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EFFECTS OF HYDRODYNAMIC TREATMENT OF WOOD PARTICLES ON MECHANICAL PROPERTIES OF WOOD-POLYMER COMPOSITES

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ABSTRACT

This study is aimed to investigate the mechanical properties of wood-polymer composites (WPC) made on the basis of polylactic acid (PLA) with the addition of mechanically activated wood particles (WPs) from larch wood (*Larix sibirica*) in a mass ratio of 70/30%. WPs were obtained by hydrodynamic treatment that lasted for 5-45 min. Size estimation and distribution of WPs were carried out using the sieve method, and an analytical sieving machine. It was found that the size of WPs decreased with increasing hydrodynamic treatment time. WPs with sizes ranging from 100 to 300 μm constituted the majority of the wood pulp. After testing, the SEM method was used to examine the WP surface and WPC samples. The mixing of WPs and PLA was carried out using the melt mixing method. WPC for the study of mechanical properties was obtained by flat pressing in a mold. Tensile tests were conducted in accordance with GOST 11262-2017, bending tests in accordance with GOST 4648-2014. It is shown that the duration of hydrodynamic treatment of WPs for 25 min provides the highest strength parameters of PLA-based WPC. This can be explained by the uniform dispersion of fibrillated WPs in the polymer matrix and the formation of their adhesive bond by mechanical engagement.

Keywords: wood-plastic composite; hydrodynamic processing; larch sawdust; mechanical property; tensile strength; bending strength; wood particle size.

INTRODUCTION

Increasing concern for sustainability compels humanity to reduce environmental problems, including those caused by the use of polymers from fossil raw materials (Korhonen *et al*., 2018, Friedrich, 2020, Accorsi *et al*., 2014). Synthetic polymers based on petroleum products are very slowly destroyed in the environment, which can take hundreds of years. As a result, numerous plastic waste has already accumulated in the earth, oceans and atmosphere (Panaitescu *et al*., 2020). Scientific research in recent decades has helped to find a solution to this problem – using environmentally friendly biopolymers, such as polylactic acid (PLA), in everyday life, and creating polymer composites that do not harm the environment and are recyclable (Partanen *et al*., 2016, Naghdi, 2021, Teymoorzadeh *et al*., 2015, Petchwattana *et al*., 2020, Narlıoğlu *et al*., 2021).

Among biodegradable polymers, PLA is most widely used in the production of packaging materials, consumer goods, biomedicine, etc. (Partanen *et al*., 2016). PLA is a

biodegradable polymer derived from renewable sources such as cane, corn and other plant materials. It is carbon-neutral, compostable, does not emit hazardous decomposition products, and has high mechanical properties. However, pure PLA is a fragile material and needs modification, for example, with the help of plasticizers and reinforcing particles (Farah *et al*., 2021).

Natural plant fibers from renewable raw materials have a number of advantages, such as improved biodegradability, low cost, low abrasion, high specific strength, low density, etc. (Friedrich, 2020). The addition of solid dispersed or fibrous natural plant particles into the polymer matrix is carried out in order to change the physical and chemical, mechanical, thermal, frictional and other properties of materials, but the main task is to improve the physical and mechanical properties (Lipatov, 1977, Sánchez-Safont *et al*., 2018, Özdemir *et al*., 2022, Akpan *et al*., 2018, Mahesh *et al*., 2020).

Wood is widely used as a filler in wood polymer composites (WPC). Such filler can be relatively inexpensive woodworking waste. The wood filler also improves the properties of the composite. The authors Wan *et al.* (2019, 2018) concluded that wood flour (WF) of 150 μm < particle size < 180 μm executed the optimal improvement to mechanical properties of PLA/WF/PMMA. Tensile strength, flexural modulus and Young's modulus of PLA/WF/PMMA (150-180 μm) were enhanced 10.02%, 37.21% and 32.80% in relation to neat PLA, respectively. Reinforcement of polycaprolactone (PCL) with 0.5 to 15 wt% of WF results in the tensile strengths of PCL/5 wt% WF and PCL/15 wt% WF being 16.0 MPa and 15.0 MPa, respectively. The tensile strength of pure PCL is 13.0 MPa (Cintra *et al*., 2022).

The shape of the filler particles is one of the most important factors affecting the mechanical properties of the WPC (Murayama *et al*., 2018). Recent studies reported that WPCs with fibrous surface wood particles obtained by mechanical milling have better mechanical properties (Isa *et al*., 2014, Isa *et al*., 2016, Stark and Rowlands, 2003, Salasinska and Ryszkowska, 2015, Tanpichai and Wootthikanokkhan, 2018).

Several studies noted that making WF by wet milling significantly improves the WPC properties. Wet milling of wood can destroy the cell walls with fibril delamination on the surface. This improves WPC mechanical properties such as tensile strength, impact resistance, and fatigue life (Delviawan *et al*., 2019, Isa *et al*., 2014, Isa *et al*., 2016, Haque *et al*., 2019).

The study (Qiang *et al*., 2018) of bleached softwood kraft pulp obtained by wet grinding in a ball mill found that the tensile strength of the PLA-based composite is the highest when the grinding time is the longest (reinforcing particle size is the smallest). Gravelsins *et al.* (1988) studied the wet milling of wood in a roller mill. It was shown that wet milling is more efficient than dry one. For example, milling of pine sawdust-water slurry at 75 kW·h/t SEC yields 350 μm particle size while milling air-dry and wet sawdust in a gas environment at the same SEC yields 570 and 990 μm particle size, respectively. For example, the SEC required to mill aspen chips down to 400 μm is 100 kW·h/t while the SEC for dry milling is three times that amount (320 kW·h/t). Therefore, wet milling is a promising process for making wood fillers used in biopolymer-based composites.

Our previous studies (Ermolin *et al*., 2019) indicate the production of wood particles dispersed to the state of fibrous material with fibrillated surfaces in the process of hydrodynamic processing. Thus, the purpose of this work is to study the influence of morphological characteristics of wood particles crushed by hydrodynamic method on the mechanical properties of wood-polymer composites.

MATERIAL AND METHODS

Materials

Sawdust from Siberian larch (*Larix sibirica*), 85 ± 12% moisture content, obtained from sawing logs was investigated. Polylactic acid (PLA) was used as a matrix (NatureWorks Ingeo 4043D).

Wood particles preparation

The sawdust was collected at the Krasles sawmill (Krasnoyarsk Territory, Russia). A lab-grade rotary-pulse hydrodynamic homogenizer was used (Figure 1). We mixed the sawdust with water at 8 to 10 ^{o}C (1). The sawdust content in the mixture was 10%. After mixing sawdust with water, the apparatus was switched on and repeated mass processing was carried out by passing through the hydrodynamic homogenizer (3) (rotor and stator). The rotation frequency of the rotor was 2950 rev/min. The treatment lasted for 0, 5, 15, 25, 35, 45 minutes (Delviawan *et al*., 2020).

After the hydrodynamic treatment, the wood particles (WPs) were frozen in a vacuum and lyophilized in a Lyoph Pride LP10 dryer (ilShin Bio Base Co., Ltd., Korea) to preserve the wood structure close to its original state with maximum swelling and hydration values. The WPs were then ground using a mixer (MMBP 1000; Robert Bosch GmbH; Germany).

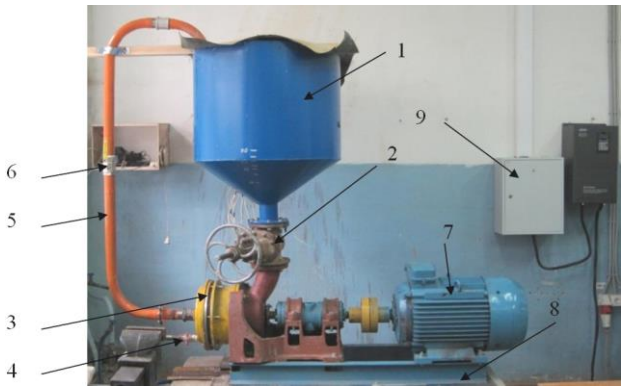


Fig. 1 The general arrangement of the test bench: 1: tank; 2: gate valve; 3: hydrodynamic homogenizer; 4: drain ball valve; 5: circulation pipe; 6: ball valve; 7: motor; 8: support frame; 9: control panel.

Characteristics of wood particles

The WP particle size distribution with a Retsch AS 200 Control analytical sieve shaker (Retsch AS 200 control, Retsch GmbH, Haan, Germany) was estimated. There were three measurements of 10 minutes each. The dry sample weight was 6.655±1.318 g. We used a laboratory balance with an accuracy of 0.001g. We then examined the WP surface with a scanning electron microscope (SEM) (Hitachi TM4000Plus, Japan). The WPs sample was placed on a table (without any additional sample preparation). During the image capturing process accelerating voltage of 15 kV was used.

Wood-Polymer Composite Manufacturing

WPs and PLA granules were pre-dried for 12 hours at 70 ± 2 °C before preparing the composite. We blended the dry PLA/WP at 70/30 wt% (Lima *et al*., 2020, Khan *et al*., 2020, Farrokhpayam *et al*., 2021). The dry PLA/WPs were blended at 170 to 185°C, 13 rpm using a twin-screw micro-compounder (LTE 12-36 12, LabtechEngineeringCo. Ltd., Thailand), and then granulated.

For obtaining the dumbbell-shaped and rectangular samples, the granules were molded and pressed in a LabPro 1000 hydraulic press (FontijnePresses, Netherlands) at 200 °C with a pressure 7.5 MPa for 5 minutes, then cooled to room temperature.

The dimensions of the dumbbell-shaped samples for tensile tests were ~ 80 × 5 × 4 mm as specified in the GOST 11262-2017 standard Type 5 (ISO 527-2:2012). The dimensions of the rectangular samples for bending tests were ~ 80 × 10 × 4 mm according to the GOST 4648-2014 (ISO 178:2019)] standard *(*Figure 2). The density of the composite samples was 1252 ±23 kg/m^3.

Fig.2 Mechanical test specimens.

All samples were conditioned at 20 °C and 65% RH for 1 week prior to each test. The moisture content of the WPC samples was ~ 0.5%. The composite samples were labeled W5, W15, W25, W35, and W45 to indicate the duration of WP hydrodynamic treatment of 5, 15, 25, 35, and 45 minutes, respectively.

Mechanical properties of wood-polymer composite

Tensile and bending tests to estimate the WPC mechanical properties were performed. The tensile test procedure was per GOST 11262-2017, and the bending test procedure was per GOST 4648-2014.

A universal testing machine (UTS-110MN-30-0U, Testsystems, Russia) for the tensile strength and 3-point bending strength tests was used.

Each mechanical test was performed on three samples.

The fracture surfaces of the WPC samples after tensile and bending tests using SEM (Hitachi TM4000Plus, Japan) were examined. No additional sample preparation was carried out. The WPCs were carefully fixed on aluminum stubs. Accelerating voltage was 10kV.

Statistical processing

Microsoft Excel 2010 for Windows 8 was used for statistical processing. Each test was repeated three times. The mean value, standard deviation and confidence intervals were calculated. The significance level was 0.05.

RESULTS AND DISCUSSION

Evaluation of wood particles

Figure 3 shows the WP particle size distribution. WP sizes decrease with an increase in the duration of hydrodynamic treatment. However, the particle size distributions for the 35 and 45-minute treatments are almost identical. The number of particles smaller than 20

μm increases with treatment time. For a 45-minute treatment, the number of particles smaller than 20 μm is double that of a 5-minute treatment. WPs between 100 and 300 μm make up the majority of the wood pulp treated for 10 to 45 minutes. It is supposed to be the most optimal WP dimensions for WPC with the best mechanical properties. (Murayama *et al.*, 2019, Delviawan *et al.*, 2020, Haque *et al.*, 2019).

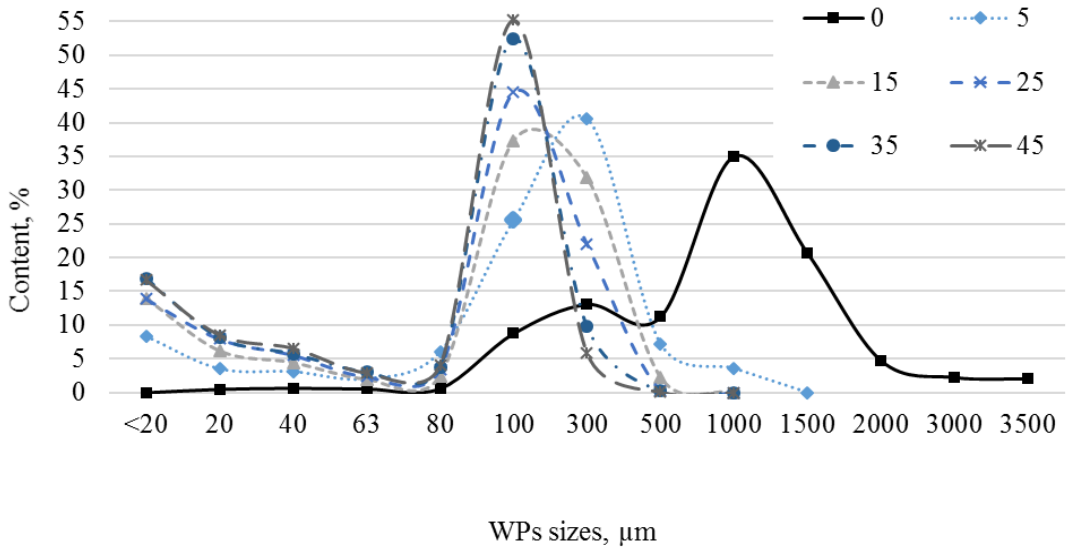


Fig. 3 WP size distribution vs. hydrodynamic treatment periods.

Hydrodynamic treatment significantly changes the morphological structure of WPs. The SEM images clearly demonstrate such changes. Figure 4 shows the microphotographs of the original WP samples and samples subjected to hydrodynamic treatment in a disperser for different periods. The WP before treatment (Figure 4a, x300 magnification) appears as small fragments of wood tissue with cellular structure. After 5 minutes of treatment, the surface lamination of the particles is clearly visible (Figure 4b). The number of small ribbon-like particles (partially destroyed tracheids with fibrillated surfaces) increases. Longer treatment increases the amount of fibrous elements and increases the fibrillated fine fraction (Figure 4c-e).

After hydrodynamic treatment in a disperser for 45 min (Figure 4f), most of the wood pulp is dispersed into fibers. The x300 magnification images show that individual fibers are fragments of cell walls with a high pore content which determines their high specific surface area.

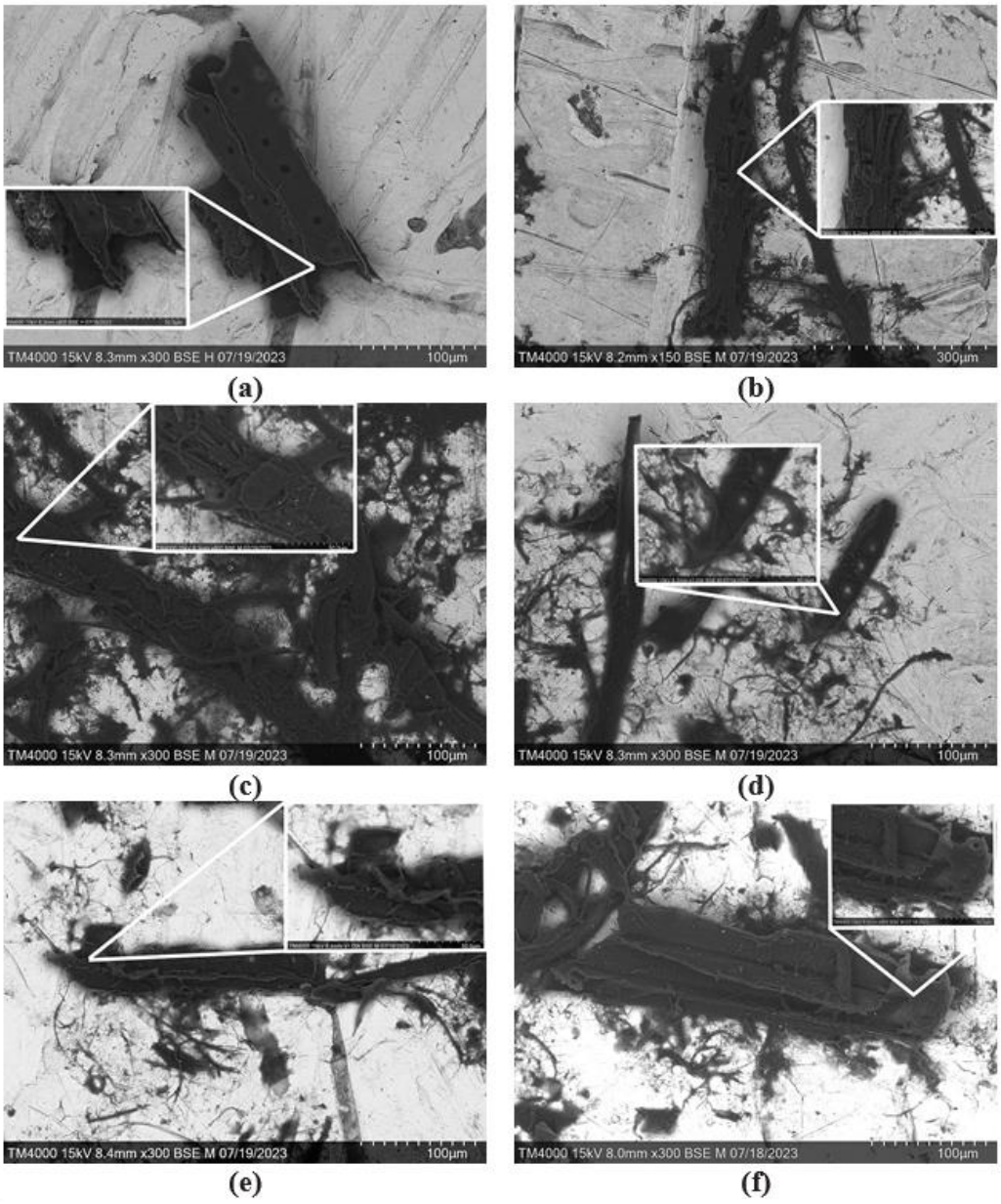


Fig. 4 The SEM images of wood particles hydrodynamically treated for (a) 0, (b) 5, (c) 15, (d) 25, (e) 35, and (f) 45 min.

Morphological and mechanical studies

SEM was used to examine the effects of hydrodynamic treatment duration on the condition of the composite sample cross-sectional surface after bending and tensile tests. Figure 5 shows the SEM images of the fracture surface filled with PLA wood particles hydrodynamically treated for 5, 15, 25, 35, and 45 min, respectively.

The SEM images show that the wood particles are well dispersed in the matrix, and the composite is homogeneous. Note the wood particles highlighted by the dashed lines. Many of these are fractured near the sample surface, i.e., more particles are fractured than elongated. This fracture pattern is consistent with the results reported in (Shah *et al.*, 2008). The authors indicate an improvement in the mechanical properties of a PLA-based wood-polymer composite when wood particles are added. They claim that the morphology of the composite fracture surface indicates fiber breakage, not elongation. This is a strong indication of good compatibility between the matrix and the wood filler.

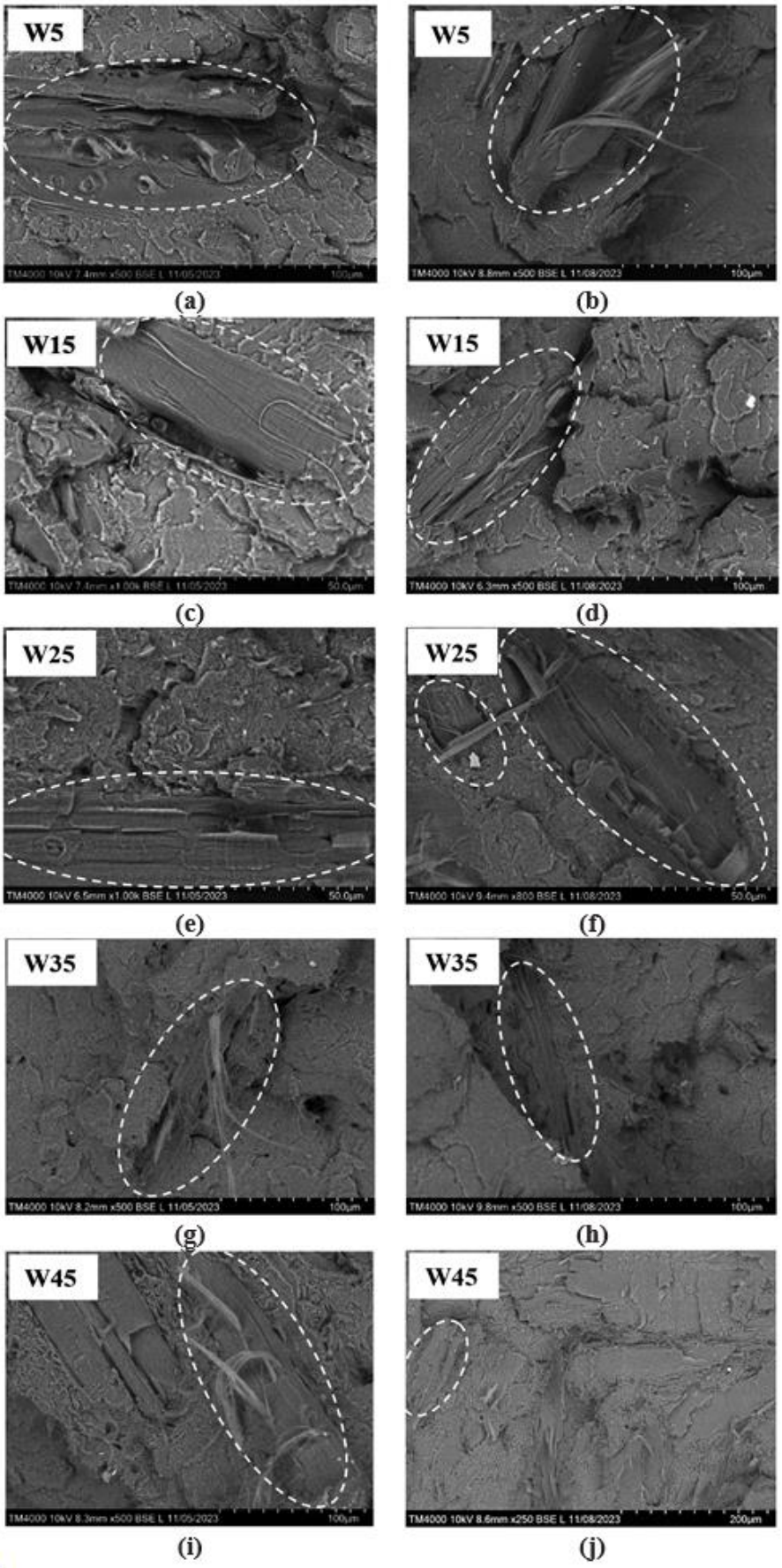
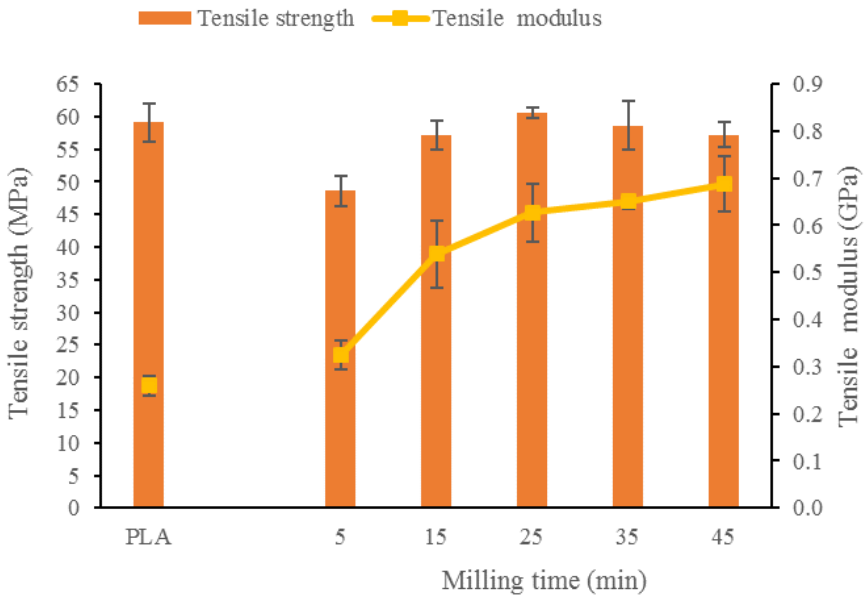


Fig. 5 The SEM images of wood particles samples after bending (a, c, e, g, i) and tensile (b, d, f, h, j) tests.

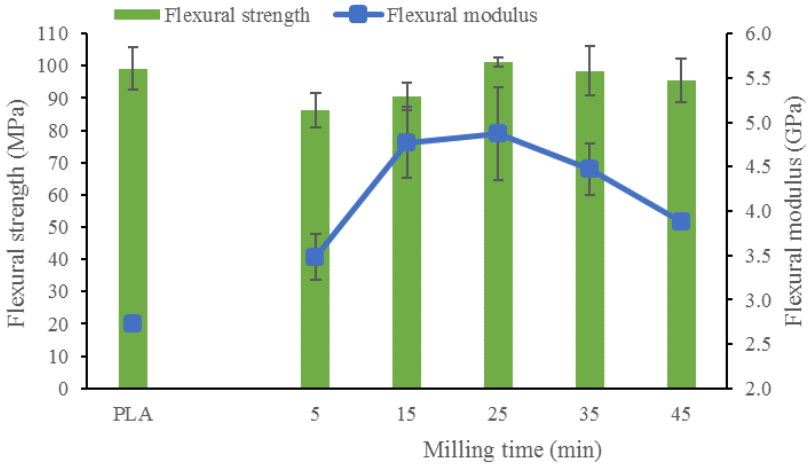
The images of the fracture surface (Figure 5) show more clearly the partial delamination of the cell walls along the reinforcing elements (fibrils) due to the anisotropy of the physical and mechanical properties of the wood. The longitudinal strength is much higher than the transverse strength. The results show that the hydrodynamically treated wood particles contribute significantly to the mechanical properties of the composite. They transfer stress to the polymer matrix and reduce stress concentration. This can be explained by the increase of the interphase contact area between the PLA matrix and wood particles as the filler fibrillation occurs during the hydrodynamic treatment. Also, the study (Panaitescu *et al.,* 2020) proposes that hydrophobic lignin present in WPs promotes adhesive interaction with the biopolymer matrix. The proposed process of WP mechanical activation can contribute to better mechanical properties of the resulting WPC.

Figure 6a demonstrates the effect of the hydrodynamic treatment duration on the WPC tensile strength. The results indicate that adding wood particles with a minimum processing time to a composite leads to a more than 40% reduction in the tensile strength compared to the initial PLA. Our previous studies have shown that the specific surface value of wood particles does not change significantly at this treatment time (Ermolin *et al.*, 2019). This determines the small interphase contact area between wood and polymer.

If the treatment time is less than 25 minutes, the WPC tensile strength increases with the treatment time. The 25 min treatment time yields the highest tensile strength of the composite, comparable with that of PLA, while the elastic modulus of the composite increases by more than 2.5 times.



(a)



(b)

Fig. 6 The effect of the hydrodynamic treatment duration on the WPC (a) tensile and (b) flexural strength.

The SEM image (Figure 5 e, f) above shows that the fracture surface contains fibrous cell wall elements of wood particles exposed to hydrodynamic treatment for 25 min, probably due to the adhesive bond between the WPs and the PLA matrix.

Mechanical locking explains adhesion: the matrix penetrates pores, holes, gaps, or other irregularities in the substrate and is mechanically locked to it. Mechanical locking occurs between the matrix and the rough surface of the natural fiber (Liu *et al.*, 2012). If the surface is rougher, the binding area between the matrix and the WPs increases, as the bond strength at the interface (Mohammed *et al.*, 2022, Gogoi and Manik, 20221).

If the treatment time exceeds 25 minutes, the WPC tensile strength decreases with treatment time. A 45 min. hydrodynamic treatment results in pronounced fibrillar elements with the cellular structure of the wood particle surface (Figure 4f).

The authors Isa *et al*. (2014) report that WPCs containing fibrillated wood particles have higher strength than WPCs with smooth surface particles.

The tensile strength of a WPC containing wood particles treated for 45 min is lower than that of a WPC containing particles treated for 25 min.

Particles treated for 45 min, compared to 25 min, are characterized by a high degree of destruction of the cell wall wood substance. This contributes to an increase in the adhesive interphase area. In this regard, it can be assumed that the adhesive bond between the WPs and the PLA matrix is stronger for a 45-minute treatment. However, there are clusters of small wood particles on the fracture surface of the WPCs containing particles treated for 45 min. These results indicate that the size of the WPs in the WPC has a greater effect on tensile strength than the fibrillated structure of the WP surface.

Figure 6b shows the effect of hydrodynamic treatment duration on the WPC bending strength.

It demonstrates correlation similar to the WPC tensile strength. A 25 min. treatment time gives the highest bending strength parameters of the WPC.

It is also proved that the bending modulus decreases with the addition of wood particles processed for more than 25 minutes. An explanation for this may be the fact that the increased processing time of WPs leads to the breakdown of many bonds between the supramolecular structures of the components, which causes fibrillation of cell walls. Such particles have a low stiffness and consequently decrease the bending modulus (Gacitua *et al.*, 2010).

CONCLUSION

This study was aimed to investigate the mechanical properties of wood-polymer composites (WPC) made on the basis of polylactic acid (PLA) with the addition of mechanically activated wood particles (WPs). WPs were obtained by hydrodynamic treatment. As a result, it was found that hydrodynamic treatment significantly changes the morphological and anatomic structure of WPs. The mechanical properties of the WPC obtained using PLA depended on the morphological characteristics of the WPs. The duration of the hydrodynamic treatment of WPs for 25 minutes provides the strength parameters of WPC without binders, commensurate with the strength of PLA. This method made it possible to grind WPs to an average size of 100-300 μm and ensure fibrillation of their surface. It increased the interphase contact area between the PLA matrix and wood particles and, ensured their adhesive interaction without binders. A 25-minute treatment demonstrates the formation of WPs between 100 and 300 μm (about 45%), which have a substantial effect on the mechanical properties of WPC.

Furthermore, the effect of the mass ratio of the components on the physical and mechanical properties of WPC containing hydrodynamically treated WPs will be investigated.

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