

PHYSICO-MECHANICAL PROPERTIES OF HOT WATER MODIFIED MILKWEED WOOD (*CALOTROPIS PROCERA*) REINFORCED RECYCLED POLYPROPYLENE COMPOSITES

*¹Okah Kayode Peter, ²P.A.P. Mamza, ²C.E. Gimba, ³Joseph Jacob, ⁴Marietje Lutz

¹Department of Chemistry, Federal University, Gashua, Yobe State, Nigeria

²Department of Chemistry, Ahmadu Bello University, Zaria, Nigeria

³Department of Chemistry, Air Force Institute of Technology (AFIT), Kaduna, Nigeria

⁴Department of Chemistry and Polymer Science, Stellenbosch University, South Africa

*Corresponding Author Email Address: petokason@gmail.com

ABSTRACT

This study reports the use of hot-water-treated Milkweed wood (*Calotropis procera*) particles as reinforcement in recycled polypropylene (rPP) to produce wood-plastic composites (WPCs). The incorporation of *C. procera* filler significantly improved the performance of the composites, particularly in samples containing treated wood particles. The highest tensile strength values were obtained at 20% filler loading for both untreated and treated composites of which their particulate sizes of 75, 150 and 300 μm gave the tensile strength values for the untreated to be 19.19, 18.8 and 17.41 MPa respectively and the treated values to be 26.62, 22.75 and 21.41 MPa respectively as well, an average increased value of 27% for each particle size. The flexural strength follows the same trend as tensile strength, with the optimum value occurring at 20% weight filler loading at 75 μm . The flexural strength obtained for 75, 150, and 300 μm for untreated composites was 20.15, 19.41, and 17.24 MPa, respectively, while the corresponding treated composites were 24.89, 21.85, and 20.15 MPa. Other mechanical properties increased with an increase in filler loading, while elongation at break decreased with increasing filler loading for both treated and untreated composites. Water absorption investigation revealed that treated wood/rPP composites were more resistant to water intake than the untreated milkweed wood/rPP composites. The water absorption (%) at 20% wt filler loading for each particulate size of 75, 150, 300 μm was: 8.31, 9.52, and 12.4% (untreated) respectively, and the corresponding treated wood/matrix were: 1.17, 1.45, 1.65% after 32 days immersion in water. The hot water treatment of milkweed wood enhanced the mechanical properties and water absorption resistance of milkwood/rPP composites. This study shows that hot water treatment is an eco-friendly, low-cost cost and sustainable pretreatment method that can significantly enhance the performance of wood-plastic composites, especially for outside door services without the extensive need for chemical modification

Keywords: *Calotropis procera*, Recycled Polypropylene, Wood-Plastic Composites, Hot water Treatment, Water Absorption.

INTRODUCTION

Natural fibre-reinforced polymer composites have drawn increasing attention in recent years owing to their low density, renewability, biodegradability, and favorable mechanical properties relative to cost and environmental impact (Jiuping *et al.*, 2018). As industries are seeking more environmentally sustainable alternatives to synthetic fibre reinforced materials, composites

using agro-waste, non-wood fibres, and under-utilized plant sources are becoming promising candidates (Pokhrel *et al.*, 2021). However, realizing their full potential depends critically on issues such as filler content, particle/fibre size, surface treatment, matrix compatibility, and moisture sensitivity. *Calotropis procera* is a small perennial tree of the *Apocynaceae* family, and it is native to Africa, the Arabian Peninsula, Western Asia, and India (Mazaherifar *et al.*, 2022). This tree was introduced to Australia, Latin America, and the United States because of its economic benefits (Lottermoser, 2011; Hassan *et al.*, 2015; Kaur *et al.*, 2021; Al-Rowaili *et al.*, 2020). It is commonly known as the giant apple of Sodom, or calotrope (Mazaherifar *et al.*, 2022), grows slowly and is drought-resistant and evergreen with softwood, having thick branches that may grow up to 6m (Kaur *et al.*, 2021). *Calotropis* species (e.g. *Calotropis procera*, *Calotropis gigantea*) have been investigated for use in natural fibre composites. Velusamy *et al.* (2018) studied *Calotropis gigantea* fibre-reinforced epoxy composites and showed that increasing fibre content and length led to increased water absorption and reductions in mechanical strength under wet conditions. Characterization work has further shown that fibre loadings above certain thresholds (e.g. 40% wt) can result in a decline in tensile, flexural, and impact strength, likely due to fibre agglomeration and weak interfacial bonding. *Calotropis procera* is an abundant, under-utilized plant in Nigeria, most especially in the northern part of the country, whose fibres and woody components have attracted growing attention as low-cost fillers and reinforcements for polymer composites. Its morphological and chemical characteristics make it a promising candidate for valorization in polymer matrices, but published work also shows high sensitivity of mechanical performance to water uptake, fibre length/particle size, and processing conditions. Ramesh *et al.* (2021) also studied the fabrication by compression molding of laminates/composite specimens using *Calotropis* fibres (randomly oriented) in polymer resin (epoxy). The tensile, flexural, impact, and water absorption tests were investigated; they reported a high percentage range of water absorption and showed that water uptake reduces mechanical performance.

Thermal and hydrothermal modifications (including hot-water or steam treatments) alter the chemical composition of lignocellulosic materials principally by reducing hygroscopic hemicelluloses and modifying lignin and accessible hydroxyl groups (Hosseinaei *et al.*, 2012; Pelaez-Samaniego *et al.*, 2013; Chang *et al.*, 2015), which commonly reduces water uptake and can improve dimensional stability and interfacial compatibility with nonpolar polyolefin matrices. Consequently, heat/hot water treated (HWT) wood

fractions often produce wood-polymer composites (WPCs) with improved weathering resistance and reduced moisture-induced deterioration versus untreated wood, while also changing stiffness and strength trends in non-trivial ways. Despite the expanding literature on *Calotropis* fibres and on heat-treated wood in WPCs, a systematic study that simultaneously evaluates hot water treatment of Milkweed wood filler (versus untreated filler), a wide range of filler loadings, distinct particle size fractions, and a recycled polypropylene (rPP) matrix (with a practical compatibilizer content) is still lacking. Such an integrated approach is essential to identify the practical optimum combinations that maximize strength and hardness while preserving acceptable toughness and ductility for real-world applications. Accordingly, this work investigates the physico-mechanical response (tensile and flexural strength and modulus, impact resistance, elongation at break, and hardness) of recycled polypropylene composites reinforced with hot water-treated and untreated wood (*Calotropis procera*) fillers across controlled particle size fractions and filler loadings. The study seeks to quantify how hot-water treatment influences water absorption behavior and mechanical performance, to clarify the interactions between particle size and filler loading that control strength and hardness, and to determine the optimum combination of filler loading and particle size under practical compatibilization conditions for structural and semi-structural applications. The findings will give both fundamental understanding and pragmatic material design for circular-economy (reuse, repair, and recycle) polymer composites.

MATERIALS AND METHODS

The recycled Polypropylene (rPP) used in this study was PP black, Batch No: 05PP011, supplied in May 2014, by MYPLAS (Pty) Ltd, Stellenbosch, South Africa. The tree was harvested and pruned as logs from Gashua Forest in Yobe State, Nigeria. The logs were milled into chips and pulverized into a small particle size. This wood particle was subjected to hot water treatment (HWT) at 140 °C temperature and pressure of 2.5 bar for 2 hours. The wood-to-water ratio in a stainless-steel cylindrical reactor was 1:4 in a closed system. After 2-hour duration, the heating was stopped, allowed to cool down and the content was filtered and oven dried at 70 °C for 24 hours.

Samples Preparation

The untreated and treated woods were further milled, using Jaw crusher and ball milling machines (Retsch Masch, Nr 70992 GMbH & CO. and Kera b.v. Soeter berg Overveld 057748 Holland) respectively, then sieved using impact Laboratory sieve (ISO 3310-1:2000, bs 410-2000) to obtain 75, 150, and 300µm particle sizes as fillers, weighed according to the planned percentage weight (10, 20, 30, 40 and 50%) and the corresponding complementary percentage weight of rPP polymer as shown in Table 1 were weighed and bagged respectively. Subsequently, the amount of maleic anhydride grafted polypropylene (MAPP) was equally weighed and bagged and kept as well for the next stage (extrusion process).

Table 1: rPP and Wood Formulations

| S/N | Composite Sample code | Plastic forms (%) | Plastic content (%) | Wood flour content (%) | Coupling agent (%) |
|-----|-----------------------|-------------------|---------------------|------------------------|--------------------|
| 1 | rPP100 | recycle d | 100 | 0 | 0 |
| 2 | rPP85utW 10 | recycle d | 85 | 10 | 5 |
| 3 | rPP75utW 20 | recycle d | 75 | 20 | 5 |
| 4 | rPP65utW 30 | recycle d | 65 | 30 | 5 |
| 5 | rPP55utW 40 | recycle d | 55 | 40 | 5 |
| 6 | rPP55utW 50 | recycle d | 45 | 50 | 5 |

Where, r = Recycled, PP = Polypropylene, u = Untreated, t = Treated, W = Wood

Compounding and Extrusion

The recycled polypropylene, treated and untreated *Calotropis procera* wood of 75, 150, and 300µm particles, respectively, were mixed and compounded using a customized single screw extruder machine. The extruder machine is made up of a single screw length of 55cm and a diameter of 2.5 cm, i.e., the length to diameter ratio was 22:1 with a rotational speed of 100rpm. The Screw has a conical tip towards the die with reducing flight depth. The machine has two heating zones that can be controlled separately. The extruding temperatures used were 180 °C and 160 °C for the compounding. The polymer, the wood sample, and MAPP were fed into the Hooper; the materials were simultaneously transported to the heating zone (180 °C at the rear end and 160 °C at the front) where the polymer was melted and mixed with wood content and later forced through the die to form pellets of WPC. The collected pellets from the machine die were reintroduced into the Hooper for proper mixing, and the process was repeated 3 times to obtain the final homogenous composite pellets. The same procedure was repeated for all the weighed samples.

Compression Molding

The extruded pellets of wood/rPP at particle sizes 75, 150, and 300µm were taken to a two-roll mill machine according to ASTM 15-672 by introducing the extruded mixture into the heated rollers at 180 °C, where it melts and flows after mixing for 5 minutes. The mixed material was collected and subsequently placed in a metal mold 150 x 100 x 5 (mm) length, width, and thickness, respectively, and then pressed on a compression molding machine at 180 °C for five (5) minutes. Thereafter, cold-pressed at room temperature for three (3) minutes and a pressure of 4Pa. the composites were removed from the machine and kept for future analysis. The pressing was carried out in the polymer processing laboratory at the Department of Polymer Technology (Nigerian Institute of Leather and Technology, Zaria). Composite Sheets were thereafter cut into various dimensions for characterization.

FTIR Analysis

The Fourier transform infrared (FTIR) spectroscopic analysis of the *Calotropis procera* wood particles was studied using FTIR Thermo Scientific Nicolet (iS10). Both untreated and treated samples were

subjected to this test in order to investigate the effect of hot water treatment on peak intensity and some functional groups of the *Calotropis procera*'s wood structure.

Proximate Analysis

The treated and untreated wood samples of *Calotropis procera* were taken to the National Animal Production Research Institute (NAPRI) laboratory, Shika, Zaria, where the quantitative and qualitative analysis of the wood was conducted and recorded as shown in Table 2.

Water Absorption Test

Water absorption tests were conducted in accordance with ASTM D570-98, in which each of the specimens was weighed, recorded, and immersed in water in a container for 24 hours at a temperature of 23 ± 2 °C. The sample was removed, the surface water was wiped off using dry cloth, and weighed again to determine the weight gain by the samples using a weighing balance machine. The sample was returned to the water container, and these procedures were repeated for 32 days. Percentage water absorption was calculated and recorded using this formula in equation (1).

$$\text{Water absorption (\%)} = \frac{(W_f - W_i)}{W_i} \times 100 \quad (1)$$

Where W_i = Initial weight and W_f = final weight of the specimen.

Tensile Strength

Tensile properties of each composite were determined according to ASTM D 638 standard with a gauge length of 40mm and load force of 100KN. The sample's dimensions of $100 \times 10 \times 5$ mm were tested using a Tensile Strength Testing machine with a crosshead speed of 2mm/min, Model TM2101-T7, Manufactured by Shanghai Rixin Electronic co, Ltd, Shanghai Rixin Technology, China. The reading was automatically recorded and calculated by the instrument's software. At least 3 specimens were tested for each composite blend and property. Tensile properties obtained from this test were tensile strength, elongation at break, and tensile modulus.

Flexural Strength

Three-point bending test was performed in accordance with ASTM D 790 standard to measure the flexural properties of the composites using Tensiometer: Lonroy (LC008). A maximum load of 100KN and cross-head speed of 5 mm/min were applied, and loading continued until fracture, and values of flexural strengths were recorded automatically. The dimension of the sample was $100 \times 30 \times 3$ mm in length, width, and thickness, respectively. The tests were conducted by positioning the specimens horizontally in the sample compartment of the machine, with at least 3 specimens tested for each composite blend.

Impact Test

The Impact energy test was conducted with a Charpy Impact Testing Machine, Cat. No. 412 (Model No. 6957), capacity of 15 Joules) According to the ASTM-E23 standard, a notch depth of 0.5 mm and a notch base radius of 0.25 mm. Samples were tested at room temperature by a single swing of the pendulum hammer using Norwood. The specimen size was $100 \times 11 \times 5$ (mm). Each sample was placed on the vice and clamped firmly. The pendulum hammer was raised to the required height and then released and strike the sample at once. Then, the impact energy absorbed by the specimen was recorded. The impact energy of the samples in

Joules per square meter was recorded with 3 specimens conducted for each sample blend of the composites.

Hardness Test

The hardness test was conducted using (Microvickers Hardness Tester MV1-PC, Mh-v CM. Seria No: 07/2012-1329) with a maximum capacity of 0.3 Kgf (150 HV) in accordance with ASTM E-384. The test was carried out at a temperature (23 ± 2 °C). The specimen dimension was $30 \times 10 \times 3$ (mm). The specimen was mounted on a specimen compartment, and the indentation point was focused. thereafter three different points were indented on each specimen, and the hardness values were recorded. The average of the three readings was calculated and recorded.

RESULTS AND DISCUSSION

Characterization of *Calotropis procera*

FTIR Analysis of the untreated and treated Milkweed wood particles was evaluated, and the effect of chemical modification on the Milkweed was observed using FT-IR Spectroscopy and proximate analysis. The comparison of the FT-IR spectra of the untreated and the treated Milkweed wood in Figure 1 shows a reduction in O-H stretching intensity. The shifting of the peaks 3315 cm^{-1} of untreated to peak 3304 cm^{-1} in the treated Milkweed wood was observed. This may be attributed to the disruption of the hydrogen bond between the O-H groups of cellulose and hemicellulose and the reduction of the OH group associated with some of the removed hemicellulose present in the wood structural particles (Lawal *et al.*, 2023; Gumel and Tijjani, 2015). Peaks at 2922 cm^{-1} for the untreated and 2917 cm^{-1} for treated Milkweed wood spectra may have emanated from C-H stretching of the aliphatic group, and the reduction in their stretching intensity indicates the removal of hemicellulose (Jayamani *et al.*, 2020). The absorption peak at 1732 cm^{-1} in the untreated wood was observed to have reduced to 1727 cm^{-1} in the treated spectra was associated with carbonyl C=O stretching of acetyl and attributed to the asymmetric deformation of lignin in the treated wood. The band at 1244 cm^{-1} is attributed to the stretching vibration of C-O groups present in lignin and hemicellulose structure. Weakening of this peak, 1240 cm^{-1} in the treated spectra, confirmed the removal of hemicellulose (Jin *et al.*, 2022). This observed peak decreased as a result of hemicellulose removal, had typically supported the reduction observed in the proximate analysis result shown in Table 2, where the reduction of hemicellulose was found to be from 10.72% in the untreated to 8.62% in the treated wood. However, peaks 1646 cm^{-1} and 1642 cm^{-1} in the untreated and treated Milkweed wood, respectively, were attributed to the C=C peak, which also showed an intensity reduction. Tjeerrdsma and Miltitz (2005) reported that it is possible to hydrolyze hemicellulose preferentially in a mild heat treatment of wood in an acid medium because hemicellulose is hydrolyzed more easily than cellulose. They added that the course of acidity is attributed to the formation of carbonic acid in the thermolysis process and is a function of time and temperature. The carbonic acid formed catalysis the depolymerization of hemicellulose (Kubovsky *et al.*, 2020). This process of using HWT to remove hemicellulose often reduces the hygroscopicity but improves durability, dimensional stability and resistance to biological degradation of the treated woods (Zhang *et al.*, 2017).

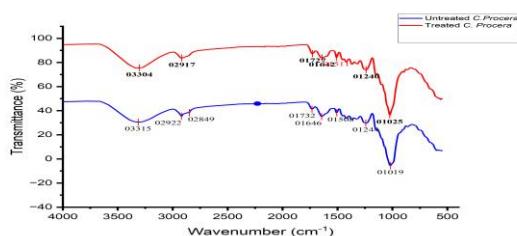


Figure 1: Fourier Transform Infrared Spectroscopy (FTIR) Spectra of untreated and treated *Calotropis procera*.

Proximate Analysis

Table 2 presents the chemical composition of *Calotropis procera* wood, showing the percentage distribution of lignin, hemicellulose, and cellulose before and after hot water treatment. Prior to treatment, lignin, hemicellulose, and cellulose accounted for 17.11%, 21.02%, and 31.47% of the wood composition, respectively. After the hot water treatment, their proportions changed to 23.44%, 8.04%, and 43.11%, respectively. The reduction in the hemicellulose content of *Calotropis procera* indicates that the treatment significantly removed this component. The observed decline from 21.02% to 8.04% suggests that the hot water extraction effectively degraded or dissolved part of the hemicellulose fraction. This selective removal likely enhanced the relative proportion of lignin and cellulose in the treated samples. The trend is consistent with the findings of Palaez-Samaniego *et al.*, (2013), who observed that the lignin content in the bark of pine wood increased after hot water extraction due to the elimination of hemicellulose. Hot water treatment at elevated temperatures (120–

140 °C) promotes the partial depolymerization of hemicellulose while leaving lignin largely unaffected (Teng-chun *et al.*, 2017). The reduction in hemicellulose consequently increases the relative lignin content (as shown in Table 2), thereby improving the hydrophobicity of the treated wood. Since lignin contains aromatic rings with nonpolar characteristics, its enrichment enhances the material's resistance to moisture uptake and improves the overall compatibility between the wood filler and the polymer matrix. These structural modifications result in improved water resistance, higher physical integrity, and better thermal stability of the treated *Calotropis procera*/rPP composites compared to the untreated ones. Arwinfar *et al.* (2016) also reported similar findings with beech wood that was thermally treated using steam at varying temperatures (120–180 °C) for 30–120 minutes and later employed as filler in polypropylene composites with a coupling agent. Their study concluded that thermal treatment induced structural rearrangements in the fibre, creating a thin layer of amorphous lignin that facilitated better bonding between the fibre and polymer. This modification, characterized by fewer fibre ends and reduced voids, promoted stronger interfacial adhesion and enhanced mechanical properties. Treatments at approximately 150 °C for 30 minutes were found to yield optimal compatibility between the wood fibres and polymer matrix. Consequently, such heat or hot water treatments that modify or mobilize lignin (making it more amorphous) improve fibre–matrix interaction, reduce surface defects, and enhance the strength and durability of wood–plastic composites.

Table 2: Proximate analysis results for Treated and Untreated Milkweed Wood

| S/N | Milkweed Type | Description | | | | | | | | | |
|-----|---------------|-------------|------|------|-------|-------|-------|-------|-------|---------------|---------------|
| | | %DM | %ASH | %EE | %CF | %CP | %NDF | %ADF | %LIGN | %HEMI CELL | %CELU LOSE |
| 1 | Untreated | 90.72 | 2.43 | 8.43 | 31.25 | 13.25 | 69.6 | 48.58 | 17.11 | 21.02 | 31.47 |
| 2 | Treated | 91.51 | 0.29 | 5.02 | 67.49 | 6 | 74.59 | 66.55 | 23.44 | 8.04 | 43.11 |

Water Absorption

Figure 2 revealed the water absorption behavior of the treated Milkweed/rPP composites. It was observed that the percentage of water intake increased with increasing particle size and filler loading. This logical behavior is not unexpected since, generally, lignocellulose is hygroscopic; higher content dictates a greater volume of water interaction, hence a higher percentage of water absorption capacity. For instance, the water absorption of the Milkweed/rPP composites at 10, 20, 30, 40, and 50% filler loading for 75 µm particle size is 1.04, 0.94, 2.57, 5.14, and 7.01%, respectively. This result and study are supported by earlier research, such as that of Kraiem *et al.* (2020). Their study, which was based on the effect of wood particle size on water absorption of wood–plastic composites, reported that composites made with recycled HDPE and higher wood sawdust percentages showed increased water absorption within the immersion period. They also noted that particle size had a similar effect, as increasing particle size also increased water absorption. The increased water

absorption with increasing particle size can be attributed to the increased porosity associated with higher irregularities of larger particles compared to smaller ones. Figure 3 illustrates the effect of hot water treatment on the water absorption of the Milkweed/PP composites. For clarity, the water absorption for composites with 75 µm particle size at 20% filler loading for both treated and untreated samples has been juxtaposed on the graph. The treated samples showed higher resistance to water uptake compared to untreated samples with the same Milkweed/rPP composite composition. This observation can be attributed to the depletion of hemicellulose during the hot water treatment process, which is also evident in Table 1, where the hemicellulose content was reduced from 21.02% (untreated) to 8.04% (treated) in the wood structure. This treatment reduced the accessible -OH moieties responsible for retaining water molecules by infiltration as a result of hydrogen bond formation between the wood's structural -OH groups and water molecules, which consequently led to lower water intake (Pelaez-Samaniego *et al.*, 2013).

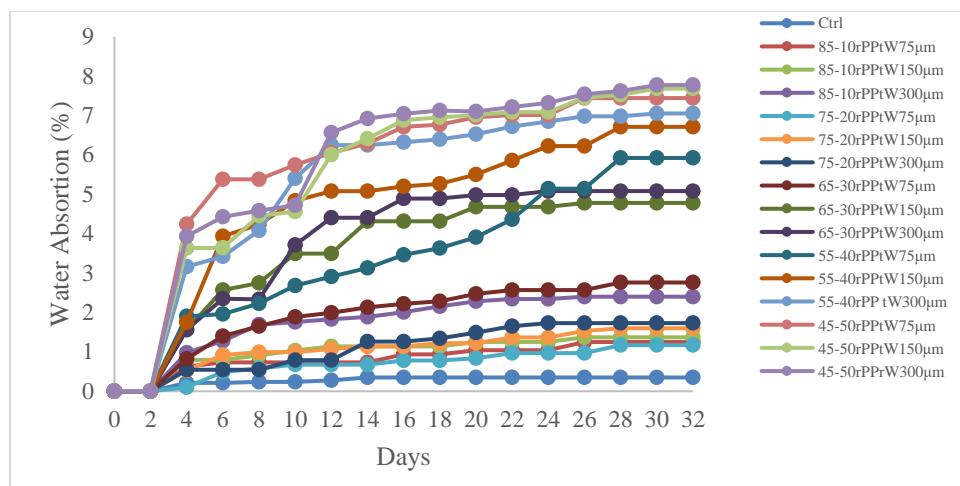


Figure 2: Water Absorption on treated Milkweed/rPP Composites

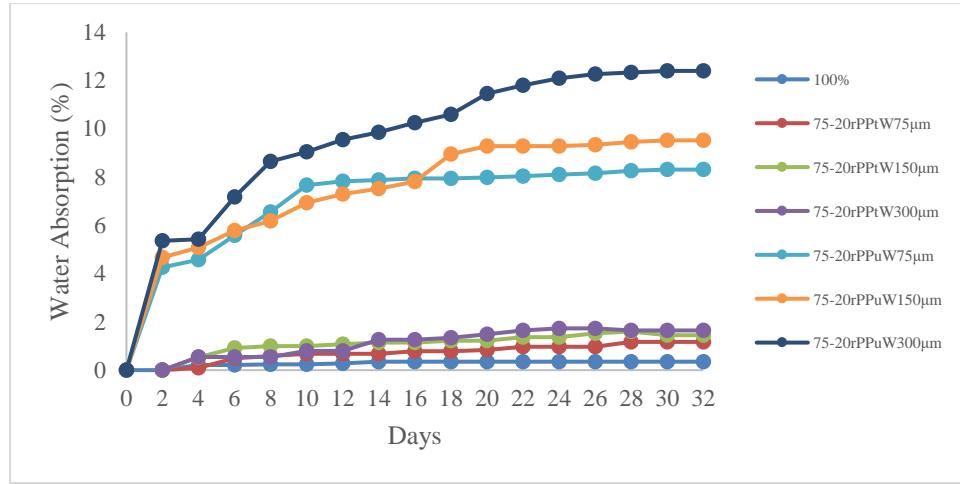


Figure 3: Water Absorption on treated and untreated at 20% filler loading For Milkweed/rPP composites

Tensile Strength

Figure 4 illustrates the effect of hot water treatment (HWT) of the Milkweed wood filler (140 °C, 2.5 bar) on the tensile strength of recycled polypropylene (rPP) composites. Composites made using HWT-fibres consistently exhibited higher tensile strength values than those made with untreated wood particles. This improvement may be attributed primarily to the reduction of hydroxyl (–OH) groups caused by hemicellulose removal during the hot-water treatment, which in turn increases the hydrophobic nature of the wood and improves interfacial bonding between the wood particles and the polymer matrix (Pelaez-Samaniego *et al.*, 2013). As the data show, composites with filler loading exhibited higher tensile strength than the control (0 % filler) up to a loading of 20 wt%, but for filler loadings above 20 wt% (30, 40, and 50 wt%) the tensile strength decreased for both treated and untreated wood-composite systems. Specifically, at 20% wt filler loading, the tensile strength values were 17.46 MPa for the untreated composite and 21.41 MPa for the treated, corresponding to a 23 % increase due to hot water treatment. At 50 wt% filler loading, the treated composite reached 13.09 MPa, whereas the untreated composite registered 9.06 MPa, corresponding to a 44.5 % increase. The

rising trend in tensile strength up to 20 wt% filler can be attributed to effective stress transfer between the well-dispersed wood particles and the rPP matrix, made possible by improved fibre–matrix compatibility following HWT. Beyond 20 wt% loading, however, the decline in tensile strength may be attributed to several factors: the onset of filler particle agglomeration, poor filler–matrix wetting, increased stress concentrations, and possible matrix discontinuities, all of which reduce effective stress transfer and promote premature failure. These mechanisms are consistent with prior observations in natural-fibre polymer composite literature (Kamarudin *et al.*, 2022; Islam *et al.*, 2024). Importantly, the incremental benefit of the treatment increases with filler content, while at 10 wt% filler, the tensile-strength increment was 14.4 %, at 50% wt, it reached 44.5 %. This suggests that the treatment becomes increasingly beneficial as the number of filler–matrix interfaces increases, where improved interfacial adhesion effectively mitigates the otherwise deleterious effects of higher filler loading. Furthermore, the decline in tensile strength at high filler contents (above 20 wt%) emphasizes the need to optimize filler loading in such composites. The optimum loading in this case (20 wt%) appears to represent a balance between filler

reinforcement and matrix continuity. This behaviour aligns with broader findings in wood-plastic composites and other natural-fibre reinforcements: beyond a certain loading threshold, mechanical performance inevitably declines unless filler dispersion and interfacial bonding are carefully managed (Pelaez-Samaniego et al., 2013; Khoaele et al., 2023).

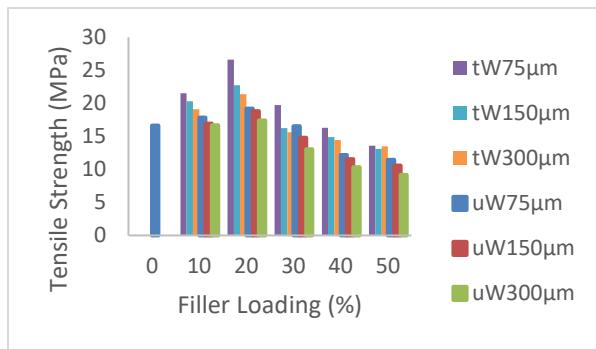


Figure 4: Effect of filler loading and particle size on tensile strength for treated and untreated composites.

Tensile modulus

Figure 5 illustrates the effect of HWT on tensile modulus; the values obtained from HWT samples were observed to be higher than the untreated sample composites, and all the composite samples with filler have higher values of tensile modulus than the control (0% filler) composite sample. Generally, tensile modulus was found to increase with an increase in filler loading and increase in particle size. In the treated sample, the highest tensile modulus obtained was 4.98 GPa at 50 wt% filler loading for 75μm, while the lowest was 1.9 GPa at 10 wt% for 300μm. The corresponding highest tensile modulus obtained for the untreated composite was 3.74 GPa, and the lowest was 1.8 GPa at the same filler loading. It is clear that the treated values are higher than the untreated composites owing to better interfacial bond between Milkweed wood and rPP matrix as a result of hemicellulose and wax contents removal, which accompanied HWT. Tensile modulus is an indication of the stiffness of a material; the incorporation of fillers into the polymer matrix increases the stiffness of the composites. This could be attributed to the even dispersion of the filler in the matrix, which efficiently hinders chain movement during deformation. On the other hand, smaller particles enhance better dispersion within the matrix than larger particles, hence the decreased tensile modulus with an increase in particle size. Similar behavior has once been reported by Onuegbu and Igwe (2011). However, Njoku et al. (2011) reported that periwinkle particle size within the range of 400 – 1000 μm has a negligible effect on the tensile modulus of Polyester-periwinkle shell particle composites in their study on the effects of variation of particle size and weight fraction on the tensile strength and modulus of periwinkle shell reinforced polyester composite. In this study, a larger particle size was observed to enhance tensile modulus; contrary to the aforementioned scholars, this effect may be attributed to improved stress distribution within the material, as larger particles generally increase the stiffness of materials. This is in line with the study made by Edoziuno et al. (2025), who reported the study of Mechanical and structural performance of hardwood charcoal-reinforced polyester composites, effects of particle size and filler

loading. In their findings, they showed that larger particle sizes of charcoal filler gave tensile modulus of 906.7 MPa for the 250 μm filler at 10 wt% while 651.6 MPa was obtained for the 150 μm filler at 20 wt%. They concluded that tensile modulus generally increases with larger filler sizes, although there may be some fluctuations.

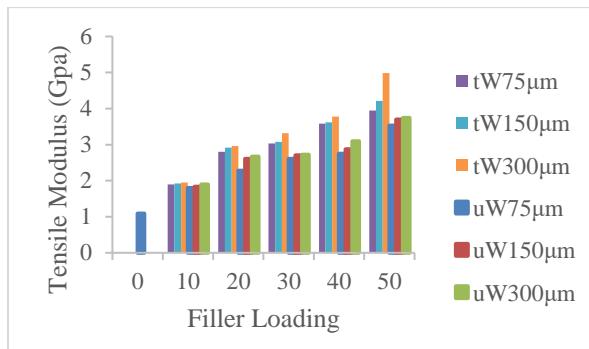


Figure 5: Effect of filler loading and particle size on tensile modulus for treated and untreated composites.

Flexural Strength

As illustrated in Figure 5, the flexural strength exhibited a pattern similar to the tensile strength trend. Treated composites generally demonstrated higher flexural strength than the untreated composites. The flexural strength increased progressively with filler loading up to about 20 wt%, after which a gradual decline was observed at higher filler contents (30–50 wt%). This behavior indicates that a moderate filler concentration enhances stress transfer between the matrix and the filler, thereby improving the composite's bending resistance. However, excessive filler loading resulted in reduced strength, likely due to particle agglomeration and poor interfacial adhesion between the Milkweed filler and the polypropylene matrix. Comparable findings have been reported by Onuoha et al. (2017), who noted that improved particle dispersion and wettability at lower filler levels promote stronger interfacial bonding and efficient stress transfer during flexural loading. In contrast, higher filler content tends to cause non-uniform distribution and weak bonding zones, leading to premature failure under bending stress. The effect of particle size was also significant. Composites containing smaller particle sizes (75 μm) recorded higher flexural strength compared to those reinforced with larger particles (300 μm). The smaller particles provided greater surface area for adhesion and better interaction with the matrix, minimizing void formation and stress concentration points. Conversely, the larger particles created micro-gaps and interfacial discontinuities that weakened the overall structural integrity of the composite. A related observation was made by Lawal et al., (2023), who found that date seed particle-reinforced polypropylene composites with smaller filler sizes exhibited superior flexural strength. They attributed this to enhanced filler dispersion and more effective filler–matrix interfacial bonding, which facilitated improved load transfer within the composite.

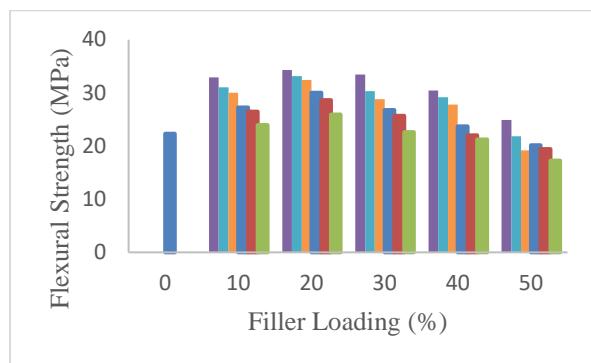


Figure 6: Effect of filler loading and Particle size on Flexural Strength for treated and untreated composites

Elongation at Break

Figure 6 illustrates that the neat recycled polypropylene (100 % rPP) exhibited the highest elongation at break (15.21), indicating the greatest ductility. This is followed by the untreated *Calotropis procera* (milkweed)/rPP composites, which recorded elongation values of 12, 11.14, and 10 for wood particle sizes of 75, 150, and 300 μm , respectively. The hot-water-treated composites showed slightly lower values of 9.41, 8.78, and 8.75 for the same particle sizes. The reduction in elongation after filler addition suggests a restriction in the polymer chain mobility caused by wood particle–matrix interactions. The further decline observed in treated samples can be associated with the partial removal of hemicellulose and other amorphous constituents during hot water treatment, which increases fibre crystallinity and stiffness, thereby reducing flexibility. The treatment also improves the surface roughness of the fibres, enhancing adhesion with the recycled polypropylene matrix and thus facilitating better stress transfer. However, the improved interfacial bonding often leads to reduced deformability before fracture. Similar findings were reported by Chen *et al.* (2024), who noted that thermal or hot water treatment of lignocellulosic fibres typically results in decreased elongation due to increased rigidity and reduced molecular motion. In this study, a general downward trend in elongation at break was observed with increasing filler loading and particle size for both treated and untreated samples, as presented in Figure 6. This behavior can be attributed to the restricted movement of polymer chains and the formation of micro-voids and weak interfacial zones at higher filler content. When the filler content is excessive, or the particle size is large, stress concentration sites develop around the fibre–matrix boundaries, promoting crack initiation and propagation, which further lowers elongation. Comparable reductions in elongation with filler loading and particle size have also been observed by Lawal *et al.* (2023), who attributed the effect to reduced chain mobility and the formation of stiffer composite structures.

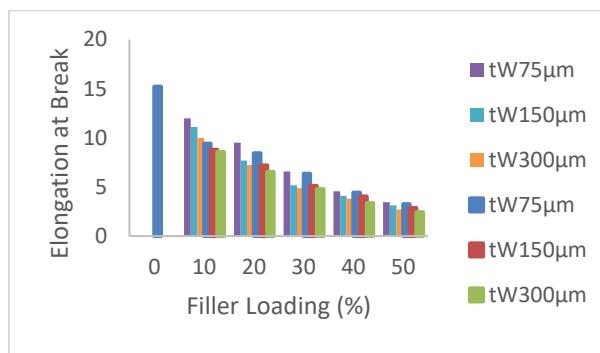


Figure 7: Effect of filler loading and Particle Size on Elongation at Break for treated and untreated composites

Impact Energy

The impact behavior of fibre-reinforced composites reflects their capacity to absorb energy during sudden loading or shock. Generally, incorporating wood particles into a polymer matrix enhances impact resistance due to improved energy dissipation at the filler–matrix interface. However, the extent of improvement is largely influenced by the surface condition of the particles and the quality of bonding with the matrix. In this study, the impact strength of polypropylene composites reinforced with untreated and hot-water-treated milkweed fibres was evaluated. Figure 7 shows that the hot-water-treated milkweed wood composites consistently exhibited higher impact strength values than the untreated fibre composites across all fibre loadings. This improvement can be linked to the removal of surface impurities (such as waxes and pectin) during the hot-water treatment at 140 °C and 2.5 bar, which exposed cellulose fibrils and enhanced fibre roughness. The resulting fibre surface modification promoted better mechanical interlocking with the polypropylene matrix, hence improving impact energy absorption. The slight reduction in impact strength observed at higher particle loadings may be attributed to particle agglomeration and reduced matrix mobility, which disrupts stress transfer during impact. This behavior aligns with findings reported by Lawal *et al.* (2019), who observed enhanced toughness in Date seed particle-reinforced thermoplastic composites following sodium hydroxide (NaOH) surface treatment, which was due to improved interfacial bonding. Overall, the results confirm that hot-water treatment is beneficial for boosting the impact performance of milkweed-reinforced composites. Improved particle–matrix bonding, which facilitates effective stress transfer and increases the composite's ability to withstand sudden impact loads. Thus, incorporating treated milkweed fibres into polypropylene presents a promising route toward lightweight composite materials with strengthened impact resistance for packaging, automotive, and eco-product applications. The impact energy is the measurement of the toughness of a material, i.e. determination of resistance to fracture of a material that is subjected to a sudden and intense blow or mechanical shock. As depicted in Figure 7, impact energy decreases with increased filler loading and particle size. This could be attributed to the irregular orientation of large particles, which resulted in less interaction within the composite's composition. This observed decrease may also be a result of large wood particles and agglomeration of excessive filler that are oriented in the form of layers, which are incapable of supporting stress transmission from the polymer matrix to the filler. This observation was also reported by Lawal *et al.* (2023). The hot water-treated wood

composite samples possessed higher impact energy values than the untreated wood composite samples. The impact energy range values obtained for untreated composites were: 0.75-0.4, 0.72-0.36, and 0.68-0.35 J/m², and for treated composites, the range was: 0.65-0.34, 0.65-0.38, and 0.57-0.3 J/m² at 75, 150, and 300 µm, respectively. This is because the hot water treated wood particles were more compatible with the hydrophobic nature of the polymer matrix, HWT may have depleted some OH-moiety along with hemicellulose removal that tends to improve the interfacial bond between the filler/matrix, hence increased the values of the HWT samples over the untreated samples as also observed in other tests (Tensile, flexural and hardness).

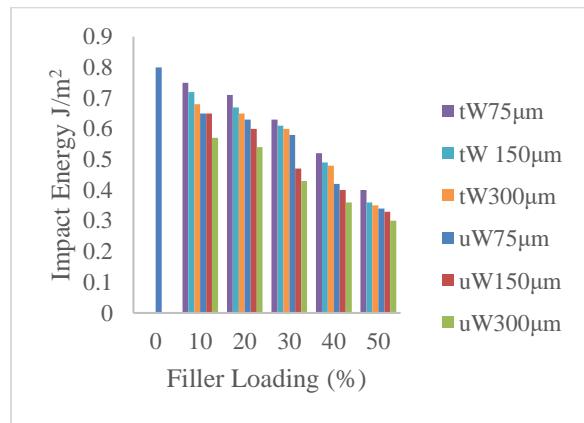


Figure 8: Effect of filler loading and Particle Size on Impact Energy for treated and untreated composites

Hardness Properties

Figure 8 illustrates the hardness characteristics of the fabricated composites. In general, the hot-water-treated (HWT) milkweed wood particulate composites exhibited higher hardness values than the untreated milkweed wood–wood-polypropylene composites. The improvement in hardness for the treated samples is attributed to enhanced compatibility between the Milkweed particulates and the polypropylene matrix, which promotes stronger interfacial bonding. The Vickers hardness (HV) of the control sample (100% rPP) was 22.8 HV. For the untreated milkweed wood particulate-reinforced rPP composites with particle sizes of 75, 150, and 300 µm, the hardness values ranged from 30.52–60.29, 30.52–71.77, and 37.54–76.69 HV, respectively. In comparison, the HWT milkweed particulate composites recorded hardness values of 56.4–79.3 HV, 60.3–84.73, and 63.63–79.46 HV, for the same particle sizes. The results show that increasing filler content led to a corresponding rise in hardness, and a similar increasing trend was observed with larger particle sizes. The maximum hardness value (79.46 HV) occurred at 300 µm particle size and 50% filler loading for the treated composite, while the lowest value (23.21 HV) was recorded in the untreated composite containing 10% filler at 75 µm particle size. The improvement in hardness with higher particulate loading indicates the reinforcing effect of the milkweed wood particles, which reduce polymer chain mobility and increase stiffness (Mohd *et al.*, 2018). Additionally, larger particle sizes are more capable of bearing surface loads due to their tendency to distribute near the surface of the composite, thereby contributing to higher hardness values. Similar findings regarding the influence of particle size on composite hardness have been reported by Lawal *et al.* (2019).

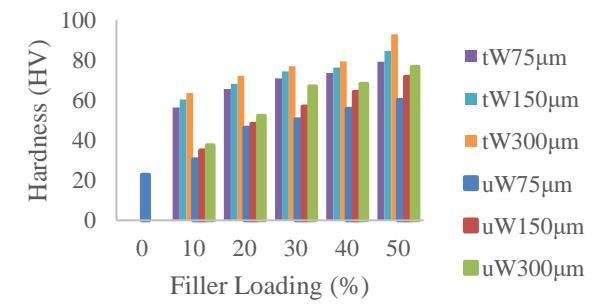


Figure 9: Effect of filler loading and Particle Size on the Hardness test for treated and untreated composites

Conclusion

This study established that milkweed wood particulates serve as effective reinforcement for recycled polypropylene (rPP), with hot-water treatment (HWT) yielding the most favorable performance outcomes. The HWT milkweed–rPP composites consistently exhibited enhanced tensile strength, tensile modulus, flexural strength, impact strength, and hardness relative to untreated composites. These improvements are attributed to better surface cleanliness, increased roughness, and improved interfacial bonding between the particulates and the polymer matrix. The reduction in elongation at break observed in the treated composites indicates increased rigidity, which is characteristic of stiffened polymer structures. Furthermore, the lower water absorption of the HWT composites confirms improved fibre–matrix compatibility and reduced microvoid formation, enhancing durability in moisture-prone environments. Overall, the findings demonstrate that HWT milkweed wood particulates significantly improve the mechanical properties and moisture resistance of wood/rPP composites, positioning the composite as a viable, sustainable material for medium-to-high performance applications. Potential utilization includes automotive interior parts, construction and building panels, household and consumer products such as table surfaces, and environmentally friendly biocomposite manufacturing, thereby contributing to economic growth through the valorization of *Calotropis procera* (Milkweed) wood and plastic waste.

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