

Research Article

Drainage in a Screw Press and Utilization of the Recovered Fibres after Thermo-Hydrolytic Disintegration of Waste Fibreboards

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Abstract

A thermo-hydrolytic disintegration process qualifies as a promising option for recycling the waste MDF and preserving the fibrous morphology of the recovered lignocellulosic fibre material. This study aims to include a drainage process between the thermo-hydrolytic disintegration and the further utilization of the recovered fibres (RF) obtained using a screw press for removing the disintegration water (DW). In this context, the chemical properties of the RF (pH, nitrogen content, formaldehyde emissions) and the DW (pH, formaldehyde, reducing sugars and equivalents and nitrogen contents) were analyzed. Moreover, the RF material was utilized to produce recycled MDF panels, solely containing the RF (100%) and hence supplanting 50% of the virgin fibres (VF). The recycled MDF portrayed significant reductions in the internal bond strength (IB), and flexural properties (MOR, MOE): in the case of MDF made from 100% recycled fibres, about half the strength was reduced, and in the case of MDF made from 50% recycled fibres, the strength was reduced by 20-25%. The Thickness swelling (TS) of the recycled MDF panels was similar, while the water uptake (WA) was higher than that of the original MDF. The recycled MDF panels also exhibited a higher content of formaldehyde and emission. The findings recommend the application of a screw press process for prompter drainage of the RF and to utilize the RF obtained in combination with the VF to



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achieve adequate mechanical properties rather than using the RF separately for the manufacturing of the recycled MDF panels.

Keywords

Fibreboards; MDF; wood-based panels; waste; thermo-hydrolytic disintegration; screw press; mechanical properties; physical properties; formaldehyde release

1. Introduction

Fibreboards are defined as a dry-formed panel product manufactured from ca. 80-90% of fine lignocellulosic fibres are obtained from the thermo-mechanical pulping (TMP) from a refiner combined with a synthetic adhesive (generally urea-formaldehyde, UF resin). The global production of medium-density fibreboards (MDF) along with high-density fibreboards (HDF) reached a total of 98.6 million m³ in 2018 [1]. On completion of their service life, these panels turn into waste, and a majority are deposited in landfills or are burned. In Europe, waste wood materials consisting of more than 5% of organic materials have been prohibited from landfilling since 2005 [2]. Recycling of the MDF wastes presents an opportunity for extending the application of wood resources, reduction in the consumption of new resources, energy, landfilling as well as the expenses avoided through purchase/disposal fees, and helps in the creation of “green” jobs [3]. The thermo-hydrolytic disintegration process would be a promising option to preserve the fibrous morphology of the recovered lignocellulosic fibre material during the release of the fibres from the thermosetting resin matrix. Thermo-hydrolytic disintegration processes involve the application of liquid water, steam, heat and occasionally pressure for cleaving the existing adhesive bonds in waste MDF [2]. Toward the end of the disintegration process, recovered fibres (RF) and a resin-water mixture, termed disintegration water (DW), are obtained. The implementation of an appropriate de-watering process for separating the RF from the DW is an important aspect for reducing the drying time of the RF.

The effects of using RF on the properties of the newly produced MDF after repeated thermo-hydrolytic disintegration processes have been investigated [4]. This study aims to implement a drainage process between the previously researched thermo-hydrolytic disintegration process and the further utilization of the obtained RF using a screw press for removing the DW. In this context, the analysis of the chemical and morphological properties of the RF and the chemical properties of the DW was made. Moreover, the RF material is utilized for producing recycled MDF panels solely containing the RF (100%) and supplanting 50% of these by virgin fibres. The paper also explains the physicommechanical as well as chemical (such as formaldehyde release) properties of these MDF panels.

2. Materials and Methods

2.1 Materials

Three types of uncoated industrial fibreboards (medium-density fibreboard-MDF and high-density fibreboard-HDF) having different densities, thicknesses, as well as binder types and amounts,

were used for the thermo-hydrolytic disintegration process (Table 1 and Figure 1). Moreover, the nitrogen content (NC) of the panels was determined using the Kjeldahl method as described previously [5]. The UF content of the MDF panels was calculated keeping in mind that the UF resin contained roughly 30% nitrogen, neglecting the very low NC of the fibres (<0.1%) used for producing the panels [4] (Table 1). However, due to the complexity in formulation, the UF content for Board-1 could not be calculated from the nitrogen content alone.

Table 1 Properties of the uncoated industrial fibreboards.

Fibreboard properties		Board-1	Board-2	Board-3
	Fibreboard type	MDF	MDF	HDF
	Density (kg m ⁻³)	792	739	862
	Thickness (mm)	6.0	19.0	6.6
Binder type	Urea formaldehyde (UF) amount (%)	5.5	9.9	14.7
	Melamine urea formaldehyde amount (%)	10.5	0	0
	Analyzed nitrogen content (%)	6.0	3.6	5.0
	Calculated UF content (%)	n.c.	11.5	16.0

n.c. = "not calculated".



Figure 1 Uncoated industrial fibreboards used for the thermo-hydrolytic disintegration process.

2.2 Thermo-Hydrolytic Disintegration of Waste Fibreboards

Firstly, each fibreboard type was cut into pieces having dimensions of 25 × 25 mm² in cross-section by using a circular saw. The thermo-hydrolytic disintegration process was carried out separately for each type of fibreboard. Hence, for every disintegration, 9 kg of these fibreboard pieces were immersed in 30 liters of water at 95 °C in the autoclave Zirbus Z3 (Zirbus Technology GmbH, Bad Grund, Germany) for 30 min (Figure 2a). After every disintegration process, the

recovered fibres, along with the disintegration water, were stored in barrels made of high-density polyethene and was sent to Maschinenfabrik Reinartz GmbH & Co. KG (Neuss, Germany).

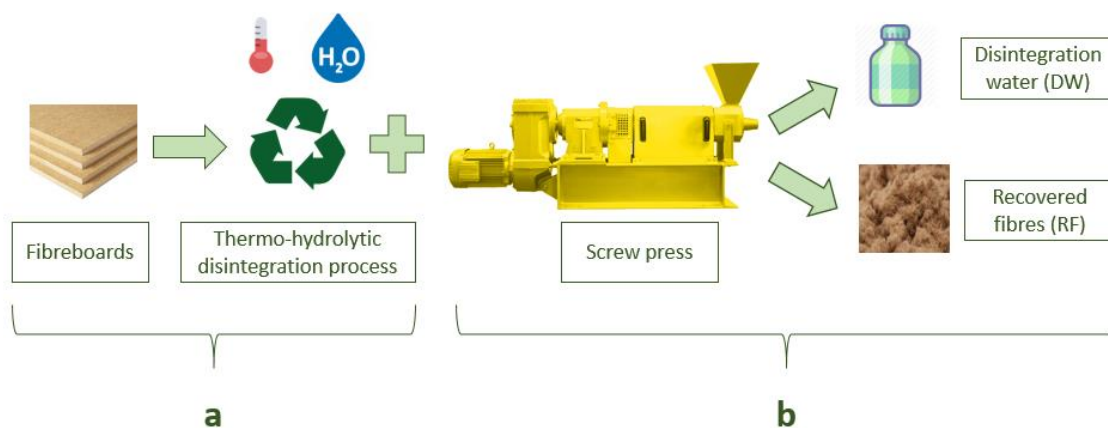


Figure 2 Schematic illustration of a) Thermo-hydrolytic disintegration of waste fibreboards, b) Drainage of recovered fibres using a screw press.

2.3 Drainage of Recovered Fibres Using a Screw Press

The excess water (disintegration water -DW) was removed using a screw press developed by Maschinenfabrik Reinartz GmbH & Co. KG (Neuss, Germany). Thus, the RF and DW were obtained separately (shown in Table 2 and Figure 2b). To determine the chemical and morphological properties, the RF was conditioned in a climate chamber at 20 °C with 65% relative humidity.

Table 2 Labeling system of the recovered fibres (RF) obtained and disintegration water (DW) samples after the disintegration processes.

Disintegrated fibreboard	Obtained recovered fibre	Obtained disintegration water
Board-1	RF1	DW1
Board-2	RF2	DW2
Board-3	RF3	DW3

2.4 Determination of the Chemical Properties of the Recovered Fibres

The pH value was measured after the extraction of fibres from cold water (4 specimens per fibre type); 2g of sample material was placed in a 100 mL Erlenmeyer flask, and 60 mL of distilled water was added. Afterwards, the Erlenmeyer flask was placed on a shaker present at room temperature, and the measurements were made after periods of 50 min, 6 h, and 24 h, respectively, with the help of pH electrode InoLab Level 2 (WTW GmbH, Weilheim, Germany). Due to minor differences, only the average pH value was reported for each type of fibre. The Nitrogen content was determined using the Kjeldahl method to assess the remaining UF resin content on the RF2 and RF3 after thermo-hydrolytic disintegration (4 specimens per fibre type), as previously described in [5]. Through the calculation of the remaining resin content on the RF, it was assumed that the UF resin contains roughly 30% nitrogen. The proportion of initial contents of the binder, which had remained

on the RF, was calculated by relating the NC of the RF to the NC of the respective MDF. Formaldehyde emissions from the fibre mass were determined based on the basic principle of the standard EN 717 3, which used approximately 2g of the fibre material in a bleached tea bag (4 specimens per fibre type), and the values were recorded after 3 h of emission [6]. All the results were compared to industrially produced thermo-mechanical pulp (TMP) fibres (virgin fibres-VF) as a reference.

2.5 Chemical Analysis of the DW

Analysis was made on the disintegration water samples for obtaining the pH value, including the formaldehyde content based on the acetylacetone method (EN 120 [7]) and nitrogen content based on the Kjeldahl method (4 specimens per DW sample) as described previously in [5]. The amount of reducing sugar equivalents in the DW was assessed based on the 3,5 dinitrosalicylic acids (DNSA) method (4 specimens per DW sample) [8].

2.6 Production of the Recycled MDF Panels Using RF2

Recovered fibres obtained after the disintegration of Board-2 were selected for manufacturing recycled MDF panels due to their compatibility with the thickness of the original board and resin content with the susceptible comparison of the previous research made by the authors. Recycled MDF panels (1 replica per panel) were manufactured using the RF2 obtained by the disintegration of the Board-2. Further disintegration of the RF2 was made in the hammer-mill VS1 N (Electra SAS, Poudenas, France) using a mesh size of 2.0 mm. Two different types of MDF panels containing 100% and 50% hammer-milled RF2 were produced and labelled as B100 and B50, respectively (Figure 3). The other 50% of the formulation of the B50 was made up of industrially produced thermo-mechanical pulp (TMP) fibres (virgin fibres-VF). These were blended with the RF2 after hammer-milling as performed previously. The MDF panels were produced with 10% UF resin (Kaurit 350, BASF, Ludwigshafen, Germany) based on dry fibre mass and 2% ammonium sulfate ((NH₄)₂SO₄) as a hardener based on the solid content of the resin. The resin and the hardener were pneumatically applied onto the fibres within a rotary drum using a spraying nozzle. The resinated fibre material was further hot-pressed (190 °C, 5 N mm⁻²) for 15 s mm⁻¹ to attain a target density of 700 kg m⁻³ using metal bar stops to reach a panel thickness of 16 mm.



Figure 3 Uncoated industrial Board-2 fibreboard and recycled MDF panels (B100 a B50) produced using recovered fibres obtained by the disintegration of Board-2 (RF2).

2.7 Determination of the Physico-Mechanical and Chemical Properties of the MDF Panels

All the MDF panels and the respective cut specimens were conditioned at 20 °C having 65% relative humidity until a constant mass was reached. The density of each individually cut MDF specimen was determined as per EN 323, and the overall density of the panel was calculated as the average of these samples [9]. In addition, the following properties were assessed: the moisture content (MC) according to EN 322 (36 specimens, 12 per panel), internal bond strength (IB) according to EN 319, the thickness swelling (TS) and water uptake (WU) after immersion in water for 24 h according to EN 317 while applying the sample dimensions of 50 × 50 × 16 mm³ (4 specimens per Board-2 and three specimens per recycled panel), the flexural strength (MOR) and modulus of elasticity (MOE) according to EN 310 while applying the sample dimension of 370 × 50 × 16 mm³ (two specimens per Board-2 and three specimens per recycled panel) [10-13]. The Formaldehyde content was determined as per the EN 120, perforator method) (1 specimen per panel), while the formaldehyde emission of the boards was analyzed according to EN 717-2 (gas analyzer method [14]) (two specimens of Board-2 and one specimen per recycled panel) and EN 717-3 [6], flask method) with two specimens for every panel for 3 h and 24 h [7]. The NC of the panels was determined using the Kjeldahl method (three specimens per Board-2 and two specimens for every recycled panel) as described previously in [5]. The binder content of the produced original and the recycled UF bonded MDF panels were calculated considering the UF resins contained roughly 30% nitrogen when the very low NC of the virgin fibres were ignored ($\leq 0.1\%$) (Table 1 and Table 3). All the aforementioned properties were also analyzed for the Board-2 before being disintegrated using the thermo-hydrolytic process.

Table 3 Physico-mechanical and chemical properties of industrially produced B2, 100% RF containing recycled B100, 50% recovered fibres (RF) and 50% virgin fibres containing recycled B50 MDF panels; N = specimen amount per panel, mean values (MV) \pm standard deviation (SD).

MDF properties	Board-2			B100			B50		
MDF type and used fibre type	Industrially produced, 100% virgin fibres			Recycled, 100% RF2			Recycled, 50% RF2 and 50% VF		
	N	MV	SD	N	MV	SD	N	MV	SD
Number or replica panel	8	n/a	n/a	1	n/a	n/a	1	n/a	n/a
Density (kg m ⁻³)	21	739.0	5.6	21	715.0	25.9	21	681.5	30.6
Thickness (mm)	n/a	19.0	n.c	n/a	16.0	n.c	n/a	16.0	n.c.
Moisture content (MC) (%)	12	7.8	≤ 0.1	12	7.2	≤ 0.1	12	6.8	≤ 0.1
Internal bond strength (IB) (N mm ⁻²)	4	0.3	≤ 0.1	3	0.1	≤ 0.1	3	0.2	≤ 0.1
Flexural strength (N mm ⁻²)	2	30.6	1.9	3	12.3	2.1	3	24.0	1.0
Modulus of elasticity (MOE) (N mm ⁻²)	2	3735.3	50.4	3	1610.0	306.2	3	2566.0	254.8
Water uptake (%)	4	19.0	1.1	3	35.9	1.0	3	40.7	1.4
Thickness swelling (%)	4	10.0	1.6	3	10.1	0.1	3	8.9	0.2
Formaldehyde content - Perforator method (mg 100 g ⁻¹ o.d. board)	1	2.0	n/a	1	11.0	n/a	1	13.7	n/a
Formaldehyde emission - Gas analyser method (mg m ⁻² h ⁻¹)	2	2.5	≤ 0.1	1	10.1	n/a	1	10.5	n/a
Formaldehyde emission - Flask method (mg kg ⁻¹ o.d. board) 3 h	2	2.5	≤ 0.1	2	7.3	0.8	2	7.5	≤ 0.1
Formaldehyde emission - Flask method (mg kg ⁻¹ o.d. board) 24 h	n/a	n.a.	n.c	2	104.7	5.7	2	116.1	6.7
Nitrogen content (%)	3	3.6	≤ 0.1	2	4.9	≤ 0.1	2	3.8	≤ 0.1
Calculated UF content (%)	n/a	11.5	n.c	n/a	15.7	n.c	n/a	12.2	n.c

n/a = "not applicable", n. c. = "not calculated", n. a. = "not analyzed"

3. Results

3.1 Chemical Properties of the Recovered Fibres

The moisture content of the recovered fibres (RF) was as low as 20%, indicating the effectiveness of the drainage process. As the moisture content was well below the fibre saturation point of coniferous wood such as spruce, it could be assumed that the degradation of the cell wall occurs or that the fibres were lost during processing. As bound water is held by intermolecular attraction within cell walls, this water cannot easily be removed unless the cell walls are broken [15]. The pH value of virgin fibres (VF) amounted to 4.0, while those of the recycling fibres (RF) ranged between 5.5 and 6.3 (Table 4). The higher pH of the RF may be attributed to the UF and the MUF resin itself, including the alkaline ammonia formed as a result of degradation from the resins during disintegration. Under acidic conditions in an aqueous environment, ammonia forms ammonium [16, 17], which results in the pH increment of the RF samples.

Table 4 Chemical properties of the virgin fibres (VF) and recovered fibres (RF), the mean values (MV) \pm standard deviation (SD).

Chemical properties	Virgin fibres (VF)				RF1		RF2		RF3	
	Non-milled		Hammer-milled		MV	SD	MV	SD	MV	SD
Moisture content after drainage (%)	n/a	n/a	n/a.	n/a	20.1	n.c.	22.0	n.c.	20.7	n.c.
Moisture content after conditioning (%)	7.1	≤ 0.1	8.9	≤ 0.1	17.9	2.1	11.0	0.3	11.4	0.8
pH (50 min, 6 and 24 h)	4.2	≤ 0.1	4.2	≤ 0.1	6.3	0.1	5.5	0.1	6.2	0.1
Nitrogen content (%)	0.1	≤ 0.1	0.1	≤ 0.1	2.8	≤ 0.1	1.7	≤ 0.1	3.3	≤ 0.1
Calculated UF content (%)	n.c	n.c	n.c	n.c	n.c.	n.c.	5.4	n.c.	10.6	n.c.
Amount of initial binder content remained on the fibres (%)	n/a	n.c	n/a	n.c.	46.6	n.c.	47.0	n.c	66.2	n.c.
Formaldehyde emission-Flask method (mg/kg o.d. fibre) (3 h)	5.6	0.2	9.1	0.07	57.2	0.9	29.6	5.8	36.6	7.5

While the nitrogen content (NC) of the VF used for manufacturing the B50 was negligible ($\leq 0.1\%$), the NC of the recovered fibres was as high as 2.8%, 1.7% and 3.3% for RF1, RF2 and RF3, respectively (see Table 4). Thus, the resulting calculated binder content was 5.4% and 10.6% for RF2 and RF3, respectively (Table 4). Calculations were made on the amounts of initial binder contents found on the RF2 and RF3. The results revealed that the fibreboards possessed a relatively high tendency to be hydrolyzed. Furthermore, 46.6%, 47.0% and 66.2% of the initial binder content could still be found on the RF1, RF2 and RF3, respectively, implying that roughly half of the initial binder content was hydrolyzed and dissolved in the DW samples while an insignificant amount evaporated during the processes of disintegration and drainage or even both. Due to the complexity in the formulation, the proportions of the UF and MUF for RF1 cannot be calculated using the nitrogen content alone (Table 4).

Formaldehyde emissions of the VF after 3 h were 5.6 and 9.1 mg per kg of the oven-dried (o.d.) fibres for the non-milled and hammer-milled samples, respectively (Table 4). As explained above, for NC, due to the remaining binder on the RF samples, formaldehyde emissions of the RF were 4.2 times higher than that of VF, on average. Thus, RF3, which contains higher amounts of NC (3.3%), releases higher formaldehyde (36.6 mg per kg o.d. fibre) compared to the RF2 (29.6 mg per kg o.d. fibre). The formaldehyde release was not found to be proportional to the NC content. Thus, RF1 exhibited a slightly lower NC (2.8%) compared to RF3 but released more amounts of formaldehyde (57.2 mg per kg o.d. fibre), although the binder contained MUF resin which is considered to be more stable during hydrolysis.

3.2 Chemical Properties of the Disintegration Water (DW)

The thermo-hydrolytic disintegration water samples obtained from the MDF panels exhibited higher pH values when compared to demineralized water (Table 5). This is attributed to the formation of ammonia (NH_3), which reacts with water to form ammonium hydroxide (NH_4OH) [18]. The amount of formaldehyde present in the DW after the disintegration processes was 1618.8 mg/L, 813.6 mg/L, and 1385.3 mg/L for DW1, DW2 and DW3, respectively. Significant levels of reducing sugar equivalents are detected in the DW samples (shown in Table 5), which may be products having a cleavage, mainly consisting of hemicelluloses released during the production of VF and/or the thermo-hydrolysis of the MDF panels. The nitrogen content of the DW1, DW2 and DW3 was found to be 0.8%, 1.0% and 1.2%, respectively (Table 5). On comparing the chemical properties of the DW samples to that of a previous study made by the authors [4], the screw press process was seen to have an alteration; the pH values were more alkaline, while the DW contained more dissolved UF (higher NC of the samples). Thus, higher formaldehyde content and fewer equivalents for reducing sugars were seen. A possible explanation for this alteration could be due to typically volatile ammonia, a by-product of the UF-resin breakdown, that would have been captured and not released, as the obtained recovered fibres together with the disintegration water were stored in barrels made of high-density polyethylene and were sent to Maschinenfabrik Reinartz GmbH & Co. KG (Neuss, Germany).

Table 5 Chemical properties of the disintegration water (DW) obtained after the thermo-hydrolytic disintegration processes compared to the demineralized water mean values (MV) \pm standard deviation (SD).

Chemical properties	DW1		DW2		DW3		Demineralized water
	MV	SD	MV	SD	MV	SD	
pH	5.7	≤ 0.1	5.4	≤ 0.1	6.4	≤ 0.1	5.2
Formaldehyde content (mg L ⁻¹)	1618.8	0.4	813.6	≤ 0.1	1385.3	≤ 0.1	n.a.
Reducing sugars and equivalents (mg L ⁻¹)	5.7	1.3	1.2	0.2	7.8	0.3	0.2
Nitrogen content (%)	0.8	≤ 0.1	1.0	≤ 0.1	1.2	≤ 0.1	≤ 0.1
Calculated UF content (%)	n.c.	n.c.	3.2	n.c.	3.8	n.c.	n.c.

3.3 Physico-Mechanical and Chemical Properties of the MDF Panels

It was seen that when 100% of the recovered fibres containing recycled MDF (B100) exhibited a slightly higher density, the B50 exhibited a lower density when compared to the target density (see Table 3). The recycled MDF containing 100% RF (B100) had an IB, which was nearly half as high as that of the original MDF (Board-2). The IB of the other recycled MDF (B50) was 28.5% lower than the original MDF, even though its final density was lower by 7.8%. These results are contrary to the previous studies made by the authors [4], in which increased IB results were seen when 100% of the RF was used for the production of the recycled MDF. In addition, in the present study, the amount of binder content remaining on the RF was roughly 20% higher than the study mentioned above, and the fibres were severely shortened. Thus, the reduced IB of the B100 panel could be due to the over proportional consumption of the adhesive by fines and loss of strength of the RF [19]. The flexural strength (MOR) of the recycled MDF, B100 and B50 was lower by 59.8% and 21.5% than the original MDF panel, respectively. Similarly, the modulus of elasticity (MOE) of the recycled MDF panels was also reduced by the same range as the MOR compared to the original MDF. As for the IB, it can be attributed to the shortened fibre length due to the thermo-hydrolytic disintegration conditions, by subsequent hammer-milling and through the severe drainage process in the screw press. The properties related to the strength of these recycled MDF were significantly lower compared to those of the previous studies, which used the de-watering of the RF within a spin dryer [4]. In the MDF, the length of the fibre particularly determines the flexural strength and stiffness of the boards [20, 21]. This indicates that a severe shortening of the RF occurs in the screw press. Another possible explanation for these reductions might be that the flexibility of the RF was reduced due to the thermo-hydrolytic disintegration process, making the RF more brittle. Brittleness, in turn, could lead to the shortening of fibre under the mechanical impact.

Compared to the original MDF panel (Board-2), the recycled MDF containing 50% of RF displayed an 11% decrease in the thickness swelling (TS), while the other recycled MDF (B100) showed no difference. The latter result is surprising since the IB of the B100 variant was reduced by about 50%, and the TS generally decreases with the increase in the bond quality. Water uptake (WU) after 24 h immersion, however, was increased with the average ca, two times for the recycled panels. When

compared to two recycled panels, the slightly lower WU of B100 can be attributed to the reduced wettability of the RF due to the presence of the remaining resin. In addition, hydrophilic sugars derived from the degradation of hemicelluloses might be washed out during recycling. These sugars might absorb water, particularly in the virgin boards. Thus, the NC and the calculated UF content of the B100 was higher. B100 is supposed to contain ca. Nitrogen content of 3% derived from the utilized 10% UF binder and 1.7% nitrogen is derived from the used RF. The obtained result of 4.9% NC is under this assumption, and the same applies accordingly to B50.

The formaldehyde content (2.0 mg per 100 g o.d. board) of the original MDF panel was far below the upper limit value of 8 mg per 100 g o.d. board, as per the standard EN 120 [7]. The recycled MDF, however, exhibited a very high amount of formaldehyde, which might have been derived from both the new UF resins that were used for producing the recycled MDF and the remaining binder on the RF.

Similarly, formaldehyde emissions of the recycled MDF panels were significantly higher than of the original MDF panels irrespective of the employed method (gas analyzer and flask). No difference was noted between the two recycled MDF panel types. In a previous study made by the authors [4], lower formaldehyde emissions were noted from the recycled boards compared to the virgin panels, which was attributed to scavenging of the formaldehyde due to reaction with urea, ammonia, and oligomeric decay products of the UF resin. This would result in lower formaldehyde emissions for B100 compared to B50.

However, it should also be noted that the limitations of the laboratory-scale production of the recycled MDF panels could cause some inconsistent physicomechanical and chemical results when compared to the industrially produced Board-2.

4. Conclusions

The present study aims to implement a drainage process based on the application of a screw press, which occurs in between the thermo-hydrolytic disintegration process and the further utilization of the obtained RF (e.g., for production of new panels). Compared to the previous study, which applied de-watering in a spin dryer, the screw press process produces dryer fibres but affects the chemical properties of RF as well as the DW differently. One of the most significant findings of this study is that the recycled MDF panels showed a decrease in mechanical properties compared to the original MDF panels. Although improvement in these properties was seen with the mixing of RF with VF, the strength of the panels containing only the VF were not received. Altogether, drainage in a screw press is very efficient; however, pressing too hard to achieve a low moisture content would result in severe fibre damage. Thus, either the pressing force must be reduced, or the RF should be used in combination with the VF to obtain better mechanical properties for the recycled MDF panels.

Author Contributions

F. Yağmur Bütün Buschalsky prepared the experimental design together with Carsten Mai. She performed the data collection, analysis and interpretation. Furthermore, she wrote the manuscript. Carsten Mai prepared the experimental design together with F. Yağmur Bütün Buschalsky. He assisted in data interpretation and manuscript writing.

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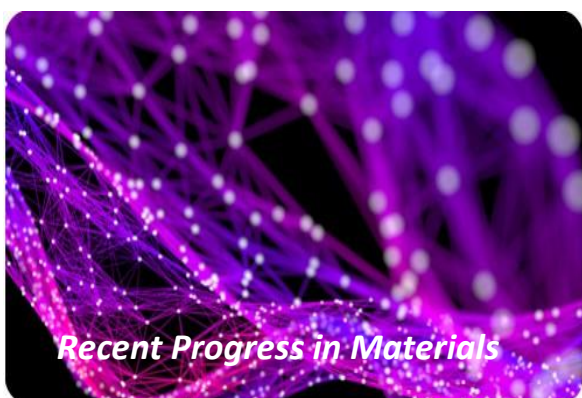
Competing Interests

The authors have declared that no competing interests exist.

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