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Effect of Using Alkoxy - Silane (ethoxy silane) Coupling Agent to Enhance the Mechanical Properties of Bio-Composites

I.C.C. Iloabachie, V.C. Nnadi, A.C. Chime

Abstract: ***This research work investigated the effect of alkoxy silane (ethoxy silane) on the mechanical properties of polyethylene/ dried powdered pine apple peels composite. The pine apple peels was washed in distilled water, sun dried for about eight hours and later oven dried at a temperature of 60ºC for about three hours. The dried pine apple peels was pulverized and sieved using a mechanical sieve arranged in descending order of fineness. The alkoxy -silane (ethoxy-silane) coupling agent (2%) was first hydrolyzed in ethanol to deliver the alkoxy functional silane to the interior of the pineapple peels and ion-free water at room temperature for 6 hours. This was followed by addition of the dried powdered pineapple peels into the hydrolyzed coupling agent solution in a reactor at a temperature of about 80 °C for 20 minutes. The formed product was oven dried at 110 °C for 3 hours and pulverized using a locally made grinding machine and sieved using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard as was reported by Rajan et al., (2007) to obtain pineapple peels coated with the coupling agent (150mµ). The recycled polyethylene waste was washed using distilled water, sun-dried and shredded in a shredding machine. The pineapple peels powder coated with alkoxy silane and the shredded recycled polyethylene waste were blended using a two-roll rheo-mixer at 50°C and a rotor speed of 60 rpm. The percentage of the powdered pineapple peels coated with coupling agent in the recycled polyethylene matrix was varied from 10% to 40% to produce four different compositions. A hydraulic pressing machine was used to compress the produced composites for about ten minutes applying a pressure of about 25 tons at 130°C. The produced composite samples were allowed to cool to room temperature under sustained pressure before being removed from the hydraulic press for various mechanical tests. The impact tests were performed according to ASTM D256 standard using Impact testing machine model EXT94064/6705CE, 300 J. Flexural test was performed using Universal Testing Machine model TUE-C-100, according to ASTM D790. The hardness tests were performed according to ASTM D785 standard using Rockwell Scale K hardness testing machine. The results showed that maximum flexural strength of 10.8MPa and 384Hv were recorded at 40wt.% reinforcement. The developed composite can be used applications where moderate strength will be required.***

Keywords: ***Ethoxy Silane, Mechanical Properties, Hydrolyzed Ethanol***

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I. INTRODUCTION

Increasing use of composite materials in various fields of industrial applications has continued to attract the interest of researchers in enhancing the properties of bio-composite materials. Bio-composites are usually produced by mixing eco-friendly ingredients – usually plant materials mixed with natural resins and binders. The necessity for combination was due to inherent deficiency of most materials to have exactly, the right properties for most engineering applications. Iloabachie (2018) opined that the availability of natural fillers and ease of fabrication have continued to tempt researchers to try locally available inexpensive agro waste materials to study the feasibility of reinforcement purposes and to what extent they satisfy the required specifications of good reinforced polymer composites for different engineering applications. Most plant materials are lignocellulosic and cellulosic in nature. Islam and Beg (2010) viewed lignocellulosic materials as the emerging generation of reinforcing materials in combination with thermoplastics considering their renewable nature. Islam and Beg (2010) further argued that the good strength characteristics and the favourable strength/weight-ratio of the lignocellulosic materials fibre may be likened to the strong cellulose backbone structure and this is considered an advantage compared to other conventional reinforcing materials. Xie et al., (2010) highlighted the numerous advantages natural fibers over conventional inorganic fillers as (1) abundance and therefore low cost, (2) biodegradability, (3) flexibility during processing and less resulting machine wear, (4) minimal health hazards, (5) low density, (6) desirable fiber aspect ratio, and (7) relatively high tensile and flexural modulus. Wambua et al., (2003) stated that natural fibers with its tough and light-weight nature produce composites with a high specific stiffness and strength when used as reinforcement in matrices. Geottler (1983) and Klason et al. (1984) however, observed that most polymers, thermoplastics in particular, are non-polar (hydrophobic) substances, and are incompatible with polar (hydrophilic) wood fibers hence, resulting to poor adhesion between polymer and wood fiber in wood fiber polymer composites (WFPC). Islam and Beg (2010) maintained that the hydrophilic nature natural fibre is its major drawback and this reduces its compatibility with hydrophobic polymeric matrices. The hydrophilic nature of natural fibers, Iloabachie (2018) argued makes it prone to moisture absorption, limiting the exterior application of the developed composites.

The moisture absorption of the lingo-cellulosic fillers may cause dimensional change of the resulting composites and weaken the interfacial adhesion. This leads to induced stress, micro-cracking and ultimate failure in service of the developed composite which to degradation of mechanical properties Takase and Shiraishi (1989) observed. Peijs et al., opined that poor environmental and dimensional stability also, limits wider application of natural fibre composites. While Rowell et al., (1986) argued that eliminating moisture absorption in composites involves application of expensive surface barriers however, Tajvidi and Ebrahimi (2003) believed that adequate wetting of the fibre by the matrix and proper fibre–matrix bonding may lower the rate and quantity of absorbed water in the composite interface region.

Woodhams et al. (1984) suggested that improvement in affinity and also, proper interaction in terms of adhesion between natural fibers and polymers (thermoplastic matrices) in production can be achieved by using chemical coupling agents. Coupling agents, Pritchard (1998) explained are substances applied in small amounts to treat a surface to achieve bonding between the surface and other surfaces. As was stated by Islam and Beg (2010) coupling agents in natural fibre reinforced plastic composites play vital role in enhancing compatibility and adhesion between polar natural fibre and non-polar polymer matrices by forming bridges of chemical bonds between the fibre and the matrix.

Islam and Beg (2010) stated that Beg et.al and John et.al studied coupling agent performance in wood-fibre reinforced high-density-polyethylene composites and observed that the improvement on the interfacial bonding strength, flexural modulus, and other mechanical properties was mainly related to the coupling agent type, functional groups, molecular weight, concentration, and chain structure. The maximum value of interfacial adhesion was achieved with 3 wt % concentration level for most maleated composites. Sonia et. al. (2007) reported that silane-modified polymer increased the interfacial adhesion between the fibre and the matrix and this effect was better than that obtained for the maleated-polypropylene-coupled composites. A silane coupling agent, according to Yun (2003) is a chemical substance capable of reacting with both the reinforcement and the polymer matrix of a composite material, acting as an interphase between the polymer phase and the fiber phase in the process and forms a chemical bridge between the two phases. Silane coupling agents have the ability to form a durable bond between organic and inorganic materials Arkles (2014). Alkoxy-silanes which give methanol and ethanol as byproducts during coupling reactions are t most commonly used. Chloro-silanes generate mainly hydrogen chloride as a byproduct during coupling reactions,hence, are generally less utilized.

Pang et al., (2005) argued that the good mechanical, chemical, and weather resistant properties of unsaturated polyester resins (UPE) when reinforced with fibers, makes them most widely used thermosetting matrix in NFRPs. However, poor interfacial bonding between the polymer and natural fillers has remained an increasing area of research interest among researchers. Epoxy composites.

In this work therefore, an attempt is made to investigate the effect of methoxy-silane coupling agent on the interfacial tensile strength and flexural strength of unsaturated polyester and epoxy bio-composites.

II. MATERIALS AND METHODS

A. Materials

The materials used in this research work were ethoxy-silane coupling agent, powdered pine apple peels, recycled polyethylene waste (matrix) and methyl ethyl ketone peroxide (catalyst), cobalt naphthanate (accelerator) supplied by Ndidiamaka Trading Company in Enugu.

B. Methods

i.Drying of Pineapple peels

The pine apple peels was sundried for eight hours to reduce the moisture content before ovum drying at 60^{o}C for three hours.

ii.Grinding and Sieving of Dried Pineapple Peels

The dried pine apple peels was ground to different particle sizes using a locally made grinding machine. Sieving was carried using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard as was reported by Rajan et al., (2007).

iii.Chemical Analysis of Pineapple Peels

The chemical analysis of the pineapple peels was carried out using Absorption Spectrometer (AAS)-Peckinhelma 2006 model.

iv.Sample Preparation

Application of Coupling Agent on the Surface of the dried pineapple peels by Coating process. The tri-alkoxy -silane (ethoxy-silane) coupling agent was first hydrolyzed in ethanol to deliver the alkoxy functional silane to the interior of the pineapple peels and ion-free water at room temperature for 6 hours. This was followed by addition of the dried pineapple peels into the hydrolyzed coupling agent solution in a reactor at 80 °C for 20 minutes. The formed product was oven dried at 110 °C for 3 hours and crushed by a locally made grinding machine and sieved using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard as was reported by Rajan et al., (2007) to obtain pineapple peels coated with the coupling agent. The recycled polyethylene waste used for this work was sun-dried and shredded in a crushing machine. The pineapple peels powder and the ground recycled polyethylene waste was blended using a two-roll rheo-mixer at 50°C and a rotor speed of 60 rpm. The percentage of the pineapple peels reinforcement in the recycled polyethylene matrix was varied from 10% to 40% to produce five different compositions. A hydraulic pressing machine was used to compress the produced composites for about ten minutes applying a pressure of about 25 tons at 130°C. The produced composite samples were allowed to cool to room temperature under sustained pressure before being removed from the hydraulic press for various mechanical tests.

v.Impact Test

The impact tests were performed according to ASTM D256 standard using Impact testing machine model EXT94064/6705CE, 300 J capacity.

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The test machine made use of a pendulum-testing machine which determined the Charpy impact strength of the composite. The impact velocity of the pendulum is 5.24m/s. The impact toughness of a composite is determined by measuring the energy absorbed in the fracture of the specimen. A stop pointer was used to record how far the pendulum swings back up after fracturing the specimen in the scale. The test was performed by noting the height at which the pendulum was released and the height to which the pendulum swung after it had struck the specimen. The height of the pendulum times the weight of the pendulum produces the potential energy and the difference in potential energy of the pendulum at the start and at the end of the test is equal to the energy absorbed.

vi.Flexural Test

Flexural test was performed using Universal Testing Machine model TUE-C-100, according to ASTM D790.The composite samples were tested at a three-point bending test at a cross head speed of 5 mm/min. In each case, three samples were taken and average value of the flexural force was recorded. The flexural stress was computed using the following equation:

$$\sigma_{max} = \frac{3P_{max}L}{bh^2} \quad \text{……. (1)}$$

Where, P_{max} is the maximum load at failure (KN), L is the span (mm); b and h are the width and thickness of the specimen (mm) respectively.

vii.Hardness Test

The hardness testing machine model used was Type DVRB-M 220/240 V. The hardness tests were performed according to ASTM D785 standard using Rockwell Scale K hardness testing machine. The proper indenter ball 1/8” for scale K was installed. The indenter was put into the pressure shaft so that flat part of the indenter cylinder was in front of the Allen screw. The Allen screw was slightly tightened and the machine switched on by the main switch in front of the panel. Loading force of 100kg was selected using the lever force on the right side of the machine. The test specimen was placed on the anvil and lifted against the indenter. The anvil was lifted with the test specimen carefully until the green light in front of the panel came on. The red light on the panel signaled the completion of the test and the machine switched off automatically.

III. RESULTS AND DISCUSSION

The flexural properties of composites Subiat and Verma (2013) argued depend critically on the microstructure of the composite and the interfacial bonding between the reinforcement and the matrix. Generally, addition of powdered pineapple peels increased the flexural strength of the composite. From Fig. 3.1 it could be observed that the flexural strength of the composite increased with addition of the pineapple peels; however, significant improvement in flexural strength of the composite was observed in the composite sample containing tri-alkoxy -silane (ethoxy-silane) coupling agent. Although, the flexural strength of the composite increased with increase in the weight percent (wt. %) of the powdered pineapple, it could be observed that addition of coupling agent in the composite sample ensured better flexural strength. Pineapple peels contains mainly cellulose and lignin, higher concentration of the powdered pineapple peels will result in higher interfacial bonding between the matrix (recycled polyethylene) and the reinforcement i.e. powdered pineapple peels and this ensured better stress transfer from the matrix to the reinforcement. An improvement of approximately 40% in flexural strength at 30wt. % of powdered pineapple peels was achieved with addition of a coupling agent. This may be attributed to the added coupling agent that helped to improve dispersion of the cellulosic powdered pineapple peels in the recycled polyethylene and promoted better stress propagation between the matrix and the reinforcement. Similar trend was reported by Ndiaye and Tidjani (2012).

Weight Percent of Particulate (%)	Flexural Strength MPa +CA	Flexural Strength MPa
10	7	5.9
20	8.2	6.3
30	9.5	6.8
40	10.4	7.6

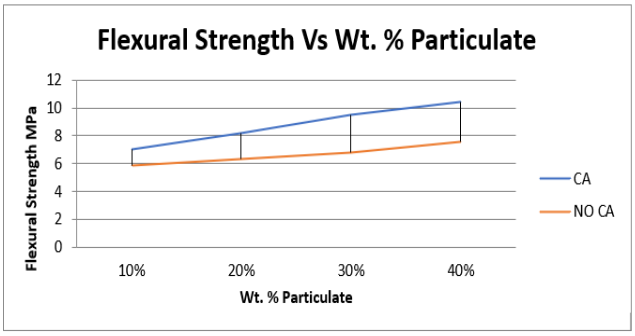

Fig. 3.1: Flexural Strength against Wt. % of Powdered Pineapple Peels in Recycled Polyethylene

A. Hardness

Generally, the stiffness of any material is a function of its hardness. As was reported by Mehdi et al. (2009) the addition of reinforcement material in polymer resins imparts stiffness in the polymeric material with a resultant increase in hardness of the emerging of composite. As can be observed in Fig. 3.1, though, addition of powdered pineapple peels resulted in increase in hardness of the developed composite, however, incorporation of a coupling agent in the composite mixture gave better hardness values compared to the composite mixture without a coupling agent. Highest hardness vale of 387Hv was obtained at 40 wt. % in the composite sample containing coupling agent. This represents about 10.26% increase in hardness when viewed against the composite sample without a coupling agent at the same composition of reinforcement. However, when compared with the virgin polymer (238Hv) without powdered pineapple peels and coupling agent, it could be observed that about 62.61% improvement in hardness value (387Hv) was achieved. Similar trend had been reported by Musa et al. (2019).

Weight Percent of Particulate (%)	HRN Value +CA	HRN Value
0	238 (No CA)	238 (No CA)
10	342	322
20	346	331
30	366	343
40	387	351

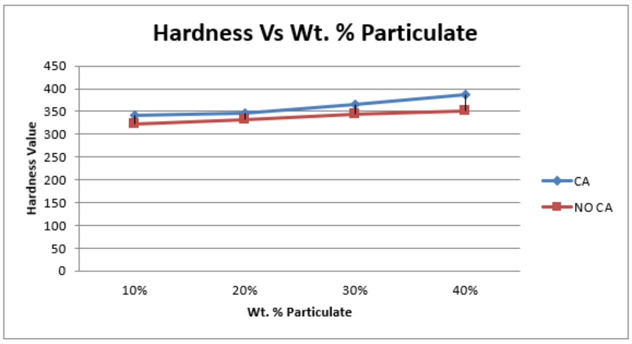

Fig. 3.2: Hardness Against Wt. % of Powdered Pineapple Peels in Recycled Polyethylene

B. Impact Strength

Jamal et al. (2006) argued that the impact strength of the impact strength of composite materials is a function of the filler material to absorb energy capable of impeding crack propagation and poor interfacial adhesion which induces micro-cracks between the filler and the matrix, thereby, precipitating easy crack propagation. As observed in Fig. 3.3, the impact strength of the composite initially increased up to 20wt. % of the powdered pineapple peels and started decreasing.

Despite this trend, the composite sample containing coupling agent maintained higher impact strength values at all compositions of the reinforcement material which indicates that the better impact strength values may be as a result of the added coupling agent.

The decrease in the impact strength of the composite as was observed may be seen as a result of the decrease in impact energy as quantity of the powdered pineapple peels increased. Poor interfacial adhesion between the matrix and the powdered pineapple peels which lead to micro-cracks as a result of reinforcement pull out from the matrix may account for the lower impact strength observed in the composites without addition of coupling agent. In addition, the slightly higher impact strength recorded by the composites containing a coupling agent may be attributed to better compatibility and favorable matrix/reinforcement interaction caused by the addition of coupling agent in the composite.

Weight Percent of Particulate (%)	Impact Energy (J) + CA	Impact Strength (J/M2) + CA	Impact Energy (J)	Impact Strength (J/M^2)
10	0.58	5800	0.53	5300
20	0.61	6100	0.57	5700
30	0.55	5500	0.49	4900
40	0.52	5200	0.44	4400

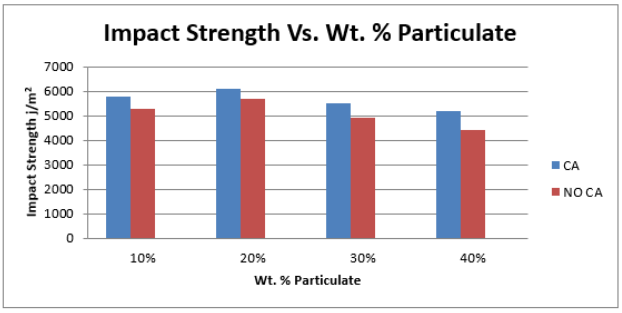

Fig. 3.3: Impact Strength Against Wt. % of Powdered Pineapple Peels in Recycled Polyethylene

From fig. 3.4a and Fig. 3.4b as was reported by Musa et al. (2019) 40% filler loading resulted in better mechanical properties. Fig. 3.4a showed even distribution and dispersion of the filler in the composite with coupling agent. This Mehdi (2009) argued has the advantage of making the reinforcing fillers to be fully embedded in the matrix thereby preventing de-bonding which leads to plastic deformation in composites

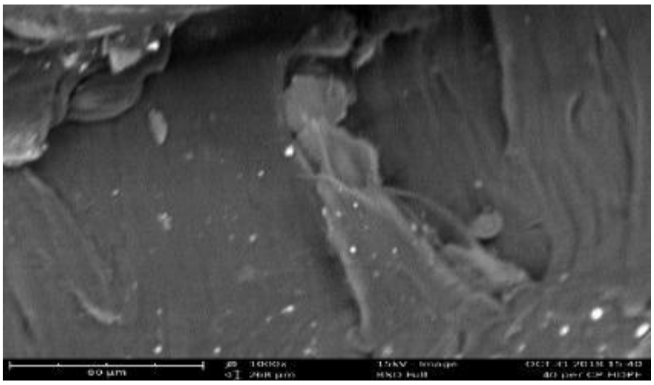

Fig. 3.4a SEM Image of CP/HDPE Composite A at 40% Filler Loading with Coupling Agent Musa et al. (2019)

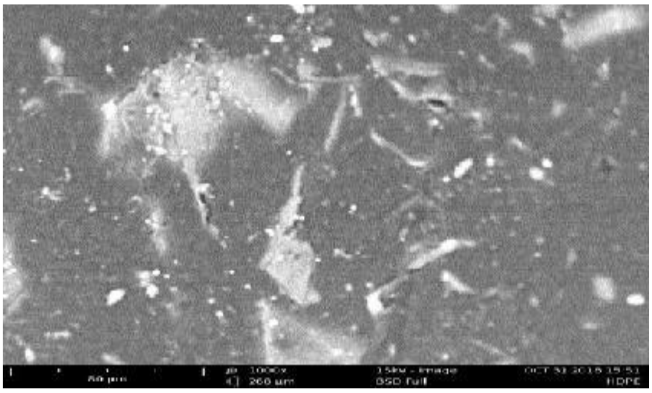

Fig. 3.4b SEM Image of CP/HDPE Composite B at 40% Filler Loading Without Coupling Agent Musa et al. (2019)

IV. CONCLUSION

- Dried pineapple peels in powdered form can be used effectively as a reinforcement material to develop a composite.

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- Tri-alkoxy – silane improved matrix (polyethylene) – reinforcement (dried powdered pine apple peels) interaction thereby, enhanced the mechanical properties like flexural strength and hardness.

DECLARATION STATEMENT

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Ethical Approval and Consent to Participate	No, the article does not require ethical approval and consent to participate with evidence.
Availability of Data and Material	Not relevant.
Authors Contribution	All the authors have equal participation in this article.

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