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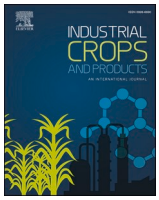

Manufacturing and characterization of sustainable and recyclable wood-polypropylene biocomposites: Multiprocessing-properties-structure relationships

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ABSTRACT

In this study, sustainable polymeric materials with a polypropylene matrix reinforced with wood waste were developed for structural applications. The new polymer biocomposites (WPCs) were evaluated for their mechanical and structural properties regarding their susceptibility to multiple processing. As thermo-mechanical degradation processes are associated with the repeated processing of plastics, which causes changes in the properties and structure of these materials. Therefore, to determine the extent to which the composites can be used under operating conditions, the composites were examined by DMA. As a result of the study observed some effects caused by the repeated effects of shear stress and temperature on the rheological and mechanical properties of polymer composites. The first of these is related to a decrease in viscosity of WPC composites subjected to the six times processing and changes in flow conditions during extrusion and injection moulding due to the degradation of the polypropylene matrix. As the viscosity of the composites decreased, a reduction in tensile strength and other mechanical properties of the polypropylene matrix was noted. On the other hand, the second effect observed leads to the conclusion that, as the composites' processing cycles increase, the WPC composite's mechanical properties increase due to an increase in the degree of homogenization of the individual components of the WPC composition. This study aims to describe the relationship between these two primary processes and to determine the relationship between the properties and the structure of the new WPCs.

1. Introduction

The sustainability of natural materials such as natural fibres and wood as a natural structural material with interesting properties such as i.e. low greenhouse gas emission and low production energy has led to a significant increase in demand for these sustainable products in several industries (Ates et al., 2020; Rana et al., 2023, 2022). There are several possibilities for environmentally friendly products and long-term carbon storage when using these natural precursors from different resources in sustainable composites (Singha and Thakur, 2008c; Singha and Thakur, 2008a; Uppal et al., 2022). Wood-based sustainable polymer composites (WPCs) are one of the primary construction materials in the building industry, used to make decking and facade panels, and also find applications in the manufacture of many other products, such as automotive products and furniture (Elamin et al., 2020; Ramesh et al., 2022; Turku et al., 2017). Sustainable composites from wood, natural fibres and their different precursors are being exploited in interior/ exterior applications (Singha and Thakur, 2008b; Singha and Thakur, 2009; Zielińska et al., 2021). They typically are the prime structural features in most buildings and other sustainable materials. Their applications include door and window sheathing, floor, I-beams, joists, components, and moulded wall panels as skin and structural elements.

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Generally, it can be said that hardly any buildings around the world do not contain wood as one of the components. In addition to this, the manufacture of WPC composites also provides opportunities to produce polymer matrix from granulates of thermoplastics, i.e. polyethene, polypropylene, poly(vinyl chloride) - PVC, poly(lactic acid) - PLA, polyoxymethylene, as an alternative to virgin plastics (Andrzejewski et al., 2019; Czarnecka-Komorowska et al., 2013, 2018, 2021b; Nukala et al., 2022; Satya and Sreekanth, 2022;). Polymer-wood composites are made of wood fibers or wood chips/particles that are embedded in a suitable thermoplastic polymer matrix. One of the major advantages of these wood-based materials is that they use waste products from the pulp and timber industries all around the globe. Hence, they are significantly less expensive than virgin timber-based products. In fact, over the past few decades, the use of wood-based raw materials has attracted rapidly growing interest from the scientific and industrial communities. The subject of many research includes issues of grinding, drying, modification, and pelleting as well as the production and recycling of wood polymer composites (Beg and Pickering, 2007; Borysiak et al., 2018; Dominkovics et al., 2007; Elamin et al., 2020; Fan et al., 2021; Krause et al., 2018; Macko and Mrozinski, 2019; Poletto, 2017; Rydzkowski and Michalska-Pożoga, 2016; Rydzkowski and Radomski, 2013; Shi and Gardner, 2006). Among many polymer matrices used to prepare wood polymer composites, polypropylene is one of the most frequently used. Polypropylene-based polymer composites are the current state-of-the-art materials, and their use is increasing exponentially in several applications, from automotive to aerospace to name a few. These composites offer exceptional mechanical characteristics such as low weight, high strength and stiffness, and low susceptibility to fatigue and corrosion. The appropriate combination of polypropylene toughness and stiffness of reinforcing fillers in wood fibres is the main reason why wood polymer composites are still becoming more popular (Bhaskar et al., 2021; Faruk et al., 2012; Liber-Kneć et al., 2006; Migneault et al., 2011; Ondiek et al., 2023; Wang et al., 2011; Zajchowski and Ryszkowska, 2009). Using high-stiffness, high-strength materials in the form of fibres moderates the tendency for premature brittle failure, allows components to be moulded at low temperatures and enables the anisotropic design to meet major load-carrying requirements. Studies show that under uniaxial tensile conditions, fibres are generally effective. Under uniaxial tension conditions, fibres have been found to be predominantly efficient; however, they suffer a variety of failures under compression that are naturally associated with fibre micro-buckling or kinking linked to the polymer matrix or interfacial issues. Generally, these kinds of failure of the polymer composites regulate the real-life usage of polymer composites and set design limits well below the anticipated intrinsic performance of the constituent fibres. Natural materials, such as wood, are fully hierarchical, with precise structural features resolved at every possible magnification. In terms of production perspectives, the use of wood fillers has a number of advantages, such as the ability to process using classic techniques such as extrusion and injection molding. In addition, adding wood fillers ensures higher dimensional stability of products, lower processing temperature, higher Vicat's temperature and finally lower production cost. (Bhaskar et al., 2021; Borysiak, 2015; Krause et al., 2018, 2017; Syduzzaman et al., 2020; Tomaszewska and Zajchowski, 2013). Unfortunately, the main and still rapidly increasing problem of using composite materials is their effective recycling (Hamdan et al., 2002; Krause et al., 2018; Selke and Wichman, 2004). As a highly filled material, the WPC composites are often treated as unrecyclable waste (Rybarczyk et al., 2020). The reuse of wood composites, or rather their recovery, is usually limited to the combustion process, mainly due to the high wood filler content (Adhikary et al., 2008; Bledzki et al., 2008; Zhou et al., 2022). Due to the circular economy (Guo et al., 2023), the approach to the form of WPC waste management through the use of recycling is changing. Thus, the overarching goal of this work is to determine the effect of multiple processing involving six cycles of extrusion with pelletization and injection moulding) on the rheological, thermo-mechanical and structural properties of WPCs. The second important aspect of the pairing is a comparative assessment of the differences between the unmodified WPC composition and commercially available blends.

2. Materials and methods

2.1. Sample preparation

Two types of polypropylene-based wood composites were prepared by the extrusion. The first one was prepared from wood fibres (fraction size of 80–150 μm) and isotactic polypropylene matrix (WPC1), the filler material was the Rettenmaier wood flour obtained from conifer trees, Lignocel C120 (prod. Rettenmaier and Söhne GmbH Co. KG, Rosenberg, Germany), the matrix was BasellOrlen polypropylene (PP) Moplen HP500J ($\rho = 0.91$ g/cm^3, MFI=3.20 g/10 min (N = 2.16 kg; T = 230 °C). The second composite (WPC2) was prepared from the Rettenmaier Compound H composition and the same polypropylene Moplen HP500J. In both polymer compositions, the optimum content of wood flour was 20 wt%. Numerous experimental studies had previously confirmed this value. A virgin polypropylene with the trade name Moplen HP500J (PP) was used as the reference material. A scheme for the preparation of the WPC composites, together with an evaluation of their characteristics and structure, is shown in Fig. 1.

WPC composites were prepared on the single screw extruder (L/D=34, the mixing zone on the end of the screw), firstly the dry-blend was prepared on the rotational mixer and then processed through the extruder, and the extrusion temperature was set to 190 °C. The extrudate was quenched in cold water and pelletized. After that, the composition was dried in a laboratory cabined dryer Memmert ULE 500 (Schwabach, Germany) at 80 °C for 24 h to decrease the moisture content. Every mentioned step was repeated 6 times, and after each processing step, the sample was taken for further research. The specimens with dimensions of 80 mm × 10 mm × 4 mm (Fig. 2) for tensile tests were injection-moulded using an Engel ES 80/20 HLS machine (Schwertberg, Austria) with a temperature of 180–190 °C, mould temperature of 35 °C and 35 s of cooling time. Sample series were prepared after each processing cycle, and all 6 series of samples were investigated employing mechanical, rheological and microscopic studies.

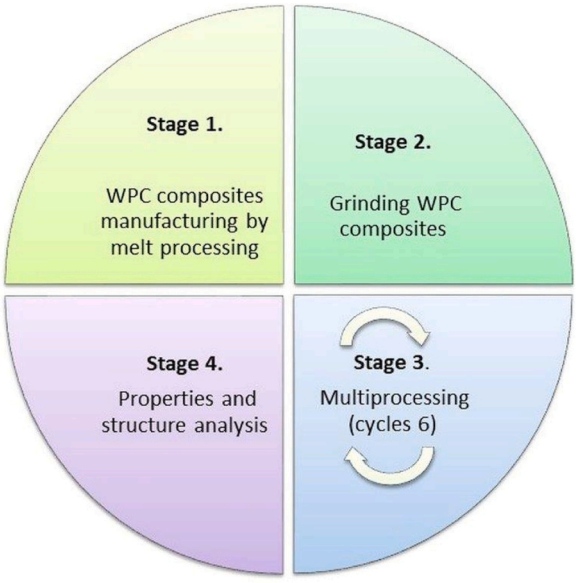

Fig. 1. Schematic of WPC biocomposites manufacturing and evaluation of their properties and structure.

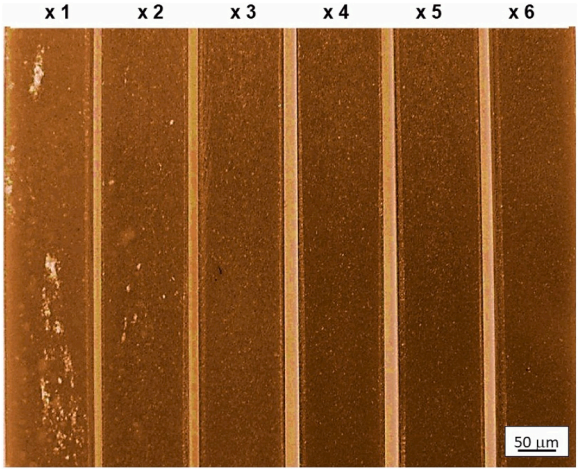

Fig. 2. General view of injection moulded samples made from WPC2 composite.

2.2. Thermal characterization

The thermal analysis measurements based on differential scanning calorimetry (DSC) were carried out using the Netzsch DSC F1 Phoenix 204 apparatus. The temperature program was comprehended with a heating/cooling rate of 10 K/min. The range of the measurement was set from room temperature to 230 °C. This procedure was conducted twice to evaluate DSC curves from the second melting. The aluminium crucibles with the pierced lid were used as the sample holders. All measurement was performed under the inert atmosphere of nitrogen.

2.3. Rheological characterization

The rheological properties of the melted WPC composite were investigated using two methods. The first technique was an Anton Paar MCR 301 rotational rheometer equipped with the cone-plate system in which the tests were performed at a constant temperature of 200 °C. The frequency sweep in this experiment varies from 0.05 rad/s to 100 rad/s, and the level of strain was 5%. The storage and loss modulus and complex viscosity measurement was realized as a function of frequency (ω). And the second one was melt flow index (MFI) measurements were realized for each of the investigated materials using a Dynisco melt flow indexer (Dynisco, USA) according to ISO 1133 standard (190 °C, 2.16 kg) (ISO 1133–1:2011 Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1). Because of the inadequate thermal resistance of the wood flour, the test temperature was set to 200 °C.

2.4. Dynamic mechanical analysis (DMA) characterization

Dynamic Mechanical analysis of the prepared wood polymer composites was carried out employing DMA, Anton Paar MCR301 rheometer (Graz, Austria), equipped with a torsion clamp system, the frequency of 1 Hz and strain amplitude of 0.01% were applied. The tests were performed on the rotational rheometer with the torsion system equipment. The sample size for this test was 4 × 10 × 50 mm (thickness/width/length). The dog-bond samples were prepared from previously injection moulded rectangular bars. The DMA test was performed at a frequency of 1 Hz and an oscillating dynamic strain value of 0.01%. The temperature ramp starts at − 50 °C and ends at 180 °C. The heating rate was kept at 2 K/min. The mechanical properties such as storage modulus (G'), loss (G'') modulus, and damping factor ($\tan\delta = G''/G'$) were recorded as a function of temperature.

2.5. Mechanical characterization

Tensile tests according to ISO 527–2:2012 Plastics — Determination of tensile properties — Part 2. Test conditions for moulding and extrusion plastics standards were carried out using the INSTRON 4481 (Instron, UK) universal testing machine with a 50 mm/min crosshead speed. Dumbbell samples, each measuring 80 × 10 × 4 mm was conditioned for over 36 h and after that time the test was performed. Tensile tests were used for samples after every processing cycle for WPC 1, WPC 2 and polypropylene (PP) specimens. Ten measurements are carried out for tensile strength (TS) and elongation at yield point (ε_y) as a function of processing cycles.

2.6. Microscopic analysis

The macro and microscopic observations after each process were also studied. Thin composite films were produced by compression moulding of the granulated product between glass plates. The microscopic observation was aimed at comparing the wood fibre's size and distribution. The observation was realized using the Carl Zeiss optical microscope with a polarizer. The morphology of the WPCs was observed using an Opta-Tech camera (Opta-Tech, Warsaw, Poland) at 32 × and 15 × magnifications.

3. Results and discussion

3.1. Differential scanning calorimetry (DSC) analysis

The differential scanning calorimetry (DSC) technique was applied to evaluate the impact of reprocessing on the resulting WPCs thermal properties (Krause et al., 2017; Zhou et al., 2022) and the relationship between cooling rate and crystallization rate in wood fibre-reinforced recycled plastic composites (Cui et al., 2010). The DSC results were collected in Fig. 3, on which the DSC curves obtained after 6 heating and cooling processing cycles were presented. DSC thermograms obtained during the measurements don't show any significant changes after all extrusion cycles (Hristov and Vasileva, 2003). The DSC curves for the particular samples show only the characteristic peaks of melting and crystallization for the polypropylene at 165 °C and 115 °C, respectively. It means the recycling process did not cause significant changes in the crystallization temperature (T_c) and entropy values (ΔH_c) of the virgin polypropylene and its composites. The melting temperature (T_m) and enthalpy values (ΔH_m) change observed are insignificant. It is typical for polyolefin materials, which are very stable and easy to reprocess. In summary, the DSC analysis didn't show the apparent changes in the crystalline structure of the polypropylene matrix. This confirms the dominant influence of processing parameters i.e., temperature on the mechanical properties (tensile stress and elongation) of the manufactured wood polymer composites.

3.2. Rheological properties analysis

In terms of processing and product properties, very important features are rheological properties. The viscosity of plasticized polymeric materials, especially composites, results from the components' properties and their fragmentation, geometry and mixing. Many relevant polymer properties can be characterized using rheological tests. The analysis of rheological properties indicates the structure of polymer chains, composite morphology and also its behaviour under processing conditions in the plasticizing system, in the extrusion die or in the injection mould (Alhussein et al., 2019; Ares et al., 2010; Hadi et al., 2018; Latef et al., 2020; Lewandowski et al., 2022; Mazzanti et al., 2014; Ou et al., 2014; Rydzkowski and Radomski, 2013; Wang et al., 2011).

In the linear viscoelasticity domain, the storage and loss modulus values are frequency-dependent (Ares et al., 2010). Fig. 4(a-b) presents the corresponding values for both composites WPC1 and WPC2. The

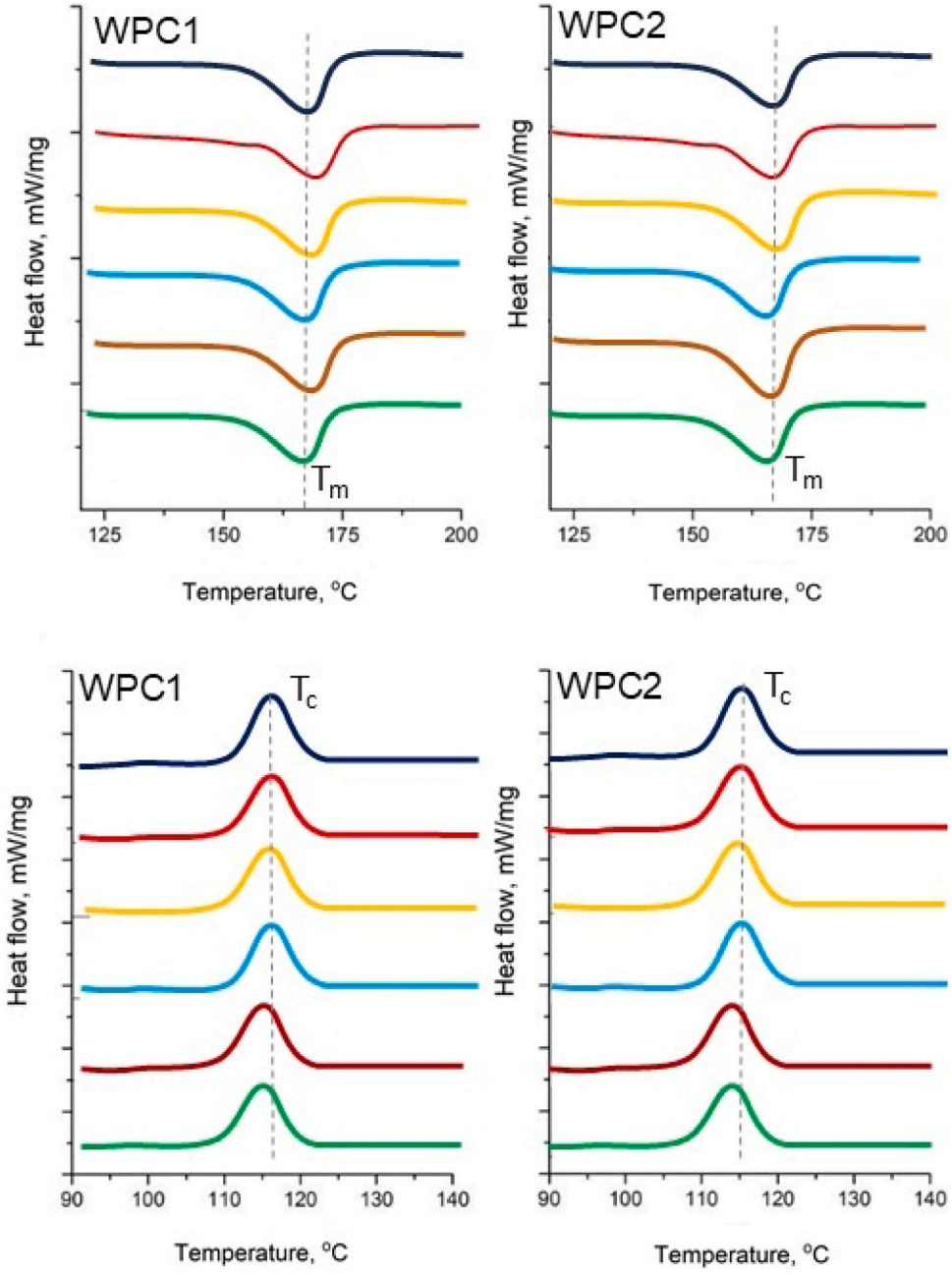

Fig. 3. DSC endothermic (up) and exothermic (down) thermograms peaks of WPC 1 and WPC 2 composites.

curves don't show final relaxation at low frequencies, which is usually observed for the virgin PP melts, also shown in Fig. 4c. The possible lowering of the modulus probably may be reached at the lower frequency or after a long time. The relaxation time is related to the interactions between particular particles of the wood and among the wood fibres and the polymeric matrix. The results show that the dynamic modulus (E′) of both WPC composites was found to decrease as a function of processing cycle numbers. The results suggest that reprocessing such composite materials causes a decrease in stiffness and probably an increase in the toughness of the WPC composite. It was also found that the difference between the curves is bigger in the case of the WPC2 composite, although for the WPC1, the differences are lower but still clear. This characteristic spread for the WPC2 composite may be caused by overheating the wood filler caused by the high temperature of the extrusion process. After several cycles of extrusion, the bonding effect is over, which can be seen during the observations of the storage modulus, when after every processing, the values of the modulus are visibly lower. The changes in storage modulus for the WPC1 composite are not as significant as for WPC2. However, the changes in the viscosity parameters caused mainly by the polypropylene degradation are still visible and should facilitate the material flow inside the processing machine channels.

MFR is an indirect assessment of thermoplastic melt behavior (viscosity) under strain. Due to the possible degradation of the wood particles at the temperature above 200 °C, the Melt Flow Rate (MFR) test was performed at 200 °C using a standard load of 2.16 kg. As can be seen in Fig. 5, the MFR value for the virgin PP and the WPC is significantly dependent on the cycle number. The results showed that the melt flow rate for the WPC1 and WPC2 composites is lower (about 50%) compared to that of the virgin PP, due to the presence of wood as filler in the PP matrix. Furthermore, it was shown that the MFR for WPC1 and WPC2 composites increases with increasing processing cycles, e.g., from 0.8 g/

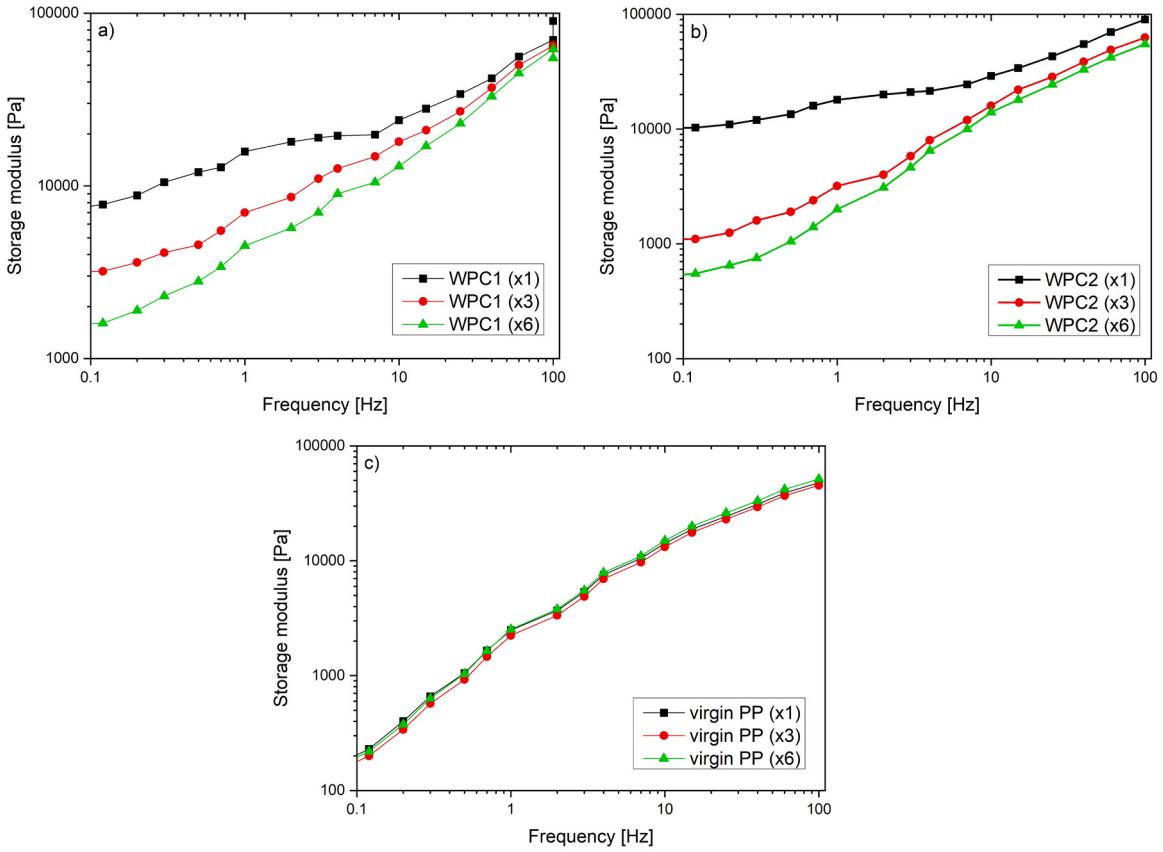

Fig. 4. Storage modulus curves of WPC 1 (a), WPC 2 (b) composites and virgin PP (c) as a function of frequency.

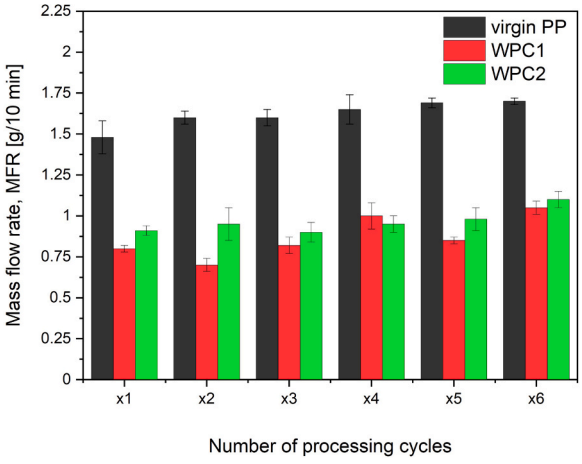

Fig. 5. Mass flow rate (MFR) of examined virgin PP and its composites as a function of processing cycles.

10 min for WPC2 to 1 g/10 min after 6 times processing). This can probably be attributed to the 'better packing' of the filler in the PP matrix due to better dispersion of shorter wood fibres with each processing cycle. Reprocessing by shredding and injection moulding six times results in improved adhesion between the polypropylene continuous phase and the wood filler (Czarnecka-Komorowska et al., 2021a).

3.3. Mechanical properties analysis

The solid-state dynamic mechanical analysis measurements were carried out to investigate the mechanical characteristics of the wood polymer composites after several cycles of extrusion processing. The rectangular samples were shaped using an injection moulding machine and tested using the DMA methodology. The result of the experimental measurement, i.e. storage modulus, is presented as a function of changing temperature. General changes between the material after the 1st and 6th processing cycles are presented in Fig. 6. First two diagrams present the storage modulus (a-b), and the last two ones are the tan δ thermograms (c-d).

As per our expectation, the pure polypropylene sample has been found to exhibit the lowest storage modus values due to the full spectra of the temperature. The modulus values of WPC 1 and WPC2 composite are higher, which can be attributed to the reinforcement influence/effect of the used wood flour. As we have expected for the pure PP, the storage modulus curves course was practically the same. Surprisingly, the tests realized on the composite materials don't show any significant changes between recycled materials. However, there have been some interesting changes between WPC1 and WPC2 compositions. After first processing, the storage modulus values under glass transition temperature (T_g) have been practically the same for both compositions. On the other hand, above the glass transition temperature and the storage modulus of WPC2 is higher (can be seen visibly), which could be confirmed by a higher E modulus in a static tension test. The course of E′ after the 6th processing cycle is different. Above the T_g temperature, the storage modulus of WPC1 is higher. The tangent δ thermograms confirm this tendency. This means that the mechanical properties of WPC1 material should be better after several processing cycles, while the WPC2 properties decrease after multiprocessing.

The stress-strain tests were used to plot the tensile strength (Fig. 7a) and elongation at yield point (Fig. 7b) diagrams as a function of multiprocessing (da Costa et al., 2005; Dickson et al., 2014; Jayaraman and Bhattacharyya, 2004; Paukszta and Zielińska-Maćkowiak, 2012). The results of the static tensile test also confirm the DMA investigation,

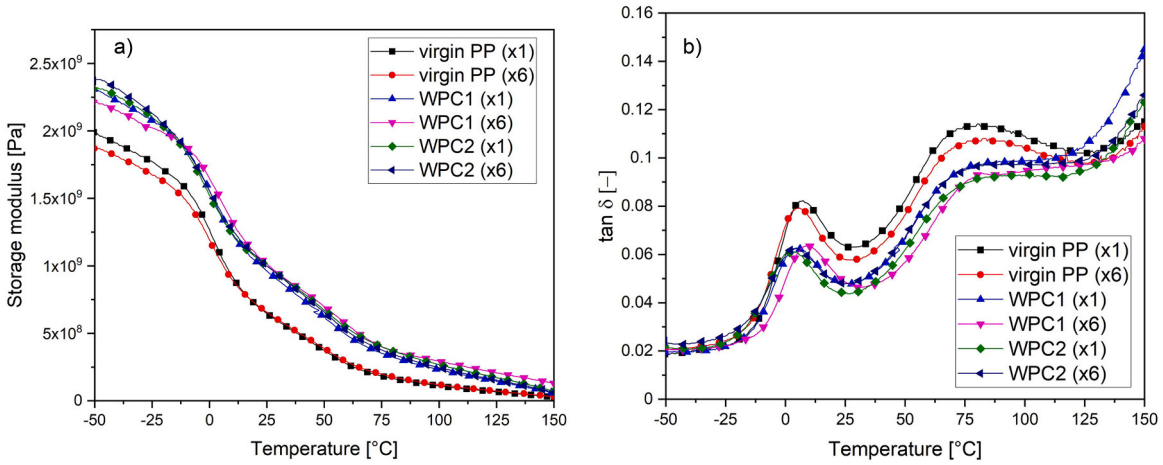

Fig. 6. (a) Storage modulus of the samples obtained after the first and 6th processing cycle. (b). Tangent δ of the samples obtained after the first and 6th processing cycle.

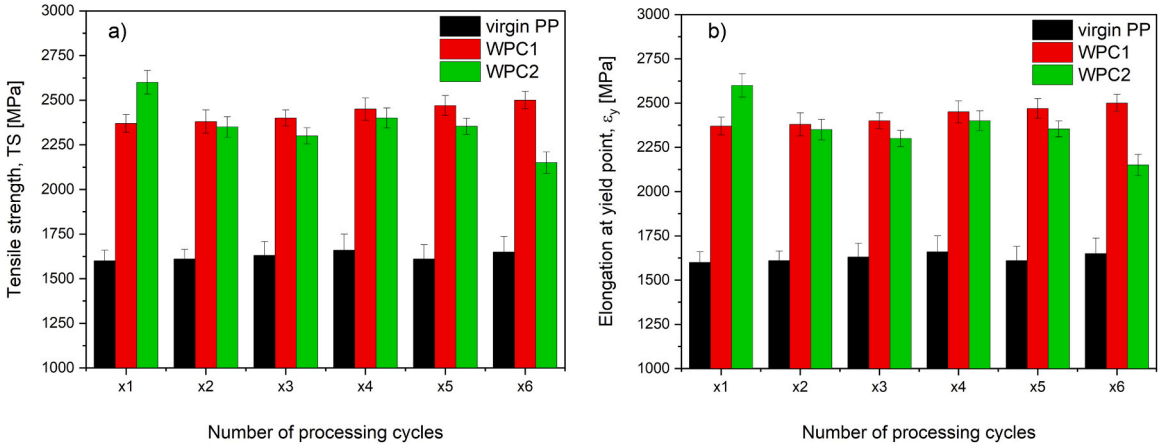

Fig. 7. a. The value of tensile strength as a function of processing cycles. b. The value of elongation at yield point as a function of processing cycles.

where the mechanical properties of WPC1 have been found to increase after multiprocessing. In contrast, the properties of the WPC 2 composite were worse because of the same processing procedure.

The mechanical properties represented by tensile strength (TS) and Young's modulus (E) were different for the WPC1 and WPC2 composite (Butylina et al., 2011). The tensile strength of WPC 1, despite a lower value than virgin PP, was growing constantly after each processing cycle. In contradiction to the first material, the WPC2 strength decreases after multiprocessing. The same tendency was observed in the tensile modulus. After the 6th processing cycle, the value of the Young modulus for WPC 1 was 10% higher than for WPC 2.

On the contrary, the elongation at yield point changes (Fig. 7) is different for virgin PP; the elongation of samples after every extrusion was lower, and the tendency for the WPC samples was inverse. Thus, the mechanical results confirm our hypothesis that the stiffness of the WPC will tend to increase due to better homogenization of the wood fibres in the polymer matrix. From another point of view, a possible partial degradation of the polymer matrix related to reprocessing may lead to higher flow ability and, consequently, better packing during injection moulding. Similarly, partial degradation of the PP causes a lower elongation, as observed for the virgin polypropylene.

3.4. Microscopic observations analysis

The microscopic observation of the WPC1 samples after the first and last processing is presented in Fig. 8. The wood particle dispersion is visibly better for the WPC1 composite; it can be directly related to the influence of multiprocessing. The same cycle of extrusion didn't show the difference for WPC2 material. The product was already well-dispersed as a market-available composite. That's why there were no aggregations of wood particles; only the distribution was worse, which was visible by the unaided eye in the following picture.

Fig. 9 shows the pictures of the macro lens (magnification, x 1.5), where the wood flour-rich areas are eliminated after every next extrusion cycle. These undistributed wood flour clusters are visible on the surface of injection moulded samples, which can be seen in Fig. 2. This defect decays after the third processing cycle.

4. Conclusions

In this paper, eco-friendly polymer composites were developed with polypropylene as the polymer matrix and wood flour as the filler of natural origin at 20 wt%. Two types of polypropylene-based wood composites were prepared by the extrusion. The first was prepared from wood fibres and isotactic polypropylene matrix (WPC1), and the second composite (WPC2) was prepared from the Rettenmaier Compound H composition and the same polypropylene matrix. The influence of reprocessing on rheological, thermo-mechanical properties of WPCs was investigated. The results show that the dynamic modulus (E′) of both WPC composites decreased as a function of processing cycle numbers. The multiple recycling process did not cause significant changes in the crystalline structure, crystallization temperature (Tc), or entropy values

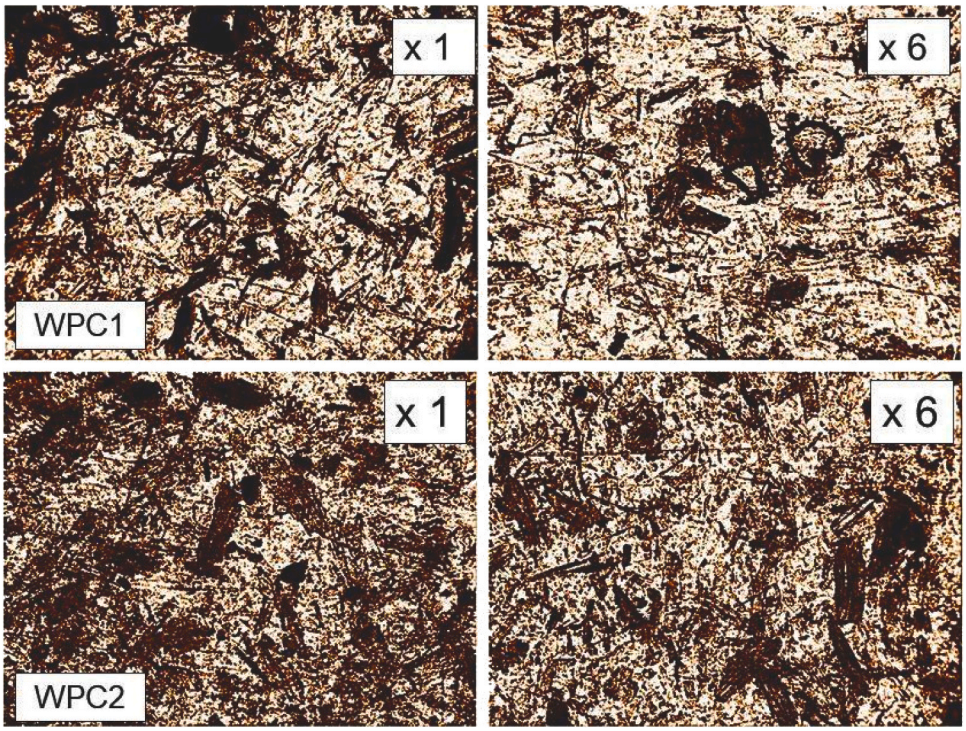

Fig. 8. Microscopic view of WPC 1 (upper) WPC 2 (lower) composites (magnification at 32 lens).

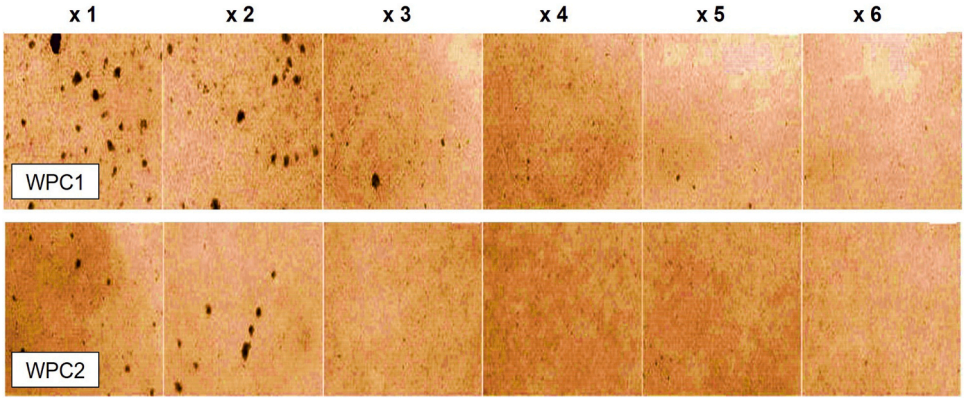

Fig. 9. Macroscopic view of WPC 1 (upper) and WPC 2 (lower) (magnification at 1.5 lens),.

(ΔH_c) of the virgin PP and its composites. The melting temperature (T_m) and enthalpy values (ΔH_m) change observed are insignificant. It is typical for polyolefin materials, which are stable and easy to reprocess. The MFR value for the virgin PP and the WPC significantly depends on the cycle number. The results showed that the MFR for the WPC1 and WPC2 composites, due to the presence of wood as filler in the PP matrix, is lower (about 50%) compared to that of the virgin PP. The storage modulus values of composites tested are higher than for pure polypropylene, which can be attributed to the reinforcement influence/effect of the used wood flour. After first processing, the values of the storage modulus under glass transition temperature (T_g) have been practically the same for both tested composities. On the other hand, above the glass transition temperature the storage modulus of WPC2 is higher which could be confirmed by a higher E modulus in a static tension test.

Above the T_g temperature, the storage modulus of WPC1 is higher. The tangent δ thermograms confirm this tendency. This means that the mechanical properties of WPC1 material should be better after several processing cycles, while the WPC2 properties decrease after multiprocessing.

WPC thermoplastic composites contain agglomerates of wood fraction, especially fine ones such as wood flour. As a result of multiprocessing and the associated mechanical loads, the agglomerates shrink and disperse.

Similar to any other materials, the mechanical and deformation properties of wood-based polymer composites are also determined by their microstructure. When it comes to reprocessing wood polymer composites, several parameters affect the overall properties of the resulting composites. Indeed, the assessment of the multiprocessing impact on WPC composite properties is a very complex subject that depends on many factors. Most of the factors are focused on the processing parameters which have a direct influence on the final mechanical properties of the resulting composite material. The observed phenomenon of improvement in material indicators like tensile modulus and strength is related to the degradation of the polymer matrix and homogenization of the WPC composition. In a particular case, the synergy effect caused the increase of mechanical properties after several cycles of processing.

CRediT authorship contribution statement

Andrzejewski J: Conceptualization, Investigation, Writing – review & editing. Czarnecka-Komorowska D.: Investigation, Supervision, Writing – review & editing. Barczewski M.: Conceptualization, Investigation, Supervision, Rydzkowski T: Investigation, Writing – review & editing, Gawdzińska K.: Writing – review & editing, Thakur V.K.: Conceptualization, Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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