



Chemical Treatments of Fibre and their Effects on the Physico-Mechanical Properties of African Fan Palm Powder Reinforced Waste Polypropylene Composites

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Abstract: The effects of chemical treatments on the mechanical properties of African fan palm powder (AFPP) filled polypropylene composites were investigated. The composites were developed via melt mixing and compression molding techniques. The fibres were treated with benzoyl chloride (BC) potassium tetraoxomanganate (VII) (KM) and sodium hydroxide (SH) solutions. Composite samples of varying weight fractions of reinforcement: 5, 10, 15, 20, and 25 % wt were characterized using physical and mechanical tests. Chemical treatments of AFPP were observed to decrease the rate of water absorption of the developed composites with BC, KM and SH treated samples having 3.72 %, 4.05 %, and 4.2 % at 25 % weight fraction of reinforcement after 240 hours of immersion respectively. Optimum tensile and flexural strength were recorded at 20 wt % of AFPP incorporated while other mechanical properties such as elastic modulus, impact strength, and hardness results were observed to increase with weight fraction of reinforcement up to 25 wt %. These compositions have the tensile strength of 98.45 MPa (55.56 MPa), the flexural strength of 55.56 MPa (50.45 MPa), and an elastic modulus of 85.67 MPa (55.67 MPa). These results indicated that chemical modification of fibers improves the physical and mechanical properties of African fan palm-reinforced polypropylene composites.

1. Introduction

Plastic wastes have been a major environmental concern in recent times because of their increased usage in many commercial sectors and improper method of disposal. Polypropylene wastes is the second most common plastic wastes in Nigeria after polyethylene. Natural fibres are used as replacement for conventional fibres such as aramid, glass, carbon, and Kevlar in composite manufacture due to their advantages such as low cost, availability, high-strength to-weight ratio and fairly good mechanical properties. Natural fibre composites have received substantial attention in the manufacturing industries because of their ease of processing, lightweight, and resistance to corrosion. However, despite the properties and applications of natural fibre-based composites their hydrophilic property poses a serious challenge to their effective use as they are not well compatible with polymer matrix which is hydrophobic (Dhakal *et al.*, 2006; Jacob and Mamza, 2020). It is important, therefore, that this problem is addressed in order that natural fibre may be considered as a viable reinforcement in composite materials.

Several researches on the application of natural fibres in composite development revealed that the susceptibility of physico-mechanical and thermal properties to water absorption could be minimized by the use of fibre surface modification (Tong *et al.*, 2014; Hossain *et al.*, 2014; Fiore *et al.*, 2015; Mohanta, 2016). The interfacial adhesion can be improved by modifying the fibres, the matrix, or both the fibres and the matrix by different physical and chemical methods. Fibre treatments involving use of various chemicals play an important role in improving the reinforcing capabilities of fibres. These treatments can be classified as fibre pre-treatments, coupling agents, compatibilizers, or dispersing agents (Joseph *et al.*, 1999; Premalal *et al.*, 2002; 2002; Zampaloni *et al.*, 2007; Paul *et al.*, 2010; Siddika *et al.*, 2014; Jacob *et al.*, 2018; Usman and Usman, 2020; Guna *et al.*, 2020; Rousseau *et al.*, 2023 and Jacob, 2023).

Pre-treatments involve the use of chemicals that remove undesirable and non-strength contributing fibre constituents such as lignin, pectin and hemicelluloses. Compatibilizers are chemicals that lower the surface energy of fibres to make them more non-polar and therefore more compatible with polymer matrices (Mohanta, 2016). Dispersing agents are used to improve the dispersion of fibres in the matrix. Coupling agents are mainly responsible for improving the adhesion between reinforcing fibres and the matrix material, but can also reduce the water uptake of the fibres and assist in fibre dispersion as well. Due to this overlap in functions and to simplify matters, all bonding agents and surfactants have been grouped as chemical treatments. This modification process eliminates undesirable fibre constituents like impurities, lignin, waxes, pectin, and hemicelluloses which bind these fibrils together. This causes inefficient fibre-matrix interaction and poor surface wetting. Elimination of these materials also gives rise to a rougher fibre surface increasing surface contact between the fibre and the matrix. This results in an improvement in mechanical interlocking between the polymer and the fibre, leading to enhanced mechanical properties such as tensile strength, flexural strength, hardness index, and impact strength. It has become pertinent, therefore, that this problem is addressed so that natural fibre may be considered as viable reinforcement in polymer composite.

2. Methodology

2.1 Sourcing and preparation of samples

The polypropylene wastes were collected from refuse dumps and plastic waste collection centres in Samaru-Zaria, Kaduna State, Nigeria. These were washed to remove dirt. The African fan palm used as reinforcement was collected from Kala'a, Hong Local Government, Adamawa State, Nigeria. It was dried at room temperature for four (4) weeks to eliminate moisture content and then pulverized into powder and sieved to 100 μm .

2.2 Experimental

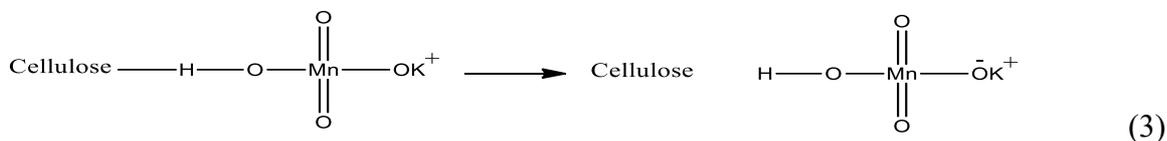
2.2.1 Fibre Surface Modification

(i) Alkalization: The African fan palm powder was immersed in 5 % sodium hydroxide solution for five hours, stirred, and filtered. It was then washed several times with distilled water until the solution becomes neutral. The resulting residue was finally dried in an oven at 80 °C for five hours by the work of (Jacob *et al.*, 2018) and that made by Usman, *et al.* (2016) as indicated in equation (1).

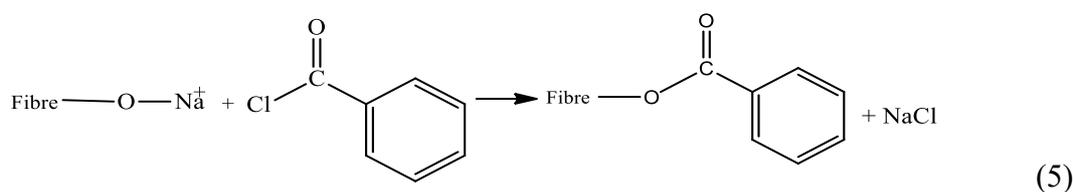


(ii) Permanganate Treatment: In permanganate treatment, the AFPP sample was initially alkaline pre-treated to activate the hydroxyl groups of cellulose and lignin in the fibre following the procedure in (i) above. The pre-treated samples were then separately soaked in 1 litre 0.125% KMnO_4 solution in

acetone for about 2-3 minutes; washed with distilled water and dried in an oven at 60 °C (Jinitha., 2016). Equations (2) and (3) show the interactions of AFPP with permanganate solution.



(iii) Benzoylation: In the treatment with benzoyl chloride, the procedure by (Dhanalakshmi and Baraju, 2015; Jacob *et al.*, 2018) was adopted. This could be represented by equations (4) and (5).



2.3 Preparation of AFP filled RPP composites

The chemically modified African fan palm powder reinforced recycled polypropylene composites were prepared according to the procedure earlier reported (Jacob *et al.*, 2019).

2.3.1 Composite production

The materials were compounded via melt mixing at a temperature of 160 °C to obtain a homogeneous mixture. The compositions of the African fan palm powder incorporated were 5, 10, 15, 20 and 25 % respectively, while that of shredded PP wastes was varied accordingly from 0, 95, 90, 85, 80 and 75 % respectively. Curing of the samples was then carried out using hydraulic press at a temperature of 160 °C and a compression pressure of 3 Pa for 10 minutes. Samples obtained were cooled and machined for physical and mechanical properties tests.

2.4 Mechanical Property Test

2.4.1 Tensile test

The tensile characteristics (tensile strength, elastic modulus, of the samples was determined using the ASTM D638 (2022) recommended method. The samples were machined to dumbbell shape and then placed in computerized Instron universal tensile testing machine 3369 model, which measured the tensile strength and elastic modulus were evaluated.

2.4.2 Flexural strength

Flexural strength was measured under a three-point bending approach using a universal testing machine according to ASTM D790. The distance between the spans was 40 mm and the strain rate was 5 mm/min.

2.4.3 Hardness test

The hardness test of composites is based on the relative resistance of its surface to indentation by an indenter of specified dimensions under a specified load (Jacob *et al.*, 2018). Samples of 30 mm x

30 mm x 5 mm were tested for shore hardness values with Durometer Shore A. Five measurements were performed on the sample at different spots and the average of the values was taken as the hardness of the sample (Jacob *et al.*, 2018; Jacob and Mamza, 2020).

2.4.4 Water absorption

Water absorption test was carried out according to ASTM D570 (2010) method. The test sample was an oven dried specimen of dimension 76 × 25 × 5 mm immersed in water at ambient temperature for 24 hours. After immersion period of 24 hours, the specimens were removed and patted dry with a cloth (lint free) and then reweighed using a Sartorius ED 224S digital Analytical balance. In order to evaluate long term moisture absorption on the composites, the process was repeated at 48, 72, 96, and 120 up to 240 hours exposure. The dried weight before ($W_{initial}$) and after weight immersion (W_{final}) were noted. Similar method has been reported (Jacob and Mamza, 2020; Jacob and Shinggu, 2021). The water absorption was determined as follows:

$$W = \frac{W_{final} - W_{initial}}{W_{final}} (\%) \quad (6)$$

3. Results and Discussion

3.1 Influence of chemical modification on the physical properties of recycled polypropylene composites. Figure 1 depicts the water absorption of untreated AFPP-RPP composites. Expectedly, the trend indicated an increase in moisture absorption with a weight percentage of AFPP and time. The highest water absorption recorded was 20.55 % at 25wt % of AFPP. The reason for the observed high percentage of moisture absorption in the untreated composite samples is not far-fetched. Higher fibre content could also result in higher voids entrapped in the composites, hence, high water accumulation at the interface between fibre and matrix (Tajeddi *et al.*, 2010). Similar results of an increase in water uptake with weight fraction of reinforcement have been reported (Jacob and Mamza, 2020; Jacob and Shinggu, 2021).

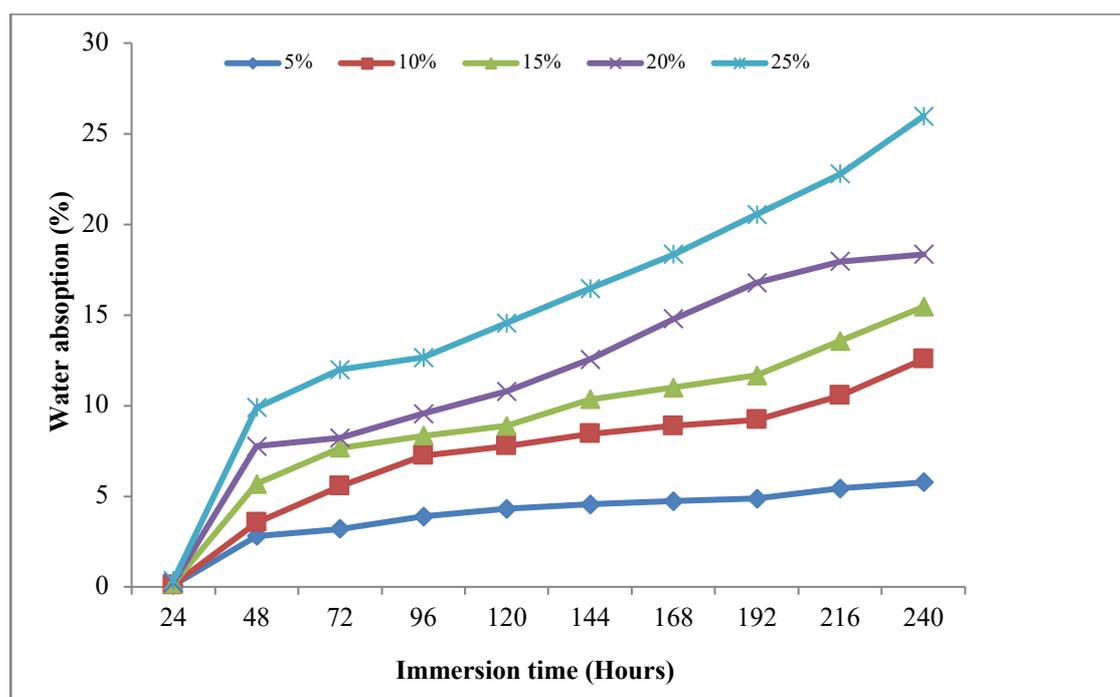


Figure 1: Percentage of water absorption of untreated AFPP-reinforced RPP composites.

Figure 2 shows the water absorption profile for benzoyl chloride-treated AFPP-RPP composites after 240 hours of immersion. It could be observed that chemical modification presented a water absorption rate that was much lower compared to the other chemical modifications considered. This could be due to improved interfacial bonding between the fibre incorporated and the recycled polypropylene matrix, thereby decreasing the number of voids created. It could also be attributed to the fact that the chemical treatment of fibres removes non-cellulosic materials like lignin, pectin, and wax which contributes to greater water uptake by natural fibre-filled composite materials. The highest water absorption of 3.72 % was recorded at 25 wt % while the least was 2.43 at 5wt % AFPP.

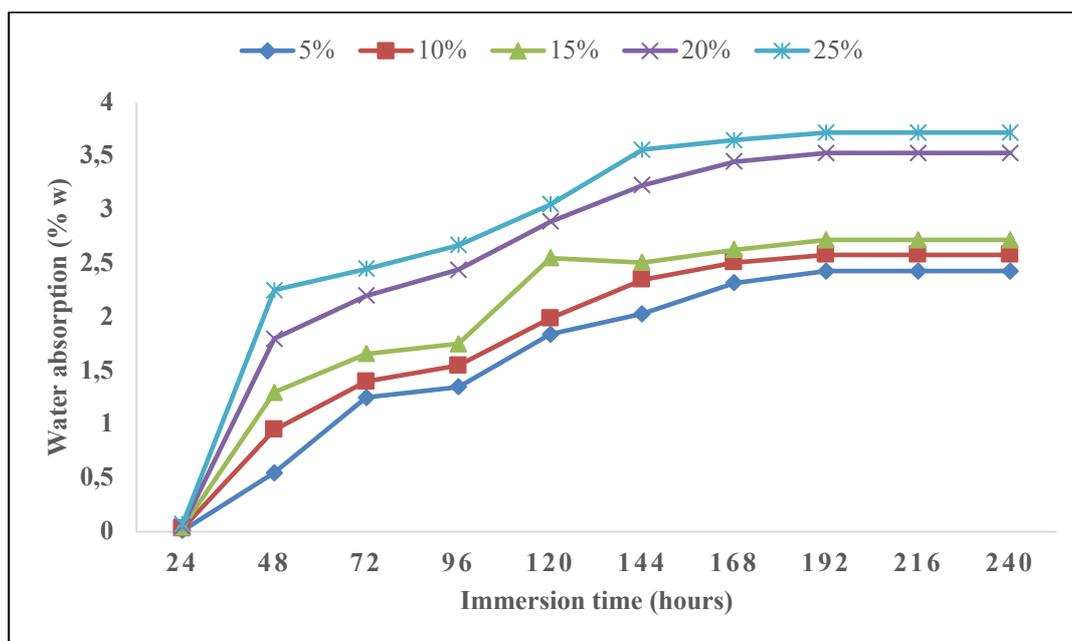


Figure 2: Percentage of water absorption of BC-treated AFPP reinforced RPP composites

Figure 3 depicts the percentage water absorption of potassium permanganate-treated AFPP-RPP composites. From the curve, it is clear that the percentage of moisture absorption increases with the weight fraction of reinforcement. This could be attributed to an increase in the surface area of reinforcement in the composites leading to an increased number of pores. The highest water absorption was observed at 25 wt% AFPP; while the least water absorption of 2.4 % was obtained at 5 wt % AFPP.

Figure 4 depicts the percentage water absorption of sodium hydroxide-treated AFPP-reinforced RPP composites. It could be observed that water absorption increases with time and also with the amount of AFPP incorporated. The highest water absorption recorded was 4.2 % for 25 wt% AFPP content after 240 hours of immersion, while the least was at 5 wt% with a 2.8 % absorption rate. This indicates that the treatment of African fan palm powder with sodium hydroxide decreases its susceptibility to moisture absorption. Generally, it was observed that beyond 240 hours (10 days) for all composites, there was no further increase in water absorption, which could be attributed to the fact that the pores created may have been saturated with water, thus giving rise to such behaviour. The observed increase in water absorption by composites at higher amounts of natural fibre could be due to the poor adhesion/compatibility between the fibres and the polymer matrix. As the amount of natural fibres increases, the micro-level processing of the composites becomes difficult and may cause the fibre layering out, this creates micro-voids and cracks within composites. This tends to cause the flow of water molecules along the fibre-matrix interface, leading to the diffusion of water from the interface to the matrix and the fiber (Sreekala *et al.*, 2002). The rate of water absorption in the developed

composites for the three chemicals investigated increases in the order: BC-AFPP<SH-AFPP<KM-AFPP<UAFPP.

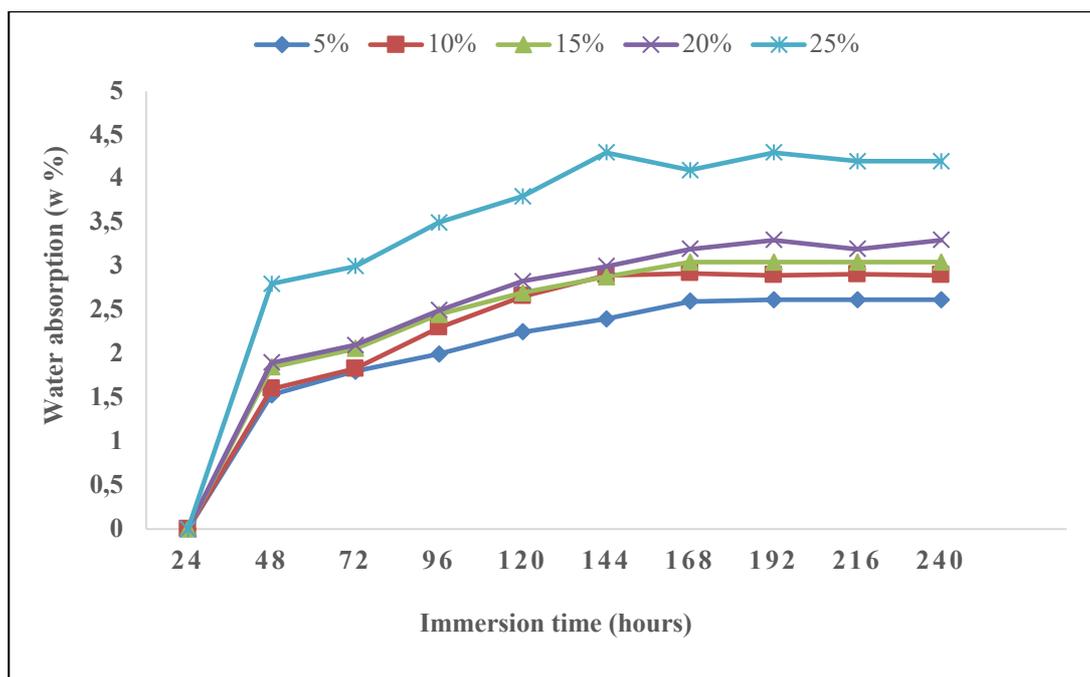


Figure 3: Percentage of water absorption of KM-treated AFPP reinforced RPP composites

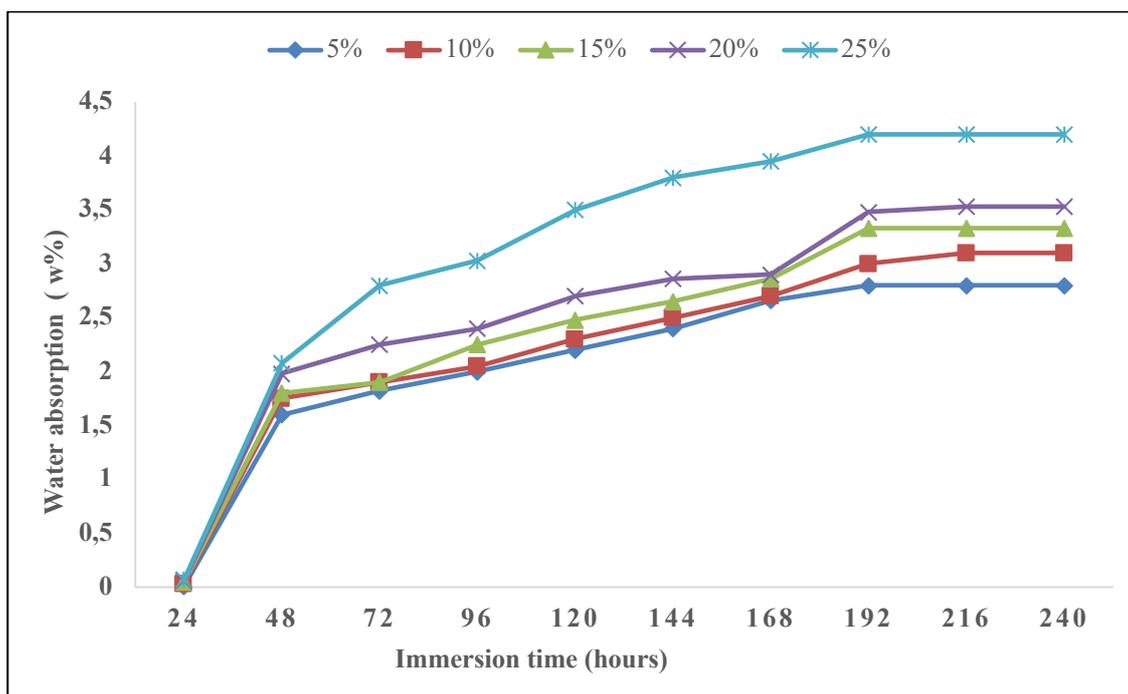


Figure 4: Percentage of water absorption of SH treated AFPP reinforced RPP composites

3.2 Influence of chemical modification on the mechanical properties of recycled polypropylene composites

Figure 5 depicts the tensile strength of AFPP-RPP composites with increasing weight of reinforcement. A gradual increase in tensile strength could be observed up to 20 % weight of reinforcement and a sudden decrease at 25 %. This could be ascribed to the debilitating nature of the

interfacial attraction of the consequent composition as the percentage of RPP is reduced with an increase in weight fraction of AFPP fibre (Jacob and Mamza, 2021) reported that a decrease in ultimate tensile strength could be attributed to poor fibre-matrix adhesion and the agglomeration of fibre particles in the consequent composition.

Figure 5 also shows the effect of chemical treatment on the UTS of AFPP-RPP composites. The strength of the untreated fibre-reinforced RPP samples was found to be lower than the raw (control) sample. This could be attributed to the presence of non-cellulosic materials like lignin, hemicelluloses, natural oils, and pectin wax which makes natural fibres incompatible with hydrophobic polymer matrix as a result of poor interfacial bonding.

However, chemically modified samples have shown improvement in UTS with benzoyl chloride treatment exhibiting the highest tensile strength of 11.53 MPa at 20 % weight fraction of reinforcement which is 40.42 % higher than the untreated fibre. The enhanced UTS observed with benzoyl chloride treatment may be attributed to the improved fibre-RPP interface resulting from better adhesion. Jinitha *et al.* (2016) also reported an increase in UTS with chemical modifications of the fibres. Meanwhile, the observed decrease in UTS with KMnO_4 treatment may be due to fibre damage at higher concentrations of KMnO_4 which could decrease the strength of the composites (Corrales *et al.*, 2007). According to Milan *et al.* (2015) a higher concentration of potassium permanganate (more than 1%) causes the removal of cementing materials within the cellulosic structure and degrades fibre properties.

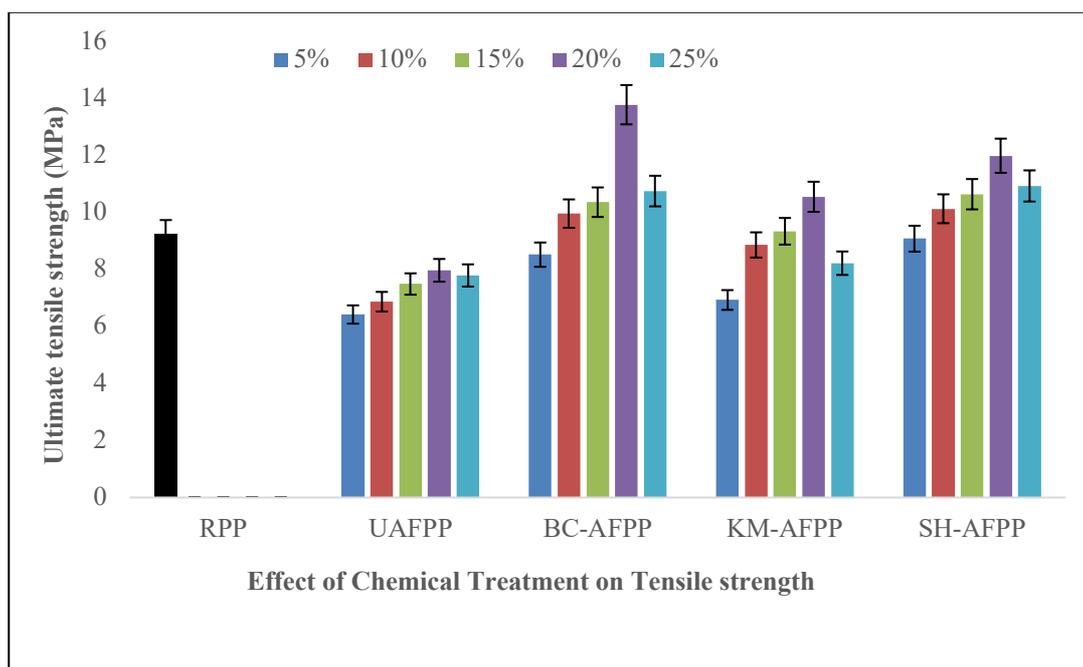


Figure 5: Ultimate tensile strength of African fan palm powder reinforced RPP composites

An increase in stiffness of the developed material with a weight percentage of reinforcement could be observed in Figure 6. The trend showed that the elastic modulus (stiffness) of the composites increased from 27.21 MPa at 5 % to 53.13 MPa at 25 % wt of AFPP. The increase in elasticity (Young's modulus) implies that the reinforcement exhibits high stiffness (modulus) compared to the polymeric material (RPP). This is because, at a high weight percentage of reinforcement, the composites would be able to withstand greater loads. Similar results have been reported by (Jacob *et al.*, 2019). Natural lignocellulosic fibres have been observed to have an elastic modulus higher than polyethylene,

polypropylene, and other polymeric materials (Wang *et al.*, 2016). Owing to this, the stiffness of the composites tends to improve with the incorporation of these fibres. The figure also depicts the influence of the chemical modification of fibres on the elastic modulus of AFPP-RPP composites. Generally, it could be observed that chemically modified composites have higher stiffness values than the unmodified samples. This is anticipated because, chemical modification of the plantain peel powder improved fibre roughness, and created an increased area of contact between the AFPP fibre and the RPP matrix. Removal of undesirable fibre constituents like lignin, natural oils, and pectin from the fibre surface could lead to better packing of cellulose chains and consequently enhanced mechanical properties.

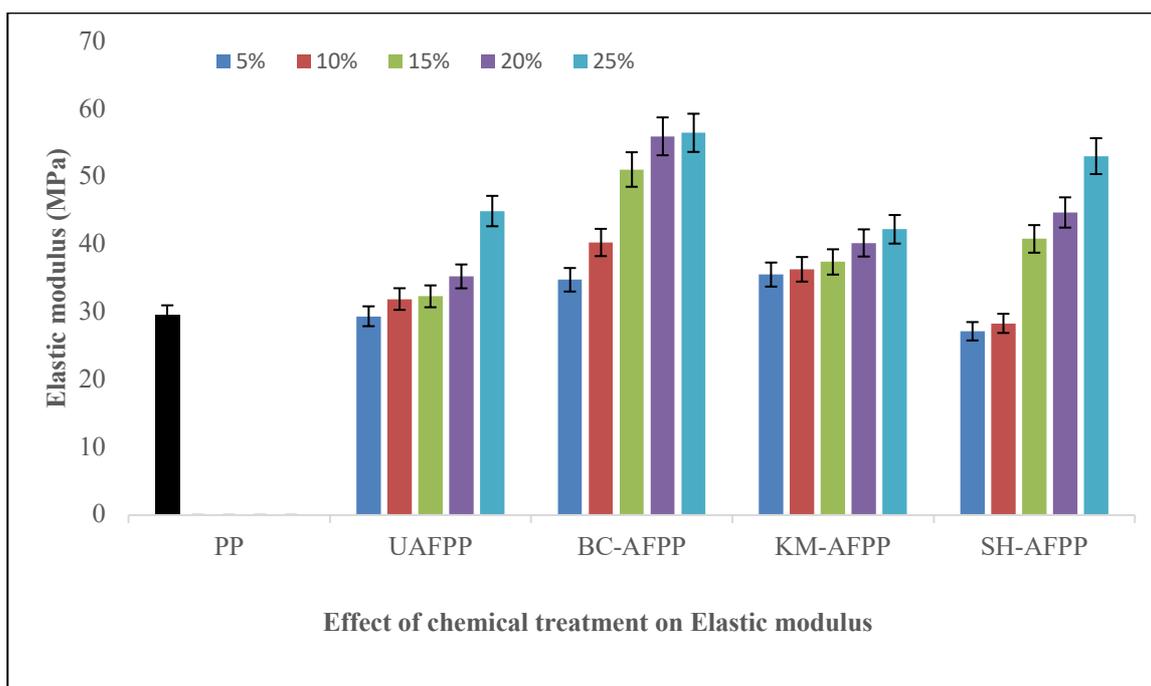


Figure 6: Elastic modulus of African fan powder reinforced RPP composites

The result of the effect of weight percentage of reinforcement and chemical treatment on the elongation at break (%) of AFPP-reinforced RPP composites presented in Figure 7 indicated a decrease in elongation at break (%) with the incorporation of AFPP. The decrease is dependent upon the amount of AFPP incorporated. An increase in the weight percentage of reinforcement culminated in the stiffening and hardening of the composites; this reduces its resilience and toughness and could lead to lower elongation at break (%) (Jacob and Thomas, 2004). This increase in hardness resulted in a decrease in the ductility of the material; hence as the weight percentage of reinforcement increases, the ductility decreases. Jacob *et al.* (2018) also reported a decrease in ductility with weight fraction of reinforcement in nanofiber-reinforced composites.

The composites produced from benzoyl chloride-treated AFPP exhibited the highest elongation at a break of 79 %. This is about 33 % higher than the untreated sample, 10 % higher than sodium hydroxide-treated AFPP-RPP composites, and 14 % higher than the potassium permanganate samples. Generally, it was observed that chemical modification of PPP fibres improved the % of the elongation at the break of the composites. In a report by Maya *et al.* (2007), NaOH was shown to be more effective than silane treatment. Chemical modification removes excess hemicelluloses, lignin, wax, and pectin. The removal of the impurities may have exposed more area of the fibre surface for better contact with the polymer matrix.

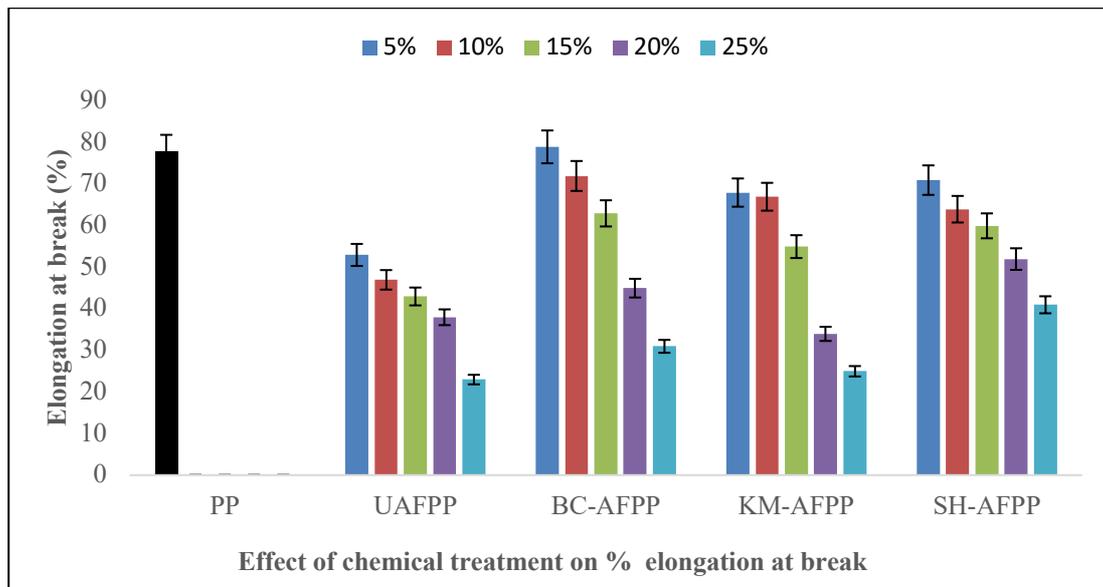


Figure 7: Elongation at break of African fan palm powder reinforced RPP composites.

Figure 8 shows the effect of the weight percentage of reinforcement and chemical treatment on the flexural strength of plantain peel powder reinforced-RPP composites. The flexural strength of the composites of AFPP-RPP was observed to be higher than the unreinforced (control) sample. The increase in flexural strength could be due to even dispersion of fibres in the polymer matrix which allows chain movement during deformation.

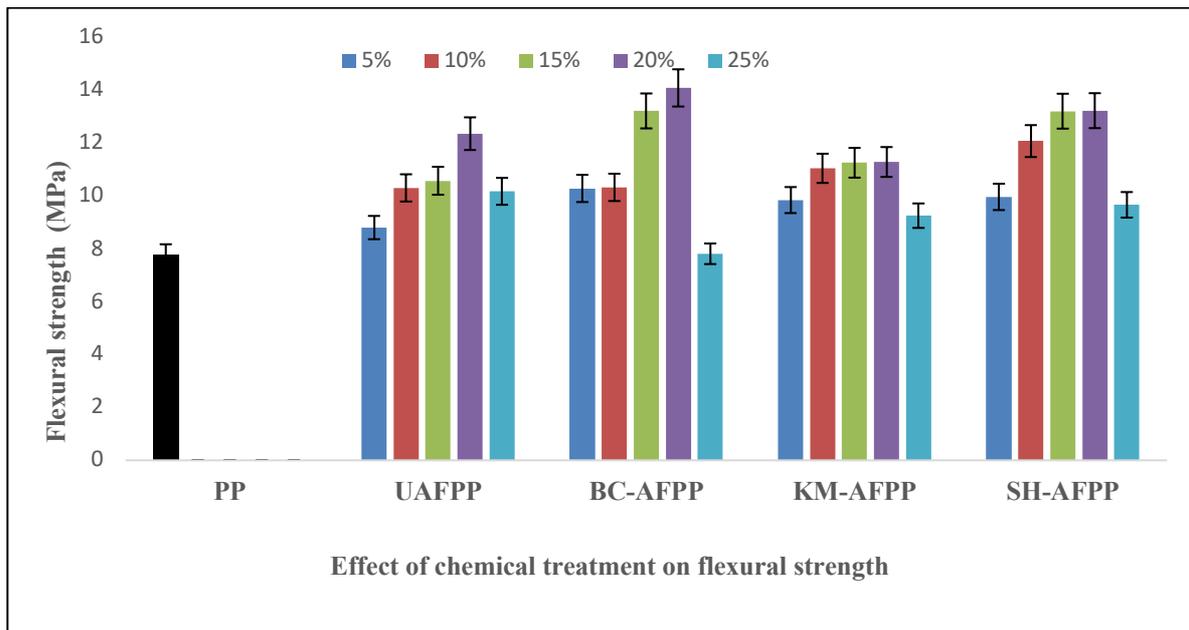


Figure 8: Flexural strength of African fan palm powder reinforced RPP composites

However, there is a decrease in flexural strength at a higher weight percentage of reinforcement (above 20 %). This is because of an increase in rigidity and stiffness of the composites with an increase in reinforcement. This is in agreement with the results of [Raju et al \(2012\)](#) who investigated the mechanical and physical characterization of groundnut shell powder-reinforced vinyl ester resin composites. The improvement in flexural strength after the different fibre surface treatments could be due to an increase in the roughness of the fibre and thus an increase in the area of contact between the modified AFPP and RPP matrix. [Figure 9](#) shows the impact strength of AFPP-RPP composites. An

improvement in impact energy of the composites with weight fraction of reinforcement could be observed from 5 wt % to 25 wt %. These values are 6.34 J/mm² and 9.87 J/mm² respectively while that of unreinforced RPP is 10.87J/mm². The increase in impact strength with weight fraction of reinforcement could be due to a reduction in deformation processes and thus improve the ability of the material to absorb and dissipate energy. However, it is pertinent to note that incorporation of AFPP into RPP decreases its impact energy in the first instance, which was observed to increase upon subsequent AFPP addition. This could be attributed to particle-particle interaction and therefore gives rise to zones of weakness which may induce failure at lower stress values. Chemical treatment of fibres was also found to improve the impact energy of composites. Benzoyl chloride-treated samples (BC-AFPP) had higher impact energy values compared to potassium permanganate-treated (KM-AFPP) and sodium hydroxide-treated (SH-AFPP) composite samples.

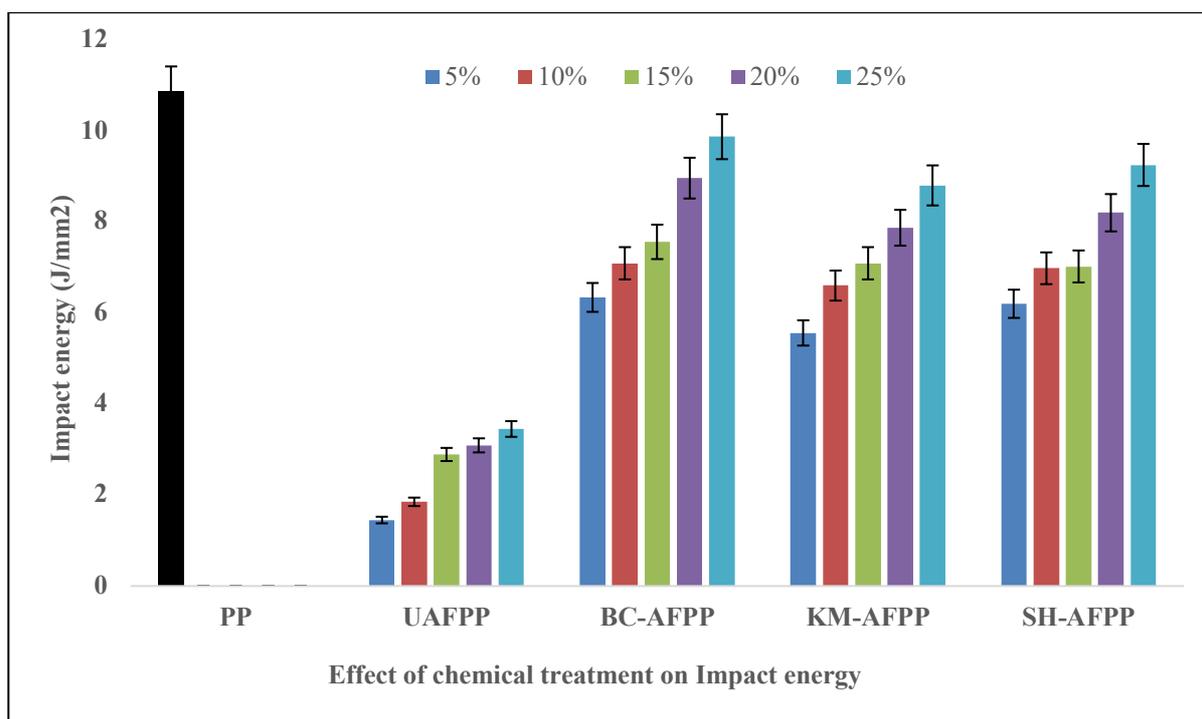


Figure 9: Impact energy of African fan palm powder reinforced RPP composites

Graphical depiction of hardness values of plantain peel powder reinforced RPP composites in [figure 11](#) shows a gradual increase in hardness for all the composites. A decline in hardness value at 15 wt% for the untreated samples and 10 wt% of the potassium permanganate treated samples, and then an increase was observed. The improvement in the hardness value of the composites could be ascribed to the brittleness of the fibres in the polymer matrix, while the point of decrease may be due to the uneven distribution of fibres in the RPP matrix ([Imoisili & Jen T-C. \(2020\)](#)). Furthermore, the incorporation of fibres may have improved the material resistance to indentation. The potassium permanganate-treated samples indicated the least hardness values at all weight percentages of reinforcement. Maximum hardness value was recorded at 25 wt% fibre which is 85.6 Shores for benzoyl chloride treated composites, while the least was noted at 5 wt% with 70.1 Shores for the untreated samples while 65 Shores were recorded as the hardness value of the unreinforced RPP material. Findings are in good agreement with those obtained recently ([Imoisili & Jen T-C. \(2020\)](#); [Abisha et al. 2023](#); [Li et al. 2016](#); [Arulmurugan & Venkateshwaran, \(2019\)](#); [Almtori et al 2021](#)).

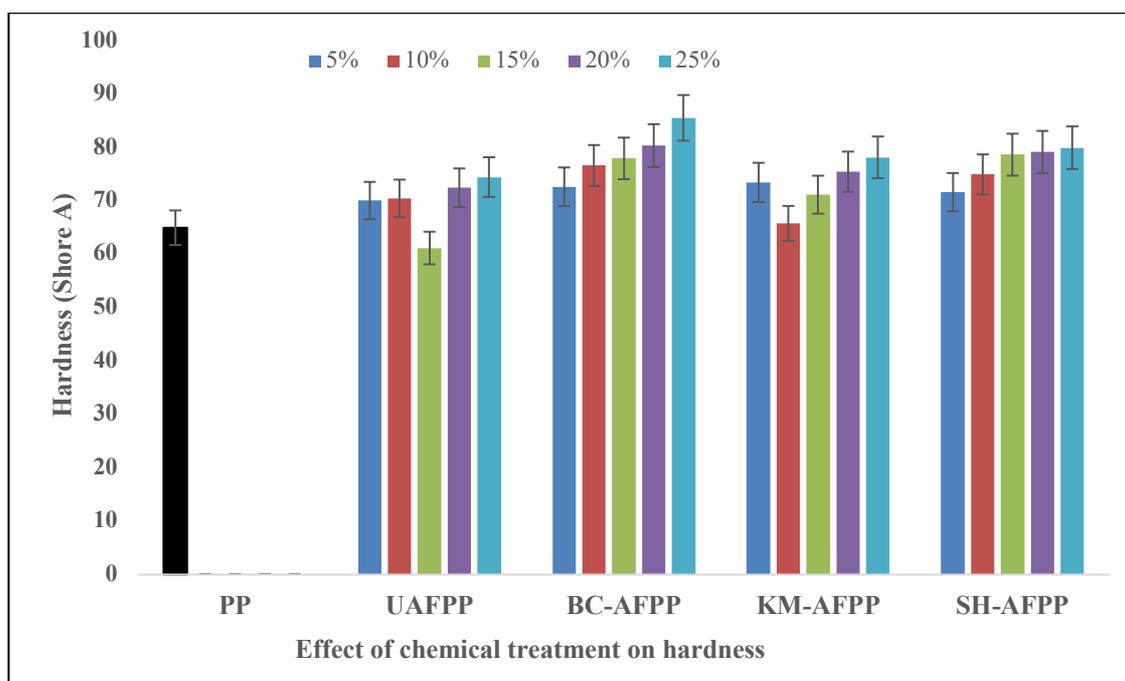


Figure 10: Hardness values of African fan palm powder reinforced RPP composites

Conclusion

Benzoyl chloride, potassium permanganate, and sodium hydroxide-treated African fan palm powder-filled polypropylene composites were developed; and from the results obtained, the following conclusions are made:

- Chemical treatments of AFPP were observed to decrease the rate of water absorption of the developed composites with BC, KM and SH treated samples having 3.72 %, 4.05 %, and 4.2 % at 25 % weight fraction of reinforcement after 240 hours of immersion respectively.
- All the mechanical properties investigated in this work were observed to increase with the weight fraction of reinforcement with the benzoyl chloride-treated samples having the highest.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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