

Effect of Chemical Treatment of Avocado Wood Flour (AWF) on the Properties of High Density Polyethylene (HDPE) for the Production of Natural Filler Polymer Composites

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ABSTRACT: This study investigated the effect of chemical treatment of Avocado Wood Flour on HDPE for the production of composites for marketable ventures. The wood flour was characterized in terms of cellulose, hemicelluloses, lignin and ash content. Sodium hydroxide, acetic acid and maleated polyethylene (MAPE) were used for surface treatment to enhance the compatibility between the filler and the polymer matrix. The mechanical and water absorption properties of the composites produced were compared to virgin polymer and composites without chemical treatment. It was evaluated that the effect of chemical treatment on the filler at varying concentrations of the filler in the HDPE matrix was significant in analyzing the mechanical, morphological and change in the structural properties of wood thermoplastic composites. Results indicated that composites with surface treatment had better mechanical properties and less water absorption than the untreated composites, which also inferred by SEM and FTIR analysis.

Key words: *Chemical Treatment, HDPE, Avocado Wood Flour, Composites, Mechanical Properties.*

1. INTRODUCTION

The usage and manufacturing of organic filler polymer composites are reigning today. This has made significant area of research interest due to the needs in the global market for enormous improve properties. Recently, the demand for this material is very high due to its usage in the global society for various purposes [1]. This is why researchers in Nigeria should utilise this opportunity to source for fillers with specified qualities to enhance organic fillers-polymer productions that can be commercialised as this can improve the dwindling economy as a result of over-dependency on oil revenue. Nigeria as a country is blessed with trees owing to its large vegetations where different natural fillers which cannot be found in any part of the world can be sourced.

Natural filler like Avocado wood is found in almost all the parts of Nigeria. The natural filler polymer composites derivable from the Avocado wood can be a tremendous addition to the production of roofing sheets, window frame, car bumpa, etc. No scholarly research has been carried out in the study of the ability of Avocado wood fillers in enhancing the properties of the polymer composites. The Avocado wood filler has a reasonable amount of cellulose content which makes it good enough for the production of polymer composites.

The use of natural filler in the production of polymer matrix composites has its advantages and disadvantages. The advantages include: good resistance to corrosion, enhance mechanical damping, improvement in toughness, good specific strength, good resistance to fire, outstanding fatigue strength, low cost etc., [2]. The disadvantages are: low compatibility between the polymer matrix and the filler, poor ability to convey stress from the matrix to the filler, poor water resistance on the composite and large water retention. The use of the appropriate compatibilizer or chemical treatment(s) of filler and matrix is a better way that should be encourage for reduction of the setback of compatibility [3,4]. Chemical treatment such as alkali (treating filler in sodium hydroxide or other alkali solution), acetylation (soaking in acetic acid solution and/or other compound with similar properties) and coupling agent (adding MAPE, MAPP to the filler etc) were introduced to improve on the compatibility of the composite [5]. A coupling agent is a compound that helps at the interface for proper adhesion between the filler and the matrix [6]. The coupling agent helps to react with hydroxyl groups of cellulose and functional groups of the matrix [8]. The importance of chemical treatment was to reduce lignin and

hemicelluloses from the organic fillers and improve cellulose content which enhances better mechanical properties of the composites [9].

High Density polyethylene is one the major thermoplastic material used in the production of thermoplastic composites. It has large tensile strength, can be used for products and packaging such as; milk jugs, detergent bottles, butter tubs, garbage containers and water pipes. It is also used to produce almost all plastic containers in the world. Throughout the world, the usage of this material has attained up to a volume above 30 million tons in the year 2007 [10].

2. EXPERIMENTAL PROCEDURE

2.1 Preparation of the Filler

The avocado wood was sourced in Federal Housing Estate Trans Ekulu in Enugu State of Nigeria. The natural fiber was sun dried for 14 days. It was crushed, ground to powder form, and was later sieved to 100 mesh.

2.2 Characterization of the filler in terms of cellulose, hemicelluloses, lignin and ash contents

The characterization of the fiber was carried at Divine Chemical and Analytical laboratory Nsukka, Enugu.

2.2.1 Determination of cellulose content of the filler

0.7g (C) of the air dried milled filler which was initially treated, and added to a 95% solution of nitric acid and ethanol mixture. The mixture was filtered and the residue was rinsed with warm water. It was later rinsed with ethanol to fully remove the residual acids. The residue was oven dried at 100°C to constant weight C₁. The test was repeated for three times and the average value was taken [11]. The percentage cellulose is calculated by:

$$\% \text{ Cellulose} = \frac{C_1}{C} \times \frac{100}{1} \quad (1)$$

2.2.2 Determination of hemicelluloses content of filler

0.7g (C) each of the air dried ground filler sample was mixed 0.5g sodium lauryl sulphate solution for 1 hour. The mixture was filtered and the residue rinsed with warm distilled water and ethanol. The residue was later oven dried to constant mass at 100°C for 8 hours. The constant mass was weighed which equals to neutral detergent fiber weight. The test was repeated for three times and the average value was recorded. Hemicelluloses content was determined as the difference in weight of neutral detergent fiber and the acid detergent fiber made from acid hydrolysis of the identical mass of sample. The test was repeated for three times and the average value was recorded [12].

$$\text{The percentage hemicelluloses was by} = \frac{NDF-ADF}{C} \times \frac{100}{1} \quad (2)$$

Where NDF is the neutral detergent fibre, ADF is the acid detergent fibre and C is the mass of sample.

2.2.3 Determination of lignin and ash content

0.7g (C) of the air dried ground filler samples was warmed with 5ml of 72% w/w H₂SO₄ solution for 4.5 hours in order for the cellulose and hemicelluloses to undergo hydrolysis. The suspension that left was filtered with a crucible and fully rinsed with boiled distilled water and ethanol to thoroughly take away acid content in it. The solid residue was dried at 105°C for 24hrs and weighed again to a mass (C₁). This residue that was left is called the acid detergent fiber. The residue was then moved to a dry porcelain crucible which was initially weighed and reheated to 600°C for 5hrs and then cooled. It was finally weighed to mass (C₂) after the mass of the crucible was deducted. The test was repeated for three times and the average value was recorded [13].

$$\text{The percentage Ash content is calculated using} = \frac{C_2}{C} \times \frac{100}{1} \quad (3)$$

$$\text{The Percentage lignin is calculated using} = \frac{C_1-C_2}{C} \times \frac{100}{1} \quad (4)$$

2.3 Preparation of High Density Polyethylene

The HDPE was produced from Indorama Petrochemical Limited Eleme, Port Harcourt; Rivers State. It was obtained at Awada, Onitsha in Anambra State of Nigeria.

2.4 Sourcing of Maleated Polyethylene

This was sourced from sigma-Aldrich Chemical Corporation.

2.5 Sourcing of Sodium Hydroxide and Acetic Acid

These reagents were obtained in Main Market, Enugu

2.6 Treatment of the Avocado Wood filler with Sodium Hydroxide

The wood flour was immersed in a 6wt% of NaoH solution at 16hrs. The flour was then rinsed with distilled water, filtered and dried for 10hrs.

2.7 Treatment of the Organic Filler with Acetic Acid

The flour was immersed in a solution of 6wt% NaoH for 16hrs and 4vol% acetic acid for 1hour. The flour was later washed with distilled water, filtered and sun-dried for 10hrs.

2.8 Treatment with Maleated Polyethylene (MAPE)

The treated filler with sodium hydroxide followed by acetic acid were added with 5wt% of maleated polyethylene.

2.9 Composite Productions

The avocado wood filler was filled in HDPE. The untreated and treated filler was mixed using HDPE. The avocado wood flour was filled at 5, 10, 15, 20, and 25% filler content. The different compositions of natural filler and the HDPE were injection molded using an injection molding machine at Ekenedilichukwu workshop, Onitsha. The composites were allowed to cool and cut into machine size for determination of mechanical and water absorption properties.

2.10 Tensile Testing of Composites

This test took place at the University of Nigeria Civil Engineering Workshop, Nsukka Enugu State of Nigeria using universal tensometer BSS1610 model no 8889 manufactured by Hounsfield tensometer limited. This test was carried using ASTM D638. The equipment has a cross-head speed between 10-100cm. The dimensions of tensile test sample size for ASTM used were 3.2mm x 19mm x 160mm. The samples were inserted into the gripping chucks of the tensometer and placed firmly. A continuous load was applied to the sample and till fracture occurs. The ultimate tensile strength, elongation and modulus were calculated.

2.11 Flexural testing of composites

The equipment used for this was universal tensometer used in tensile test above. The dimension of flexural test sample size for ASTM D790 used were 3.2mm x 19mm x 300mm. The test sample was placed and fixed firmly on 3-point support span. A continuous load was applied in the centre of the sample until fracture and constant deflection occurred. The test was stopped at this condition. The flexural strength and modulus were obtained.

2.12 Hardness testing of composites

The equipment used for this test was also universal tensometer. The dimension of hardness sample size for ASTM used was 3.2 mm × 19mm mm×19mm. The specimen was clamped into the machine. A steel Brinell bulb of diameter of 10mm was picked to obtain the indentation for this test. The sample was fixed in the equipment after

which the indentation test connections were placed in the tensometer testing machine. The sample was subject to a specific load in the machine, the indentation which corresponded to the depth of indentation was measured. The Brinell hardness was calculated by applying this relationship:

$$BHN = \frac{2F}{\pi D \left[D - \sqrt{D^2 - d^2} \right]} \quad (5)$$

Where BHN is the Brinell hardness numbers (Pa), D is the diameter of the steel ball (m), d is the depth of indentation (m) and F is the specific load (N).

2.13 Testing Impact Strength of composites

The equipment used for this test was impact tester machine located at the University of Nigeria, Nsukka, Mechanical Engineering Department Workshop, Enugu State.

The dimension of impact testing specimen size for ASTM D256 used was 3.2 mm × 19 mm × 80 mm. The specimen was clamped into the machine. The pendulum from the impact tester was released and allowed to strike through the specimen. The impact strength was determined.

2.14 FTIR Test

The FTIR-8400S Fourier transform infrared spectrometer was used for the test. The FTIR properties was analysed at the National Research Institute for Chemical Technology, Zaria. 50 mg of potassium bromide salt and 1.5 mg of the composite samples were weighed. The sample was ground to a fine particle size using a mortar and pestle. The sample was later mixed with potassium bromide salt. The mixture was passed into potassium bromide die to obtain a potassium bromide pellet. The pellet was put into the machine (FTIR) and scanned at a range of 500–4500 nm. The Spectral was exhibited on the Screen of the FTIR spectrometer, which was used to determine organic compounds and the functional groups in the composite.

2.15 SEM Test:

This analysis was carried in at the National Research Institute for Chemical Technology, Zaria using scanning electron microscope. The capacity used was 15 kv with image setting at 1000x magnification. 1 g of the sample was put into the sputter coater machine after 5 second conductivity property was obtained. The sample was later placed on a charge reduction sample holder into the machine and it was allowed to maintain for 10 seconds. The rotary knob on the machine was adjusted to clear image was captured which was moved into an electronic image mode, which gave the morphology of the sample, this morphology was saved in the machine which finally gave the SEM picture.

2.16 Water Absorption Test

The test was carried out at Divine Chemical and Analytical Laboratory, Nsukka, Enugu state. The composite sample was cut to dimension of 3.2 mm × 19 mm × 19 mm oven dried for 50°C for 30 minutes [14] according to ASTM specification and initially weighed (B_1). The sample was immersed in water for 12 weeks at room temperature using ASTM D96 – 06 and weighed again (B_2) after the left over water on the surface was removed.

The percentage of water absorption was calculated using the formular:

$$M = \frac{B_2 - B_1}{B_1} \times \frac{100}{1} \quad (6)$$

Where M is the percentage of water absorption, B_1 is the initial weight of the sample and B_2 is the weight after immersing in water.

3. Results and Discussion

3.1 Characterizations of the Filler

Table 1: Characterization result of the organic filler content

Filler	Cellulose (%)	Hemicelluloses (%)	Lignin (%)	Ash (%)
Avocado Wood	53.293	31.25	7.11	2.043

Table 1 shows the characterization result of the filler content of the avocado wood (AWF). From the result in Table 1, it can be observed that 53.293% cellulose, 31.25% hemicelluloses, 7.11% lignin and 2.043% ash content. This showed that cellulose content in the filler is reasonable enough for the production of polymer–filler composites due to its cellulose content.

3.2: Effect of Filler Content on the Properties of the HDPE/AWF Composite with Untreated and Treated

AWF

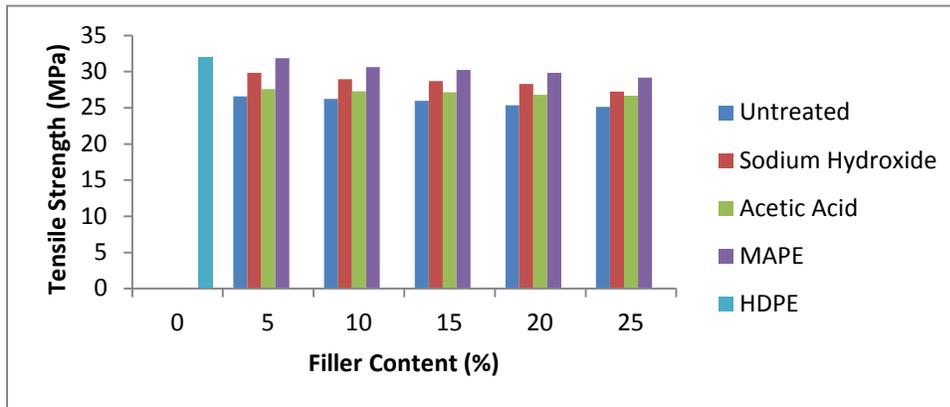


Fig. 1: Effect of filler content on the tensile strength of HDPE/AWF composites.

Fig. 1 shows the effect of filler content on tensile strength of HDPE/AWF with untreated and treated AWF. It can be noted that the increase in filler content decreased the tensile strength. This was due to increased in the interface area which reduced adhesion between the filler and the polymer phase [15]. Treating the filler with sodium hydroxide helped to activate the hydroxyl groups in the filler for proper adhesion with HDPE which further improved the strength of the composite [16,17]. Also, further treating with acetic acid also reduced the strength which may be due to pH. In Fig. 1, an increase in filler content from 5 to 25% decreased tensile strength of untreated AWF from 26.6 to 25.12MPa; followed by sodium treated AWF decreased from 29.83 to 27.23 MPa; acetic acid treated AWF decreased from 27.58 to 26.68 Pa and MAPE treated AWF decreased from 31.87 to 29.17MPa.

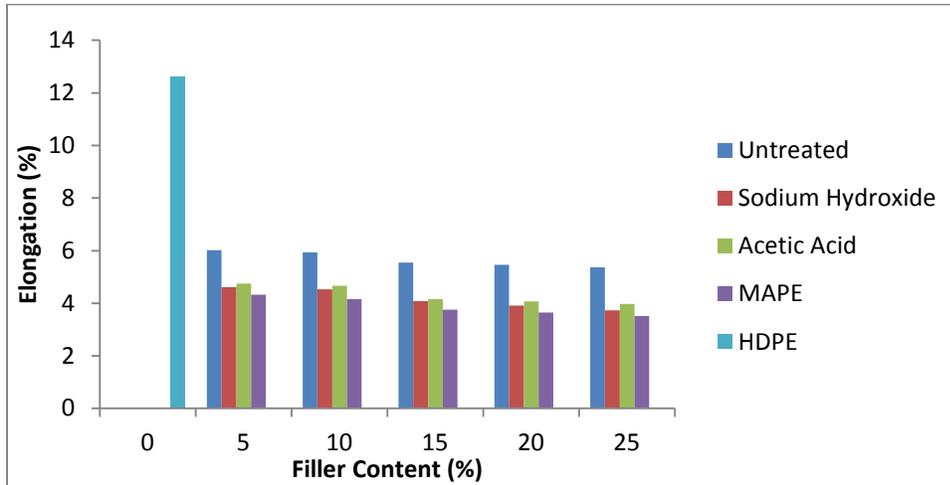


Fig. 2: Effect of filler content on the elongation of HDPE/AWF composites.

Fig. 2 shows the effect of filler content on the elongation of HDPE/AWF with untreated and treated AWF. It can be noted that the increase in filler content decreased the elongation. This was due to the incapability of the filler to allow stress to move the polymer matrix to the filler. This is in agreement with the previous authors [18, 19, 17, 14]. Also, addition of filler to the polymer matrix decreased the ductility of the polymer matrix-filler composite [14]. In Fig. 2, an increase in filler content from 5 to 25% decreased elongation of untreated AWF from 6.02 to 5.37 %; followed by sodium treated AWF decreased from 4.61 to 3.73%; acetic acid treated AWF decreased from 4.75 to 3.98 % and MAPE treated AWF decreased from 4.32 to 3.52%.

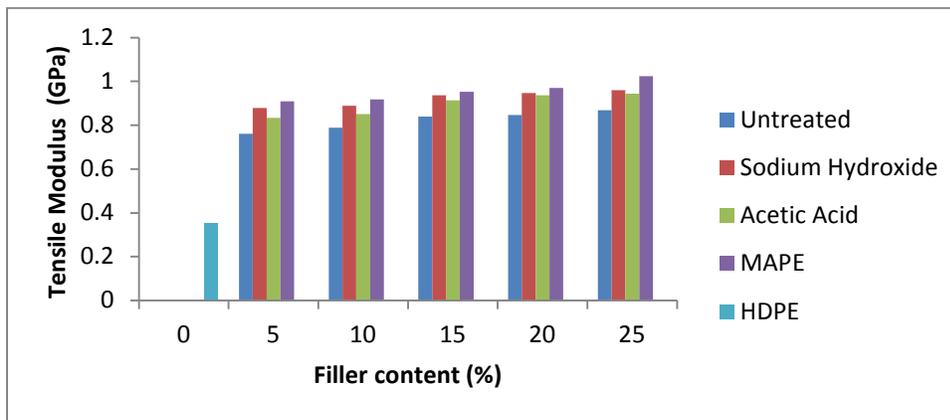


Fig. 3: Effect of filler content on the tensile modulus of HDPE/AWF composites.

Fig. 3 shows the effect of filler content on the tensile modulus of HDPE/ AWF with untreated and treated AWF.

It can be noted that the increase in filler content increased the tensile modulus. This was due to when material undergoes tensile force, there are slight separation of micro-space that are generated which prevent stress propagation at the interface, this obstruction increased addition of filler to the polymer matrix, decreasing the ductility of the polymer matrix-filler composite and increased the stiffness of the material [20,14]. The treated composites showed better result due to influence of these chemical treatments which improved the phases between the filler and polymer matrix that maintained property of the material. These related to other researchers work [20, 14, 21, 22, 23]. In Fig. 3, an increase in filler content from 5 to 25% increased tensile modulus of untreated AWF from 0.761 to 0.869GPa; followed by sodium hydroxide treated AWF increased from 0.878 to 0.96GPa; acetic acid treated AWF increased from 0.833 to 0.944GPa and MAPE treated AWF increased from 0.909 to 1.023GPa.

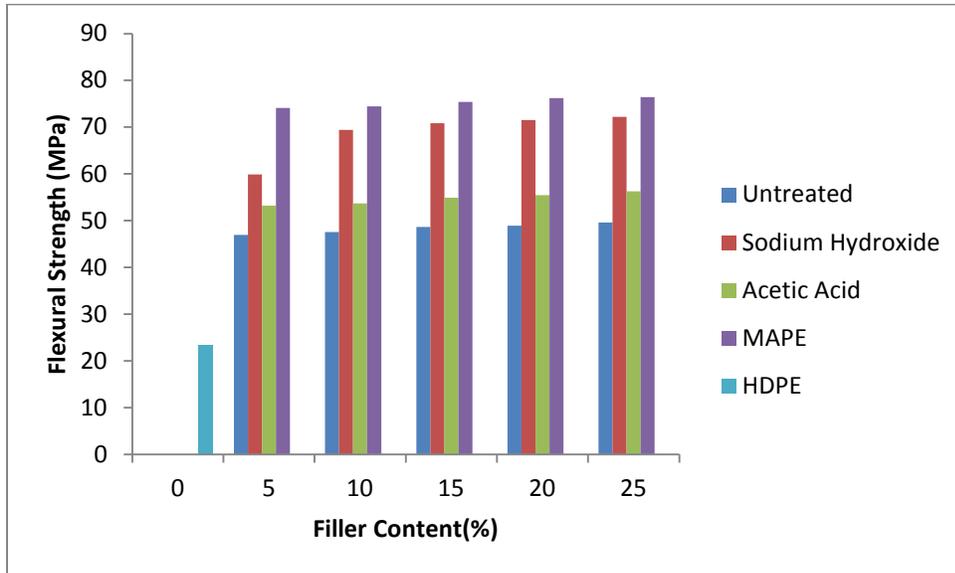


Fig. 4: Effect of filler content on the flexural strength of HDPE/AWF composites.

Fig. 4 shows the effect of filler content on the flexural strength of HDPE/AWF with untreated and treated AWF. It can be noted that increased in filler content increased the flexural strength. This was as a result of the enhancement of the bond between the filler and the polymer matrix. These results were in agreement by other researchers [24, 31, 25]. In Fig. 4, an increase in filler content from 5 to 25% increased flexural strength of untreated AWF from 46.95 to 49.62MPa; followed by sodium hydroxide treated AWF increased from 59.87 to 72.22MPa; acetic acid treated AWF increased from 53.18 to 56.27MPa and MAPE treated AWF increased from 74.11 to 76.43MPa.

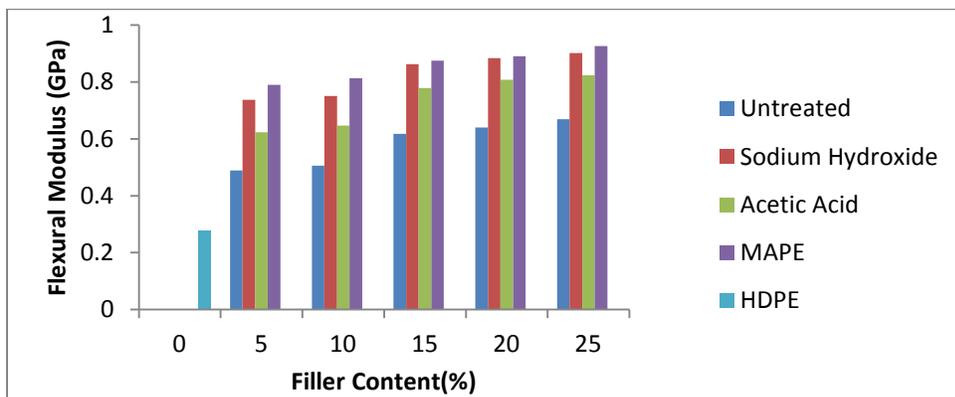


Fig. 5: Effect of filler content on the flexural modulus of HDPE/AWF composites.

Fig. 5 shows the effect of filler content on the flexural modulus of HDPE/AWF with untreated and treated AWF. It can be noted that increase in filler content increased the flexural modulus. This was as a result of the enhancement of the bond between the filler and polymer matrix that led to increase in the stiffness of the composites due to chemical modification of the filler. These results were related by other authors [4, 31, 25]. In Fig. 5, an increase in filler content from 5 to 25% increased flexural modulus of untreated AWF from 0.489 to 0.669GPa; followed by sodium hydroxide treated AWF increased from 0.737 to 0.901GPa; acetic acid treated AWF increased from 0.623 to 0.823GPa and MAPE treated AWF increased from 0.738 to 0.926GPa.

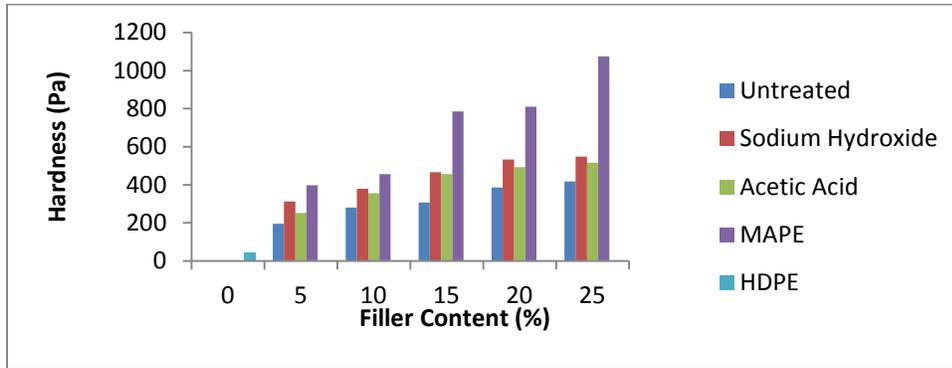


Fig. 6: Effect of filler content on the hardness of HDPE/AWF composites.

Fig. 6 shows the effect of filler content on the hardness of HDPE/AWF with untreated and treated AWF. It can be noted that the increase in filler content increased the hardness. This was as a result of the enhancement of the bond between the filler and polymer matrix that led to increase in the stiffness of the composites due to chemical modification of the filler. These results were related by works done by other researchers [24, 31, 25]. In Fig. 6, increased in filler content from 5 to 25% increased hardness of untreated AWF from 196 to 418Pa; followed by sodium hydroxide treated AWF increased from 313 to 548Pa; acetic acid treated AWF increased from 252 to 517 Pa and MAPE treated AWF increased from 398 to 1074Pa.

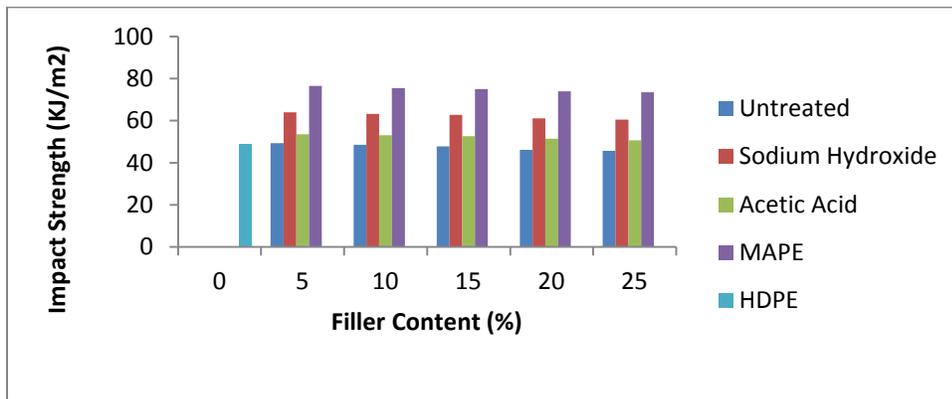


Fig. 7: Effect of filler content on the impact strength of HDPE/AWF composites.

Fig. 7 shows the effect of filler content on the impact strength of HDPE/AWF with untreated and treated AWF. This may be as a result of the organic filler which is very rigid serves as stress concentrators inside the polymer matrix which decreased crack initiation energy of the composites and impact strength [26,27]. It is also due to weak adhesion between polymer matrix and the organic filler [5]. The work relates to other authors [26, 5, 27]. In Fig. 7, an increase in filler content from 5 to 25% decreased impact strength of untreated AWF from 49.29 to 45.77 KJ/m²; followed by sodium treated AWF decreased from 64 to 60.52KJ/m²; acetic acid treated AWF decreased from 53.6 to 50.68 KJ/m² and MAPE treated AWF decreased from 76.48 to 73.46 KJ/m².

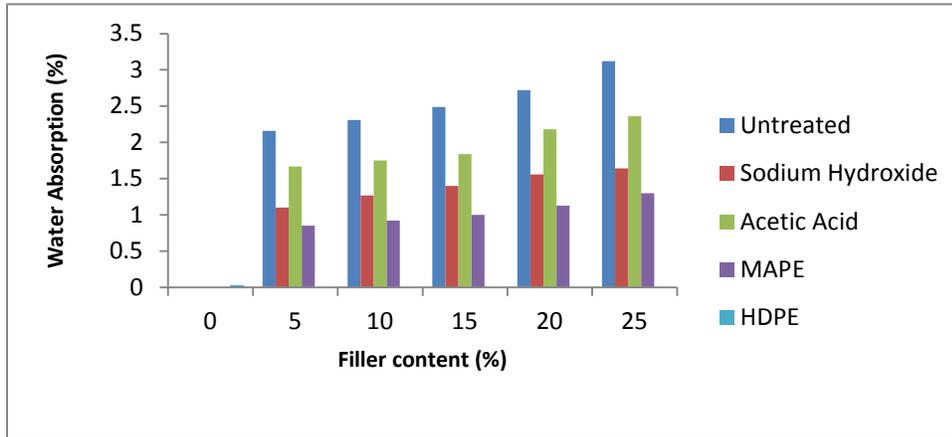
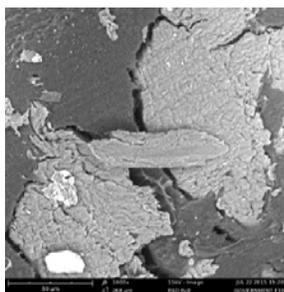


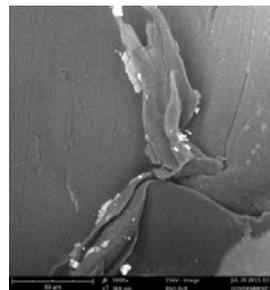
Fig. 8: Effect of filler content on the water absorption of HDPE/AWF omposites at 12 Weeks.

Fig. 8 shows the effect of filler content on the water absorption of HDPE/ AWF with untreated and treated AWF at 12 weeks. It can be noted that the increase in filler content increased the water absorption. This was as a result of the hydrophilic cellulose of the filler in the composites [9]. Treated filler showed less absorption of water in the composites due to chemical treatment to strengthen the bonds between the filler and the polymer matrix [29]. Also, treated filler showed reduction of water absorption in the composites due to free hydroxyl groups of the filler surfaces and anhydride groups in the MAPE [28]. In Fig. 8 an increase in filler content from 5 to 25% increased water absorption of untreated AWF from 2.16 to 3.12%; followed by sodium hydroxide treated AWF increased from 1.1 to 1.64%; acetic acid treated AWF increased from 1.67 to 2.36% and MAPE treated AWF increased from 0.85 to 1.3%.

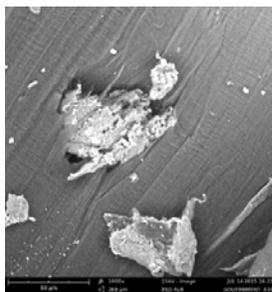
3.3. SEM Morphology of HDPE/AWF Treated and Untreated Composites



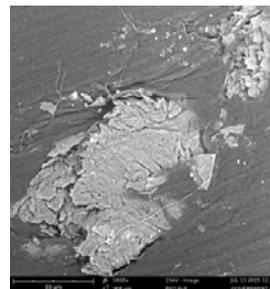
(a)



(d)



(b)

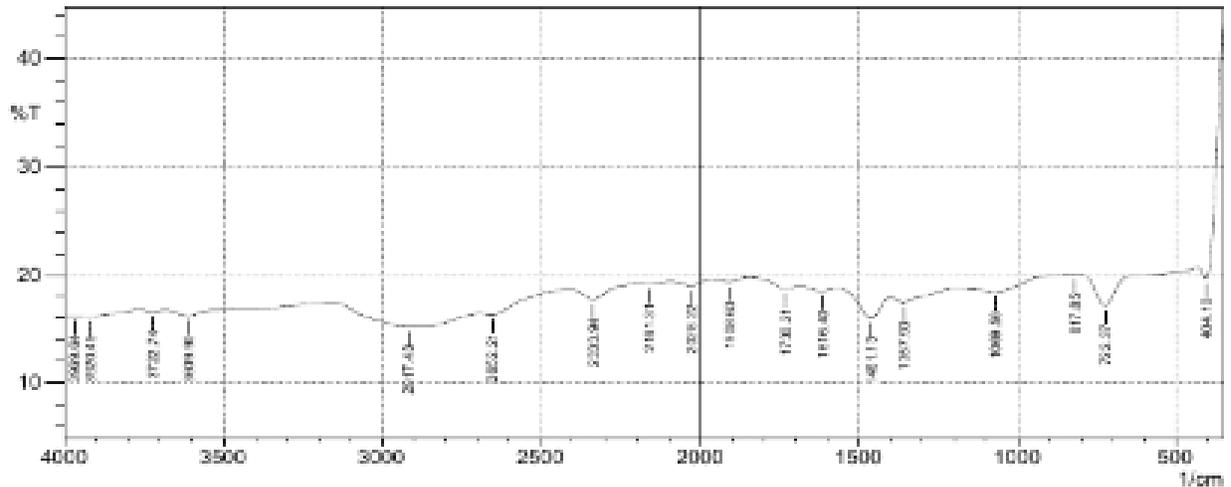


(c)

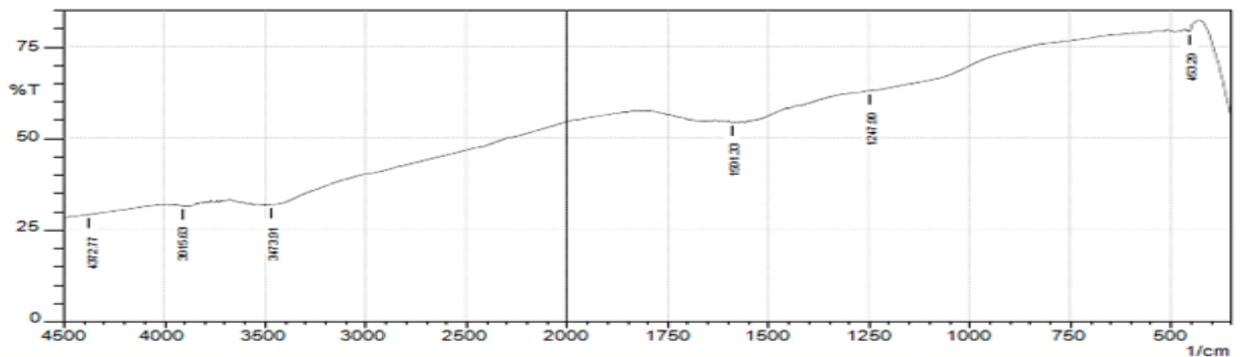
Fig. 9: SEM Morphology of HDPE/AWF Composite for: (a) Untreated (b) Treated with Sodium hydroxide (c) Acetic acid (d) Maleated polyethylene.

Fig. 9 (a-d) shows the SEM morphology of HDPE/AWF untreated, HDPE /AWF treated sodium hydroxide, HDPE/AWF treated acetic acid and HDPE/AWF composites treated maleated polyethylene. In Fig. 9 (a), indicated few white spotted filler particles, slight clear, and cracks which resulted in gaps in the surface of SEM morphology. These gaps led to feeble surface interaction between the AWF and HDPE matrix. In Fig. 9(b), revealed that there is a change in the SEM morphology as the filler is treated with sodium hydroxide. This led to enhanced surface with few white spotted types of filler on SEM morphology than the untreated composite. This clear morphology enhanced the adhesion of treated filler in the HDPE of the composite. In Fig. 9 (c), there is an alteration in the SEM morphology surface of the filler on HDPE due to the addition of acetic acid which led to the less clear surface than Fig. 9(b). This also led to increase in the dispersion of the particles of the filler in HDPE matrix. In Fig. 9(d), showed that the addition of MAPE gave more clear SEM morphology in the HDPE matrix. There was improvement in the bonding between the HDPE and the filler as a result of the addition of maleated polyethylene in the AWF. The white spotted on the surface of the composite which may affect on the properties the material. The treated filler showed improvement in the SEM morphology than untreated filler due to modification of the interface between the filler and HDPE matrix as a result of chemical treatment.

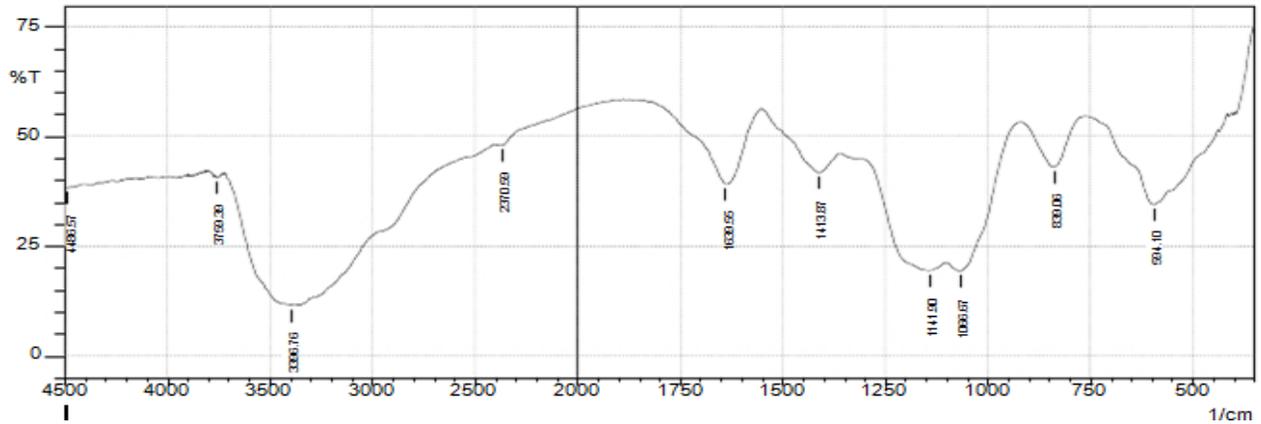
3.4 FTIR Analysis of HDPE/AWF Treated and Untreated Composites



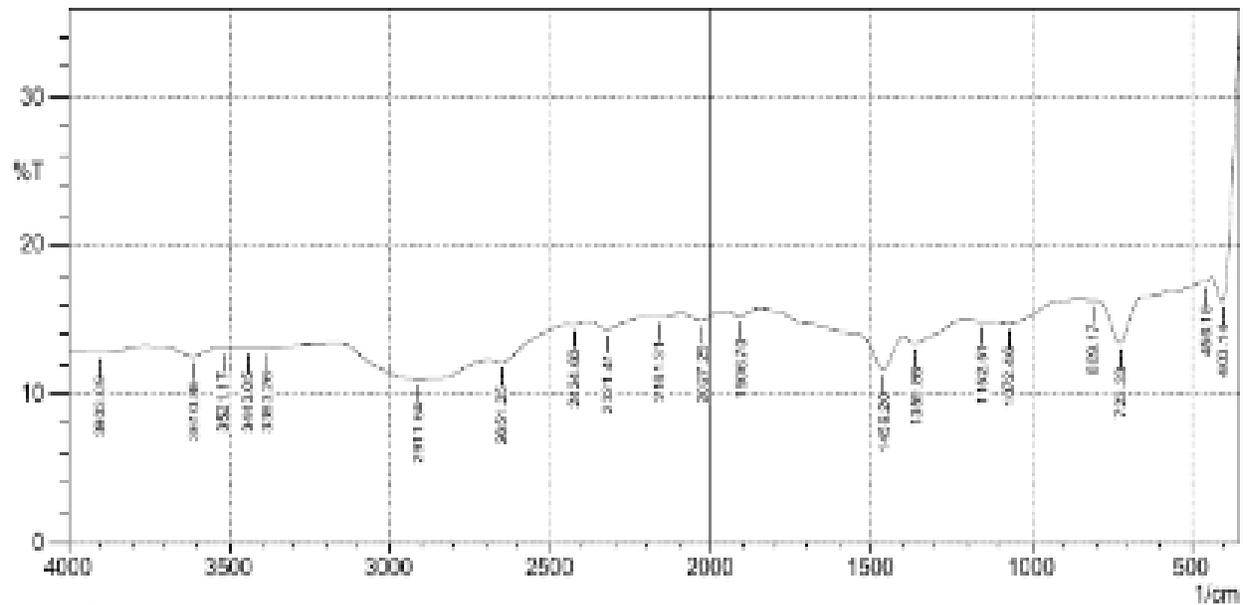
(a)



(b)



(c)



(d)

Fig. 10: FTIR of HDPE/AWF Composite for: (a) Untreated (b) Treated with Sodium hydroxide (c) Acetic acid (d) maleated polyethylene.

The FTIR spectra of HDPE/AWF untreated, HDPE, /AWF treated sodium hydroxide, HDPE/AWF treated acetic acid and HDPE/AWF composites treated MAPE as shown in Fig. 10(a-d). The different peaks in the FTIR spectra of the composites were obtained in the FTIR library [30]. In Fig. 10(a -d), the region of 4486.57 to 3383.26cm^{-1} is associated to the stretching of hydroxyl group of alcohol and phenol in the cellulose. The hydroxyl group stretching of carboxylic acid at the band of 2917.43cm^{-1} and 2651.25cm^{-1} in the filler. The peak of 2424.6 to 2161.31cm^{-1} is characterized by phosphorus acid and ester of the hydroxyl group stretching. The stretching of N-H and hydroxyl groups of primary amide centered at 2028.22cm^{-1} and 2027.25cm^{-1} . The overtone and combination bands of substitution of benzene ring aromatic compounds was consigned to 1908.63cm^{-1} and 1906.7cm^{-1} . The peak at 1730.21cm^{-1} correspond to aliphatic C=O stretching of esters. The stretching C=H group of alkenes is the properties of the band at 1639.55cm^{-1} , 1630.92cm^{-1} and 1616.4cm^{-1} which shown the present of HDPE. The peak at 1591.33cm^{-1} is assigned to C=N and C=C stretching of pyridine. The C=C skeletal vibration stretching of aromatic disubstituted benzene is at band of 1461.13cm^{-1} and 1459.2cm^{-1} . The band at 1413.87cm^{-1} is allocated to N=N stretching of azo compound. 1359.86cm^{-1} and 1357.93cm^{-1} are features of aromatic nitro compound of NO_2 symmetric stretching. The aliphatic C-O stretching of esters is related to the region of 1247.99 to 1141.9cm^{-1} . The region at 1072.46 to 1066.67cm^{-1} gave the presence of C-O stretching of alcohol and phenols. The C-H out-of-plane

bending at peaks of 839.06cm^{-1} to 722.37cm^{-1} which is characterized by alkenes present in HDPE. The C-Cl stretching of alkyl halide is at the band of 594.1cm^{-1} to 403.14cm^{-1} . In Fig. 10(a), it is characterized by a carbonyl group at 1730.21cm^{-1} which vanished in Fig. 10(b-d). This was due to removal hemicelluloses and lignin which reduced the bonding strength of the composites.

Conclusion

Surface treatment of AWF has enhanced the mechanical, water absorption, morphology and structural properties of the filler on the HDPE. From the results, MAPE treated filler demonstrate the highest mechanical properties and the least water absorption capacity. This means that treated filler showed better properties than untreated filler in the composites production.

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