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Properties of biocomposites produced with polypropylene and willow (*Salix babylonica* L.) wood/bark

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Abstract

The present study investigates the influence of various components of wood-plastic composites (WPCs) namely wood (W), inner bark (IB), outer bark (OB), and their varied percentage mixture on the mechanical behaviour. To achieve this goal, willow W, IB and OB flours were used as reinforcements at different weight percentages (17%, 27%, and 40%) in combination with polypropylene (PP) at varying weight percentages (44%, 58%, and 64%) along with a 2% compatibilizer. These constituents were processed in a twin-screw extruder with each treatment having a distinct mass proportion of reinforcement to polypropylene. Subsequently, test samples were fabricated using an injection molding machine from the obtained pellets. The mechanical properties of the resulting biocomposites were evaluated in accordance with ASTM standards. It was observed that, the flexural and tensile characteristics of the WPCs improved by the increasing inner bark content. Based on the findings of this investigation, a formulation comprising 27% wood, 27% inner bark, 44% polypropylene and 2% compatibilizing agent (W/IB/PP/MAPP) can be recommended where high mechanical properties are required. However, the other reinforced biocomposites exhibited notably lower notched impact strength compared to pure polypropylene.

Keywords: Willow wood and bark, mechanical properties, biocomposites, polypropylene

Polipropilen ve söğüt (*Salix babylonica* L.) odunu/kabuğu ile üretilen biyokompozitlerin özellikleri

Öz

Bu çalışmada, odun-plastik kompozitlerin OPK'lar) çeşitli bileşenlerinin, yani odun (O), iç kabuk (İK), dış kabuğun (DK) ve bunların çeşitli yüzdelerdeki karışımlarının mekanik davranış üzerindeki etkisi araştırıldı. Bu amaç için, farklı ağırlık yüzdelerinde (17%, 27% ve 40%) söğüt O, İK ve DK unları, değişen ağırlık yüzdelerinde (44%, 58%, 40%) ve 64%, ayrıca 2%'lik bir bağdaştırıcı ve polipropilen (PP) ile kombinasyon halinde takviye olarak kullanıldı. Bu bileşenler çift vidalı bir ekstrüderde işlendi; her işlemde polipropilene göre farklı bir kütlesel takviye oranı vardı. Daha sonra, elde edilen peletlerden bir enjeksiyon kalıplama makinesi kullanılarak test numuneleri üretildi. Elde edilen biyokompozitlerin mekanik özellikleri ASTM standartlarına uygun olarak değerlendirildi. İç kabuk içeriğinin artmasıyla OPK'larıneğilme ve çekme özelliklerinin arttığı gözlemlendi. Bu araştırmanın bulgularına dayanarak, yüksek mekanik özelliklerin gerekli olduğu durumlarda 27% odun, 27% iç kabuk, 44% polipropilen ve 2% uyumlaştırıcı madde (O/İK/PP/MAPP) içeren bir formülasyon önerilebilir. Bununla birlikte, diğer güçlendirilmiş biyokompozitler, saf polipropilenle karşılaştırıldığında belirgin şekilde daha düşük çentikli darbe dayanımı sergiledi.

Anahtar kelimeler: Söğüt ağacı ve kabuğu, mekanik özellikler, biyokompozitler, polipropilen

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1 Introduction

Thermoplastic composites benefit greatly from the inclusion of lignocellulosic materials, which offer several advantageous features such as cost-effectiveness, favourable mechanical properties, ease of machining and eco-friendliness (Kaboorani et al., 2021). Bark stands out as one of the most abundant resources at worldwide among the various lignocellulosic materials. It is usually used as fuel in ovens or landfiller. The bark content can be variable between 5 to 28% in a tree (Nyikosov, 1985; Kolozs, 2000; Guidi et al., 2008). The tree's bark protects it from insect damage, weather conditions and fungal infections, and it also has an important role in protecting wood against fire. Bark decomposes at a slower rate than wood because of its low nitrogen content in natural settings (Bersenev, 1975).

The innermost layer of the middle lamella in the bark primarily consists of pectin which makes it more prone to microbial degradation compared to the lignified fibers (Ek et al., 2009). In the case of willow, the outer bark is predominantly composed of aged phloem tissues (Panshin and de Zeeuw, 1980) and is relatively thin in younger trees. The sclerenchyma fibers of willow present significantly greater stiffness and strength compared to the standard pulp fibers derived from both hardwood and softwood (Dou et al., 2016). The bark of willow trees, which includes various species within the Salix genus, has been utilized as an indicator of air pollution in urban areas due to its tendency to accumulate heavy metals (Sawidis et al., 2011). In addition to its environmental monitoring capabilities, the bark of willow trees has historically been valued for its robust fibers which were extracted and employed in ancient times for the production of ropes and fishing nets (Bick, 2012). The amount of cellulose as an organic compound in the phloem and outer bark is determined to be relatively low while its amount reaches 40 to 50% in the wood, 18 to 25% in the phloem and only 3 to 17% in the outer bark (Ugolev, 1986). Han and Shin (2014) also compared the organic materials of the bark and wood of *Salix caprea*.

The chemical analysis of the willow bark and wood fibers shows a similar composition (Doczekalska et al., 2014; Oktaee et al., 2017) (Table 1). Muñoz et al. (2013) reported that the bark fibers of *Eucalyptus nitens* could be successfully used as a reinforcement for thermoplastic composites. The influence of pine and larch bark on the properties of polyethylene (PE) was studied by Rudenko (2010). The best combination was found in the composites with 80% PE and 20% bark. The modulus of rupture, the density, the thickness swelling and the water absorption were 41 MPa, 960 kg/m^3, 2% and 0.1%, respectively.

Table 1. Chemical compound of wood (willow) and bast fiber of bark (Doczekalska et al., 2014; Oktaee et al., 2017)

Substances soluble in	**Ethanol-benzene mixture (%)**	**Cold water (%)**	**Hot water (%)**	**1% NaOH (%)**
Willow wood	7.2	5.2	5.4	26.3
Fiber species	Cellulose (%)	Lignin (%)	Pentosans (%)	-
Willow wood	43.9	24.3	18.4	-
Fiber species	Cellulose (%)	Lignin (%)	Ash (%)	Hemicellulose (%)
Willow wood	44.8	24.3	0.7	34.5
Willow bark	44	25.8	3.3	30.8

Willow is a hardwood with low density characterized by a relatively uniform structure which can be used in the production of panels such as mixed hardwood oriented strand board (OSB) (Han et al., 2006). In this study, the bark fibers of willow were subjected to grinding and sieving processes to obtain two types of fillers which were a soft partition filler with inferior performance and a hard partition filler with superior performance. However, it is still unknown whether the enhanced performance observed with the hard partition filler would translate into other applications such as the manufacturing of wood-plastic composites (WPCs). It is crucial to consider the impact of bark contamination on the wood supplies utilized in WPC production alongside promoting improved biomass utilization by creating markets for bark (Harper and Eberhardt, 2010). The incorporation of bark in WPCs generally leads to reduced strength compared to composites composed solely of wood (Muszynski and McNatt, 1984; Blanchet et al., 2000; Makarychev, 2015). Furthermore, it has been demonstrated that the inclusion of tree bark as a filler in thermoplastics enhances thermal conductivity while composites thermal diffusivity remains unchanged (Makarychev, 2015). The inner and outer bark fibers have the potential for reinforcing thermoplastic composites. Previous studies reported that the use of bark without wood decreased the mechanical properties of thermoplastic composites. However, the objective of the study was to use willow inner and outer bark fibers combined with willow wood fiber and to characterize their properties for use in biocomposites used in outdoor applications such as decking, siding and fencing.

2 Material and Method

2.1 Material

Three fresh logs with a diameter of 1 m at breast height from the willow tree (*Salix babylonica* L.) were obtained from Saqqez joineries in Kordestan province located in the west of Iran. Saqqez was situated on the banks of the Saqqez River on the Zagros. The inner and outer of the wood bark were separated manually with a scalpel and stored at 20 °C until their use. The willow's wood and bark were meticulously sliced into tiny fragments and processed using a laboratory-grade electric rotary mill to obtain flours from the wood (W), inner bark (IB) and outer bark (OB). These flours were sieved to achieve a particle size ranging between 40 and 60 mesh. Willow wood and bark flours were dried in an oven at 65 ± 2 °C until they reached a constant weight before preparation of composite specimens.

Table 2. Compositions of the WPC formulations

Biocomposite code	**Wood flour (wt %)**	**Inner bark flour (wt %)**	**Outer bark flour (wt %)**	**PP (wt %)**	**(MAPP) (wt %)**
17%OB17%IB64%PP	0	17	17	64	2
17%W17%OB64%PP	17	0	17	64	2
17%W17%IB64%PP	17	17	0	64	2
40%OB58%PP	0	0	40	58	2
40%IB58%PP	0	40	0	58	2
40%W58%PP	40	0	0	58	2
27%W27%OB44%PP	27	0	27	44	2
27%W27%IB44%PP	27	27	0	44	2
27%OB27%IB44%PP	0	27	27	44	2
Neat PP	0	0	0	100	0

Homopolymer polypropylene (PP) was procured with a trade name of P10800 from Arak Petrochemical Company in Iran. The melt flow rate of the PP material ranged from 7 to 10 g per 10 min at a temperature of 190 °C. Maleic anhydride-grafted polypropylene (MAPP) was utilized with the trade name Aldrich 427845 to serve as a coupling agent. The compositions of polypropylene, MAPP, inner bark (IB), outer bark (OB) and wood (W) were varied according to the specified proportions outlined in Table 2. Table 2 was assessed using a complete randomized block design. Mechanical property testing was carried out with three replicates for each formulation.

2.2 Preparation of biocomposites

The constituents of each biocomposite sample including PP, MAPP, W, IB and OB were preliminarily mixed based on the proportions specified in Table 2. The homogeneous compounds were subsequently blended using a counter-rotating extruder with twin screws (Known as Dr. Collin System) operating at a speed of 70 rpm and a temperature range of 155-190 °C. Then, the resulting mixture was extracted from the mixing bowl, subjected to a cooling process in water and granulated pellets. Before the injection molding, the pellets were dried at a temperature of 85 °C for 24 hours. Finally, the dried pellets were placed in injection molding using an injection molding machine (Imen Machine Co., Iran) at temperatures ranging from 160 to 180 °C and a pressure of 10 MPa.

2.3 Mechanical testing

Before mechanical testing, all biocomposite samples were stored at a controlled temperature of 23 °C and 50% RH. Flexural testing that involves the determination of flexural strength and modulus, was performed using an Instron 1186 universal testing machine by ASTM test method D-790. The speed of the machine’s crosshead was set at 5 mm/min and the dimension of the flexural test specimens was 105 × 10 × 4 mm.

The tensile properties of the composites were evaluated using an Instron-1186 testing machine following the ASTM D-638 standard. The dimensions of the tensile test specimens were 145 × 10 × 4 mm.

Notched Izod impact testing was conducted using a Santam machine by ASTM D-256 standard. The dimensions of the specimen used for the notched Izod impact test were 60 × 12 × 6 mm.

2.4 Statistical analysis

Statistical analysis was conducted by utilizing analysis of variance (ANOVA) with the assistance of SPSS software. The variance of the 10 different formulation designs presented in Comparison of property means was conducted by using Duncan's new multiple range test at confidence levels of 99% and 95% as depicted in Figures 1, 2, 3, 4 and 5.

3 Results and discussion

3.1 Flexural modulus and flexural strength

The flexural modulus and strength values of the biocomposites ranged from 30.68 MPa to 46.83 MPa and 1207 MPa to 4426 Mpa, respectively (Figures 1 and 2). The biocomposites OB/PP exhibited the lowest flexural strength and flexural modulus. The 40% OB content presented the lowest flexural strength and was significantly varied from the treatments having lower OB content amongst the bark/PP conditions. The flexural strength and flexural modulus characteristics increase significantly by decreasing the OB content from 40% to 27% and

17%.The ideal composition for enhancing flexural and modulus strength was identified as 27% W, 27% IB and 44% PP within the IB/PP biocomposites. Also, the values for IB/PP were varied from those for neat PP. Thus, the IB acted more as a biocomposite filler rather than as a reinforcing agent. Hosseinihashemi et al. (2017) and Yemele et al. (2010) found the same trends also.

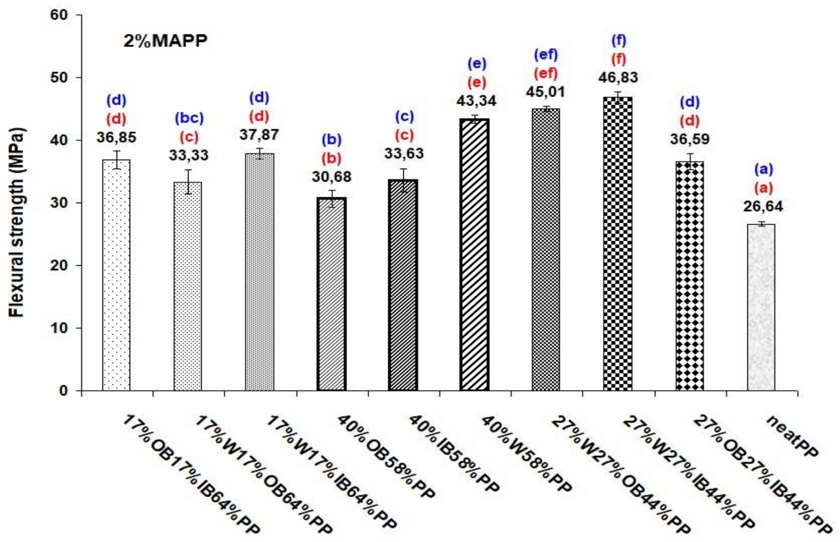

Figure 1. Mean values of flexural strength of the IB/PP, OB/PP, W/PP, IB/OB/PP, IB/W/PP, OB/W/PP, IB/OB/W/PP and neat PP composites. Multiple range tests of Duncan (in parentheses) reveal distinct alphabetical designations signifying significant differences among various treatments (composites). Red colour means at a 95% confidence level and blue colour means at a 99% confidence level.

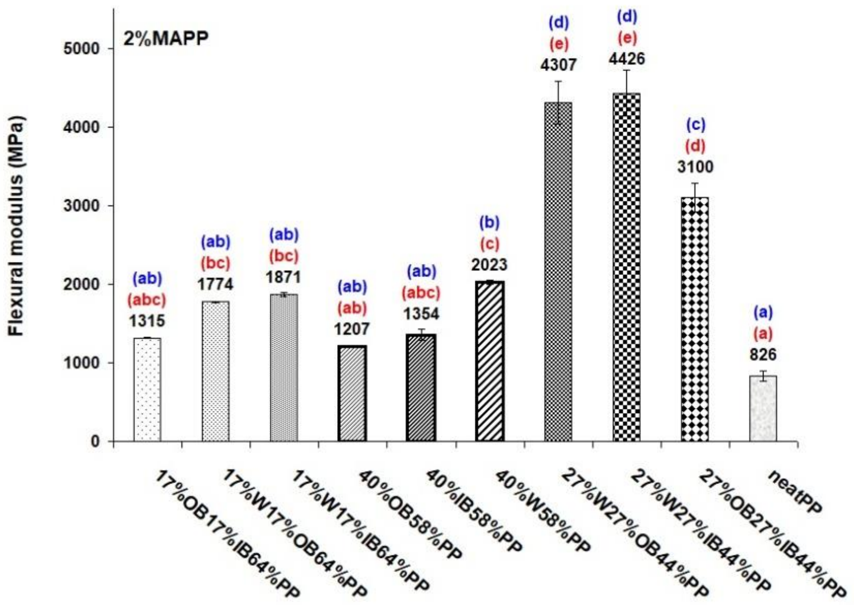

Figure 2. Mean values of flexural modulus of the IB/PP, OB/PP, W/PP, IB/OB/PP, IB/W/PP, OB/W/PP, IB/OB/W/PP and neat PP composites. Multiple range tests of Duncan that are indicated within parentheses demonstrate varying alphabetical designations highlighting significant differences among different treatments (composites). Red colour means at a 95% confidence level and blue colour means at a 99% confidence level.

The variation in flexural strength can be attributed to different factors in the polypropylene (PP) matrix because of the presence of inner bark (IB) and outer bark (OB). These factors include the length and fine content of IB and the aspect ratio (length/width) of IB and OB within the PP matrix which are observed to account for the high and low effects (Migneault et al., 2009; Dou, 2015). The lower inherent strength of fibers of bark (Yemele et al., 2010) reduced cellulose (polysaccharide) content in bark fillers compared to wood (Harper and Eberhardt, 2010) and the occurrence of delamination between fines and PP (Safdari et al., 2011; Hosseinihashemi et al., 2017) are also contributing factors. Moreover, the higher concentration of extractives in the outer bark may result in a weakened surface layer thereby diminishing the effectiveness of the coupling agent in creating cross-linking networks with cellulose (Saputra et al., 2004).

When the bark flour was blended with wood flour, the flexural modulus increased compared to the biocomposites that contained only OB. The flexural modulus strength recovered by increasing the outer bark flour content in all of the combinations. For instance, the flexural strengths did not show a significant difference with composites made of W/PP (17% WF + 17% IB + 64% PP) and (17% OB + 17% IB + 64% PP) in the composite made of W/OB/PP such as 27% OB + 27% W + 44% PP. This can be attributed to the high mechanical properties of wood. The amount of cellulose in wood is higher than in bark. As known, cellulose in wood significantly affects the mechanical properties. As the cellulose content in the bark is lower than wood, the flexural properties of the biocomposites were lower than those of the biocomposites containing wood flour. The lower quantity of fiber in the composites was offset by its superior quality and it leads to no noticeable distinctions between the two biocomposites. The biocomposites consisting of 40% W and 58% PP exhibited favourable flexural strength as observed in the results. Notably, these biocomposites demonstrated a significant difference in flexural strength compared to all other treatment groups. In contrast, the biocomposites composed of 40% OB and 58% PP displayed lower flexural strength compared to treatments with lower wood or wood/outer bark content despite containing a lower proportion of OB, This observation suggests that fiber content alone cannot be solely attributed as the determining factor in enhancing flexural strength. Other factors such as the morphological and chemical characteristics of the fibers should be taken into consideration to understand the variations in flexural strength as well as the intrinsic strength of the fibers.

3.2 Tensile strength and tensile modulus

The tensile strength and modulus spanned from 21.64 MPa to 37.55 MP and 1740 MPa to 4653 MPa in reinforced biocomposites, respectively (Figures 3 and 4). The tensile strength of those biocomposites made of OB/PP was found to be lower than other biocomposites. The biocomposites produced from W/IB/PP and W/PP showed the highest tensile strength and modulus values among all other compositions, respectively. The biocomposites having more than 17% IB (i.e. 27% IB) and about 40% W were significantly different from each other. The ideal composition for enhancing tensile strength modulus was 27% IB and 40% W among the IB/PP and W/PP biocomposites.

Previous research has indicated a positive correlation between the cellulose content of a fiber and its tensile strength (Pickering et al., 2016; Väisänen et al., 2016). However, the relatively low mechanical strength observed specifically in the biocomposite consisting solely of outer bark (OB) and the combination of wood (W) and outer bark (W + OB) suggests the presence of interactions between the bark extractives and the matrix. The elevated levels of extractives and minerals present in the fractionated bark tissues posed challenges during the extrusion processing stage. These extractives may be engaging with the polypropylene (PP) matrix potentially leading to alterations in rheological properties. However, further investigation is necessary to confirm this hypothesis.

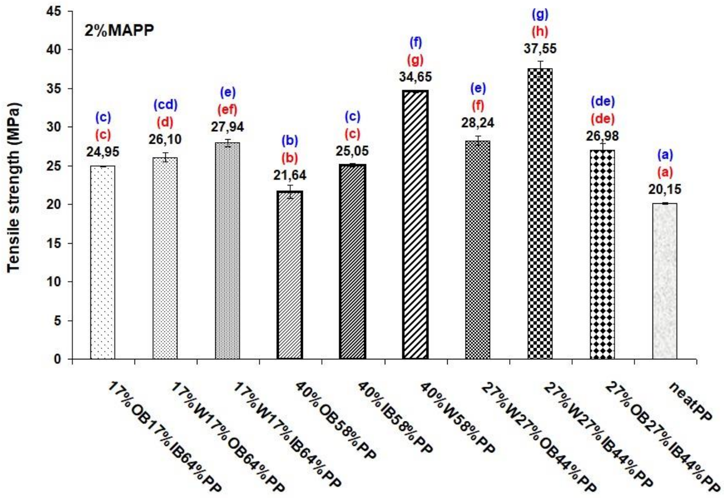

Figure 3. Mean values of tensile strength of the IB/PP, OB/PP, W/PP, IB/OB/PP, IB/W/PP, OB/W/PP, IB/OB/W/PP and neat PP composites. Multiple range tests of Duncan presented in parentheses signify distinct alphabetical designations that indicate significant disparities among various treatments (composites). Red colour means at a 95% confidence level and blue colour means at a 99% confidence level.

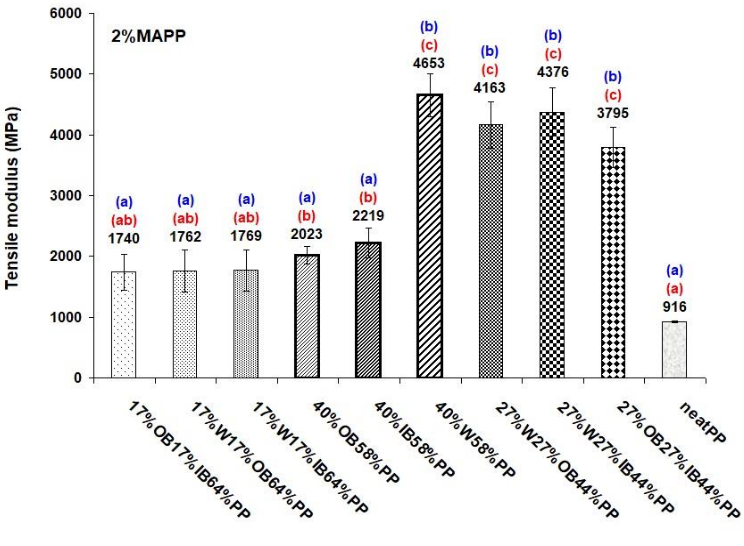

Figure 4. Mean values of tensile modulus of the IB/PP, OB/PP, W/PP, IB/OB/PP, IB/W/PP, OB/W/PP, IB/OB/W/PP and neat PP composites. Multiple range tests of Duncan presented in parentheses signify distinct alphabetical designations that indicates significant disparities among various treatments (composites). Red colour means at a 95% confidence level and blue colour means at a 99% confidence level.

In the current study, it was observed that bark flour exhibited better dispersion within a polymer matrix compared to wood flour. However, this improved dispersion did not yield any mechanical advantages (Harper and Eberhardt, 2010). The inferior performance of OB can be attributed to several factors in comparison to IB and wood. These include the low slenderness ratio of OB fibers (Stark and Berger, 1997), inadequate dispersion of fine fibers within the plastic matrix leading to stress concentration (Gamstedt et al., 2007) and lower intrinsic strength of outer bark fibers compared to IB and wood fibers. Some researchers have reported poor adhesion between the outer bark fibers and the coupling agent which could be another reason contributing to lower tensile strength (Bouafif et al., 2008; Bouafif et al., 2009).

The tensile strength and moduli of the biocomposites increased with higher W and IB contents. Generally, the length and aspect ratio of willow inner bark fibers were found to be approximately twice as high as those of willow wood fibers although variations were observed among different willow species. The increased fiber length positively impacted the bending properties of the biocomposites. The composition consisting of 27% W, 27% IB and 44% PP exhibited a higher flour content compared to the composition of 17% W, 17% IB and 64% PP. Nevertheless, the notable presence of IB in the initial composition led to a significantly higher tensile strength. This highlights the fact that the impact of fiber content on mechanical properties relies heavily on the inherent strength and dimensions of the fibers. These results are consistent with earlier studies suggesting that augmenting particle size or slenderness ratio tends to improve both flexural and tensile modulus as well as strength (Stark and Berger, 1997; Stark and Rowlands, 2003).

3.3 Impact resistance

The obtained impact strength values ranged from 35.28 $J.m^{-1}$ to 62.00 $J.m^{-1}$ as illustrated in Figure 5. Interestingly, the impact strength results contradicted the findings for other mechanical properties such as flexural strength, flexural modulus, tensile strength and tensile modulus. Biocomposite compositions containing 27% OB, 27% IB and 44% PP as well as compositions with lower flour content and higher polypropylene content (64% PP) exhibited higher impact strength. This observation can primarily be contributed to the lack of compatibility among the phases and the presence of stress concentration regions resulting from the inclusion of bio-resource fibers. These regions are prone to crack initiation with lower energy requirements in the samples (Rowell et al., 1997). The introduction of fillers led to a reduction in energy absorption by the biocomposites, consequently resulting in decreased impact resistance. As a result, the biocomposites reinforced with lignocellulosic materials demonstrated increased brittleness and lower notched impact strength.

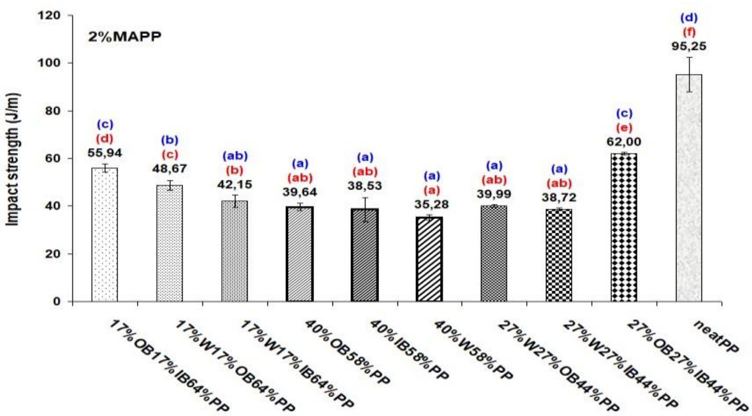

Figure 5. Mean values of impact strength of the IB/PP, OB/PP, W/PP, IB/OB/PP, IB/W/PP, OB/W/PP, IB/OB/W/PP and neat PP composites. Multiple range tests of Duncan indicated within parentheses denote distinct alphabetical designations underscoring significant disparities among various treatments (composites). Red colour means at a 95% confidence level and blue colour means at a 99% confidence level.

4 Conclusions

- The findings of this study demonstrated that the incorporation of IB and W significantly enhanced the mechanical properties including flexural strength, flexural modulus, tensile strength and tensile modulus when they were compared to neat PP. However, an inverse relationship was observed between the content of IB and W and the notched impact strength of the biocomposites. Despite the overall improvement in mechanical strength, the impact of W was substantial on the mechanical properties, yet not as prominent. Conversely, the addition of IB to the biocomposites significantly restored the mechanical characteristics. In contrast, biocomposites containing OB exhibited inferior mechanical properties compared to those incorporating W and IB. This disparity could be attributed to the distinct chemical composition between bark and wood as well as the low slenderness ratio of OB which impeded proper dispersion. Additionally, the lower strength of bark fibers contributed to this distinction compared to wood fibers.
- The fiber content effect on the mechanical strength exhibited a positive trend for biocomposites composed of W/IB/PP and W/PP while it had a negative impact on biocomposites consisting of OB/PP. Thus, the influence of fiber content on the mechanical properties is contingent upon the inherent characteristics of the fibers. Alone, OB and IB did not adequately reinforce the polypropylene composite, but more favourable outcomes were achieved when combined with W or blended together. The observed negative relation between notched impact strength and biocomposites reinforced with W and IB aligns with the behaviour typically observed in other lignocellulosic materials. This can be attributed to the increased brittleness of the polypropylene caused by the inclusion of lignocellulosic fillers resulting in a reduced energy requirement for crack initiation in the samples.

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Author Contributions

Seyyed Khalil Hosseinihashemi: Creating the research idea, writing the article, performing the statistical operations. **Ayoub Eshghi:** Conducting the laboratory work, taking the measurement data. **Younes Shirmohammadli:** Writing, original draft, reduce similarity rate, and editing.

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Conflict of interest

We confirm that there is no conflict of interest.

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