EFFECT OF NANOCLAY (HALLOYSITE) ON THE DYNAMIC MECHANICAL PROPERTIES OF RECYCLED POLYPROPYLENE FILLED NaOH TREATED MAIZE COB POWDER COMPOSITES

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ABSTRACT

This study investigates the effects of nanoclay (halloysite) on the dynamic mechanical behaviour of recycled polypropylene (rPP) composites reinforced with maize cob powder (MCP). The influence of filler loading (10–40 wt%), particle size (100–500 μm), 2 % NaOH surface treatment, and nanoclay content (2–8 wt%) on storage modulus (E'), loss modulus (E''), damping factor (tan δ), and glass transition temperature (Tg) were analyzed using a Netzsch DMA 242 dynamic mechanical analyzer. Results revealed that increasing MCP loading and decreasing particle size enhanced the stiffness and thermal stability of the composites. The addition of nanoclay further improved modulus and Tg by restricting polymer chain mobility. NaOH treatment enhanced interfacial bonding, leading to improved stiffness and damping behaviour. The highest Tg (122.6 °C) was recorded for 100 μm fillers at 40 % loading and 8 % nanoclay, demonstrating the synergistic reinforcement effect of MCP and nanoclay in rPP matrices. The improved viscoelastic properties suggest the suitability of rPP/MCP/nanoclay composites for applications requiring enhanced stiffness, thermal resistance, and vibration damping.

Keywords: *Recycled Polypropylene (rPP), Maize Cob Powder (MCP), Halloysite Nanoclay (NC), Sodium Hydroxide (NaOH) Treatment, Dynamic Mechanical Properties*

1. INTRODUCTION

The environmental and economic implications of polymer waste (Figure 1) have prompted the increasing utilisation of recycled polymers and agro-based fillers for sustainable composite materials.

Recycled polypropylene (rPP), due to its availability and favourable mechanical properties, serves as an effective matrix for biocomposites (Klyosov, 2007; Alhassan *et al.,* 2021). Maize cob powder (MCP), a lignocellulosic agricultural byproduct, offers an inexpensive and renewable filler with promising reinforcement potential (Bismarck *et al.,* 2018). However, its hydrophilic nature can hinder compatibility with hydrophobic polymer matrices, necessitating surface modifications such as NaOH treatment to enhance interfacial adhesion (Maiti and Bhowmick, 2006; George *et al.,* 2005).

Nanoclay, particularly halloysite nanoclay (HNC) (Figure 2), has attracted significant attention as a nano-reinforcement due to its high aspect ratio, layered structure, and strong interfacial interactions (Biswal *et al.,* 2017).

When incorporated in small quantities, nanoclays can significantly enhance stiffness, thermal resistance, and damping behaviour (Zhang and Wang, 2019; Kumar and Singh, 2020). Dynamic Mechanical Analysis (DMA) provides critical insight into these viscoelastic properties by quantifying storage modulus (E′), loss modulus (E″), damping factor (tan δ), and glass transition temperature (Tg) as functions of temperature and loading.

This work examines the dynamic mechanical performance of rPP composites reinforced with maize cob powder and halloysite nanoclay. The study systematically evaluates the effects of filler loading, particle size, surface treatment, and nanoclay content on the viscoelastic and thermal behaviour of these composites.

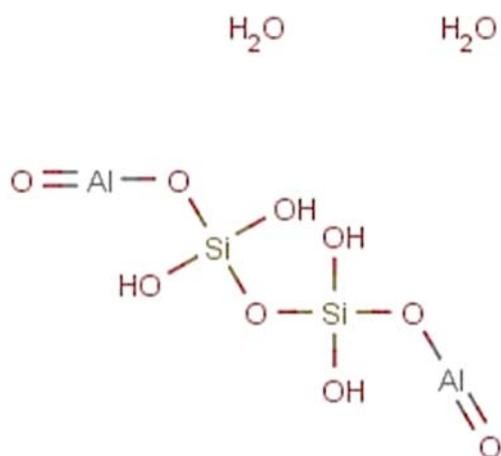Figure. 1: Plastic pollution (Source: Guardian, 2023)

Figure. 2: Halloysite nanoclay (HNC) structure
Chemical cloud.com (2025).

2. METHODOLOGY

Materials and Methods
Composite Preparation
Recycled polypropylene (rPP) was selected as the polymer matrix due to its environmental benefits and thermoplastic versatility. Maize cob powder (MCP), obtained from milled and sieved agricultural waste, was used as a natural filler. The MCP was prepared in five different particle sizes: 100, 200, 300, 400, and 500 μm. To enhance interfacial adhesion between the hydrophilic filler and the hydrophobic polymer matrix, the MCP was divided into two categories: untreated and alkali-treated. The alkali treatment involved soaking the MCP in a sodium hydroxide (NaOH)

solution to remove impurities, waxes, and hemicellulose, thereby improving surface roughness and interfacial bonding potential. The rPP and MCP were compounded using a two-roll mill machine to ensure uniform dispersion of the filler within the polymer matrix.

3.1 Storage Modulus (E)
The storage modulus reflects the stiffness and elastic behaviour of the composites. For untreated maize cob powder (UMCP)/rPP composites, E′ increased with filler loading and decreased with larger particle sizes (Table 1).

At 100 μm and 40 % loading, the E′ reached 150 MPa, compared to 121 MPa for 500 μm fillers. This improvement is attributed to better stress transfer resulting from the enhanced dispersion and interfacial adhesion of smaller fillers (Karmarkar *et al.,* 2007; Bismarck *et al.,* 2018).

The inclusion of nanoclay further enhanced the modulus. For UMCP/rPP + 8 % nanoclay composites, E′ increased to 146 MPa (100 μm, 40 %) and 115 MPa (500 μm, 40 %), compared with 105 MPa for the control (Table 2). The stiffening effect arises from nanoclay's high surface area and layered morphology, which restricts polymer chain motion and enhances load transfer (Biswal *et al.,* 2017; Alhassan *et al.,* 2021). NaOH-treated MCP/rPP + nanoclay composites showed slightly lower E′ at low filler loadings but comparable or higher stiffness at 40 % filler, indicating that chemical treatment improved filler dispersion and bonding at higher loadings (Table 4).
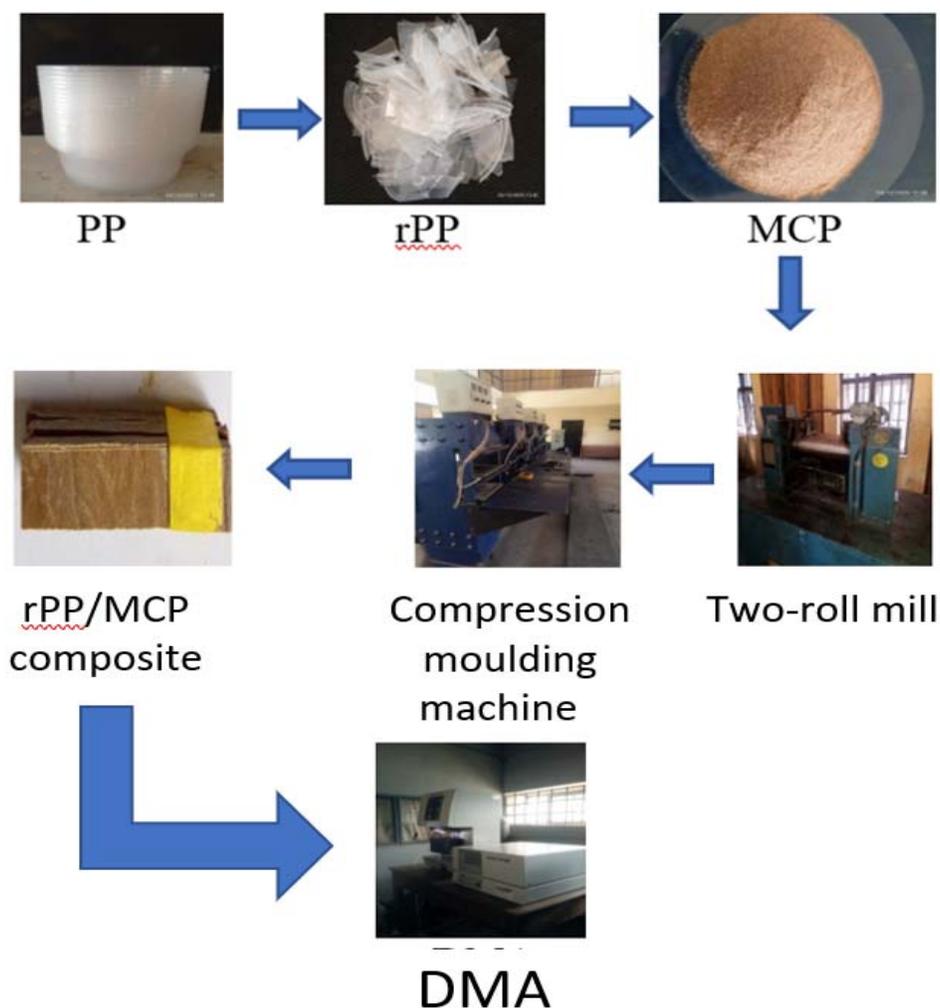
Figure. 3: Experimental steps

3.0 RESULTS AND DISCUSSION

Table 1: E`, E``, Tan ð and T_g (°C) Values of UMC/rPP Composites

S/N.	Filler Size (μm)	Filler Loading (%)	UMCP/RPP			T_g (°C)
			E′ (MPa)	E″ (MPa)	Tan δ	
1	100	10	103	904	0.114	96. 1
2	100	40	150	1027	0.146	121. 0
3	500	10	95	731	0.130	118, 1
4	500	40	121	823	0.147	118.2
Control Sample			105	963	0.109	91.8

Table 2: E`, E``, Tan ð and T_g (°C) Values of UMCP/rPP + NC Composites

S/N.	Filler Size (μm)	Filler Loading (%)	UMCCP/RPP + NC			T_g (°C)
			E′ (MPa)	E″ (MPa)	Tan δ	
1	100	10	114	1000	0.114	100.6
2	100	40	146	993	0.147	118.8
3	500	10	59	541	0.109	97.7
4	500	40	115	767	0.150	120.2
Control Sample			105	963	0.109	91.8

Table 3: E`, E``, Tan ð and T_g (°C) Values of NaOH TMCP/rPP Composites

S/N.	Filler Size (μm)	Filler Loading (%)	NaOH TMCP/RPP			T_g (°C)
			E′ (MPa)	E″ (MPa)	Tan δ	
1	100	10	92	719	0.128	97.7
2	100	40	73	468	0.156	99.3
3	500	10	68	607	0.112	100.5
4	500	40	101	716	0.141	122.0
Control Sample			105	963	0.109	91.8

Table 4: E`, E``, Tan ð and T_g (°C) Values of NaOH TMCCP/rPP + NC Composites

S/N.	Filler Size (μm)	Filler Loading (%)	NaOH TMCCP/RPP + NC			T_g (°C)
			E′ (MPa)	E″ (MPa)	Tan δ	
1	100	10	63	539	0.117	96. 1
2	100	40	65	442	0.147	122.6
3	500	10	62	517	0.120	105.4
4	500	40	88	615	0.143	121.9
Control Sample			105	963	0.109	91.8

3.2 Loss Modulus (E'')

The loss modulus indicates the energy dissipated as heat under cyclic loading. For UMCP/rPP composites, E″ increased with filler loading, from 731 MPa (500 μm, 10 %) to 1027 MPa (100 μm, 40 %), implying improved damping (Table 1) through interfacial friction (Ogunwusi, 2014). Nanoclay addition maintained or slightly enhanced E″, especially at higher loadings (Table 2).

For NaOH-treated MCP composites, E″ ranged between 442 MPa (100 μm, 40 %) and 615 MPa (500 μm, 40 %), consistent with restricted chain mobility due to the combined effects of filler and nanoclay (Table 4). Similar behaviour was reported by George *et al.,* (2005) and Bachtiar *et al.,* (2012), where increased filler content led to reduced polymer relaxation but improved energy dissipation near Tg.

3.3 Damping Factor (Tan *δ)*

Tan δ represents the ratio of viscous to elastic response, reflecting the composite's vibration absorption ability. The control rPP exhibited a tan δ of 0.109. For UMCP/rPP, tan δ values increased with filler loading, reaching 0.147 for 100 μm and 0.150 for 500 μm fillers at 40 % (Table 1). This suggests that higher filler contents enhance damping due to increased internal friction and interfacial motion (Klyosov, 2007; Ayrilmis *et al.,* 2013).

With nanoclay addition, tan δ generally increased at high loadings (up to 0.152 for NaOH TMCP/rPP + NC at 8 % nanoclay (Table 4). NaOH-treated composites exhibited the highest damping behaviour, indicating that surface treatment improved filler-matrix interaction, leading to effective stress transfer and greater viscous behaviour (Rajendran *et al.,* 2021; Nair *et al.,* 2020).

3.4 Glass Transition Temperature (*Tg*)

The Tg defines the transition between glassy and rubbery states of the polymer. The control rPP exhibited a Tg of 91.8 °C. Tg values increased consistently with filler loading and nanoclay addition. For UMCP/rPP composites, Tg increased to 118 °C at 40 % loading (100 μm) and 120 °C (500 μm) as shown in Table 1.

The addition of 8 % nanoclay further raised Tg to 122.6 °C, indicating restricted polymer chain mobility and enhanced thermal stability (Williams, 2017; Kumar and Singh, 2020). Smaller filler particles produced higher Tg due to their larger surface area and better interfacial contact (Jones and Smith, 2018; Zhang *et al.,* 2020). NaOH treatment also contributed to Tg elevation by improving filler wettability and dispersion.

These trends confirm the synergistic effect of nanoclay and chemical modification in enhancing the rigidity and thermal resistance of the composite (Table 4).

3.5 Comparative Performance of Composite Systems

The NaOH-treated MCP/rPP + nanoclay composite demonstrated superior performance overall, showing improved stiffness, damping, and Tg, confirming the beneficial role of surface modification and nanoclay synergy. This is due to the removal of cementing materials from the MCP

further supports the reinforcement and better interfacial interaction between the matrix and the filler. The addition of untreated as well as treated MCP in rPP increased the stiffness of the composites. This behaviour is primarily due to the reinforcing effect of MCP that allowed a greater degree of stress transfer. Comparatively, a higher magnitude of storage modulus (E') was observed in the case of UMCP/Rpp sample as compared with UMCP/rPP + NC, NaOH TMCP/rPP, NaOH TMCP/rPP + NC. This is in agreement with previously reported work by Gajender and Narula (2010).

Table 5: Highest E`, Highest Tan δ and T$_g$ (°C) Values of NaOH TMCCP/rPP + NC Composites

Composite System	Highest E′ (MPa)	Highest Tan δ	Tg (°C)	Key Performance Feature
UMCP/rPP	150	0.147	118	High stiffness and good damping
UMCP/rPP + NC	146	0.150	122	Enhanced thermal stability
NaOH TMCP/rPP	101	0.156	122	Excellent damping and stiffness
NaOH TMCP/rPP + NC	129	0.152	122.6	Best combination of stiffness, damping, and Tg

5.0 CONCLUSION

The incorporation of maize cob powder and halloysite nanoclay markedly improved the dynamic mechanical and thermal behaviour of recycled polypropylene composites. The results demonstrate that increasing maize cob powder content from 10 to 40 % led to a pronounced enhancement in storage modulus and damping characteristics, attributable to strengthened polymer-filler interactions. Filler particle size also played a critical role, as smaller particles (100 µm) yielded higher storage modulus and glass transition temperature values due to superior dispersion and an increased interfacial contact area.

Furthermore, alkali (NaOH) treatment of the filler significantly improved filler–matrix adhesion, thereby enhancing energy dissipation and contributing to elevated tan δ and glass transition temperature values. The inclusion of halloysite nanoclay further reinforced the composites by restricting polymer chain mobility and promoting stronger interfacial bonding, resulting in improved stiffness, damping behaviour, and thermal resistance.

The composite containing 100 µm maize cob powder at 40 % loading, treated with NaOH and reinforced with 8% nanoclay, exhibited the most favourable viscoelastic performance, achieving a glass transition temperature of 122.6 °C and a tan δ value of 0.152, thus highlighting its potential for advanced engineering applications.

REFERENCES

Alhassan, M., et al. (2021). *Polypropylene composites reinforced with agricultural waste: mechanical and thermal behavior. Journal of Polymer Research*, 28(5), 1–12.

Ayrilmis, N., et al. (2013). *Dynamic mechanical properties of wood-polypropylene composites. Composites Part B: Engineering*, 45, 1451–1456.

Bachtiar, D., et al. (2012). *Dynamic mechanical analysis of natural fiber composites. Composites Science and Technology*, 72(13), 1637–1643.

Biswal, M., et al. (2017). *Nanoclay-reinforced PP/PLF composites: Thermal and mechanical characterization. Composites Part A: Applied Science and Manufacturing*, 101, 154–162.

Bismarck, A., et al. (2018). *Natural fiber-reinforced polymer composites.* Woodhead Publishing

Chemical cloud.com (2025). Retrieved on 24/12/2025. Available at httpp/chemicalcloud.com

Gajender, S. and Narula, A. K. (2010). Effect of Particle Size and Alkali Treatment of Sugarcane Bagasse on Thermal, Mechanical and Morphological Properties of PVC-Bagase Compoites. *Journal of Reinforced Plastics and Composites,* Vol. 29. No. 5

George, J., et al. (2005). *Dynamic mechanical behavior of natural fiber composites. Composite Interfaces*, 12(6), 509–522.

Karmarkar, A., et al. (2007). *Mechanical and thermal properties of polypropylene composites reinforced with natural fibers. Composites Part A*, 38(2), 367–374.

Kumar, S., and Singh, R. (2020). *Effect of nanoclay on polymer nanocomposite thermal stability. Polymer Testing*, 86, 106479.

Klyosov, A. (2007). *Wood-Plastic Composites.* John Wiley and Sons.

Maiti, S., and Bhowmick, A. K. (2006). *Dynamic mechanical properties of filled rubber composites. Journal of Applied Polymer Science*, 102(1), 318–326.

Nair, S., et al. (2020). *Role of surface modification in polymer-clay nanocomposites. Materials Today: Proceedings*, 33, 1263–1269.

Ogunwusi, A. A. (2014). *Agricultural residues as fillers in polymer composites. Nigerian Journal of Natural Products and Medicine*, 18, 46–54.

Rajendran, R., et al. (2021). *Dynamic mechanical behavior of nanoclay-reinforced bio-composites. Materials Research Express*, 8(1), 015301.

Williams, J. G. (2017). *Viscoelastic properties of polymer composites. Journal of Materials Science*, 52(9), 5331–5343.

Zhang, J., Chen, Y., and Li, F. (2020). *Influence of filler size on thermal stability of PP composites. Polymer Degradation and Stability*, 179, 109269.

Zhang, X., and Wang, H. (2019). *Thermal and mechanical properties of PP/nanoclay composites. Composites Science and Technology*, 181, 107693.