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Oxidative Activation of Bagasse Fibers Surfaces in Medium Density Fiberboard Manufacturing

Oksidativna aktivacija površine vlakanaca u proizvodnji MDF ploča

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ABSTRACT • *This study presents the investigation of the effects of oxidant type (nitric acid and potassium dichromate), oxidant content (three different levels as 2, 4, and 6 percent) and urea- formaldehyde (UF) resin percentage (two levels as 5 and 7 percent) on mechanical and physical properties of interior grade medium density fiberboard made from bagasse fibers. Some panel properties were studied, such as modulus of rupture (MOR) and modulus of elasticity (MOE) in bending, compression-shear strength (C.S sth.), water absorption (WA) and thickness swelling (TS) after 2- and 24-hour immersion in cold water. In addition, the results indicated the best values for WA and TS after 2- and 24-hour immersion, and C.S. sth. was discerned at 7 percent UF resin content, together with 6 percent nitric acid. Furthermore, the greatest values for MOR and MOE were related to 7 percent UF resin content together with 4 percent nitric acid.*

Keywords: *medium density fiberboard, bagasse, oxidative activation, nitric acid, potassium dichromate*

SAŽETAK • *U studiji je istražen utjecaj vrste oksidansa (dušične kiseline i kalijeva dikromata), sadržaja oksidansa (2, 4 i 6 %) i postotnog udjela urea-formaldehidne smole (UF) (5 i 7 %) na mehanička i fizička svojstva ploča vlaknatica srednje gustoće, proizvedenih od bagasse vlakana. Analizirana su ova svojstva ploča: modul loma (MOR) i modul elastičnosti (MOE) pri savijanju, tlačno-smicajna čvrstoća (C.S sth.) te upijanje vode (WA) i debljinsko bubrenje (TS) nakon 2 i 24 sata potapanja u hladnoj vodi. Rezultati su pokazali najbolje vrijednosti za WA i TS nakon 2 i 24 sata potapanja, te za C.S sth. pri 7 %-tnom sadržaju UF smole i pri 6 % dušične kiseline. Nadalje, najveće vrijednosti za MOR i MOE zabilježene su pri 7 %-tnom sadržaju UF smole i pri 4 % dušične kiseline.*

Ključne riječi: *ploče vlaknatice srednje gustoće, bagasse, oksidativna aktivacija, dušična kiselina, kalijev dikromat*

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1 INTRODUCTION
1. UVOD

Faced with an increasing worldwide wood fiber shortage, environmental considerations, and in order to meet the future demand, the use of non-wood lignocellulosic fiber resources has been increased, and wood composites industry is showing a renewed interest in the production of panel products from agricultural residues (Chow, 1975; Odozi *et al*., 1986; Sampatharajan *et al*., 1992). Unfortunately, in Iran, similar to many developing Asian countries, deforestation and over harvesting have raised environmental awareness, which focused on the studies for using non-wood renewable resources in composite panel production. Non-wood based resources are getting more important as a raw material in the manufacture of composite panels. For countries like Iran, agricultural residues show excellent potential in composite manufacturing industries. Among them, sugarcane residue is one of the best raw materials for that purpose. Bagasse is abundant, unused, and can be obtained at a very low cost. Its lignin content is low and its open structure will facilitate liquid penetration (Zare-Hosseinabadi *et al*., 2008).

Composite panels, such as medium density fiberboard (MDF), are widely used in the construction and furniture industries (Maloney, 1996; Sellers, 2001; Reddy and Yang, 2005). A large amount of increasingly more expensive petroleum-derived adhesives are needed for their manufacture. For example, the production of medium density fiberboard requires a large volume of adhesive, which accounts for up to 20% of the production costs (Pierre-Louis *et al*., 2008). In addition, during production and end-use of MDF, particleboards, and other adhesively bonded products glued with formaldehyde-containing adhesives, such as urea-formaldehyde, formaldehyde emissions are a concern for the manufacturers and consumers (Maloney, 1996; Sellers, 2001). The stringent environmental and human health safety regulations have prompted research into reducing the amount of harmful and/or expensive adhesive components and replacing synthetic adhesives with more environmentally-friendly and safer alternatives (Widsten and Kandelbauer, 2008). One of the considerable potential techniques is the use of oxidizing chemicals to bond wood components (Johns and Woo, 1978). As early as 1939, Tischer (1939) reported on the use of oxidizing agents, such as potassium or sodium dichromate or nitric acid. He concluded that the use of an oxidant may be interpreted as leading to inter- fiber bonding (Tischer, 1939). Surface activation is today a common industrial process for many materials, e. g. in the paper, plastic, metal, wood and wood composites (Nussbaum, 1993). With an activated surface, a higher surface energy is obtained. This gives better bonding qualities in a subsequent operation. Hydrogen bonding and covalent bonding are thereby facilitated, resulting in much stronger bonding than the Van der Waal's forces possible between low energy surfaces (Nussbaum, 1993). A number of techniques are today available for achieving activated bonding (Nussbaum, 1993). Nimz (1974) reported on the use of hydrogen peroxide, a strong oxidizing agent, in mixtures with potassium ferricyanide and pulping residues to bond medium density particleboards. Stofko and Zavarin (1977) reported on the use of a wide variety of oxidants including chromates, nitrates, nitrites, peroxides, perchlorates, permangenates, ferric compounds, and persulfates. Excellent bonding is reported when such materials are dispersed throughout the mat of a high-density fiberboard furnish and hot pressed.

The objective of this study was to evaluate the effects of oxidative activation of bagasse fiber by nitric acid and potassium dichromate on resin consumption, as well as investigate some mechanical and physical properties of dry formed bagasse MDF boards.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Raw material
2.1. Sirovina

Moist depithed bagasse was collected from the MDF moist depithing plant at one of the Agro-Industrial Sites in Southwest of I. R. Iran. Industrial urea-formaldehyde resin (solid content 60 %, PH 6.8-7.1, viscosity 30-45 sec., density 1.28 $g \cdot cm^{-3}$, gel time 50- 65 sec., free formaldehyde max 2 %, pot life 5 hours, water tolerance 6 parts, storage life 4 weeks) was prepared from Tiran Shimi resin factory, I. R. Iran. The chemicals including HNO_3 (100456 nitric acid 65 %), $K_2Cr_2O_7$ (1048625 potassium dichromate), $C_2H_8N_2$ (800947 ethylene diamine) and NH_4Cl (1011430 ammonium chloride) were supplied by MERCK-schuchardt, Germany.

2.2 Bagasse steaming and defibering
2.2. Razvlaknjivanje *bagasse*

The bagasse was delivered to the Pulp and Paper Laboratory, Department of Wood and Paper Science and Technology, Faculty of Natural Resources, University of Tehran, I. R. Iran. A laboratory batch steaming system was used for cooking the bagasse. A sufficient quantity of bagasse was transferred into the steaming vessel and saturated steam was then injected. After a short presteaming time to equalize the steam pressure and temperature inside the steaming vessel, the exhaust valve was closed and steam pressure and temperature were raised up to the start point of steaming condition. The steaming time was started after reaching the target steaming temperature and continued for 5 minutes. One steaming temperature of 175 °C (p = 6 bar) was used. The cooked bagasse was discharged and defibered using a 25 cm laboratory atmospheric single disc refiner. The refined fibers were air dried to reach equilibrium moisture content under laboratory conditions and then fluffed using a hand mixer. Final drying to 1.5 % moisture content was achieved by drying at 110 °C in a laboratory tray dryer. Finally, dried fibers were stored in sealed plastic bags until used.

2.3 Experimental layout
2.3. Provedba eksperimenta

The fibers were then treated by oxidants and resinated by urea-formaldehyde resin according to full factorial experimental design with three factors and 2*3*2 levels shown in Table 1.

Table 1 Full factorial experimental design with three factors and 2*3*2 levels
Tablica 1. Dizajn eksperimenta s tri čimbenika i 2*3*2 razine

Type of oxidant *Vrsta oksidansa*	**UF resin level, %** *Razina UF smole, %*	**Oxidant level, %** *Razina oksidansa, %*	**Treatment No.** *Obrada br.*
Nitric acid *dušična kiselina*	5	2	1
		4	2
		6	3
	7	2	4
		4	5
		6	6
Potassium dichromate *kalijev dikromat*	5	2	7
		4	8
		6	9
	7	2	10
		4	11
		6	12

A three-factorial experiment with a completely randomized design was used for the analysis of variance (ANOVA) of the data, and Duncan's Multiple Range Test (DMRT) was used for differentiation and classification of the average values.

2.4 Chemical treatment
2.4. Kemijska obrada

Oxidative activation of bagasse fibers were carried out before gluing. For this purpose, each of the two different types of oxidant were used at three levels of 2, 4 and 6 percent (based on fiber dry basis). Nitric acid and potassium dichromate were diluted by distilled water and separately added to dry bagasse fibers by spraying in a laboratory rotary drum blender and then stored in sealed plastic bags for 2 hours, after homogenous mixing.

2.5 Panel manufacturing
2.5. Proizvodnja ploča

The panel characteristics and constant parameters for making MDF panels have been presented in Table 2.

Two different levels of 5 and 7 percent of urea-formaldehyde (UF) resin (based on the dry fiber content) were used. An amount of solid ammonium chloride, ethylene diamine (as a cross linking agent), and distilled water were mixed into the liquid UF resin to dilute the resin and achieve the target mat moisture content. Neither paraffin nor other water repellent additives were used. The diluted glue was sprayed onto treated fibers with consistent parameters using a laboratory rotary drum blender consisting of an internal spray nozzle. Then, the resinated fibers were manually formed into mats using a wooden frame. All the fiber mats were hot-pressed under the same hot-pressing parameters (Table 2).

Table 2 Panel manufacturing constant parameters
Tablica 2. Parametri pri proizvodnji ploča

Processing parameter *Proizvodni parametar*	**Value** *Vrijednost*
Ammonium chloride content, % UF o.d.b. *sadržaj amonijeva klorida, % UF o.d.b.*	1.5
Ethylene diamine content, % UF o.d.b. *sadržaj etilen-diamina, % UF o.d.b.*	1.5
Mat moisture content, % fiber o.d.b. *sadržaj vode u tepihu, % fiber o.d.b.*	14
Target density, $kg \cdot cm^{-3}$ *ciljana gustoća,* $kg \cdot cm^{-3}$	0.75
Dimension, mm x mm *dimenzije, mm x mm*	350x350
Nominal thickness, mm *nominalna debljina, mm*	10
Press pressure, $kg \cdot cm^{-2}$ *tlak prešanja,* $kg \cdot cm^{-2}$	35
Press closing time, $mm \cdot s^{-1}$ *brzina zatvaranja preše,* $mm \cdot s^{-1}$	4
Press temperature, °C *temperatura prešanja, °C*	175
Press time, min *vrijeme prešanja, min*	5

According to Table 1, 12 combinations, and three panels per each combination were made, which resulted in a total of 36 treated bagasse MDF panels.

2.6 Panel testing
2.6. Ispitivanje ploča

After cold stacking, to reach equilibrium moisture content, all treated MDF panels were kept in a conditioning chamber at 20±3 °C and 65±1 % *MC* for 2 weeks, in accordance with ASTM standard method (ASTM D 1037-99, 2005). The properties of density, compression-shear strength (*C.S. sth*.), modulus of rupture (*MOR*) in bending and modulus of elasticity (*MOE*) in bending in dry condition, thickness swelling (*TS*) and water absorption (*WA*) after 2 and 24-hour immersion in cold water were measured in accordance with EN standard methods (EN 310: 1993; EN 319: 1993; EN-317: 1993).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The average properties of bagasse medium density fiberboard panels have been presented in Table 3. The results of ANOVA test on the effect of different variables, including the resin percentage, oxidant type, and oxidant content on physical and mechanical properties of test panels, have been summarized in Table 4.

Table 3 Average values of the properties of bagasse MDF boards
Tablica 3. Prosječne vrijednosti svojstava MDF ploča izrađenih od *bagasse* vlakana

Treat. No.	***MC*** %	***D*** $g\cdot cm^{-3}$	***MOR*** MPa	***MOE*** MPa	***C.S sth.*** MPa	***WA***, %		***TS***, %	
						2 h	24 h	2 h	24 h
1	8.1	0.70	9.7	1579	0.65	160	177	74	82
2	8.3	0.714	9.97	1513	0.73	125	137	52	58
3	8.3	0.712	9.87	1798	0.74	101	110	38	42
4	8.2	0.72	12.3	2013	0.89	94	104	42	48
5	8.1	0.709	12.7	1988	0.92	86	95	38	43
6	8.3	0.72	11.6	1896	0.93	79	86	27	30
7	8.4	0.70	10	1611	0.67	143	157	68	78
8	8.4	0.716	9.1	1465	0.67	149	163	71	84
9	8.2	0.70	8.2	1373	0.63	150	166	69	81
10	8.4	0.71	10.7	1604	0.78	123	137	52	59
11	8.1	0.71	12.3	1884	0.83	117	131	52	60
12	8.3	0.7	12.3	1884	0.82	114	131	53	61

MC – moisture content / *sadržaj vode*; D – density / *gustoća*; *MOR* – modulus of rupture / *modul loma*; MOR – modulus of elasticity / *modul elastičnosti*; *C.S sth* – compression-shear strength / *tlačno-smicajna čvrstoća*; *WA* – water absorption / *upijanje vode*; *TS* - thickness swelling / *debljinsko bubrenje*

Table 4 The results of ANOVA test on the effect of variables on MDF properties
Tablica 4. Rezultati ANOVA testa svojstava MDF ploča

Independent variable *Nezavisna varijabla*	**Dependent variable**[b] / *Zavisna varijabla*								
	MC	***D***	***MOR***	***MOE***	***C.S. sth.***	***WA***		***TS***	
						2 h	24 h	2 h	24 h
A	NS	NS	S*	S**	S**	S**	S**	S**	S**
B	NS	NS	S**	S**	S**	S**	S**	S**	S**
A×B	NS	NS	NS	NS	NS	S*	S*	NS	NS
C	NS	NS	NS	NS	NS	S**	S**	S**	S**
A×C	NS	NS	NS	NS	NS	S**	S**	S**	S**
B×C	NS	NS	NS	NS	NS	NS	NS	NS	NS
A×B×C	NS	NS	S**	S**	NS	S**	S**	NS	NS
C.V. (%)	2.14	1.85	7.53	8.19	10.72	7.78	7.60	13.67	13.07

A – oxidant type / *vrsta oksidansa*; B – resin percentage / *udjel smole*; C – oxidant level / *razina oksidansa*; [a]S* – significant at 5 percent level / *značajno na razini 5 %*; S** – significant at 1 percent level / *značajno na razini 1 %*; NS – non-significant / *nije značajno*; C.V. – coefficient of variance (standard deviation/mean) / *koeficijent varijacije (standardna devijacija/srednja vrijednost)*; [b]*MC* – moisture content / *sadržaj vode*; *D* – density / *gustoća*; *MOR* – modulus of rupture in bending / *modul loma pri savijanju*; *MOE* – modulus of elasticity in bending / *modul elastičnosti pri savijanju*; *C.S. sth.* – compression-shear strength / *tlačno-smicajna čvrstoća*; *WA* – water absorption / *upijanje vode*; *TS* – thickness swelling / *debljinsko bubrenje*; 2h – after 2-hour immersion in cold water / *nakon dva sata potapanja u hladnoj vodi*; 24 h – after 24-hour immersion in cold water / *nakon 24 sata potapanja u hladnoj vodi*

The analyses showed that nitric acid was superior to potassium dichromate. Furthermore, the physical and mechanical properties of test panels improved with increasing of resin percentage and oxidant content (Table 3 and 4). Resin percentage showed a strong effect on both physical and mechanical properties of MDF boards. The effect of resin percentage on MDF properties was significant at 99 % confidence interval ($\alpha \leq 0.01$) (Table 4). The best values were attained for 7 % UF resin. For example, the values of 11.99 MPa, 1872 MPa, and 0.861 MPa were measured for *MOR*, *MOE* and C.S. sth., respectively, and 102.16 %, 114 %, 44 % and 50 % were measured, respectively, for *WA* and *TS* after 2 and 24-hour immersion in cold water. The highest effect was observed on thickness swelling. After 2 and 24-hour immersion for 7 % UF resin TS was 71 and 70 percent, respectively, of the TS for 5 % UF resin. This is in agreement with the results of Palardy *et al*. (1989), Chow *et al*. (1996); Gomez-Bueso *et al*. (2000) and Halvarsson *et al*. (2008).

In general, the properties of wood-based particleboards and medium density fiberboards are strongly dependant on the average density and to some extent on the amount of UF resin (Suzuki and Kato, 1989; Hague *et al*., 1999; Wong *et al*., 2000; Shi *et al*., 2005). This might be ascribed to increase fiber surface wettability, fiber surface resin coverage and fiber-fiber contact point, that create cross links (interbonds) between resinated fibers, which leads to the increased forces holding the fibers followed by the increase of resin percentage and consequently also the increase of furnish moisture content (Gomez-Bueso *et al*., 2000; Halvarsson *et al*., 2008).

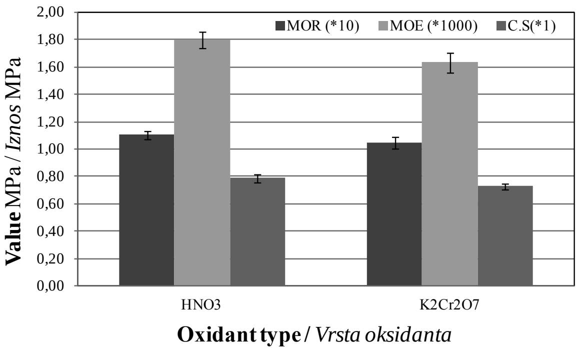

Figure 1 Effect of oxidant type on modulus of rupture (*MOR*), modulus of elasticity (*MOE*) and *C.S* strength
Slika 1. Utjecaj vrste oksidansa na modul loma (*MOR*), modul elastičnosti (*MOE*) i tlačno-smicajnu čvrstoću ploča (*C.S*)

Referring to the results in Table 4 and Figures 1 and 2, significant differences can be observed between physical and mechanical properties of two types of oxidants at 1 % level. The properties of nitric acid were better than those of potassium dichromate. The values of 11.028 MPa, 1798 MPa and 0.811 MPa were achieved for MOR, MOE and C.S. sth., respectively (Fig 1), and 107.6 %, 118 %, 45 % and 51 % for *WA* and *TS* after 2 and 24-hour immersion in cold water was achieved by nitric acid, respectively. The highest effect was observed on thickness swelling. After 2 and 24-hour immersion, *TS* for potassium dichromate was, respectively, 74 and 72 percent of that for nitric acid (Fig. 2). Nitric acid or nitrates introduce nitro-groups onto lignin or polyethylene, causing a reduction in their glass transition temperature up to 20 °C. Nitric acid may react primarily with phenolic groups, but will add nitro-groups at double bonds (Back and Danielsson, 1987). Nitric acid had a more positive effect than chromium nitrate, ferrous sulphate, or periodates (Back and Danielsson, 1987).

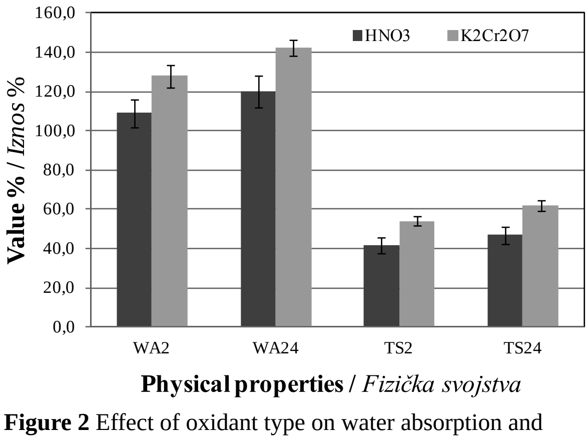

Figure 2 Effect of oxidant type on water absorption and thickness swelling after 2 and 24-hour immersion in cold water
Slika 2. Utjecaj vrste oksidansa na upijanje vode i debljinsko bubrenje ploča nakon dva i 24 sata potapanja u hladnoj vodi

It can be clearly seen from Table 4 that while all measured mechanical properties increased with the increase in oxidant content, no significant difference was shown at 95 % confidence interval ($\alpha \leq 0.05$). It is also clear that the effect of oxidant content was significantly correlated to physical properties at 99 % confidence interval ($\alpha \leq 0.01$).

Water absorption and thickness swelling decreased with increasing oxidant content from 2 to 6 percent. This is in agreement with the results of Shen (1974) and Back (1991). Moreover, the highest effect was observed on thickness swelling, as for 6 % oxidant content it was up to 20 % less than that of 2 % (Fig. 3). Rowell (1986; 1987) pointed out that modifying the cell wall polymers to make them more hydrophobic or bulking them with bonded chemicals would reduce the tendency of wood to swell and shrink by change in moisture content. This may be the reason why *WA* and *TS* were generally reduced by an increasing in oxidant content. Another reason for this may be attributed to high wettability of the oxidized fibers due to functional groups increasing, consequently good penetration of water-soluble urea-formaldehyde resin and its better performance. Also, the use of an oxidant may be interpreted as leading to inter-fiber bonding (Johns and Woo, 1978).

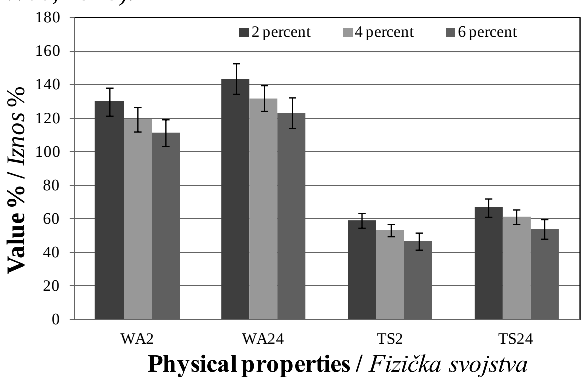

Figure 3 The influence of oxidant percentage on water absorption and thickness swelling after 2 and 24-hour immersion
Slika 3. Utjecaj udjela oksidansa na upijanje vode i debljisko bubrenje nakon dva i 24 sata potapanja u hladnoj vodi

Finally, the results showed that the effects of interaction of oxidant type, oxidant content and resin percentage on *MOR*, *MOE* and *WA* were significant at 99 % confidence interval ($\alpha \leq 0.01$) (Table 4). In addition, the highest values for *MOR* and *MOE* were related to the combination of 2 and 4 percent nitric acid along with 7 percent UF resin content (12.7 MPa and 2013 MPa, respectively) (Tables 3 and 4). The best value for *C.S.* strength was attributed to the combination of 6 percent nitric acid along with 7 percent UF resin content (0.93 MPa). The lowest values of *WA* and *TS* were also achieved for the combination of 6 percent nitric acid along with 7 percent UF resin content. The values of 79 %, 86 %, 27 % and 30 % were measured, respectively, for *WA* and *TS* after 2 and 24-hour immersion in cold water (Tables 3 and 4). Oxidative attack on lignin and especially on hemicelluloses or cellulose can also lead to some chain scission. While this process produces adequate wet strength and water swelling resistance, dry strength can be reduced (Allan and Neogi, 1971; Stenberg, 1978). One of the reasons for very high water absorption and thickness swelling might be ascribed to the fact that no water repellent additive was used. On the other hand, the natural mixture of cellulose, lignin and hemicelluloses in wood material possesses a better resistance to water and water absorption than expected for annual plant materials (Halvarsson *et al*., 2009). Consequently, the fiberboard produced of annual plant materials might have even worse water-resistant properties than fiberboards made of wood material (Sauter, 1996; Markessini *et al*., 1997; Han, 2001; Mantanis and Berns, 2001; Wasylciw, 2001; Ye *et al*., 2007). The ability of water absorption into the oxidized lignocellulosic materials will also increase and contribute to a higher water sensitivity of low resin fiberboards (Halvarsson *et al*., 2009).

Even though the addition of nitric acid and potassium dichromate improved the fiberboard properties, none of the manufactured bagasse fiberboards met the European wood-based MDF standard (EN 622-5: 2006). The water swelling properties were adversely affected, so *TS* and *WA* were several times higher than specified by the MDF standard.

4 CONCLUSIONS
4. ZAKLJUČAK

According to the results, mechanical and physical properties of chemically treated bagasse MDF boards were strongly depended on resin percentage. The higher resin contents, the better mechanical and physical properties. Nitric acid showed better results than potassium dichromate, especially for thickness swelling. Furthermore, the properties of bagasse MDF boards improved with increasing oxidant content. Mechanical and physical properties of medium density fiberboards made from low resin oxidized bagasse fibers were not acceptable according to the requirements of the EN standards for MDF. Further studies should focus on methods for improving physical properties and mechanical strength of medium density fiberboards.

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