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Full Length Research Article

THE EFFECT OF ALKALI TREATMENT ON THE MECHANICAL PROPERTIES OF DATE SEED PARTICULATES WASTE POLYPROPYLENE FILLED COMPOSITES

*[1]Lawal N., [1]Danladi A., [1]Dauda B., [1]Kogo A., [1]Baba M.A. and [2]Busuguma U.

[1]Department of Polymer and Textile Engineering, Ahmadu Bello University, Zaria, Nigeria
[2]Ramat Polytechnic, Maiduguri

*Corresponding Author Email Address: lawalnuhu1978@gmail.com

ABSTRACT
Date seeds collected as waste were reduced to 63 µm particles and treated with NaOH (aq) at 1, 3, 5, 7 and 10 %. The extent of chemical modification was assessed using FTIR analysis. The waste polypropylene (wPP)/date seed particles (DSP) composites filled with untreated and treated particles at 20 % filler loadings were fabricated using compounding and compression moulding techniques by means of two rolls mill. Tensile, flexural, impact tests and hardness (HV) were conducted and evaluated. Subsequently, a morphological study of the fractured surface of the composites was achieved using SEM. The application of alkali treatment enhanced various properties of the composites at the optimal concentration of 10 % w/v. Specifically, the tensile strength, flexural strength, elongation at break, impact strength, and hardness were improved, resulting in values of 28.86 MPa, 140 MPa, 172.5 %, 0.92 J/m², and 23.8 Hv, respectively. While tensile and flexural moduli showed a decrease with increasing alkali concentration. The SEM of the fractured surface of the treated composite revealed a better dispersion of filler particles within the matrix with no cluster seen compared to the untreated.

Keywords: *Alkali treatment, composites, date seed, dispersion and waste polypropylene*

INTRODUCTION
A composite is a combination of two materials, in which one of the materials, called the reinforcing phase, is in the form of fibres, sheets, or particles, and embedded in the other, called a matrix phase. The reinforcing material and the matrix can be metal, ceramic, or polymer. Composites typically have a fibre or particle phase that is stiffer and stronger than the continuous matrix phase that serves as the principal load-carrying member. The matrix acts as a load transfer medium between fibres, and in less ideal cases where the loads are complex, the matrix may even have to bear loads transverse to the fibre axis. The matrix also serves as protection for fibres from environmental damage before, during and after composite processing. When properly designed, the new combined material exhibits better strength than the individual material (Elanchezhian *et al*., 2018). Traditional fibre-reinforced composites consisting of carbon or glass fibres in thermosetting or thermoplastic polymers are for varieties of applications like consumer products for casing and packaging etc. Most plastics used are from petroleum products and are largely non-biodegradable. The problems associated with plastics for the inability to degrade have generated interest in developing bio-degradable materials (Kumar *et al*., 2023).
Alkaline treatment of lignocellulosic fillers involves the use of solutions such as sodium hydroxide (NaOH) to primarily alter the surface properties of these natural materials. This modification increases the compatibility of the polymer matrix in composite materials, resulting in improved interfacial adhesion due to the presence of additional hydroxyl groups on the surface (Oushabi, 2019). These hydroxyl groups promote stronger hydrogen bonding and chemical interactions with the polymer matrix. Consequently, the enhanced interfacial adhesion leads to greater strength and stiffness in the composite (Nurazzi *et al*., 2021).
Furthermore, the alkaline treatment has several additional benefits. It reduces the hydrophilicity of the lignocellulose fillers, reducing moisture absorption and enhancing the overall dimensional stability of the composite (Ahmad *et al*., 2019). It also contributes to improved thermal stability by eliminating certain thermally sensitive components like hemicellulose and partially degraded lignin (Zhang *et al*., 2015). Additionally, this treatment aids in the uniform dispersion of the filler within the polymer matrix, preventing clumping and ensuring consistent mechanical properties throughout the composite. It's important to note that the specific effects of this treatment depend on various factors, such as the type of filler used, the polymer matrix, treatment conditions, and the intended application of the composite (Wu *et al*., 2023)
A polypropylene was chosen as the matrix material for several beneficial reasons. Its low density contributes to a significant reduction in the overall weight of the composite (Shah *et al*., 2022). It displays resistance to chemicals, especially those with higher melting points, and demonstrates good thermal stability, enabling it to endure moderate temperatures without substantial degradation (Chauhan *et al*., 2022), (Aslani and Wang, 2019). Additionally, polypropylene is relatively cost-effective, enhancing the economic feasibility of the composite. Furthermore, its processing capabilities and recyclability align with the growing focus on sustainability and the circular economy (Jagadeesh *et al*., 2022). While it may not be the strongest material available, polypropylene offers a favorable balance of strength, particularly when combined with lignocellulosic materials like date seeds in composite applications (Faiad *et al*., 2022).
The date (*Phoenix dactylifera* L.) has been a valuable crop in arid and semiarid regions of the world. It has always played a significant part in the economic and social lives of the people of these regions. Pits of date palm (seed) are waste products of many industries. In some date-processing countries, such as Tunisia, date seeds are discarded or used as fodder for domestic farm animals (Souhel *et al*., 2004). In Nigeria, date palms are abundant in the Northern region and serve as a source of income for farmers.
The elements present in the date seed material were carbon (C), oxygen (O), sulfur (S), potassium (K), and iron (Fe), comprising

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81%, 17.6%, 1.5%, 1.3%, and 1.44% by weight, respectively. These elements were incorporated into the base material to improve the mechanical characteristics of the composites (Elkhouly *et al.,* 2020). Despite the abundance of date seeds, their use as fillers in composites has not been widely published. Therefore, date seeds as bio-filler can add value to it.

MATERIALS AND METHODS

Materials

Waste polypropylene (used as packages) from dump sites was collected and cleaned. The filler (date seeds) used in this study was from the Zaria City market at 'Yan dabino. Kaduna State, Nigeria.

Methods

Samples preparations

- Polypropylene (wPP) was cleaned in water, air dried and then cut into smaller pieces (flakes form) and kept in the laboratory for future work.
- Date seeds were cleaned in water to remove impurities, sundried and then ground using jaw crusher and ball mill machines (Retsch Masch. Nr 70992 GMbH & CO. and Kera b.v. Soeter berg Overveld 057748 Holland) respectively. The date seed particles were then sieved using Impact Lab. Sieve ISO 3310-1:2000, bs 410-1:2000) to obtain particle sizes of 63 µm, after which they were alkali treated at 1, 3, 5, 7 and 10 % w/v by immersing the particles for 1 hour at ratio of 1:10 date seed particles/NaOH solution. The treated samples; were washed several times until the neutral pH. It was finally dried in an oven at 70 ^{0}C for 24 hr (Elnaid *et al.,* 2020). The FTIR analysis was carried out on untreated and treated date seed particles to determine the extent of chemical modification on the date seed particles.

Fabrication of the composites

Fabrication of the composites was carried out by compounding and compression moulding techniques at 20 % filler loading using two rolls mill and compression moulding machines. A steel mould of 150 × 120 × 5 mm was used. The control sample was the unfilled waste polypropylene. The matrix and filler were mixed for 5 min in two rolls mill to achieve a homogenous mixture. The mixture was placed in the mould treated with releasing agent. The maximum pressing temperature, pressure, time and cold pressing or pressure holding time were 160 ^{0}C, 5 N/mm^2, 15 min and 5 min respectively (Lawal *et al*., 2019). After cooling, the composite was removed and conditioned in an oven at 70 ^{0}C for 48 hr before testing.

FTIR Analysis

The Fourier Transform Infrared Spectroscopic analysis (FTIR) of the date seed particles (DSP) were studied using FTIR Agilent Technologies 4000-650. Happ-Genzel.

Plate I: Some of the (wPP)/date seed composites (samples) ready for mechanical tests

Tensile Test

The test was conducted by ASTM D638 standard with a gauge length of 40 mm and load force of 100KN. The samples dimension of 100 x 10 x 5 mm was tested using a YG026D Multifunctional Electronic Fabric Strength Machine at a cross-head speed of 10 mm/min.

Flexural Test

Three-point bending test was performed in accordance with ASTM D790M Test Method I procedure A to measure the flexural properties of the composites. The samples measured approximately 100 x 20 x 5 mm in length, width and thickness respectively. The span length was 80 mm apart at a 0.5 mm/min strain rate.

Impact Test

The impact strength of the composite samples was carried out according to ASTM E23 (Notched) using Norwood Charpy Impact Tester (Model no. 6957, capacity of 15 joules). The specimen size was 100 x 10 x 5 mm.

Hardness Test

The indentation test for composite samples was carried out using *vickers* hardness tester (HV) according to ISO 6507. Each sample was loaded on the machine while in the compressed moulds form of 30 x 10 x 5 mm

Morphological Studies

The Scanning Electron Microscopy (SEM) of the composites were studied using Scanning Electron Microscope (SEM) Joel JSM 7600F at an accelerating voltage of 5.0Kv and magnification of 500x.

RESULTS AND DISCUSSION

FTIR Analysis of the untreated and treated date seed particles

The effect of chemical modification on the date seed particles was observed using FT-IR Spectroscopy. The comparison of the representative FT-IR spectra of the untreated date seed particles and the treated date seed particles in Figure 1 to 6 show a reduction in O-H stretching intensity. Shifting of the peaks from 3287 cm^{-1}, 3347 cm^{-1}, 3466 cm^{-1}, 3470 cm^{-1}, 3488 cm^{-1} and 3642 cm^{-1} could be observed for the untreated date seed particles, 1, 3, 5, 7 and 10 % (Figure 1 to 6) treated date seed particles, respectively. This is likely attributed to the disruption of hydrogen bond between the O-H groups of cellulose and hemicellulose present in the date seed particles (Gumel & Tijjani, 2015; Usman *et al.,* 2021). Peaks at 2922 cm^{-1} and 2855 cm^{-1} in all the spectra predominantly arise from C-H stretching of the aliphatic group and

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the reduction in their stretching intensity indicate the removal of hemicellulose (Jayamani *et al.,* 2020). The absorption peak at 1744 cm^{-1} was associated with carbonyl C=O stretching of acetyl groups of hemicellulose. This peak also indicates the presence of lignin and other non-cellulosic component such as pectin. However, this peak was detected in the alkaline-treated date seed particles (5, 7 and 10 %) very weakly this weakening indicates the removals of hemicellulose (Liu *et al.*, 2019), lignin and pectin. The absorbance peak at 1438 cm^{-1} was assigned to $–CH_3$ and the reduction in the intensity of this peak from 1438 cm^{-1} for untreated to 1420 for the treated ones was attributed to the asymmetric deformation of lignin. The band at 1241 cm^{-1} attributed to the stretching vibration of C-O groups present in lignin and hemicellulose structure. Weakening of this peak confirmed the removal of lignin and hemicellulose (Jin *et al*., 2022). Important modifications were observed at peaks 1148 cm^{-1} for the untreated date seed particles indicating C-O for stretching of primary alcohol, reduction in the peaks from 1148 cm^{-1} for the untreated date seed particles compared to that of the treated 1141 cm^{-1} to 1118 cm^{-1} indicates the removal of lignin and hemicellulose (Salisu *et al*., 2015).

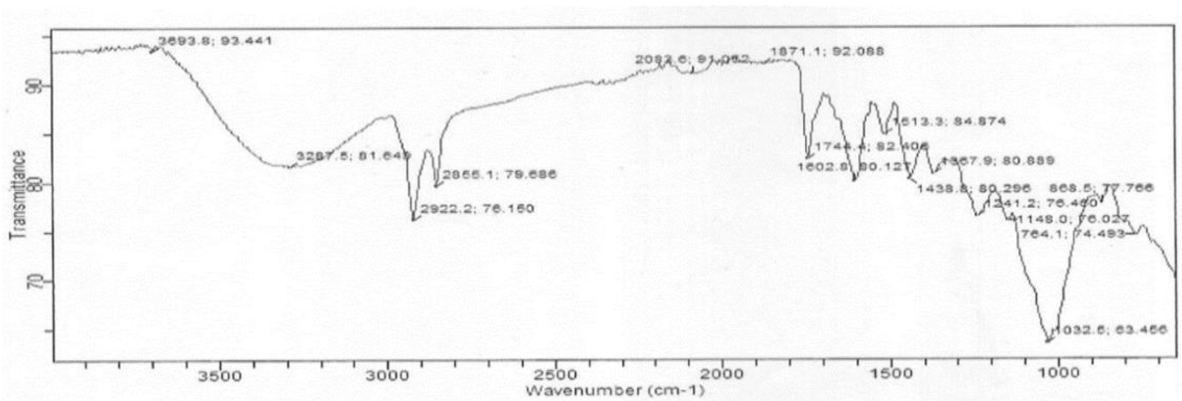

Figure 1: FTIR spectra of untreated date seed particles (DSP)

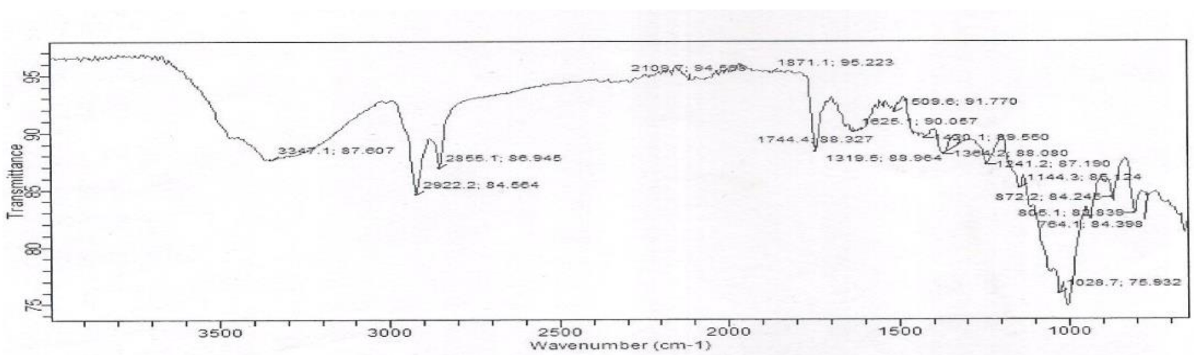

Figure 2: FTIR spectra of 1 % treated date seed particles (DSP)

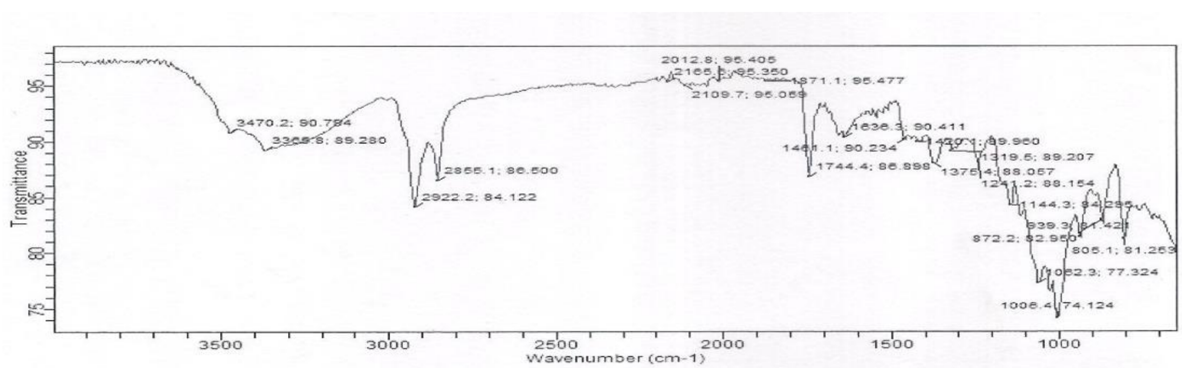

Figure 3: FTIR spectra of 3 % treated date seed particles (DSP)

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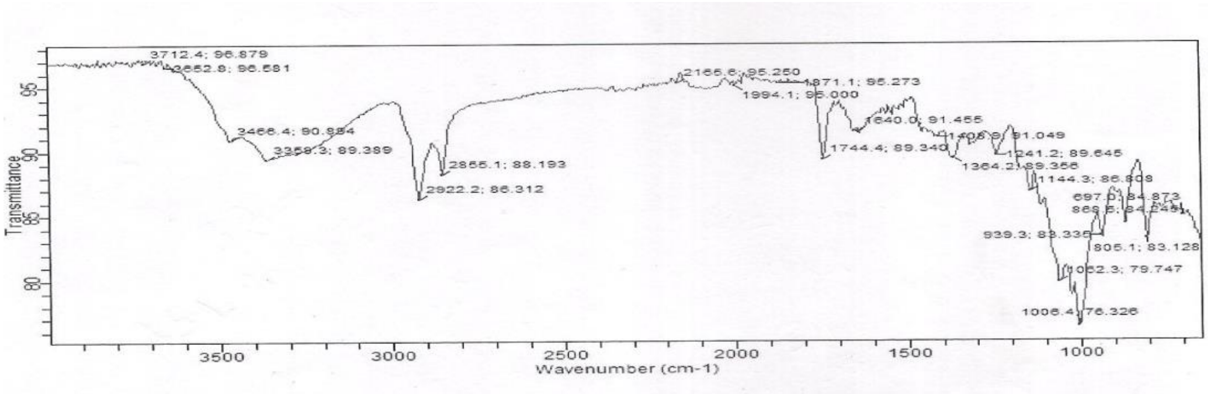

Figure 4: FTIR spectra of 5 % treated date seed particles (DSP)

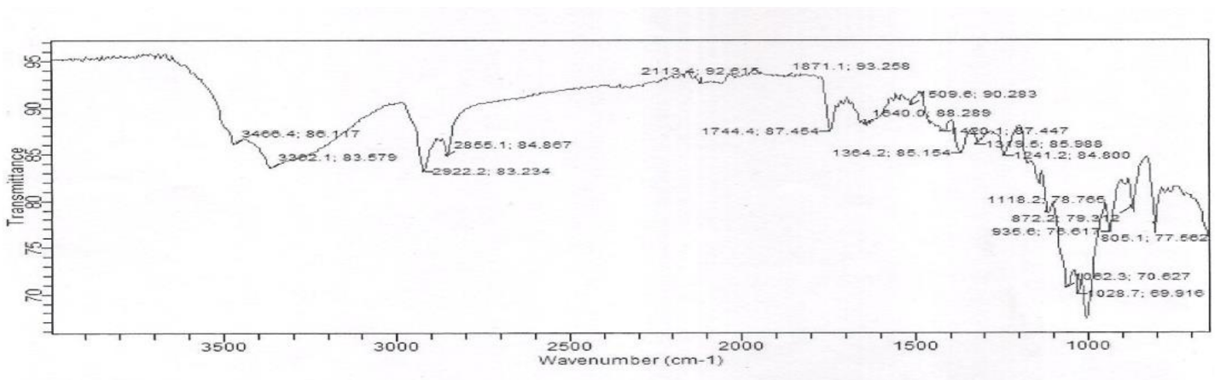

Figure 5: FTIR spectra of 7 % treated date seed particles (DSP)

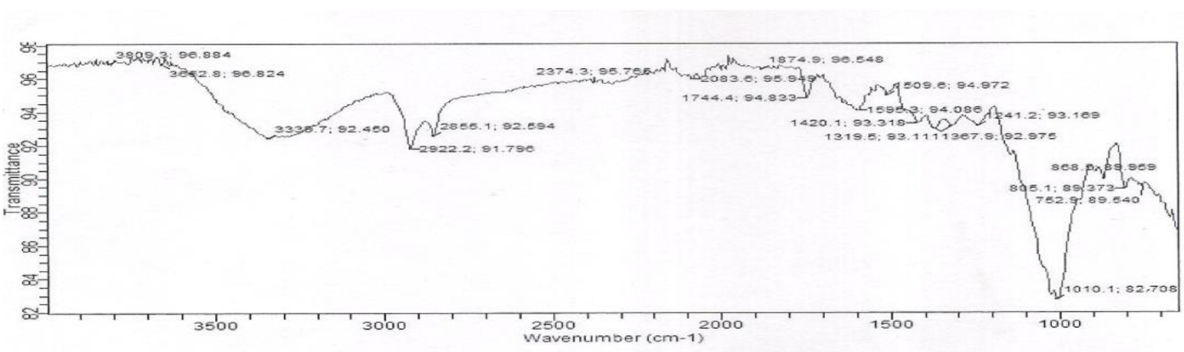

Figure 6: FTIR spectra of 10 % treated date seed particles (DSP)

Tensile Strength

Figure 7 shows that the graph of tensile strength of the composites produced with untreated and treated date seed particles at different sodium hydroxide concentrations 1, 3, 5, 7 and 10 %. It was shown that increase in sodium hydroxide concentration led to an increase in tensile strength. The highest tensile strength was observed with 10 % at 28.86 MPa and the lowest was observed with 0 % at 23.15 MPa. This increase is attributed to the removal of cementing materials such as lignin, hemicellulose, and pectin capable of preventing bonding between the filler and the matrix. Alkaline treatment cleans up the filler by removing impurities which in turn improved the date seed particles/waste polypropylene interfacial adhesion. This agrees with the results reported by (Olcay & Kocak, 2021). Alkaline treatment results in freeing the hydrogen bonds making them more reactive, increased compatibility between the filler and the matrix as revealed by the FT-IR analysis.

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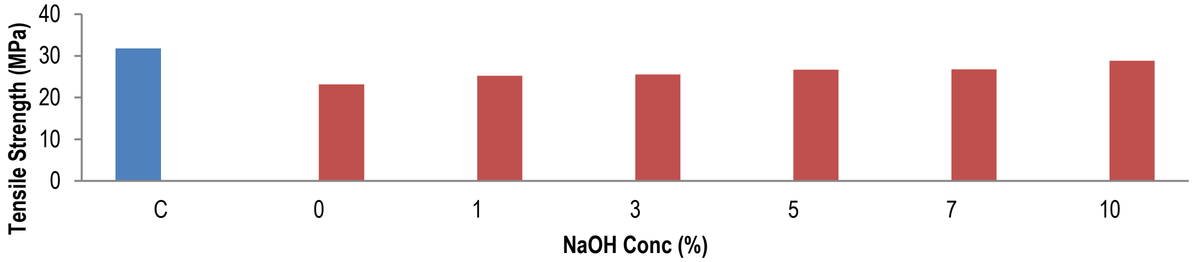

Figure 7: Tensile Strength of wPP / DSP composites against NaOH Concentration

Tensile Modulus
The tensile modulus results in Figure 8 show that the tensile modulus decreased with increased in sodium hydroxide concentration with those of the treated being lower than those of the untreated composites. The highest tensile modulus was 1.12 GPa at 0 % and the lowest was 0.81 GPa at 10 %. This could be related to the changes in the cellulose crystallinity index during treatment. The changes in the crystallinity of the cellulosic molecular structures of the treated filler is due to the partial conversion of cellulose I into cellulose II, producing more amorphous cellulose structure in the filler (Razali *et al.*, 2022).

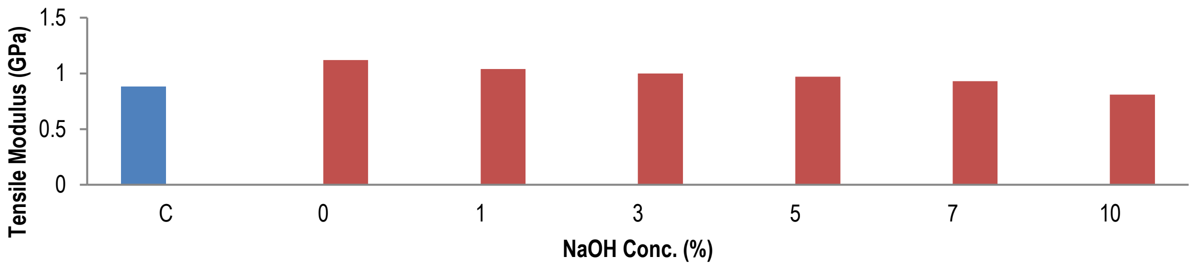

Figure 8: Tensile Modulus of wPP / DSP composites against NaOH Concentration

Elongation at Break
The elongation at break of the composites showed an increased with increase in sodium hydroxide concentration from 0-10 % (Figure 9). It was observed from the results that the EB of the treated composites were higher than that of the untreated composites. The highest EB was seen with 10 % at 172.55 % and the lowest was seen with 0 % at 139.62 %. This increase is due to the removal of impurities preventing bonding at filler/matrix interface. It could also be due to the reduction in the number of gaps at matrix/filler boundary regions which has also been observed by Olonisakin *et al.*,2022.

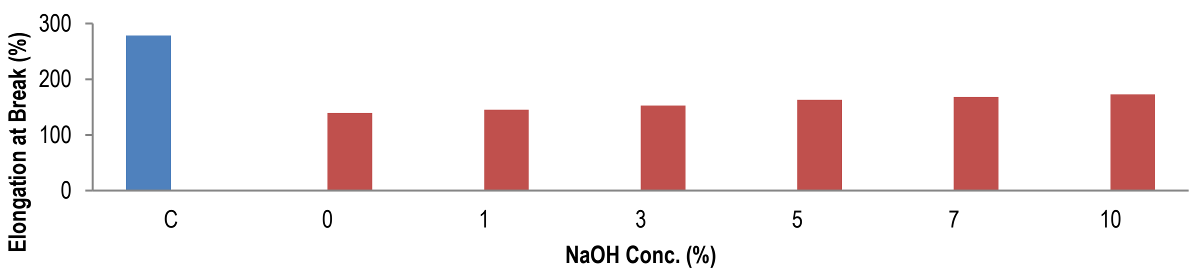

Figure 9: Elongation at break of wPP / DSP composites against NaOH Concentration

Flexural Strength
The flexural strength results as seen in Figure 10 showed an increase with increase in sodium hydroxide concentration. The highest flexural strength was obtained with 10% at 140.0 MPa while lowest was seen with 0% at 100.0 MPa. This increase in flexural strength is due to better interfacial adhesion between the hydrophobic matrix and the hydrophilic date seed particles as a result of increase in compatibility on the date seed particles. Sodium hydroxide treatment enhanced compatibility between filler and the matrix by removing free hydroxyl group at the filler cell wall rendering it more hydrophobic. The leaching of cementing materials and other organic extractives from the filler could be the reason for this increase as revealed by FT-IR analysis (Song *et al.*, 2023).

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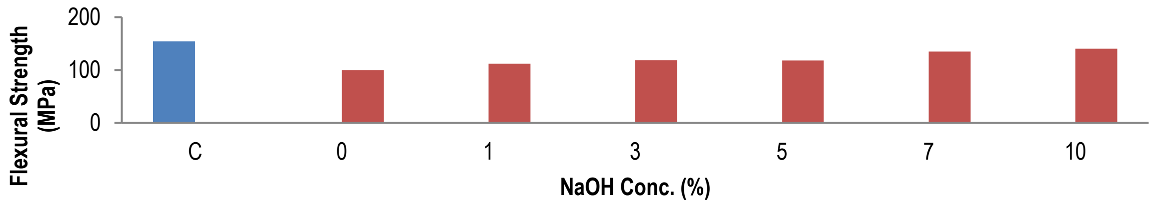

Figure 10: Flexural Strength of wPP / DSP composites against NaOH Concentration

Flexural Modulus

Figure 11 showed a result of flexural modulus decreased with increase in sodium hydroxide concentration. 0% was seen with highest flexural modulus at 1.22 GPa while the lowest was seen with 10% at 0.84 GPa. The decrease in flexural modulus could be due to deep pores at the cell wall of the date seed particles as a result of significant leaching of cementing materials (lignin, hemicellulose, pectin etc.) and or cell wall thinning (Teli & Terega, 2019) as confirmed by FT-IR analysis.

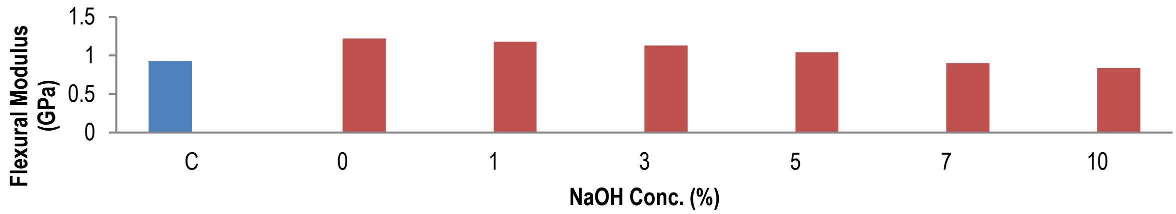

Figure 11: Flexural Modulus of wPP / DSP composites against NaOH Concentration

Impact Strength

The impact strength results in Figure 12 indicate an increase with increase in sodium hydroxide concentration. 10% had an impact strength value of 0.92 J/m^2 while 0% had impact strength of 0.7 J/m^2. Higher impact strength with 10% was attributed to the reduction of gaps/flaws at filler/matrix boundary region by sodium hydroxide treatment while the lowest impact strength with 0% was due to weaker interfacial interaction between the filler and the matrix. The impact strength for sodium hydroxide treated composites was higher than those of the untreated composites. This trend was also observed by Rajeshkumar *et al.*, 2021.

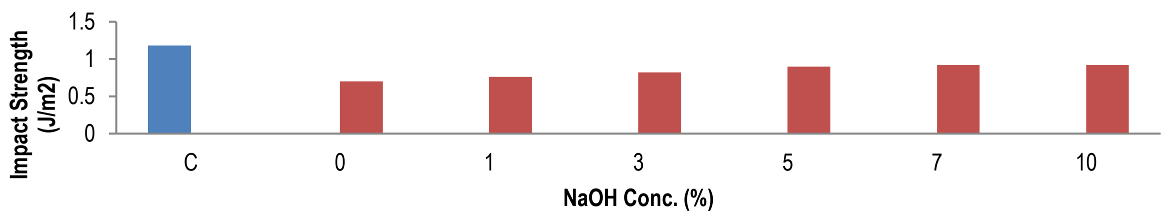

Figure 12: Impact Strength of wPP / DSP composites against NaOH Concentration

Hardness

Hardness results in Figure 13 show that the hardness for the treated composites increased with increase in sodium hydroxide concentration. The hardness value for 10% was 23.8HV while 0% had a hardness of 10.8HV. This increment was as a result of better bonding between the date seed particles and the polypropylene matrix yielding more compacted materials (Rajeshkumar, 2022).

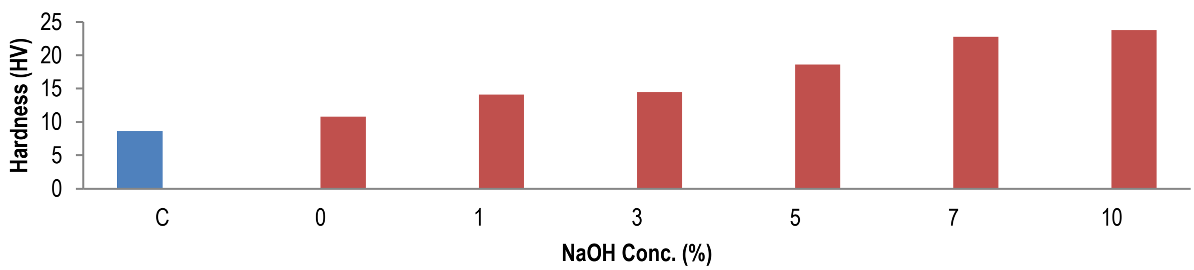

Figure 13: Hardness of wPP / DSP composites against NaOH Concentration

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Morphological Studies

Interfacial appearances of date seeds and waste polypropylene matrix are explained through scanning electron microscopy of the fractured surface of the composites as presented in Plate1. Plate II (b) shows agglomerations and clusters of the untreated date seeds. The de-bonding observed in this region may stem from the incompatibility between hydrophilic date seeds and hydrophobic polypropylene, leading to insufficient interfacial adhesion (Birniwa *et al*., 2023). This weakened interface results in reduced energy absorption during impact, diminishing the material's capacity to endure abrupt loads or impacts. The behavior is illustrated in Figure 12, where impact strengths for composites treated with sodium hydroxide were higher than those for untreated composites (Gholampour & Ozbakkaloglu, 2020). The limited interfacial adhesion might arise because untreated date seed particles possess more surface lignin and wax, hindering the infiltration of waste polypropylene into the particle core. The overall drop in tensile strength for the untreated composites may be attributed to stress concentration around the agglomerations of fillers, as depicted in Plate II (b), causing premature failure (Nurazzi, *et al.,* 2021). Conversely, the scenario differs for composites filled with treated date seeds, showing a superior dispersion of filler particles within the matrix.

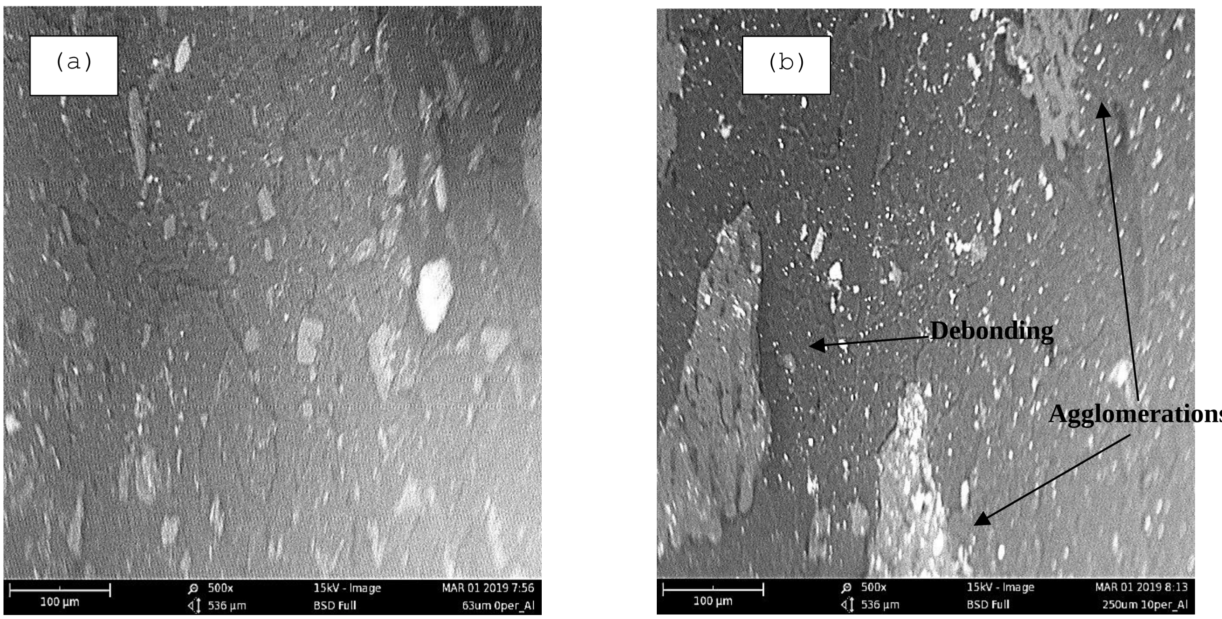

PLATE II: SEM photographs of (a) treated and (b) untreated waste-polypropylene/date-seeds particles (63 µm) x 500 magnification

CONCLUSION

Production of waste polypropylene/date seeds particle composites was successful, utilizing waste polypropylene as environmental control. Alkaline treatment using NaOH removed the superficial lignin and hemicellulose of the date seeds. Sodium hydroxide treatment also enhanced tensile strength, flexural strength, impact strength, elongation at break and hardness values of the composites. While tensile and flexural moduli decreased with an increase in sodium hydroxide concentration. Incorporating date seed particles into the polypropylene matrix has added value to the date seed instead of disposal, and the composites produced can be utilized where moderate mechanical properties are required e.g. car interiors, table tops and furniture.

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