

Effects of NanoClay and Coupling Agent on Mechanical Properties of *Picea* Flour/Polypropylene/NanoClay Composite

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The effects of nanoclay and the coupling agent Maleic Anhydride Grafted Polypropylene (MAPP) on mechanical properties of wood plastic composite produced from *Picea* flour/ polypropylene/ nanoclay were examined. For this propose, *Picea* wood flour at a constant level of 40%, MAPP in two levels of 2% and 4%, and nanoclay in 4 levels of 0%, 1%, 3%, and 5% were used. Next, wood plastic nano composite was constructed by using of injection moulding method, and its mechanical tests including tensile, bending, and impact performed on samples were evaluated. Results showed that tensile strength and bending strength, and bending modulus of composite were enhanced by increasing nanoclay and MAPP contents. Structural studies of the wood-plastic nanocomposite using X-ray diffraction demonstrated that the distribution of nanoclay particles within the polymer matrix involved intercalation, with an increase in the distance between layers observed at higher levels of nanoclay. However, this increase may also be indicative of agglomeration effects that could influence the overall material properties.

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INTRODUCTION

Wood-plastic composites (WPCs) are an innovative category of materials that have gained significant traction in many advanced countries due to their versatility and eco-friendly characteristics (Wang *et al.* 2021). These composites are composed of a combination of polymer and cellulose-based materials, offering a sustainable alternative to traditional wood products. The WPCs utilize a variety of polymers, including polypropylene (PP), polyethylene (PE), and polyvinyl chloride (PVC). These are commonly used petrochemical-based plastics that, when combined with cellulose fillers such as wood flour and fibers, can improve certain mechanical properties of the composites. However, it is important to note that the presence of biodegradable components such as lignocellulosic materials (*e.g.*, linen, straw, bamboo, chaff, and stubble) does not inherently render these plastics biodegradable. While these additives may enhance certain features, the overall biodegradability of conventional plastics remains limited. Thus, these composites should be approached with caution in terms of their environmental impact (Gardner *et al.* 2015; Elamin *et al.* 2020).

The mechanistic understanding of how nanoclay and coupling agents enhance the properties of WPCs is crucial. Previous studies have shown that the dispersion of nanoclay

within the polymer matrix leads to improved interfacial bonding, which enhances mechanical performance. The presence of nanoclay can also modify the crystallinity and thermal stability of the polymer, impacting overall composite strength (Rao *et al.* 2021; Amari *et al.* 2021). Moreover, the interaction between the hydrophilic nature of cellulose fibers and the hydrophobic polymer can be effectively mediated by coupling agents such as maleic anhydride grafted polypropylene (MAPP), which improves compatibility and results in better stress transfer within the composite (Goda *et al.* 2024).

The inherent properties of cellulose fibers, which are hydrophilic (polar), create challenges when mixed with non-polar plastics (Mohammed *et al.* 2023). This disparity can lead to poor adhesion and cohesion between the polymer matrix and the wood fibers, resulting in compromised mechanical properties (Suriani *et al.* 2021). To address this issue, chemical coupling agent are introduced during the production process (Oyelere and Wu 2025). These coupling agent, such as MAPP, facilitate the formation of cohesive surfaces, improving the bond between thermoplastics and cellulose fibers (Dairi *et al.* 2017). Research by Elamin *et al.* (2020) has shown that adding coupling agent can significantly enhance the mechanical properties of WPCs. Their findings indicated that the mechanical weaknesses often observed in poorly bonded composites were largely due to inadequate connections between the matrix and the fibers. By incorporating MAPP, they achieved notable improvements in tensile modulus, ultimate strength, and impact strength (Elamin *et al.* 2020).

Polymer nanocomposites represent a cutting-edge area of research, both in Iran and international levels (Kiaei *et al.* 2018; Kaymakci 2022). The incorporation of low amounts of nanoclay (2% to 5%) into polymer matrices has been shown to enhance mechanical properties and thermal durability (Stephen *et al.* 2020). These nanocomposites not only exhibit improved impermeability but also reduced flammability, making them suitable for a wide range of applications, including construction, automotive, and consumer products (Gardner *et al.* 2015). Unlike traditional composites, polymer nanocomposites based on nanoclay are recoverable, offering an added environmental benefit (Hussain *et al.* 2024). This recoverability is especially important in the context of sustainability, as it allows for the recycling of materials at the end of their lifecycle (Elamin *et al.* 2020).

Recent advancements in WPC technology have led to the exploration of bio-based polymers and additives, further enhancing the sustainability of these materials. Research into natural fibers, such as hemp and jute, is becoming increasingly prominent, as these materials can improve the mechanical performance and reduce the carbon footprint of WPCs (Elamin *et al.* 2020). Furthermore, the application of WPCs has expanded into various sectors, including outdoor furniture, decking, and automotive interiors, where their durability and aesthetic appeal are highly valued (Wang *et al.* 2021).

The growing demand for sustainable materials is driving innovation in this field, leading to the development of new formulations and processing techniques. In conclusion, the WPCs represent a promising solution for sustainable material needs, combining the advantages of both wood and plastic. On going research into MAPP, nanocomposites, and bio-based materials continues to enhance their performance and applicability.

This research aims to clarify the relationships between the incorporation of nanoclay and maleic anhydride grafted polypropylene (MAPP) in wood-plastic composites (WPCs). It specifically investigates how varying concentrations of nanoclay and types of MAPP influence mechanical performance and structural characteristics. By providing insights into mechanisms that have not been thoroughly explored in existing literature, this study contributes to the advancement of WPC technology and offers valuable guidance on

the optimal formulation of these composites, particularly regarding the balance between nanoclay content and compatibilizer use to achieve desired mechanical properties.

EXPERIMENTAL

In this research, polypropylene produced by Arak Petrochemistry Company, with a melt flow index of 18 g/10 min and a density of approximately 0.9 g/cm³, was used as the primary polymer material. Maleic anhydride grafted polypropylene (MAPP) from Arak Petrochemistry Company, with a melt flow index of 64 g/10 min (brand: Priex 20070), was used as the coupling agent. *Picea* wood flour and nanoclay powder (brand: Cloisite 30B) were also incorporated. To evaluate the effects of Cloisite 30B nanoclay and MAPP on the properties of WPCs, the formulation included 40% *Picea* wood flour and 60% polypropylene. Nanoclay was added at levels of 0%, 1%, 3%, and 5%, while the MAPP was included at 2% and 4%, resulting in a total of eight treatments (see Table 1).

The mixing was carried out using a Hakee internal mixer (HBI System 90, USA) at a temperature of 180 °C and a speed of 60 RPM. First, polypropylene was fed into the mixing chamber. After the polypropylene melted, MAPP was added. At the two-minute mark, the wood flour and nanoclay were introduced, and the total mixing time was 10 min. The compounded materials were then ground using a pilot-scale grinder (WIESER, WGLS 200/200 Model). The resulting granules were dried at 105 °C for 4 h. Test specimens were prepared by injection molding using an Eman machine (Iran). Finally, the specimens were conditioned at a temperature of 23 °C and a relative humidity of 50% for at least 40 h, according to ASTM D618-99, prior to testing. Mechanical properties were evaluated, including bending strength and modulus according to ASTM D 474, tensile strength following ASTM D 638, Impact resistance as per ASTM D 256.

Each test was performed in triplicate for reliability. Data analysis was conducted using Excel and SPSS 17 software. The effects of nanoclay and MAPP percentages on strength and modulus were examined through variance decomposition, with a significance level of 5%. Duncan's test was used for averaging. X-ray diffraction (XRD) was employed to identify the intercalation and exfoliation structures of the nanoclay in the composites and to assess its distribution in the polymer matrix. X-ray transmission was performed with an XRD machine, measuring diffraction angles (2θ) in the range of 0° to 10°. Samples for this analysis were prepared with dimensions of 2 × 2 × 0.2 mm³.

Table 1. Percentages of Components in Nano Composite Treatments

Treatment	<i>Picea</i> Wood Flour (%)	Polypropylene (%)	NanoClay (%)	MAPP (%)
1	40	58	0	2
2	39	58	1	2
3	37	58	3	2
4	35	58	5	2
5	40	56	0	4
6	39	56	1	4
7	37	56	3	4
8	35	56	5	4

RESULTS AND DISCUSSION

Bending Strength

Variance decomposition results from Table 2 indicate that there was a significant effect of nanoclay content on the bending strength (MOR) of the WPCs at a confidence level of 95%. The data show that while the wood flour content was held at a constant level, variations in nanoclay concentration influenced the MOR. As shown in Fig. 1, the highest bending strength of the WPCs corresponds to a nanoclay content of 3% and a coupling agent concentration of 4%.

Table 2. Variance Analysis of the Independent Effects and Interplay of Coupling Agent and NanoClay on MOR of WPCs

Dependent Variable: MOR

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	215.774	7	30.825	6.496	.001
Intercept	52859.136	1	5.286E4	1.11E4	.000
Nano	56.412	3	18.804	3.963	.027
MAPP	114.276	1	114.276	2.40E1	.000
Nano * MAPP	45.086	3	15.029	3.167	.053
Error	75.922	16	4.745		
Total	53150.832	24			
Corrected Total	291.696	23			

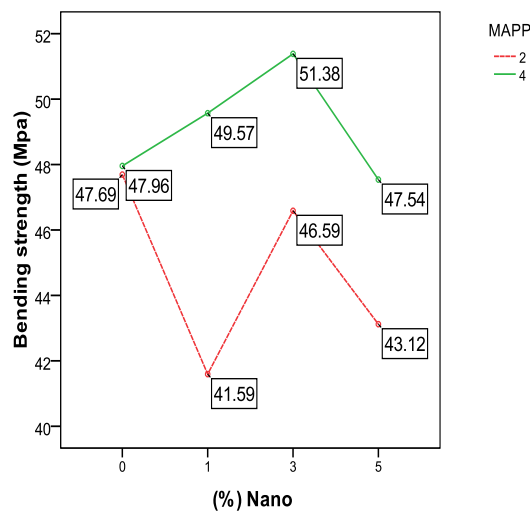


Fig. 1. Interplay of coupling agent and nanoclay on bending strength of WPCs

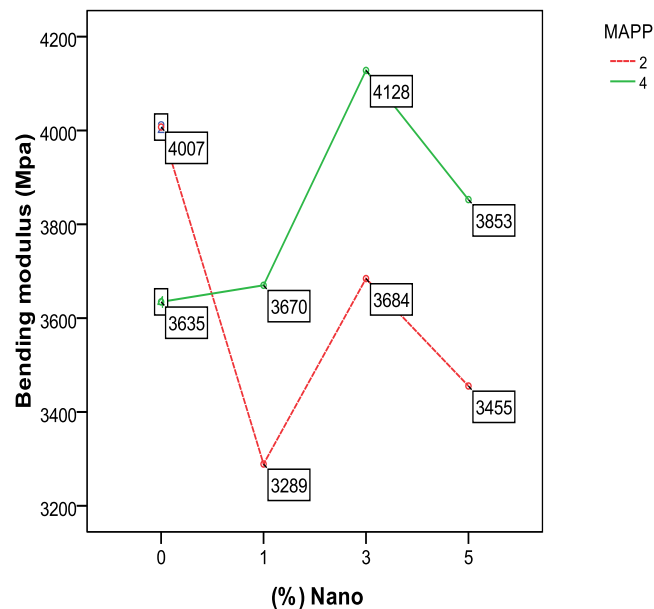
Bending Modulus

Variance decomposition results from Table 3 indicate a no significant difference in the interplay between nanoclay and the coupling agent on the bending modulus (MOE) of the WPCs at a confidence level of 95%. As shown in Fig. 2, the highest bending modulus was associated with 3% nanoclay and 4% coupling agent.

Table 3. Variance Analysis of the Independent Effects and Interplay of Coupling Agent and NanoClay on MOE of WPCs

Dependent Variable: MOE

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.600E6	7	2.286E5	4.7E0	.005
Intercept	3.313E8	1	3.313E8	6.8E3	.000
Nano	641771.000	3	2.139E5	4.4E0	.019
MAPP	270512.667	1	2.705E5	5.6E0	.031
Nano * MAPP	687620.333	3	2.292E5	4.7E0	.015
Error	768967.333	16	4.806E4		
Total	3.336E8	24			
Corrected Total	2.369E6	23			

**Fig. 2.** Interplay of coupling agent and nanoclay on bending modulus of WPCs

Tensile Strength

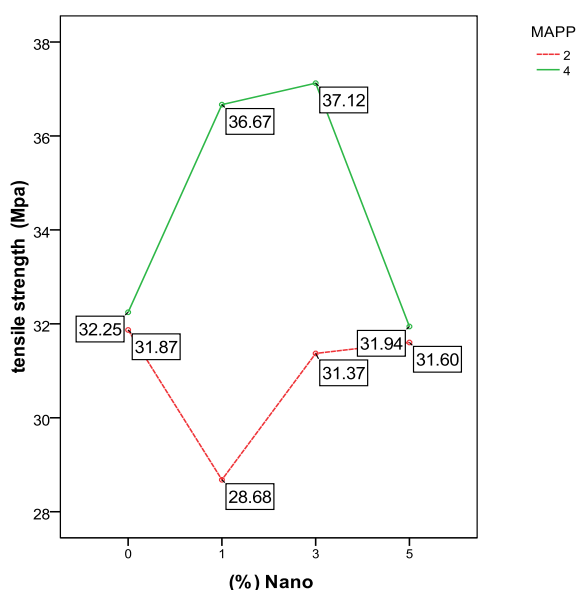
Table 4 indicates a no significant difference in the interplay between nanoclay and the coupling agent on the tensile strength of the wood-plastic composite at a confidence level of 95%. As shown in Fig. 3, the highest tensile strength is associated with 3% nanoclay and 4% coupling agent, while the lowest tensile strength corresponds to 1% nanoclay and 2% coupling agent.

As shown in Figs. 1 to 3, the mechanical properties of the composites exhibited distinct trends with varying nanoclay content. Specifically, while initial increases in nanoclay concentration (up to 1%) did not lead to substantial improvements in tensile strength, bending strength, and bending modulus, whereas the results indicated significant enhancements at 3% nanoclay content. These findings suggest that there is an optimal range for nanoclay incorporation, where mechanical properties improve due to better interfacial bonding and dispersion of nanoclay within the polymer matrix.

Table 4. Variance Analysis of the Effects and Interplay of Coupling Agent and NanoClay on Tensile Strength of WPCs

Dependent Variable: TensileStrength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	167.694	7	23.956	4.570	.006
Intercept	25643.998	1	2.564E4	4.89E3	.000
Nano	21.977	3	7.326	1.397	.280
MAPP	78.373	1	78.373	1.49E1	.001
Nano * MAPP	67.345	3	22.448	4.282	.021
Error	83.873	16	5.242		
Total	25895.565	24			
Corrected Total	251.567	23			

**Fig. 3.** Interplay of coupling agent and nanoclay on tensile strength of WPCs

The mechanism behind the enhanced mechanical properties can be attributed to the effective dispersion of nanoclay particles at lower concentrations, which improves stress transfer at the interface. However, exceeding the optimal level of nanoclay leads to agglomeration, which negatively affects the material's integrity (Rostami *et al.* 2022). Furthermore, the role of MAPP as a coupling agent is critical, as it improves compatibility between the hydrophilic wood flour and the hydrophobic polypropylene, facilitating better load transfer and reducing stress concentration points in the composite material.

The increase in nanoclay content and the presence of intercalation and exfoliation morphology in the nanocomposite—resulting from the interaction of organic chains with nanoclay particles—enhanced the strength of the composite (Amari *et al.* 2021). Additionally, the heterogeneous structure and high surface area-to-volume ratio of the nanoclay, in conjunction with the organic material, contributed to the reinforcement effect. Nanoclay particles acted as amplifiers, increasing the interfacial bonding between the two

phases. The strength of the composite was further enhanced by the addition of nanoclay due to the expansion of nanoclay layers and the strong cohesion between the polymer and the nanoclay (Rao *et al.* 2021).

Impact Strength

The results from Table 5 indicate that there were significant effects related to the interplay between nanoclay and the coupling agent on the impact strength of the WPCs at a confidence level of 95%. As shown in Fig. 4, the highest impact strength was associated with 1% nanoclay and 2% coupling agent, while the lowest impact strength corresponded to 5% nanoclay and 4% coupling agent.

Table 5. Variance Analysis of the Independent Effects and Interplay of Coupling Agent and NanoClay on Impact Strength of WPCs

Dependent Variable: ImpactStrength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.006	7	.001	.754	.632
Intercept	4.067	1	4.067	3.3E3	.000
Nano	.005	3	.002	1.3E0	.282
MAPP	.001	1	.001	.500	.490
Nano * MAPP	.001	3	.000	.204	.892
Error	.019	16	.001		
Total	4.093	24			
Corrected Total	.026	23			

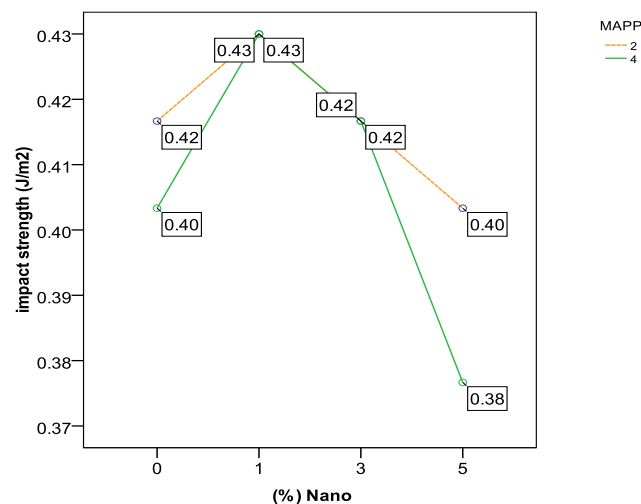


Fig. 4. Interplay of coupling agent and nanoclay on impact strength of WPCs

The presence of nanoclay particles can create areas of stress concentration, which serve as initiation points for failure (Rostami *et al.* 2022). As the amount of nanoclay increases, the impact strength of the composite decreases. This reduction occurs because the addition of nanoclay at first enhances the energy absorption capability of the composite. However, an excessive amount of nanoclay leads to the formation of regions within the polymer matrix that concentrate stress, facilitating crack propagation from these areas.

Consequently, while nanoclay can improve certain mechanical properties, an increase in its content can result in decreased impact strength due to these adverse effects. Increasing the percentage of coupling agent enhances the interfacial bonding between cellulose and polymer fibers, leading to improvements in tensile strength, MOE, and MOR, while decreasing impact strength. In other words, the coupling agent facilitates the creation of a more harmonious structure within the composite material. As the material structure becomes more homogeneous, stress distribution improves when a static load is applied, resulting in reduced stress concentration in specific regions of the product. Consequently, the stress tolerance capacity is increased. A study by Goda *et al.* (2024) indicated that enhancing cohesion between fibers and the matrix, as well as strengthening their internal connections, results in a decrease in the impact strength of multi-structured materials. Greater cohesion between the fiber and matrix increases the stress at the interface, which, in turn, diminishes the impact strength (Goda *et al.* 2024).

The observed decrease in impact strength at higher nanoclay contents can be attributed to the formation of stress concentration points within the polymer matrix. As more nanoclay is added, these particles can agglomerate, leading to localized regions of weakness that facilitate crack propagation. This highlights the importance of optimizing nanoclay content to balance enhanced energy absorption with structural integrity.

Structural Study by X-ray Diffraction Test

The X-ray diffraction spectra results of the WPCs in this study indicated that the composite structure was of the intercalation type. The results also demonstrated that the distance between the silicate layers of the nanoclay particles increased when 3% nanoclay filler is used. However, the distance between the silicate layers decreased when the nanoclay content was increased to 5%.

In Fig. 5, the X-ray diffraction spectra of the wood-plastic nano composite with 1% nanoclay and 2% MAPP are presented for an angle range of 0° to 10° . The dissipation peak of nanoclay in this spectrum was observed at an angle of $2\theta=1.85$. The distance between the silicate layers of nanoclay at this angle is $d_{11}=45.69 \text{ \AA}$. The peak observed in the spectrum corresponds to the crystalline area of the nanoclay, which does not disappear completely, indicating an intercalation structure in the wood-plastic nano composite.

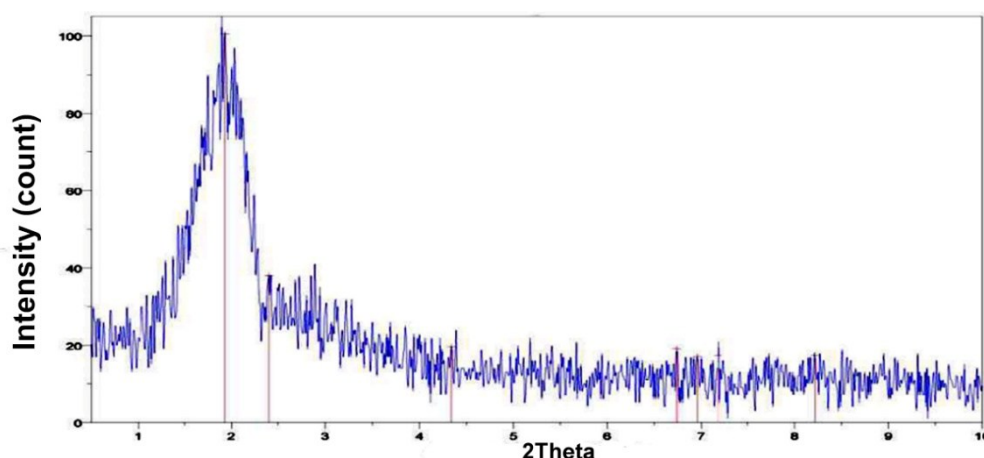


Fig. 5. X-ray Spectra of Wood-Plastic Nano Composite with 1% NanoClay and 2% MAPP

In Figure 6, the X-ray diffraction spectra of the wood-plastic nano composite with 3% nanoclay and 2% MAPP are presented. The distance between the silicate layers of nanoclay at this angle was $dA_1=47.68 \text{ \AA}$, indicating that the distance between the silicate layers increased with the addition of nanoclay up to 3%.

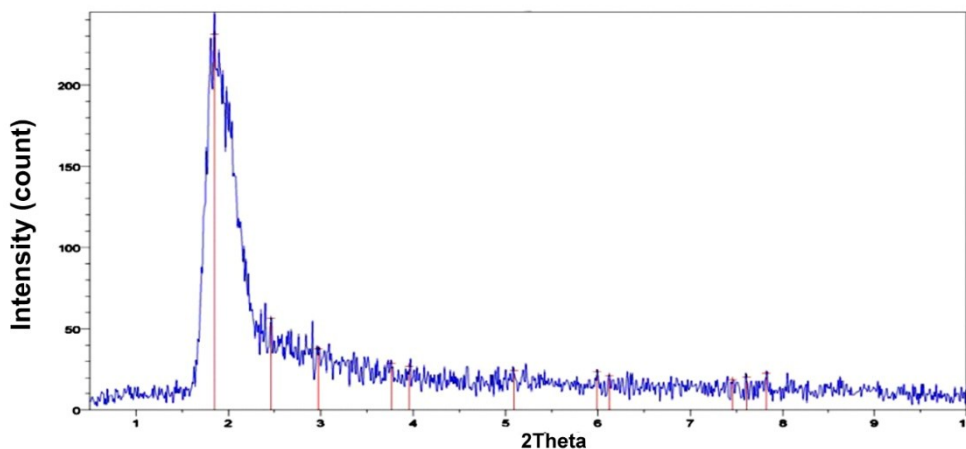


Fig. 6. X-ray spectra of wood-plastic nano composite with 3% nanoclay and 2% MAPP

In Figure 7, the X-ray diffraction peak of nanoclay was observed at an angle of $2\theta=1.85$. The distance between the silicate layers of nanoclay at this angle was $dA_1=47.67 \text{ \AA}$. The spectrum indicates that the distance between the silicate layers of nanoclay decreased with the increase of nanoclay content to 5%.

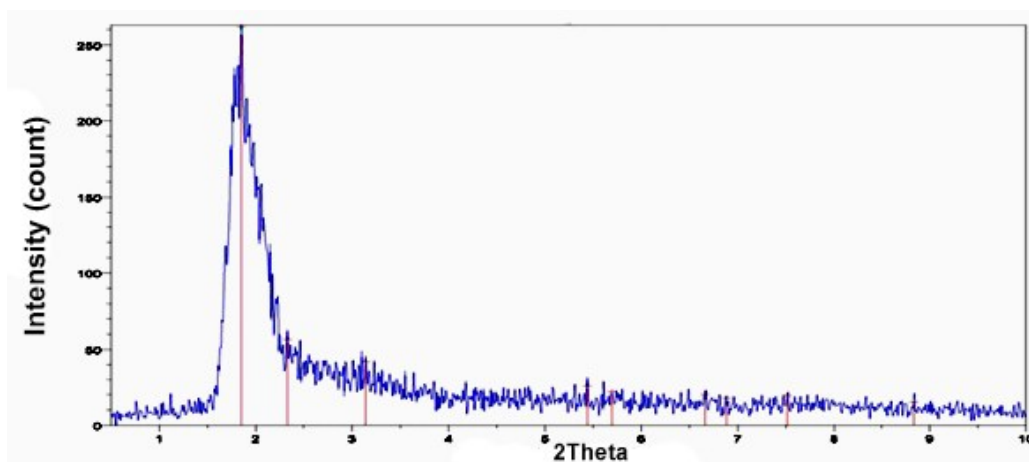


Fig. 7. X-ray spectra of wood-plastic nano composite with 5% nanoclay and 2% MAPP

Structural studies of the wood-plastic nano composite using X-ray diffraction methods indicate that the distribution of nanoclay within the polymer matrix exhibits an intercalation structure. Furthermore, the distance between the layers increases with the addition of more nanoclay particles (Goda *et al.* 2024).

CONCLUSIONS

In this research, the effect of nanoclay particles and a coupling agent on the mechanical properties of wood-plastic composites made from polypropylene and wood

flour was investigated. The results showed that:

1. The tensile strength, bending strength, and bending modulus exhibited notable improvement at 3% nanoclay content, indicating enhanced mechanical performance and a significant interaction between nanoclay and MAPP.
2. At the 1% nanoclay level, while there were initial gains in impact strength, the tensile and bending strengths did not demonstrate significant improvement compared to the control samples, highlighting the importance of optimal nanoclay content.
3. The introduction of 5% nanoclay resulted in decreased tensile strength, bending strength, impact strength, and bending modulus, which was attributed to the aggregation and compression of nanoclay particles, emphasizing the need for careful control of nanoclay levels.
4. The findings underscore the crucial role of MAPP as a compatibilizer, enhancing the interactions between nanoclay and the polymer matrix, which significantly improves the overall mechanical properties of the composites.

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