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Thermal Stability of Wood Fibers Produced from Recycled Medium Density Fiberboards

Toplinska stabilnost drvenih vlakana proizvedenih od recikliranih srednje gustih ploča vlaknatica

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ABSTRACT • *In this study, thermal stability of fibers obtained from recycled MDF was investigated and compared with virgin fibers by using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Two different methods, including electrical heating and hydrothermal treatment, were used for recycling the MDF wastes. Electrical heating method was performed at two different times (2 and 4 min) and hydrothermal method was done at three different temperatures (105, 125 and 150 °C). Chemical structure of wood fibers was also studied. TGA and DSC analysis showed higher weight loss of recycled fibers as compared to virgin fibers in a similar degradation region, which means that thermal stability of recycled fibers is lower than virgin fibers. In fact, thermal behavior of recycled fibers was medium between wood and UF resin. DSC analysis showed two exotherms at around 340 and 475 °C. The transition at around 340 °C in fibers thermogram was considered to be due to polysaccharides thermal deterioration and the exotherm at 475 °C was related to lignin carbohydrate complex deterioration. The results of chemical structure analysis showed that lignin and hemicellulose content of recycled fibers was significantly lower than that of virgin fibers, which resulted in decreased thermal stability.*

Keywords: *thermal stability, recycled fibers, MDF, electrical heating, hydrothermal*

SAŽETAK • *U ovom je istraživanju proučavana toplinska stabilnost vlakana dobivenih od recikliranih MDF ploča te je uz pomoć termogravimetrijske analize (TGA) i diferencijalne skenirajuće kalorimetrije (DSC) uspoređena s izvornim vlaknima. Za recikliranje otpadnih MDF ploča primijenjene su dvije različite metode – električno zagrijavanje i hidrotermička obrada. Metoda električnog zagrijavanja provedena je tijekom dva različita vremenska trajanja (2 i 4 min), a hidrotermička metoda provedena je pri tri različite temperature (105, 125 i 150 °C). Proučavana je i kemijska struktura drvnih vlakana. Analize TGA i DSC pokazale su veći gubitak mase recikliranih vlakana u usporedbi s izvornim vlaknima u sličnoj fazi razgradnje, što znači da je toplinska stabilnost recikliranih vlakana niža od toplinske stabilnosti izvornih vlakana. Zapravo, toplinsko ponašanje recikliranih vlakana nalazi*

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se između drva i UF smole. DSC analizom dobivena su dva egzotermna pika na oko 340 i 475 °C. Prijelaz na oko 340 °C u termogramu pripisuje se propadanju polisaharida, a egzotermna reakcija na 475 °C posljedica je propadanja spojeva ugljikohidrata i lignina. Rezultati analize kemijske strukture pokazali su da je sadržaj lignina i hemiceluloze u recikliranim vlaknima znatno manji od njegova sadržaja u izvornim vlaknima, što je rezultiralo smanjenjem toplinske stabilnosti.

Ključne riječi: *toplinska stabilnost, reciklirana vlakna, MDF ploče, električno zagrijavanje, hidrotermička metoda*

1 INTRODUCTION
1. UVOD

Nowadays, wood based panels, especially medium density fiber boards, are considered the main raw materials used in the furniture industry. However, increased production and consumption of MDF boards leads to generation of large volumes of waste and this trend will continue in the future. The generated waste is usually disposed in land filling or burned, which introduces some adverse environmental effects, especially CO_2 and CH_4 emissions (greenhouse gases) (Lykidis and Grigoriou, 2008). These methods have already been forbidden in many European countries due to environmental considerations (Athanassiadou *et al*., 2005).

In addition, the depletion of wood fiber resources and continuous rising cost of raw materials initiated the search for alternative sources of raw materials, among which wood and panel board wastes such as MDF wastes (Wolff and Siempelcamp, 2000; Michanickle and Boehme, 2003) and recycling started being considered the most environmentally friendly method of managing wood wastes (Lykidis and Grigoriou, 2008). Reuse of fibers contained in MDF wastes can return part of raw material to the production and improve the efficiency of board production and processing plants.

The use of recycled fibers for manufacturing MDF boards requires a comprehensive understanding of their characteristics as compared to virgin fibers. The properties of recycled fibers might be different from virgin fibers, which can effectively impact the quality of manufactured MDF boards. The recycled fibers are exposed to severe conditions in two different processes including board manufacturing and hydrothermal recycling treatment. These fibers undergo gluing and pressing steps when they are used for board manufacturing. The effect of possible residues of resins on the surface of fibers might seriously affect the quality of recycled fibers and this should be investigated. Feng *et al*. (2012) studied the influence of urea formaldehyde resins on pyrolysis characteristics of wood based panels. They found that UF resin used in wood based panels accelerated the degradation of wood based panels at lower temperatures and prevented the degradation at higher temperatures.

Chen *et al*. (2015) investigated the influence of urea formaldehyde resin on pyrolysis characteristics and gas evolution of waste MDF. The results of their study showed that the process of MDF pyrolysis could be divided into three main phases. The degradation rate of MDF was higher than that of wood fibers due to the influence of UF resin.

Additionally, changes in chemical structure of recycled fibers occurred during heating processes, which could influence the thermal stability of fibers (Lykidis and Grigoriou, 2008). One of the most important properties of wood fibers used in MDF manufacturing is thermal stability, which is changed in recycled fibers. Thermal stability of fibers means the resistance against high temperature during hot pressing and influences the chemical structure of fibers, which can seriously affect the physical and mechanical properties of manufactured MDF boards.

In this study, thermal stability of recycled fibers was investigated and compered with that of virgin fibers. The chemical composition of recycled fibers was also measured and compared with virgin fibers.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

For this study, MDF wastes (trimmings) and virgin fibers were collected from Pars Neopan MDF plnt located in Nashtarood, Mazadaran, North of Iran. Then, the MDF wastes were cut to smaller size, and chipped using a laboratory drum chipper.

The recycled MDF chips were defibrated applying two different methods; hydrothermal and electrical heating methods.

In hydrothermal method, the chips were steamed using laboratory autoclave at 105 ˚C for 150 min, under four bar pressure. Then, the steamed heated and softened chips were defibrated using 25 cm single disc refiner to produce fibers. Electrical heating of MDF chips was performed using laboratory scale device designed and manufactured especially for this study (Figure 1). The system consists of an isolating transformer and a heating cell.

The heating cell is a polypropylene rectangular shaped chamber with the dimension: length of 30 cm; width of 6 cm and height of 10 cm. Two removable stainless steel electrodes with thickness of 0.2 cm are installed inside the longer walls of the chamber. For heating the MDF chips by electrical heating, first the chips were crushed and then soaked in warm water for 30 minutes. Then, the mixture of wet chips and electrolyte (saltwater) was poured into the chamber. The device was turned on and electrical current was passed through the mixture starting the heating treatment. The temperature of the mixture during the heating treatment was adjusted at ultimate temperature of 100 °C. Two heating times of 2 and 4 minutes were selected to find the best time for treatment. The fibers were dried at 100±3 °C in an oven to reach the target moisture content (3-4 %).

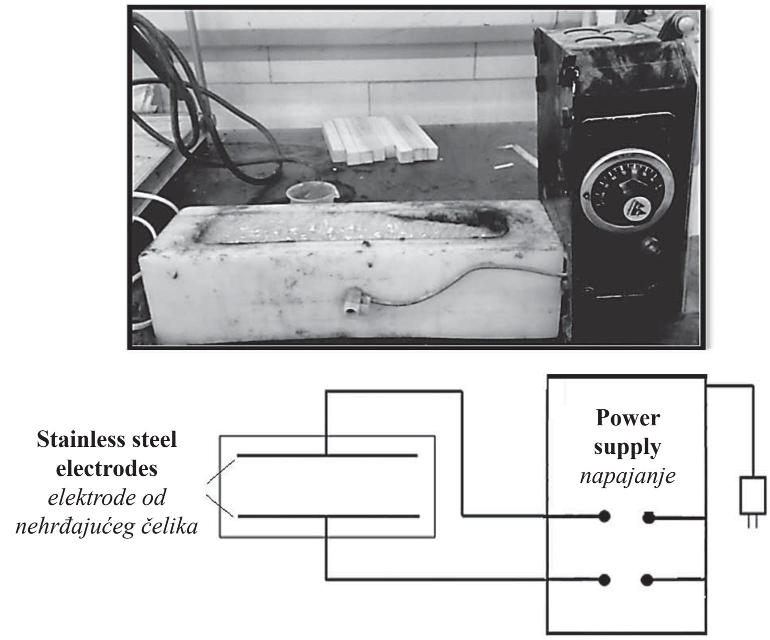

Figure 1 Laboratory electrical heating device for recycling MDF wastes
Slika 1. Laboratorijski električni grijač za recikliranje otpadnih MDF ploča

Chemical analyses

The samples for chemical analysis were taken randomly from dried fibers of each treatment. The chemical analysis was performed in three replications and the mean values for each component was reported. Relevant Tappi test methods used for chemical analysis were as follows: Sample preparation; T267-om 85: Extractives soluble in organic solvent; T207-om 97: Cellulose; T264-om 88: Lignin; T222-om 97: Holocellulose; Useful method 249- um 75.

Thermal stability analysis

Thermal behavior of recycled and virgin fibers was determined using Simultaneous Thermal Analysis (TGA/DSC) in a Mettler Toledo Analyzer, which was fully supported by computer – controlled software options for control and data handling. Ten mg of each sample was placed on a balance located in the furnace and heating was applied over the temperature range from room temperature up to 600 °C. The analysis was run under a dynamic nitrogen atmosphere flow at 20 mL/min and at a scanning rate of 20 °C/min. The derivatives of the weight loss vs. temperature thermograms were obtained to better show the different decomposition processes.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The results of measurement of chemical composition of fibers are shown in Table 1.

As can be seen in Table 1, extractives and hemicelluloses contents of recycled fibers (RF-1, RF-2 and RF-3) were significantly lower than those of virgin fibers. Lignin content was also changed during the recycling process, especially for fibers recycled via hydrothermal method (RF-3). However, cellulose content of fibers was not significantly reduced after the recycling process.

During thermal treatments, hemicelluloses usually undergo the highest degree of degradation as compared to other chemical compositions of wood due to their amorphous structure (MC Kendry, 2002; Rao and Sharma, 1998). Hemicelluloses show lower thermal stability among wood constituents due to the presence of acetyl groups (Hill, 2006). Hence, in mild thermal treatment and acidic conditions, hemicelluloses can easily hydrolyze (Tjeerdsma and Militz, 2005).

Lignin can be degraded in a wide temperature range, which begins at low temperatures (Nassar and Mackay, 1984). However, thermal degradation of cel-

Table 1 Chemical constituents of virgin and recycled fibers (electrical heating 2 min: RF-1; electrical heating 4 min: RF-2; Hydrothermal heating: RF-3)
Tablica 1. Kemijski sastav izvornih i recikliranih vlakana (električno zagrijavanje u trajanju 2 min: RF-1; električno zagrijavanje u trajanju 4 min: RF-2; hidrotermičko zagrijavanje: RF-3)

Fiber type *Vrsta vlakana*	**Lignin** *Lignin* %	**Hemicellulose** *Hemiceluloza* %	**Cellulose** *Celuloza* %	**Extractives** *Ekstraktivi* %
Virgin / *izvorno*	23.4	20.14	46.15	3.8
RF-1	22.45	15.42	46.2	1.57
RF-2	22.3	15.2	45.12	1.4
RF-3	19.5	14.44	46.18	1.22

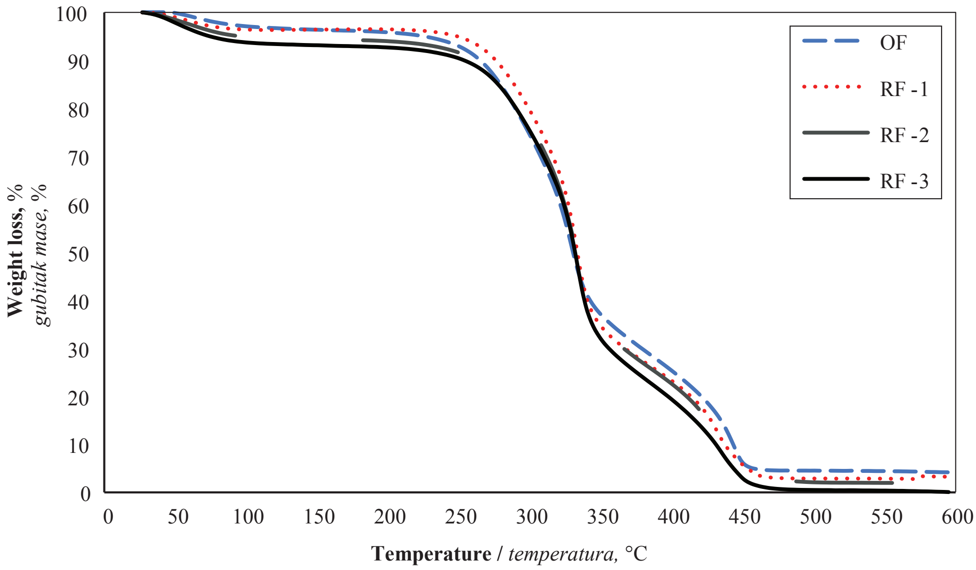

Figure 2 TGA curves of virgin fibers and recycled fibers; ohmic heating 2 min (RF-1), ohmic heating 4 min (RF-2) and hydrothermal heating (RF-3)
Slika 2. TGA krivulje izvornih i recikliranih vlakana: omsko zagrijavanje u trajanju 2 min (RF-1), omsko zagrijavanje u trajanju 4 min (RF-2) i hidrotermičko zagrijavanje (RF-3)

lulose occurred at higher temperatures (above 210 °C) (Bodirlau *et al*. 2009; Yang *et al*., 2007; Wang *et al*., 2007; Lumming *et al*., 2013). It is obvious that the chemical structure of recycled fibers ought to be different from virgin fibers due to several heating treatments. Chemical constituents are considered as main factors affecting the properties of wood fibers and such changes have an obvious influence on the quality of manufactured boards.

Figures 2-3 show TGA thermograms of virgin and recycled fibers, respectively. According to TGA results, three regions can be determined for the approximate starting and ending points of thermogravimetric analysis curve. The thermal degradation mechanism of woods generally includes three regions. The first region (60 to 100 °C) shows evaporation of water and extractives (Feng *et al*., 2012; Bodirlau *et al*., 2009). The second region (130 to 345 °C) is due to decomposition of major wood chemical constituents and the highest weight loss occurs in this step (Feng *et al*., 2012; Aydemir *et al*., 2011). The third region begins around 400 °C in which decomposition of wood occurs. The thermal degradation steps found in our experiment are in agreement with the results reported by Aydemir *et al*., 2011. Summary data of TGA are presented in Table 2.

The results illustrated in Table 2 show that the weight loss of virgin fibers was lower than that of recycled fibers. However, it is necessary to focus on details. The trend of changes in the amount of weight loss from the first phase to the third phase confirmed that in the first phase (60-130 °C), i.e. at lower temperature, the weight loss was 3.63 % in virgin fibers and about 5-6 % in recycled fibers, which showed that at this low

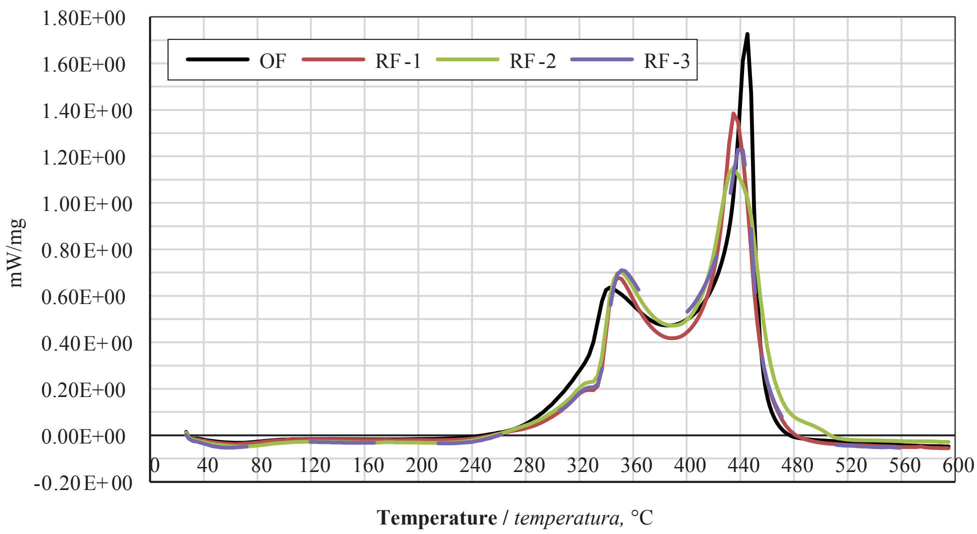

Figure 3 DSC thermograms of virgin fibers (OF), recycled fibers by ohmic heating method for 2 min (RF-1), ohmic heating method for 4 min (RF-2) and hydrothermal method (RF-3)
Slika 3. DSC termogrami izvornih i recikliranih vlakana dobivenih omskim zagrijavanjem u trajanju 2 min (RF-1), omskim zagrijavanjem u trajanju 4 min (RF-2) i hidrotermičkim zagrijavanjem (RF-3)

Table 2 TGA results for virgin and recycled fibers
Tablica 2. TGA rezultati izvornih i recikliranih vlakana

Samples *Uzorci*	T_i-T_f, °C	T_m, °C	W_{TI}, %	W_{max} (second region) W_{max} *(drugo područje)*, %	W_{TI}, %	**Weight loss in the first phase** *Gubitak mase u prvoj fazi*, %	**Weight loss in the second phase** *Gubitak mase u drugoj fazi*, %	**Weight loss in the third phase** *Gubitak mase u trećoj fazi*, %	**Residues** *Ostatci*, %
virgin	130-147	440	3.63	58.35	33.94	3.63	58.35	33.94	4.23
RF-1	130-480	420	3.64	62.87	30.21	3.62	62.87	30.21	3.26
RF-2	130-500	420	5.52	62.63	30.08	5.52	62.63	30.08	1.79
RF-3	135-515	425	6.69	62.86	30.33	6.69	62.86	30.33	0.1

T_i-T_f - temperature corresponding to the beginning and ending of decomposition, respectively / *temperatura na početku i na kraju razgradnje*; T_m - temperature corresponding to the maximum rate of mass loss / *temperatura pri maksimalnom gubitku mase*; *W* - weight loss / *gubitak mase*

temperature, the degradation of recycled fibers is faster in recycled fibers as compared to virgin fibers. In the second phase (130-345 °C), along with rising temperature, the difference between the amount of weight loss in recycled and virgin fibers was decreased and they were nearly equal; (58.35 % weight loss in virgin fibers and about 62 % in recycled fibers). Finally, in the third phase (345-600 °C), i.e. at a higher range of temperature, the weight loss in virgin fibers (33.94 %) was obviously higher than that in recycled fiber (30 % in average), which shows that at higher temperatures the presence of UF resin prevents further degradation of fibers. The reason for this variable effect of UF resin was referred to its behavior during pyrolysis at different temperatures. These results are in accordance with the findings of Feng *et al*. (2012) and Chen *et al*. (2015). Overall, it was found from the amount of residues that the presence of UF resin totally decreased the thermal stability of fibers, because maximum weight loss occurred in the second phase and it was higher in recycled fibers.

It was also observed that decomposition temperature (T_d) in the second region was lower in virgin fibers as compared to recycled fibers. Similar result was reported by Chen *et al*. (2015). It can be concluded that the rate of degradation of virgin fibers was lower in comparison with recycled fibers.

The final mass residues of virgin fibers (4.23 %) was higher than that of recycled fibers and the lowest mass residues of recycled fibers was observed in fibers under hydrothermal treatment (0.1 %). The residues of RF-1 an RF-2 were 3.26 % and 1.79 %, respectively, which shows higher thermal stability of virgin fibers. Feng *et al*. (2012) and Peng *et al*. (2011) also reported that the pyrolysis of wood material in combination with UF resin is not independent from decomposition of wood and UF resin. They concluded that UF resin accelerates the chemical reactions in wood based panels at a lower temperature and inhibits the degradation at higher temperatures during the pyrolysis process. The presence of UF resin residues on the surface of recycled fibers resulted in a lower amount of residues after decomposition of recycled fibers as compared to virgin fibers, which was reported on wood based panels by Chen *et al.* (2015). The greater weight loss shows the lowest thermal stability (Aydemir *et al*., 2011).

Generally, it can be concluded that the thermal stability of fibers was reduced after the recycling process and thermal behavior of fibers confirmed the existence of UF resin residues on the surface of recycled fibers, which can increase the thermal stability of fibers at the temperature above 400 °C.

Figure 3 shows the results of DSC analysis on recycled and virgin fibers.

In DSC curves of virgin and all recycled fibers, two main peaks can be observed at 330 to 360 °C and 470 to 480 °C, respectively, related to carbohydrate degradation and lignin-carbohydrate complex degradation. (Islam *et al*., 2011, Tsujiyama, 2001; Tsujiyama and Miamouri, 2000).

The change in the peak shape at 320 to 325 °C observed in recycled fibers is related to the existence of UF resin residues, which degraded completely in this temperature range (Raval *et al*., 2005).

In recycled fibers, the second peak, which is related to lignin-carbohydrate complex, appears at the temperature lower than that of virgin fibers (440 °C instead of 470 °C) as a result of lignin decrease and its effect on the peak (Tsujiyama and Miamouri, 2000; Reh *et al*., 1986). According to the results obtained from chemical structure analysis, recycled fibers con-

Table 3 Results of DSC analysis of virgin and recycled fibers
Tablica 3. Rezultati DSC analize izvornih i recikliranih vlakana

Fibers *Vlakna*	**Exotherm peaks,** °C *Egzotermni pikovi,* °C		**Reaction enthalpy ΔH,** J/gm *Entalpija reakcije ΔH,* J/gm	
	2st	1st	2st	1st
virgin	444.86	342.53	5957.73	4364.72
RF-1	436.30	349.98	5491.45	3938.86
RF-2	434.58	350.51	5769.30	4139.96
RF-3	439.69	352.81	5197.72	4111.30

tain lower amounts of lignin as compared to virgin fibers due to degradation reactions but no change has been observed in cellulose. The results presented in Table 3 show that the temperature of the first peak is lower in virgin fibers compared to recycled fibers, while the second peak is quite the opposite.

The reaction enthalpy in virgin fibers is higher than that of recycled fibers. The intensity of peaks shows that the level of degradation at the lower intensity corresponds to higher stability (Islam *et al*., 2011). Accordingly, it can be said that stability of recycled fibers is lower than that of virgin fibers, which confirms the results obtained from TGA.

The thermal properties of the lignocellulosic fibers are mainly influenced by their composition; cellulose, hemicelluloses and lignin content (Ornaghi jr. *et al*., 2014; Poletto *et al.*, 2012b). The results of Table 1 revealed that recycled fibers have a lower content of hemicellulose, lignin and extractives in comparison with virgin fibers, which can be the reason for decreased thermal stability of recycled fibers. The degradation of wood due to high temperatures can lead to the loss of wood mechanical strength and also produces MDF with low mechanical properties (Poletto, 2016). Indeed, the chemical structure of fibers and their thermal stability have a mutual effect on each other. Changes in chemical structure lead to decreased thermal stability of fibers and decreased thermal stability results in the change of chemical structure of fibers. This interaction might be the reason for the inferior quality of recycled MDF boards.

4 CONCLUSIONS
4. ZAKLJUČAK

The results showed that the existence of UF resin residues accelerated the thermal degradation of fibers at lower temperatures, while in the final step (complete degradation), the level of degradation of virgin fibers was higher than that of recycled fibers. The thermal behavior of recycled fibers was medium between wood and urea formaldehyde resin. Finally, the amount of residues of recycled fibers was much lower than that of virgin fibers.

The results obtained from DSC confirmed the results of TGA. The changes in the position of the second peak in recycled fibers showed the decrease of lignin content. Generally, it can be concluded that the chemical structure of recycled fibers has been changed due to the primary manufacturing process of MDF boards and recycling process. The structure of recycled fibers is completely different from that of virgin fibers. Gluing, pressing and heating processes, followed by heating and defiberation steps in recycling these fibers from MDF wastes, result in weakened fibers and cause a notable change in the chemical structure of fibers as compared to virgin fibers. Additionally, the existence of UF resin residues on the surface of recycled fibers seriously affects thermal stability. Consequently, the thermal stability of recycled fibers significantly decreased in comparison with virgin fibers and the recycled fibers could not endure high temperatures during hot pressing in MDF board manufacturing due to their damaged chemical structure and deceased thermal stability. It is very important to consider the different properties of recycled fibers when these fibers are used in MDF board manufacturing.

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