

**Original Research Article****Mechanical Behavior of Agricultural Waste Fibers  
Reinforced Vinyl ester Bio-composites****ABSTRACT**

Agricultural waste fibers have great potential in composite due to its high strength, eco-friendly nature, low cost, availability and sustainability. The agricultural waste is one of the most important problems that must be resolved for the conservation of global environment. In this study the potential of agricultural wastes such as bagasse, oil palm, coconut, cornhusk, groundnut shell and rice husk fibers as reinforcements in vinyl ester composites was investigated. The necessity of this work is to respond to the social demands for the disposal of environmentally problematic agricultural wastes and property improvement. Hence, the effects of four levels of fiber loadings (5, 10, 15 and 20 wt. %) on the mechanical properties of the composites were studied. For overall trend, as the percentage of fiber loadings increased the ultimate tensile strength, tensile modulus and hardness of the composites substantially improved, whereas the tensile strain decreased compared with the pure vinyl ester matrix with a verge point value at 10 wt. % reinforcement. In general, oil palm fibrous waste showed superior mechanical properties due to its chemical characteristics. This study has shown that the ultimate tensile strength, tensile modulus, tensile strain and hardness of the composites varied substantially based upon the type of fiber utilized and the fiber loadings, with maximum value at 10 wt. % agro fiber content.

**Keywords:** Mechanical properties, Agricultural waste, Vinyl ester, Reinforcements, Composites.

**1. Introduction**

Plenty of wastes are generated as a result of the increased activity in the modern agricultural sector which represents a serious threat to the environment. Meanwhile, dwindling supply of raw materials is causing concern and in this context the agro waste can be seen as a good alternative material for the local timber industry to produce value added product, such as bio-composites. Utilization of natural fibers especially agricultural waste fibers needs further development as a long term strategy to develop the tremendous wealth of natural plant fiber that are currently under utilized [1].

Cellulose fibers from palm pressed fibers have been used as fuel [2]. The corn husk has also been used for biodegradable film [3], heat insulator from coconut fibers [4], rice husk ash and coconut fibers in concrete [5]. Natural fibers from banana's tree as fillers into polymers composites [6]. Rice straw and bagasse fibers used as writing and printing papers [7]. In addition, oil palm fronds, bamboo fibers, coconut fibers, rice-husks and sugar cane-dregs are used to make cement boards [8, 9]. In the past few years, several studies have reported natural fibers as a reinforcing material in bio-composites thermoplastics and thermoset matrices. Coir, banana and sisal agricultural wastes can be used as reinforced polymer composites for commercial use [10-14].

Dealing with the growing demand for the renewable resources, agricultural and plantation wastes are considered as the promising and the suitable material. Biomass material is one of the important sources of alternative material for the production of bio-composites products [15, 16, 14, and 17]. An increasing global awareness about environmental issues is acting as the driving force behind the utilization of biomass material as valuable products.

Thousands of tons of agricultural waste materials are produced globally on annual basis. These wastes could be used as the potential resources for reinforcing materials in bio-composites applications. The use of such resources will not only provide the sustainable and less expensive material but at the same time will contribute to the waste disposal management as well as overcoming the environmental problems [18].

As a result of the world wide demand for fibrous materials, global decline of trees in many locations, and environmental consciousness, research into the development of composites prepared with various agro waste resources is being actively pursued. Among the promising

substitutes is the development of composites utilizing agricultural wastes (such as stalks of most cereal crops, rice husks, coconut fibers, bagasse, corncobs, peanut shells, and other wastes) is presently at the focus of interest[19,20,21,22 and 17].

Although there are some useful studies in the literature on agricultural wastes in composites [23, 24, 25 and 21), there are still many gaps in information and knowledge of composites from agricultural wastes, which must be closed in order to encourage commercial production of these novel materials.

The scope of the present work is to utilize sugarcane bagasse, oil palm, coconut, corn husk, groundnut shell and rice husk agro wastes to evaluate and compare their suitability as reinforcing materials for composite applications. Aside the importance of property improvement, an additional incentive was to respond to the social demands for the disposal of environmentally problematic agricultural wastes.

## **2. Experimental**

### **2.1 Materials**

Six types of agricultural residuals were used in the study viz.:- sugarcane bagasse, oil palm, coconut coir, cornhusk, groundnut shell and rice husk fibers. The important chemical components and fiber morphology of agricultural waste fiber materials used in this study are given in Tables 1 and 2 respectively. These parameters are important as they influence the resulting mechanical properties of the composites.

#### 74 2.1.1 Preparation of sugarcane bagasse fibers

75 The sugarcane bagasse fiber (SBF) was sourced locally within the Abakaliki town from  
76 the sellers. Cleaned and dried bagasses were initially washed with water to remove the sand and  
77 other impurities. Subsequently, the bagasses were dried under the sunshine for three days to  
78 ensure that it was well dried. The dried fibers were pulverized using Denver laboratory ball mill.  
79 The particles from the process were sieved with sieve shaker 16155 Model into 75  $\mu\text{m}$  sieve size.

#### 80 2.1.2 Preparation of groundnut shell fibers

81 The groundnut shell was sourced locally within the Abakaliki metropolis. Clean and  
82 dried groundnut shells were initially washed with clean water to remove the sand and other  
83 impurities. Subsequently, the shells were dried under the sunshine for three days to ensure that it  
84 is well dried. The dried shells were ground and sieved with sieve shaker 16155 Model into 75  
85  $\mu\text{m}$  sieve size.

#### 86 2.1.3 Preparation of rice husk fibers

87 Finely milled rice husks were collected from the Abakaliki Rice Mill in Ebonyi State,  
88 Nigeria. The milled rice husks contain many impurities like dust, small rice particles, and fine  
89 sand particles. Therefore, it needs to be cleaned in order to get pure rice husk. After cleaning  
90 with water, the rice husks were dried directly under the sun for 8 h. The dried fibers were  
91 pulverized using Denver laboratory ball mill. The particles from the process were sieved with  
92 sieve shaker 16155 Model into 75  $\mu\text{m}$  sieve size.

#### 93 2.1.4 Preparation of oil palm fibers

94 The oil palm fiber (OPF) was collected from the rural farmers of Ebonyi State, Nigeria.  
95 The fibers were soaked in hot water with detergent for three days in order to remove the residual

oil and other impurities. The fibers were dried in the sun for one week to obtain a dry mass. A 40-mesh Wiley grinder was used to reduce the fiber to smaller particles. The particles from the process were sieved with sieve shaker 16155 Model into 75  $\mu\text{m}$  sieve size.

#### *2.1.5 Preparation of cornhusk fibers*

Corn husk fibers (surrounding the ear of corn/maize) were obtained from a local farmer Market in Kpririkpiri Market, Abakaliki, Ebonyi State, Nigeria. The corn husk was dried, ground and sieved with sieve shaker 16155 Model into 75  $\mu\text{m}$  sieve size.

#### *2.1.6 Preparation of coconut coir fibers*

Coconut fibers were extracted from exocarp washed and dried under the sun for three days. After being ground in a mill and sieved with sieve shaker 16155 Model into 75  $\mu\text{m}$  sieve size. Furthermore, the fibers were washed with clean water and dried in an oven at 100°C for 24 h.

#### *2.1.7 Vinylester resin*

Vinyl ester uses a polyester resin type of cross-linking molecules in the bonding process and is tougher and more resilient than polyesters. The ester groups in vinyl ester molecules are vulnerable to water degradation by hydrolysis, which means that vinyl esters exhibit better resistance to water and many other chemicals. A vinyl ester resin has excellent physical and mechanical properties and is well known for its versatility as a composite matrix. With the development of a promising room temperature molding technique, the processability of vinyl ester resins at low temperatures has attracted considerable attention from the composite industry. The vinyl ester resin used in this work was procured from Juneng Nig. Ltd. in Enugu, Enugu State, Nigeria. The density of the vinyl ester is 1.05g/cm<sup>3</sup> with heat distortion temperature of 125°C. The specification of the vinyl ester used in the study is shown in Table 3.

## 2.2 Composite preparation

The prepared agro waste fibers were mixed with the vinyl ester resin for one hour by using stirrer. The accelerator used was methyl ethyl ketone peroxide (2% of weight for each composite) and catalyst cobalt naphthalate (1% of weight for each composite) was added after stirring process. Once accelerator and catalyst was added, the curing reaction started immediately at room temperature. The mixture was transferred to a silicon rubber mold size and polyethylene sheet in dimension of  $300 \times 300 \times 5$  mm. After the curing process, the material was taken into the compression molding machine. The weight percentage of fiber reinforcement was varied as (0%, 5%, 10%, 15% and 20%) shown in Table 4. The mixture was stirred for about 5-7 minutes until there was proper wetting and soaking of the particles by the vinyl ester resin. The homogenous slurry was poured into the mold and pressed at 10,000 psi pressure at 90°C for 15 min and allowed to cure at room temperature for 24 h. Finally, the composites were placed in an oven at 100°C for 2 hours for post curing before the mechanical tests were carried out.

Table 1: Chemical composition of selected agricultural waste fibers

Type of fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ref
Bagasse	57.4	24.5	26.3	26
Oil palm	65.1	10.2	17.5	27
Coconut coir	47.7	25.9	17.8	28
Corn husk	40.3	32.2	21.5	29
Groundnut shell	35.7	18.7	30.2	30
Rice husk	31.3	24.3	14.3	31

136 Table 2: Dimensions of selected agricultural waste fibers

Type of fiber	Fiber length (mm)	Fiber width ( $\mu\text{m}$ )	Aspect ratio (L/D)	Ref
Bagasse	1.24	22.9	54	26
Oil palm	1.3	21.7	60	27
Coconut coir	1.22	24.4	50	28
Corn husk	1.18	25.1	47	29
Groundnut shell	0.8	17.8	45	30
Rice husk	0.5	12.5	40	31

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138 Table 3: Specification of the Vinyl ester Used in the Research

Materials	Specifications
Vinyl ester	Density = $1.05\text{g/cm}^3$ HDT = $125^\circ\text{C}$

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140 Sample preparation calculations:

- 141 1. Density of vinyl ester ( $\rho$ ) =  $1.05\text{g/cm}^3$
- 142 2. Volume of the mold (V) =  $300 \times 300 \times 5\text{mm}$   
143  $= 450000\text{mm}^3$   
144  $= 450\text{cm}^3$
- 145 3. Mass of resin (m) = Volume of mold x density of resin  
146  $= 450\text{cm}^3 \times 1.05\text{g/cm}^3$   
147  $= 472.5\text{g} \approx 500\text{g}$

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149 Table 4: Samples Preparation Calculation for agro residuals/Vinyl ester Composites

Sample	% wt of fiber	% of resin	Mass of fiber	Mass of resin	Total mass
A	0	100	0	500	500
B	5	95	25	475	500
C	10	90	50	450	500
D	15	85	75	425	500
E	20	80	100	400	500

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## 2.3 Mechanical Testing

Tensile mold of gauge length 25 mm of a dumb-bell shape was used for the production of tensile samples. Following the molding of the composites, samples were prepared for tensile and hardness tests. These tests were carried out as follows:

*2.3.1 Determination of the tensile properties of the materials-* In the present study, tensile tests was performed on INSTRON 1195 at a fixed crosshead speed of  $10 \text{ mm min}^{-1}$ . Samples were prepared according to ASTM D412 (ASTM D412 1983), and tensile strength of the standard and conditioned samples were calculated. Five specimens for each sample were tested and the tensile strength and tensile modulus were expressed as:

$$\text{Tensile strength (MPa)} = P/bh \quad 1$$

$$\text{Tensile modulus (MPa)} = \sigma/\epsilon \quad 2$$

Fig. 1 shows the specimens prepared for tensile test. The testing is done using UTM to measure the force required to break a polymer composite specimen and the extent to which the specimen stretches or elongates to that breaking point.

*2.3.2 Determination of the hardness property of the materials -* The samples was indented using micro hardness tester following ASTM procedure No.D2240. The reading is noted from the calibrated scale. Five readings were taken for each sample and the average value was used.



Fig. 1: Tensile test specimens



## 2.4 Morphological study

Studies on the morphology of the composites were conducted using a TESCAN model WEGA-II scanning electron microscope (SEM). The fracture surfaces of the specimens after tensile test were sputter-coated with gold before analysis in order to eliminate electron charging.

## 3. Results and discussion

### 3.1 Tensile properties

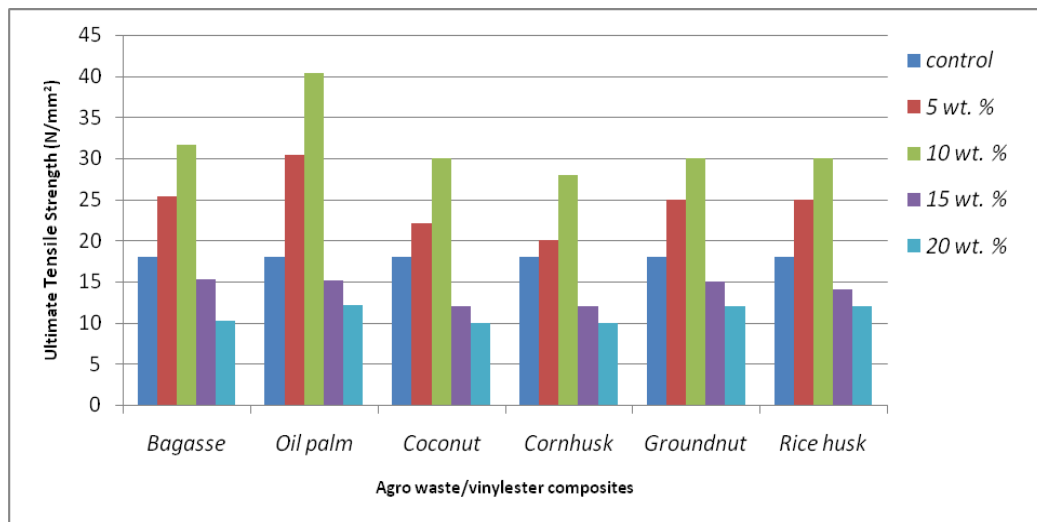


Fig. 2: Comparison of ultimate tensile strength of composites as function of fiber weight content

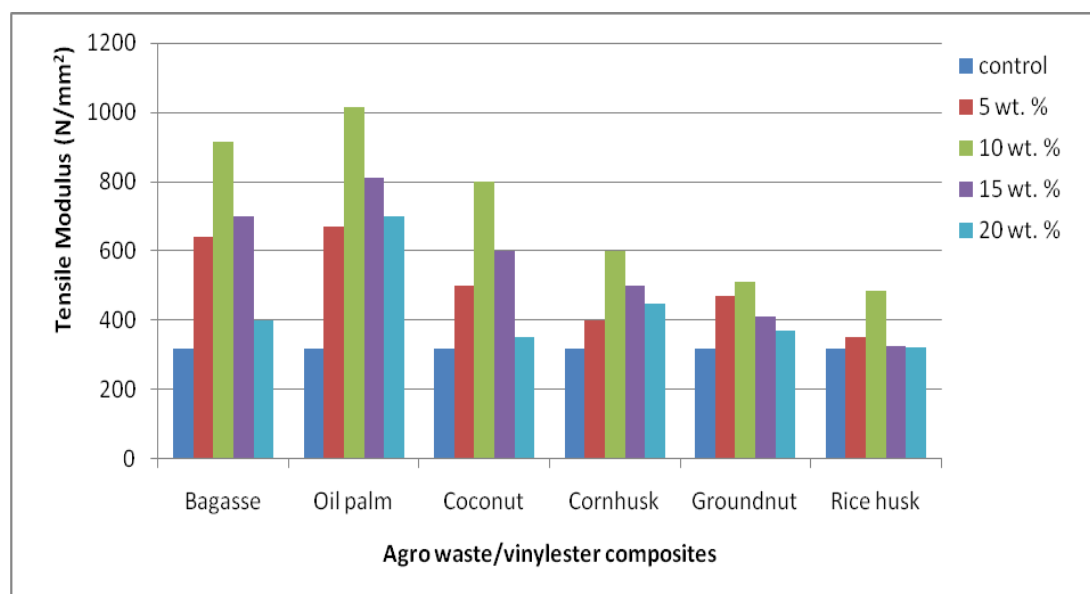


Fig.3: Comparison of tensile modulus of composites as function of fiber weight content

Figures 2 and 3 illustrate the ultimate tensile strength and modulus respectively, of fiber/vinylester composites made with various fiber types. Maximum tensile strength and modulus of the fiber/vinylester composites was observed at 10 wt. % of fiber loading for all the composites. In other words, the ultimate tensile strength (UTS) increased as the fiber weight content increased up to a verge point of 10 wt% before experiencing reduction. This was expected to happen because as the fiber content increased, the propensity for the fiber/matrix bonding strength to decrease was high. As shown, 5-10 wt% reinforcement gave better results than 15-20 wt% for the UTS because at low fiber content, the fibers are wetted properly by the vinyl ester and there is little or no fiber in contact with one another. However, at higher fiber content, the reverse was the case, the fibers were touching one another thereby reducing proper fiber wetting and bonding between the fibers and the vinylester matrix. This actually results to the reduction of the strength of the composites at this higher fiber content.

The result of the effect of fiber content on the tensile modulus was shown in Figure 3 where similar trends to that of the UTS were observed. However, there were slight differences in

the trend as the modulus for the 15 wt% fiber reinforced sample has higher value than the 5 wt% reinforced sample. The tensile modulus for 10 wt% reinforced sample emerged as the best with a value of 1015.1 N/mm<sup>2</sup> for oil palm fiber compared to unreinforced vinyl ester matrix with a value of 318.30 N/mm<sup>2</sup>.

The boost in the UTS and modulus at the presence of cellulosic fibers was expected as the mechanical properties of the composites are determined by several factors, such as nature of the reinforcement fiber, fiber aspect ratio, fiber-matrix interfacial adhesion, and also the fiber orientation in the composites. One of the most important parameters controlling the mechanical properties of short fibers composite is the fiber length or more precisely its aspect ratio (length/width). A high aspect ratio is very crucial to fiber reinforced composites, as it indicates potential strength properties. As can be seen from Table 2, oil palm fiber has high fiber length and aspect ratio compared to the other cellulosic fibers.

At high weight fractions of fibers, above 10 wt. % tensile strength decreases due to the filler high volume incorporated into the vinyl ester matrix. The agglomeration and the poor dispersion of the fibers into the vinyl ester matrix had a significant effect on the mechanical properties of the composites compared to the neat matrix strength.

### *3.2 Tensile strain*

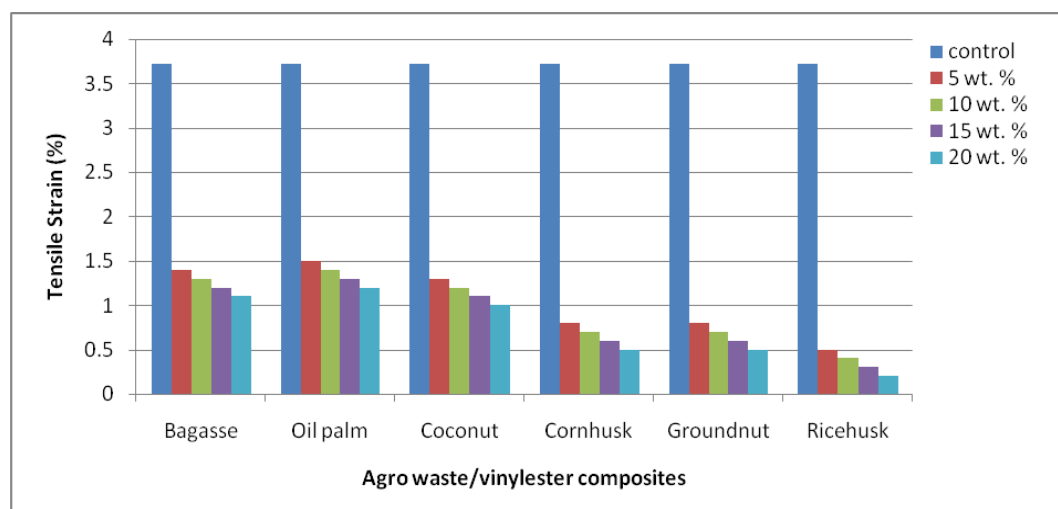


Fig.4: Comparison of tensile strain of composites as function of fiber weight content

Figure 4 shows the tensile strain result. It was observed that the unreinforced vinyl ester matrix has the highest tensile strain property of 3.72 % followed by 5wt% fiber content reinforced sample with a value of 1.5 % for the oil palm fiber/vinylester composite. It was observed that the tensile strain property reduced as the fiber content increases from 5-20 wt% for all the composites. The agro waste fibers provided reinforcements effects in the vinyl ester matrix because the stiffer the material, the greater the strength and modulus as revealed in Figures 2 and 3 and hence the lower the tensile strain. A decrease in strain as the filler content increased was observed indicating the presence of a poor interfacial adhesion between the hydrophilic fiber and the hydrophobic vinylester which does not allow efficient stress transfer between the two phases of the bio-composites.

### 3.3 Hardness

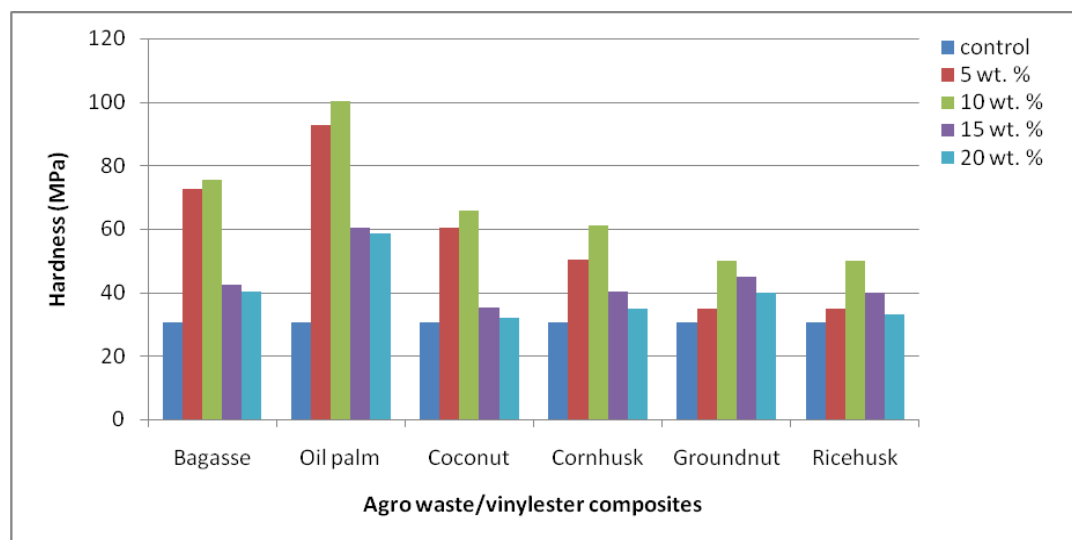


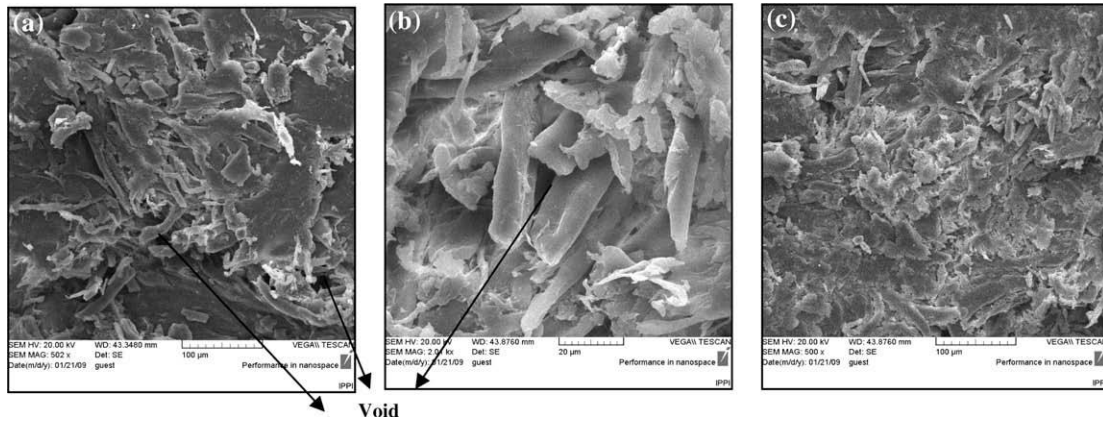
Fig. 5: Comparison of hardness of composites as function of fiber weight content

Hardness property is a measure of the resistance of the materials to surface indentation and wear. Figure 5 shows the variation of this property with the samples. It was noticed that the reinforcements lead to the enhancement of the hardness property in all the samples produced. The trend was similar to the UTS result. This shows that both the UTS and the hardness were enhanced in the same manner. The result shows that 5-10 wt% reinforced samples gave the best hardness property where the 10 wt% reinforced sample exceeded the 5 wt% reinforced sample with values of 100.2 MPa and 92.7 MPa for oil palm fiber respectively compared to the unreinforced vinyl ester matrix with a value of 30.40 MPa. Improvement of mechanical properties was possible due to adequate wetting and bonding between the fibers and the vinyl ester.

### 3.4 Morphology characteristics

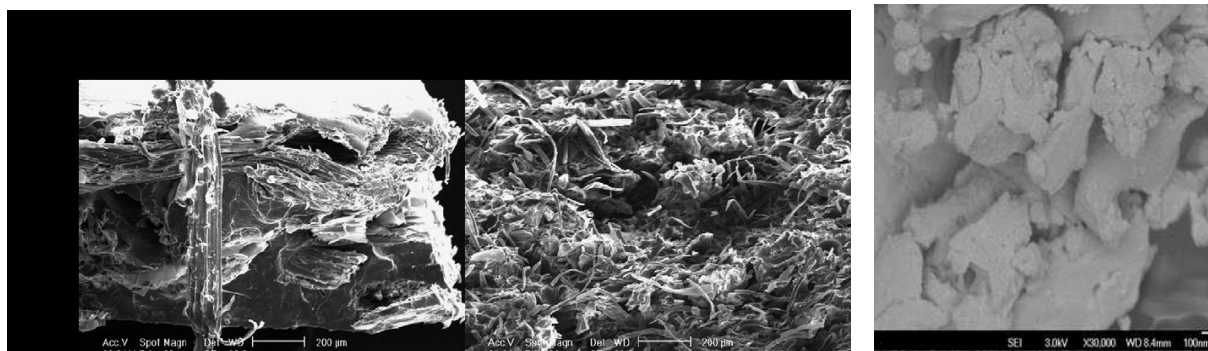
SEM is an effective method for the morphological investigations of the composites. Through SEM study the distribution and compatibility between the fibers and the matrix could be observed. The tensile fracture surfaces of the composites at 10 wt. % fibers loading are shown

in Fig. 6-11 respectively. In the case of the composite made with oil palm, the filler particles are well dispersed in the matrix polymer, as compared with the composites made with the bagasse, groundnut shell, coconut coir, corn husk and rice husk. There are some voids where the fibers have pulled-out. The presence of these voids means that the interfacial bonding between the fiber and the matrix is weak.



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Fig.6:10 wt % rice husk fiber      Fig. 7:10 wt % corn husk fiber      Fig.8:10wt % groundnut shell



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Fig.9:10 wt % coconut fiber      Fig.10:10 wt % oil palm fiber      Fig.11: 10wt% bagasse fiber

## Conclusion

In the present study, morphological and mechanical properties of vinyl ester eco-friendly composites reinforced with agricultural waste fibers have been examined. From the results and discussion presented above, the following conclusions can be made:

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i) This work shows the doing well manufacture of vinyl ester and the agro waste fibers composites by compounding and compression molding.

ii) The micro structural differences of the polymer composite are the most important factor responsible for the improvements in the mechanical properties.

iii) Based on the results, it is suggested that these composites can be used in the manufacture of low strength automobiles and other structural applications.

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