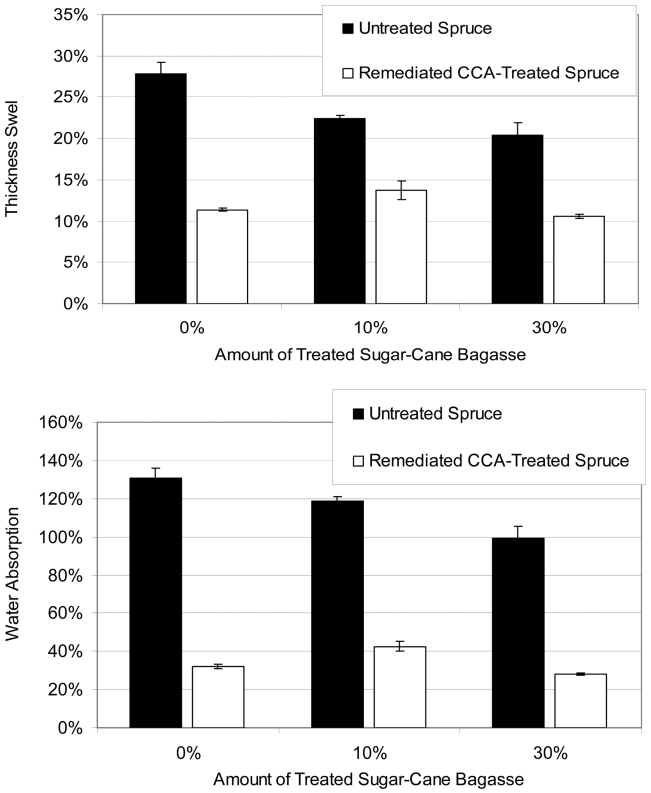

Figure 3. Water resistance properties of MDF made from untreated and remediated CCA spruce fibers, individual or in blends with sugar cane bagasse fibers.

previous observation that higher degradation of hemicelluloses chains occurs during extraction at pH 1.4. As can be noticed that extraction at pH 7.0 is not desirable, both for provided the thermal stability for fibers and removing the CCA preservatives. The values of activation energies in agreement with the mean hydrogen bond strength (MHBS) of IR measurements.

Physical and Mechanical Properties. From the previous analyses it can be concluded that remediation at pH 2.4−3.0, with 1% oxalic acid for 6 h and 80 °C, successfully provided refined fiber for application in production of MDF. Figures 3 and 4 show the properties of fiberboard made from remediated preservative-treated wood compared to those of fiberboard made using unremediated wood fiber. Further, the results clearly show that it is possible to combine and blend un- and remediated wood fibers with nonwood fiber derived from sugar cane bagasse for use in production of enhanced MDF. These enhanced MDF properties are illustrated in Figures 3 and 4. It can be seen that (Figure 3) the MDF produced from the remediated wood absorbed significantly less water and had less swell than boards made from untreated wood (59% and 75.5% reduction WA and TS, respectively). The addition of bagasse fibers to untreated spruce fibers also significantly reduced the water absorption and thickness swelling by about 24% and 27%, which is desirable for MDF panels. Also, blending 30% bagasse fibers with 70% remediated wood provided MDF with lower water resistance properties than those resulted from unremediated wood, where the reduction in WA and TS reached to about 79% and 62%, respectively. An analysis of variance (ANOVA) of the thickness swell and water absorption data indicates that remediation and the amount of bagasse, as well as their interactions, affect the thickness swell of the composites. Pairwise comparisons using Tukey method indicate that the thickness swell is less for both 10 and 30% bagasse added to untreated spruce as compared to untreated spruce alone. However, no significant difference was seen between 10 and 30% bagasse. Pairwise comparisons using Tukey method indicate that the water absorption was not statistically different for untreated spruce without bagasse and with 10% bagasse but that untreated samples with 30% bagasse had less water absorption than untreated samples with 0 or 10% bagasse. Furthermore, no statistical difference in the thickness swell or water absorption was observed among the remediated samples.

Figure 4 shows that boards containing sugar cane bagasse blended either remediated CCA treated or control wood fibers had improved internal bonding (IB). The IB was greatest for samples containing 30% bagasse fibers, in which case the improvement reached about 175% and 244% for the case of using un- and remediated spruce fibers, respectively. An ANOVA indicates that there is no significant difference in the IB strength between untreated and remediated samples. Although, at first glance, the untreated samples appear to have lower IB strength than remediated samples, the variability among the data is quite high, so there is no statistical difference. The addition of 30% bagasse, however, significantly increased the IB strength of the untreated and treated composites.

Figure 4 shows a comparison of the modulus of rupture (MOR) and elasticity (MOE) among all samples. The ANOVA revealed that the amount of sugar cane bagasse added was not found to significantly affect the bending MOE of the boards. On the other hand, an ANOVA revealed that the amount of bagasse added significantly affected the MOR. There was a statistical difference in MOE between remediated and untreated samples but not of the MOR. The MOE of boards from remediated fibers was slightly less than untreated. However, because of the values were close and because the trends were nuanced, any conclusions made about these differences in tenuous.

Economic Potential. While the technical feasibility of using remediated preservative-treated wood, treated bagasse, and possibly other alternative fiber sources seems clear, the economic viability remains unclear. Spelter and colleagues estimate that for alternative fiber sources to be profitable in the United States, they must be deliverable to the mill for $55 per dry ton.[31] Determining an accurate cost for remediating preservative-treated wood is complicated and beyond the scope of this grant, but the cost analysis are likely to depend on a number of factors including 1) the cost to collect waste wood; 2) the ability to recover and reuse or sell extractant chemicals; 3) any offsetting costs that would result from landfilling the waste wood, especially in locations that classify CCA-treated wood as toxic, requiring expensive disposal fees. Technical barriers have not yet been overcome to recover and reuse preservative chemicals, so economic feasibility may depend on the development of such recovery technology. Furthermore, since environmental regulations are continually changing, the economic feasibility of using remediated preservative-treated wood may be impacted by legislative mandates regarding the handling of preservative-treated wood waste.

■ CONCLUSIONS

Remediated CCA treated spruce wood blended with 30% sugar cane bagasse, as famous alternative nonwood fibers for wood application in Egypt, significantly improved the IB strength and water resistance of MDF. Blending bagasse with untreated

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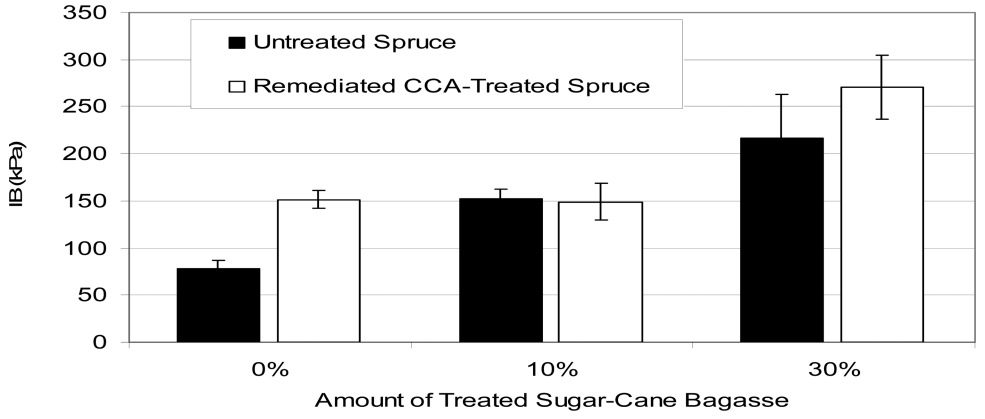

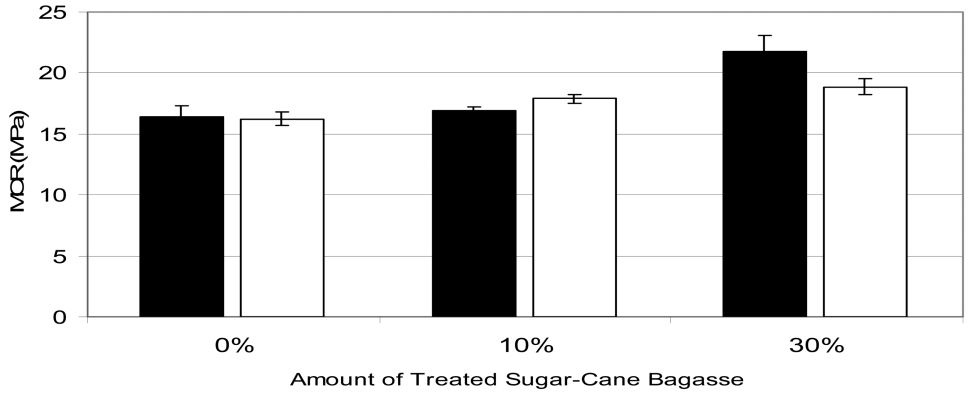

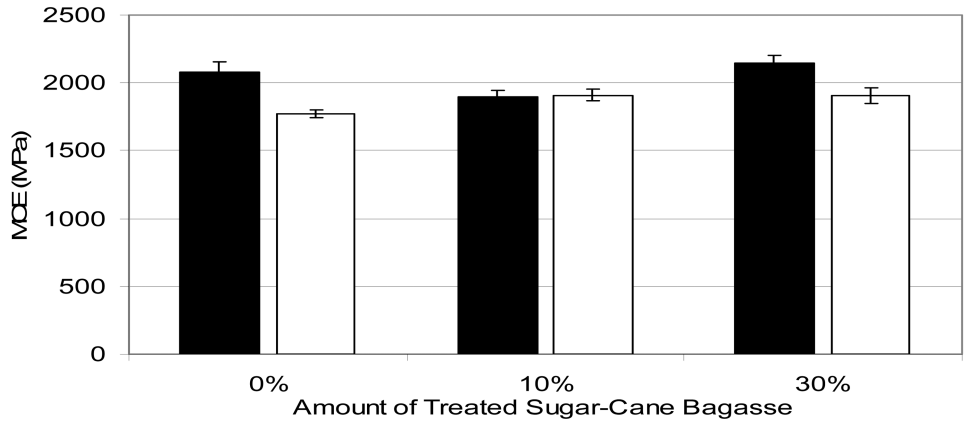

Figure 4. Internal bond (IB) and Static bending strength (MOR and MOE) of MDF made from untreated and remediated CCA spruce fibers, individual or in blends with sugar cane bagasse fibers.

spruce wood has a slight positive effect on the static bending properties (MOR and MOE) of MDF. Properties of MDF made from blending 30% with bagasse fibers, except for IB, can meet ANSI standards for interior MDF.

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Notes

The authors declare no competing financial interest.

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