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	RESEARCH
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Mechanical, Spectroscopic and Micro-structural Characterization of Banana Particulate Reinforced PVC Composite as Piping Material

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ABSTRACT

A banana particulate reinforced polyvinyl chloride (PVC) composite was developed with considerabley low cost materials having an overall lightweight and good mechanical properties for potential application as piping material. The specimen composite material was produced with the banana (stem) particulate as reinforcement using compression molding. Results showed that density and elastic Modulus of the composite decreases and increases respectively with increasing weight fraction of the particulate reinforcement. The tensile strength increased to a maximum of 42 MPa and then decreased steadily. The composition with optimum mechanical property (42 MPa) was determined at 8, 62 and 30 % formulation of banana stem particulates (reinforcement), PVC (matrix) and Kankara clay (filler) respectively with corresponding percentage water absorption of 0.79 %, Young's Modulus of 1.3 GPa, flexural strength of 92 MPa and density of 1.24 g/cm3. Fourier Transform Infrared (FTIR) analysis of the constituents showed identical bands within the range 4000-1000 cm-1 with renown research work. Scanning Electron Microscopy (SEM) result showed fairly uniform distribution of constituents' phases. X-Ray Fluorescence (XRF) confirms the Xray diffraction (XRD) result of the presence of minerals of kaolinite, quartz, rutile and illite in the kaolin clay. Comparison with conventional piping materials showed the composite offered a price savings per meter length of 84 % and 25 % when compared with carbon steel and PVC material.

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1. INTRODUCTION

Conventional steel pipes used in the petroleum industries are plagued by high cost of maintenance, corrosion and lower life cycles. Metallic pipe corrosion costs the world

approximately \$20 billion a year. The total annual cost of corrosion in the oil and gas industry is estimated at \$1.372 billion, with \$589 million representing pipeline and facility costs, downhole tubing expenses consuming \$463 million and \$320 million capital

expenditures for corrosion control [1]. Research in piping material is very significant as the networks of pipes in the US, Europe and Russia run to about 1.200,000 kilometers [2].

The use of composite pipe is expected to greatly reduce the economic losses (due corrosion and high cost of maintenance) and provides new investment opportunities. A composite material primarily consists of matrix and reinforcement and in addition may contain a third component referred to as 'filler'. The filler is mixed with the matrix during fabrication and may necessarily improve the mechanical properties rather aspects of some desired considerations. Examples of fillers are hollow glass microspheres (for reduced weight), clay or mica particles (for reduced cost) and carbon particles (for protection ultraviolent radiation). Fillers in essence add an important dimension to the design flexibility we have in composite today. However, other literatures use fillers and reinforcements interchangeably to mean the same thing. Application of composite are found in the automotive, aerospace, marine, architectural structures and some consumer products such as golf clubs, skis and tennis rackets [3].

Several works have been carried out on development and characterization of composites for various application. Samotu et al. [4] used carbonized palm kennel shell (CPKS) particulate and iron fillings with recycled empty water sachet polyethylene and developed polyethylene/palm kernel shell-iron composite for automotive application. Bello et al. [5] reviewed the potential of epoxy resin in the production of composite material. The benefit of using epoxy resin is attributed to its good chemical, thermal, electrical, dimensional stability and mechanical property. Hassan et al. [6] developed an eggshell particulate reinforced polyester composite with the eggshell in uncarbonized and carbonized state for application in the electronic, auto and building industries.

Atuanya *et al.* [7] investigated the suitability of the use of recycled low density polyethylene (RLDPE) for potential application in wood board manufacturing. Madakson *et al.* [8] characterized coconut ash as a constituent for metal matrix composite for potential application in automobile sector. Chattopadhyay *et al.* [9]

studied the effect of 40 mm loading and resin modification on the physical, mechanical, thermal and morphological properties of composites evaluated using SEM, TGA and FTIR.

Demand for engineering material with low density, high specific property, minimal corrosivity and low cost are on the increase. Materials used in this research work were chosen with respect to availability, resistance to corrosion, cost and physical property (weight). Kankara kaolin clay is in abundance in Kankara, Katsina state of Northern Nigeria. The clay is available and accessible in most market in Northern Nigeria. Naturally clay types are corrosion resistant. The clay is very cheap as a 50 kg bag costs about N2.000 (\$10). The clay is considerably less in density when compared with calcium carbonate and aluminum (both having densities of 2.7 g/cm³). Kaolin is one of the most abundant minerals in soil and sediments and is a common weathering product of tropical and sub-tropical soils [10,11].

Natural and synthetic constituents are used extensively in development of composite Natural reinforcements material. advantages over synthetic reinforcements as a result of the natural alignment of the carboncarbon bonds and also its significant strength, stiffness [12], low density, low cost and biodegradability thev offer. Natural reinforcements also provide specific properties, better structure insulating properties, and low energy consumption during their growth or processing [13,14]. Natural fibres are in great abundance in Nigeria from variety of sources. Most of the fibres are usually obtained free or at a very low cost. Polyvinyl chloride (PVC) is a widely used plastic, one of the most valuable products of the chemical industry and the second-largest thermoplastic commodity produced worldwide after polyethylene [15].

In this study, Nigerian banana stem particulate was used as the reinforcement in the thermoplastic poly vinyl chloride (PVC) matrix. It was alkali treated to improve fibre matrix interaction of the produced composite. The composite is a three-constituent composition consisting PVC as matrix, banana stem as reinforcement and *Kankara* kaolin clay as corresponding filler. Reinforcement and matrix are varied while the kaolin clay was kept constant.

Inclusion of the filler was to reduce cost as it is cheaper than PVC. Compression molding was used to produce the composite samples.

The research work seeks to develop the composite as piping material that can have a potential application in the oil industry (distribution pipe networks) and household water piping application. The piping material constituents were selected to minimize the effect of corrosion and weight compared to conventional steel pipes used in the oil industry.

2. EXPERIMENT

The experiment carried out in this work is detailed as follows.

2.1 Materials

Materials were selected based on availability, weight and corrosion resistance. Materials used are *Kankara* kaolin clay (200 g), Polyvinyl chloride (PVC, 500 g), Banana stem (200 g), Sodium hydroxide (NaOH), and distilled water (5 litres).

2.2 Preparation

The banana stem was cleaned and dried in the sun. The fibres were then manually removed by scrubbing on a rough surface and then cleansed with 1.5M sodium hydroxide in accordance with Kalia *et al.* [16] to enhance the fibre-matrix interface adhesion and later on dried in the sun [14,17]. The fibres were then ground and sieved with a sieve size of $130\mu m$. The *Kankara* clay was also sieved with same sieve size.

Table 1. Composition of constituents by weight.

	Compositions (g)			
Samples	Banana particulate	PVC	Kankara Clay	
Sample 1	0	44.0	11.0	
Sample 2	4.4	39.6	11.0	
Sample 3	8.8	35.2	11.0	
Sample 4	13.2	30.8	11.0	
Sample 5	17.6	26.4	11.0	
Sample 6	22.0	22.0	11.0	

Density of the banana particulate and Kankara kaolin clay (in powdery state) were determined using PVC [18] as the reference with known

standard true density of 1.35 g/cm³ in accordance with Dan-asabe *et al.* [19]. These were determined as 0.6 and 1.8 g/cm³ for the banana particulate and kaolin clay respectively. The composition of banana particulate and PVC were varied. The particulate was varied from 0 %, 8 %, 16 %, 24 %, 32 % and 40 % for samples 1 – 6 respectively. PVC was varied accordingly from 80 %, 72 %, 64 %, 56 %, 48 % and 40 % respectively. The Kankara kaolin was kept constant at 20 %. *The* composition of the constituents by weight is given in Table 1.

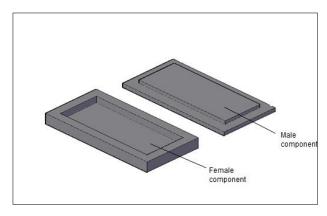


Figure 1. Composite male and female part of the mold.

The shape of the mould was fabricated in such a way that after hot compression the composite will be reduced to half its initial volume to ensure excellent compaction (devoid of pores between the constituents) as shown in Fig. 1. Material used for the mold is medium carbon steel. The size of the groove for the female component is 100x40x10 mm. The size of the male protrusion part is 99x39x5 mm. Each sample was put into the female mold to fill it and the excess put off.

2.3 Compression moulding process

This was carried out with Carver-3851 compression machine. Each sample was pressed at a temperature of 250 °C and a compression pressure of 20.7 MPa for 20 minutes. This temperature was used because preliminary trials with temperatures above it produced burnt product and temperatures below it produced less compacted product. The compression pressure (20.7 MPa) was the maximum pressure reached for the first test sample (reducing mold volume by half) and was adhered to for the remaining samples. Samples

obtained were cooled and machined in preparation for mechanical, microscopic and spectroscopic tests.

3. CHARACTERIZATION

The characterization tests carried out in this work are detailed as follows.

3.1 Spectroscopic analysis

FTIR was carried out on the banana particulate constituent at National Research Institute of Chemical Technology (NARICT), Zaria, Kaduna State, Nigeria. Samples specimen of approximately 2 grams (1 % particulate specimen and 99 % KBr were mixed) was pressed to about 12MPa and then subsequently placed in the Perkin Elmer Frontier FTIR machine. The result gives the FTIR spectra of the sample from 400 to 4000 cm⁻¹.

XRD analysis of the *Kankara* clay was carried out at the Defence Industries Corporation (DIC) in Kaduna, using the Pananalytical BV–XRD machine. The sample quantity of approximately 2 grams was formed into pellets and placed into chamber of the machine. The sample was rotated at approximately an hour and a half of the angular speed of the slit to maintain a contact angle between the incident and the reflected beam [20]. The machine provides the phase distribution of the clay recorded from Bragg's angle of 10° to 70° .

XRF analysis of the *Kankara* clay was carried out in the Defence Industries Corporation (DIC) in Kaduna using Mini Pal4 ED–XRF. The sample quantity of approximately 2 grams was formed into pellets with a hydraulic press and placed into chamber of the machine and sealed [21]. The machine was allowed to run for 1000 sec at a voltage and current of 30 KV and 50 μA respectively.

3.2 Physical and mechanical

The density of the composites was determined by measuring its respective mass and volume. Sample specimens of dimensions 20x20x5 mm were produced for the test. The mass was determined with the aid of a computerized weight balance machine to an accuracy of four decimal places. The volume of each sample was

found using Archimedes' principle. Water absorption test was carried out according to ASTM D570 [22] with oven-dried test specimen of dimension 76X25x5 mm immersed in water at ambient temperature for 24 hours until equilibrium. The specimen was removed and patted dry with a cloth (lint free) and then weighed using a digital weighing balance. The dry weight before $(W_{initial})$ immersion and the weight after (W_{final}) immersion were noted. The water absorption was determined as follows:

$$W = \frac{W_{final} - W_{initial}}{W_{final}} (\%)$$
 (1)

where $W_{initial}$ =initial weight before immersion and W_{final} =final weight after immersion.

Elastic Modulus was determined using an Electronic Tensometer ER–3 according to ASTM E8 standard [23]. Sample specimen dimensions of 120x8x5mm with dumb bell shape outside the gauge length were produced for the test. The dumb bell part was clamped to jaws of the machine and the extension was produced within the gauge span of the specimen. The elastic modulus was calculated by determining the slope of force-extension curve along the elastic region and then substituting in the following equation:

$$E = \frac{Stress}{Strain} = \frac{F}{e} \times \frac{l}{A} = Slope \times \frac{l}{A} (GNm^{-2})$$
 (2)

where F = force, e = extension, l = original length and A = cross-sectional area.

The UTS was extracted from the plot of the force-extension diagram from the electronic tensometer as obtained.

Flexural strength was determined according to ASTM D590 [24] using Universal (digital) flexural testing machine (EnerPac P-391) in the Strength of Materials laboratory in the Department of Mechanical engineering, A. B. U. Zaria. Sample specimen dimensions of 100x10x5 mm were produced for the test. The test sample was placed between two rollers and force (hydraulic handle) was applied until the sample ruptures. Flexural strength is the ability of the composite to withstand bending. This was calculated using equation 3.6:

$$F = \frac{3PL}{2hd^2} (MPa) \tag{3}$$

where P =is the maximum load, L =gauge length, b =width of specimen and d =maximum deflection.

Hardness test carried out using the electronic Microvickers MV1–PC hardness tester (maximum capacity of 500 gf) in the Shell Chair Laboratory of Mechanical Engineering Department. Sample specimens of dimensions 20x20x5 mm were produced for the test. Load (500 gf) was applied onto the test sample to produce a diamond shaped profile. The profile area was then interpreted by the computerized machine (hardness tester) to give the hardness value.

3.3 Morphology

A JOEL Field Emission Electron microscope JSM-7600F was used to carry out the microstructural analysis of the composite samples. Sample specimen of dimension 20x20x5 mm was produced for the test. The samples were polished and firmly held with the aid of a sample holder in the analysis chamber of the SEM. The scanning was commenced when the vacuum reading reaches 9.634×10^{-5} . The experiment was carried out in the Chemical Engineering Department of Ahmadu Bello University, Zaria.

4. RESULT AND DISCUSSION

4.1 Spectroscopic analysis of constituents

The FTIR of the banana particulate is depicted in Figure 2 denoting 4 major bands which is indicative of a low molecular weight material [25]. The spectrum depicts major absorption bands of lignocellulosic components (lignin, cellulose and hemicellulose) composed of alkynes, aromatic groups and various oxygen functional groups i.e. ester, ketone and alcohol [26]. Cellulose molecules arranged in microfibrils acts as the reinforcing material in the cell wall with extensive hydrogen bonding (OH stretching) producing strong crystalline structure [27]. The broad band region 3600 - 3100 cm⁻¹ is indicative of O-H stretching vibration. The band at 2940 cm⁻¹ corresponds to C-H stretching from CH and CH₂, indicative of an organic material.

The band at 1640 cm⁻¹ corresponds to C=C bond in the lignin component. The band region 1105 – 1155 cm⁻¹ is due to C-C and C-O-C asymmetrical stretching of the polysaccharide that is largely cellulose [28]. The absence of the 1740 cm⁻¹ band of carbonyl C=O from the spectrum indicated the disappearance of the hemicelluloses component

[29]. This could possibly be due to fibre treatment with sodium hydroxide. The spectrum was compared with that characterized by Chattopadhyay *et al.* [9] in Fig. 3 for the treated banana fibre and it showed identical peaks in within the range 4000–500 cm⁻¹.

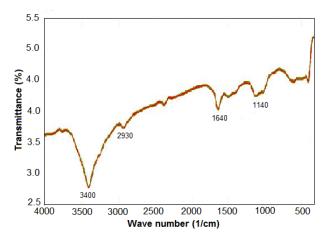


Fig. 2. FITR spectrum of the treated banana stem particulate.

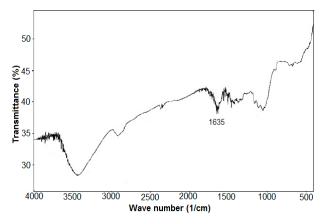


Fig. 3. FTIR spectrum of treated banana fibre [9].

The FTIR transmittance of the PVC is presented in Fig. 4 indicating its main characteristic bands. The broad band region 3400-3500 cm⁻¹ corresponds to O-H hydroxyl bond, attributed to water absorbed by the PVC. The band at 2920 cm-1 corresponds to C-H stretching vibrational mode. The band at 1250 cm⁻¹ corresponds to C-H deformation wagging. The band region 1000–1100 cm-1 is due to C-C stretch bond of PVC backbone chain [30]. The band region 960cm⁻¹ corresponds to C-H wagging vibrational mode. The band region 840cm⁻¹ corresponds to C-Cl gauche bond. The band region 600 - 650 cm⁻¹ corresponds to C-H stretching mode [31]. The spectrum was compared with that characterized by Ramesh et al. [32] as shown in Fig. 5 and it showed identical peaks within the range 4000-1000 cm⁻¹.

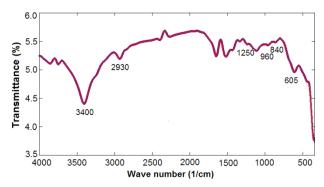


Fig. 4. FITR spectrum of PVC.

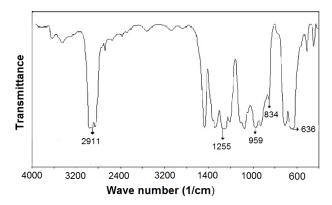


Fig. 5. FITR spectrum of pure PVC [31].

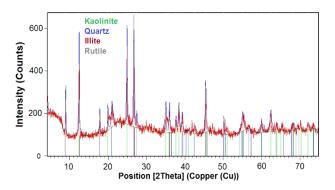


Fig. 6. XRD spectrum of the Kankara kaolin clay.

XRD analysis of the Kankara clay is presented in Fig. 6. The matched XRD pattern of the clay provided four major components- kaolinite, quartz, rutile and illite. The characteristic peaks of kaolinite could be read at Bragg's angle 12.35, 19.89, 20.38, 24.88, 34.94, 35.95, 36.06, 38.35, 45.24, 54.88 and 62.37 °. The peak at 26.6 ° was due to quartz also known as crystalline silica [33,34]. It can be seen that the quartz's peak has the highest intensity of about 640 counts followed by that of the kaolinite having not greater than 400 counts while those of rutile and illite are so minimal that they are barely visible. This showed that quartz content was much higher in the clay. The result was compared with that of Salahudeen [10] in Fig. 7 for the starting (raw) clay and it showed similar peaks pattern for the respective brag angle position though the quartz content is higher for Salahudeen (slightly above 1000 counts).

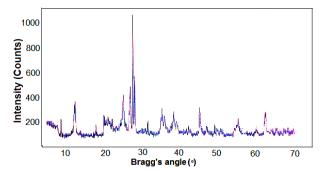


Fig. 7. XRD pattern for starting clay [10,11].

Table 2. Comparison of the XRF with known result

Compound major elements	Compound name	Concentration (%) Present work	Concentration (%) [11]
AlO ₃	Kaolinite	41.20	28.43
SiO ₂	Quartz	51.00	54.21
K ₂ O	Illite	3.00	3.90
TiO ₂	Rutile	0.08	0.11
Total		95.28	86.75

XRF analysis of the *Kankara* clay carried out gave the elemental configuration of the clay as given in Table 2. The major compounds of the kaolin clay are aluminum oxides, silicon oxides and potassium oxides forming the bulk of its composition of about 95 %. The tabular result also confirms the XRD result of the presence of minerals of kaolinite, quartz, rutile and illite in the clay. It also confirms the larger content of quartz in the clay. The result was also compared with that of Salahudeen *et al.* [11] and it also confirms the quartz's content was higher for Salahudeen et al. (Table 2).

4.2 Physical and mechanical properties

Graphical depiction of the density with increasing weight fraction of the particulate (reinforcement) indicated decrease in density of the composite (Fig. 8). The Figure also shows the water absorption of the composite with increasing weight fraction of the particulates. The percentage water absorption of the composite increases as the weight fraction of the particulate is increased. Figure 9 depicts the modulus of elasticity of the composite with increasing weight fraction of the particulates. The trend of the modulus of elasticity (stiffness) of the composites increases from 1 GPa to 2.4

GPa. The Figure also shows the ultimate tensile strength (UTS) of the composite with increasing weight fraction of the particulates. However the tensile strength increases and then decreases steeply. This could be due to weakening of the interfacial attraction of the constituent composition as the fraction of the PVC is reduced with increasing weight fraction of reinforcement.

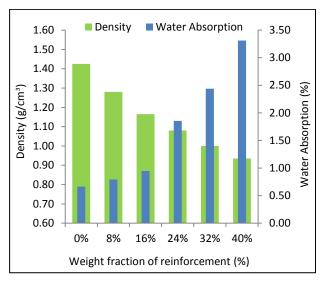


Fig. 8. Effect of density and water absorption on weight fraction of reinforcement.

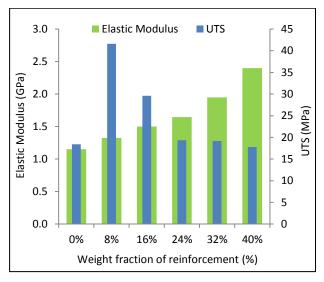


Fig. 9. Effect of elastic Modulus and UTS on weight fraction of reinforcement.

It is interesting to note that maximum is achieved at 8 % weight fraction of the reinforcement. Figure 10 shows the flexural strength of the composite with increasing weight fraction of the particulates. The flexural strength increases and then decreases steadily. The decrease could be as a result of the weakening of the interfacial attraction of the constituent

composition as the fraction of the PVC is reduced with increasing weight fraction of reinforcement. It is interesting also to note that the maximum is achieved at 8 % weight fraction of the reinforcement.

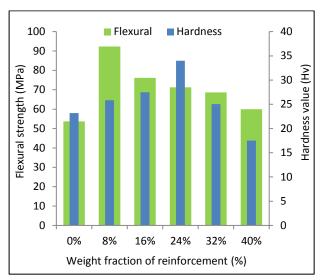


Figure 10. Effect of flexural strength and hardness on weight fraction of reinforcement.

However the hardness value slightly increases, reaches a maximum at 24 % fraction of reinforcement and then steadily decreases as shown in Figure 10. This could be as a result of decrease in the strength of the composite above 24 % of weight fraction of reinforcement. It can be deduced that the optimum mechanical property was found at 0.79 % corresponding to a density of 1.24 g/cm3, UTS of 42 MPa and flexural strength of 92 MPa with appreciable water absorption of 2.7 % and hardness value of 29.5 Hv.

4.3 Morphology

Scanning electron microscopy (SEM) result of the composite was conducted on sample with optimum property (8 % reinforcement) as shown in Figs. 11, 12 and 13. Plate 1 depicts a fairly uniform distribution of the constituent materials at 1000 magnification. At higher magnifications (Figs. 12 and 13), the kaolin amorphous crystalline component can be reasonably observed. The continuous flow (grey colour) vividly indicated the polymer (PVC) as the matrix, the non-glossy particles indicated the banana particulate as the reinforcement and the glossy semi-crystalline particle (white colour) indicated the Kankara clay as the filler. Agglomeration can be observed (Figs. 12 and 13) which can be avoided by proper mixing of

the constituents. Particle sizes of the kaolin and banana particulates were observed to be at most 4 and 2 μ respectively (Fig. 13). The dark colour indicates the background. Constituents' phases' distribution is fairly uniform.

4.4 Comparative price and weight analysis with piping materials

Price and weight per meter length of the developed composite was compared with conventional raw piping materials of PVC and carbon steel. Densities of carbon steel [35] and PVC are given as 7.8 and 1.5g/cm³ respectively. Price estimates of respective Kankara clay and banana stem particulate were estimated at \$0.2/kg and \$0.8/kg inclusive of preparation cost, treatment, fibre removal and grinding.

The estimate for the price of PVC [36] and carbon steel [37] were determined per kilogram weight at international market respectively. Prices of respective Kankara clay, banana and doum palm particulate were estimated. Weight per meter length was calculated as mass of the piping material per unit length of the pipe as (eq. 8):

$$m = \frac{\rho \times V}{I} \tag{4}$$

$$m = \frac{\rho \times V}{l}$$
 (4)
$$V = \pi (R^2 - r^2)l = \pi l(R - r)(R + r)$$
 (5)

But pipe thickness,

$$t = (R - r) \tag{6}$$

Implying,

$$V = \pi lt (2r + t) \tag{7}$$

Therefore,

$$m = \rho \pi (t^2 + 2rt) \tag{8}$$

where R = outer radius, r = inner radius, t = inner radiusthickness of pipe, V = volume of pipe materialand l = length of pipe.

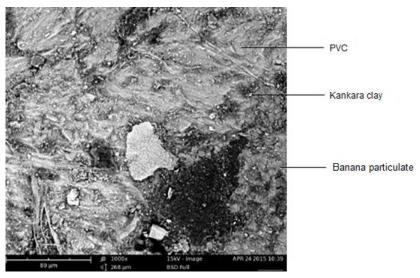


Fig. 11. Microstructure of composite of banana particulate at 1000 magnification.

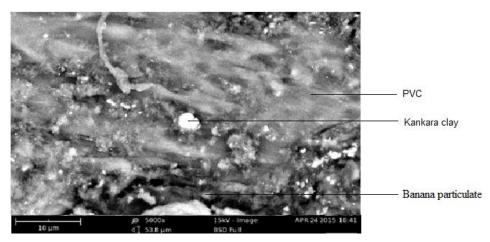


Fig. 12. Microstructure of composite of banana particulate at 5000 magnification.

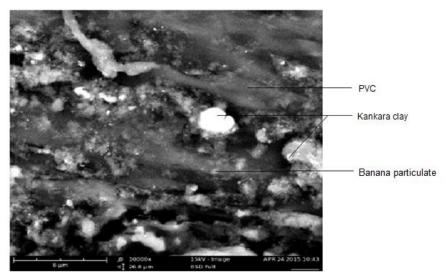


Fig. 13. Microstructure of composite of banana particulate at 10.000 magnification.

Table 3. Materials cost estimates for the piping materials.

S/N		Materials	Percentage Composition	Constituents Price/Kg	Price/Kg	Comments
1	Composite of Banana					
	(i)	Banana	8 %	1.00	1.49	Inclusive of preparation cost,
	(ii)	PVC	72 %	1.90		treatment and grinding
	(iii)	Kankara Clay	20 %	0.20		
2		PVC			1.90	Price of raw PVC at international market [36]
3	Steel				1.75	Price of raw steel coil at international market [37]

Table 4. Price and weight per meter length.

S/N	Piping Material	Density	Weight/Kg	Price/m
1	Composite of Banana particulate	1.24	0.27	0.40
2	PVC	1.35	0.32	0.62
3	Carbon Steel	7.80	1.69	2.95

Comparison of weight per meter length at piping thickness of 2.5 mm was made for the three piping materials as shown in Fig. 14. Carbon steel produced the highest cost and the largest weight per meter length thus implying higher mobility and installation cost. Material of composite of banana particulates gives the least weight per meter and at a cheaper price than PVC material. Material weight savings per meter length of 84 % and 25 % were achieved for the composite as compared with carbon steel and PVC material. Material price savings per meter length of 84 % and 17 % were achieved for the composite as compared with carbon steel and PVC material.

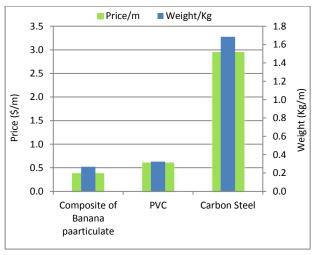


Fig. 14. Price and weight per meter length of piping materials.

Considering that the composite is viscoelastic in nature, its potential for piping application is possible as it is not a direct load carrying component as in ties, columns and beams where great amount of stresses are generated as a result of tension, compression and bending. In addition, transportation of petroleum products at long distances is done extensively along a horizontal profile, the effects of weight of the moving fluid on the pipe is virtually negligible. Thus the composite can provide alternative potential material for piping application.

5. CONCLUSION

The composite was developed with considerabley low cost materials having an overall light-weight and good mechanical property. The optimum mechanical property was determined at 8, 62 and 30 % formulation of banana stem particulates (reinforcement), PVC (matrix) and *Kankara* clay (filler) respectively, providing a corresponding density of 1.24 g/cm³, Young's Modulus of 1.3 GPa, negligible water absorption of 0.79 % and tensile strength of 42 MPa.

FTIR analysis on the respective banana particulate showed a characteristic of plant fibres spectral profile, though with varying constituent composition of cellulose, lignin and other minor constituents vis-a vis pectin, waxes and water soluble components [27]. XRD conducted on the Kankara clay showed it contained four major components namely kaolinite, quartz, rutile and illite. XRF confirms the XRD result of the presence of these minerals. The XRD and XRF were compared with known research work and the result showed similar peaks pattern and compositions respectively. SEM result of the composite indicated a continuous flow (grey colour) as PVC, the nonglossy particles as the banana particulate reinforcement and the glossy semi-crystalline particle (white colour) as the Kankara clay. There is also fairly uniform distribution of constituents' phases.

The composite was compared with carbon steel and PVC conventional piping materials. Material price savings per meter length of 84 % and 25 % were achieved for the composite as compared with carbon steel and PVC material. Material

weight savings per meter length of 84 % and 17 % were achieved for the composite as compared with carbon steel and PVC material.

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