

## The Influence of Chemical Treatment on Mechanical properties and Biodegradability of Sugarcane fibre Filler Epoxy Resin

Abdulafeez O. Akorede<sup>1</sup>, Benjamin Dauda<sup>2</sup>, Modupe. M. Adeyemi<sup>1</sup>

<sup>1</sup>Department of Chemistry, Nigerian Defence Academy, P.M.B 2109, Kaduna, Nigeria.

<sup>2</sup>Department of textile Science and Technology, Ahmadu Bello University, P.M.B 2555, Zaria, Kaduna State.

Corresponding Author : Abdulafeez O. Akorede

**ABSTRACT:** Natural fibres appears to be the material which will become a feasible replacement for non-renewable and expensive synthetic fibres due to its large quantity availability, light weight, and most of all biodegradable. Investigation were made to improve the adhesion between sugarcane bagasse fibre and epoxy resin by chemically treating the bagasse with sodium hydroxide (NaOH), acetic acid and grafting of methacrylic acid (MAA). Prior to treatment, the bagasse were washed, boiled in water for 30 minutes, sundried for 7 days and pulverized to obtain particle size of 0.60 – 2.36 mm. After each treatment, epoxy resin composite was fabricated by Hand lay-up method on which mechanical and biodegradation tests was carried. The result showed that sample treated with Sodium Hydroxide (SHTB) has the highest strength (9.73 MPa) compared to other treatment and greatest degradation (7.63 %) in compost condition. Sample treated by grafting methacrylic acid on the bagasse fibre (MAAGB) has a longer elongation (1.42 mm), higher water absorption (4.72 %), higher energy absorption (73.50 J) and greatest degradation (1.50 %) in soil burial condition while acetic Acid Treated Bagasse (ACTB) shows the lowest strength (3.64 MPa), lowest flexibility resistance (3.22 %) and slowest degradation in soil burial (0.26 %) and compost condition (1.33 %). It can be concluded that particulate bagasse is a filler and not a reinforcement which resulted in degradation of the composites.

**KEYWORDS:** Sugarcane fibre, Epoxy resin, Chemical treatment, Grafting, Mechanical properties, biodegradation.

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### I. INTRODUCTION

The increasing environmental concern throughout every field of studies has made material Scientist to research on Natural Fibre Reinforced Polymer Composites (NFRPCs) that are environmental friendly. Environmental friendly in terms of renewability, biodegradability, non-emitter of greenhouse gases and also the NFRPCs absorbs the same amount of carbon dioxide it emits<sup>[1]</sup>. Natural fibres have different origins such as wood, pulp, cotton, bark, bagasse, corncobs, bamboo, cereal straw and vegetable (such as, flax, jute, hemp, sisal and ramie)<sup>[2]</sup>. These fibres are mainly made of cellulose, hemicelluloses, lignin and pectins, with a small quantity of extractives such as fatty acids, resin chemical, waxes and so on<sup>[3]</sup>.

A composite material is a material made from two or more constituent materials (called the filler and matrix) with significantly different physical and chemical properties that, when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure<sup>[4]</sup>. The use of cellulose fibres as fillers in composite materials based on polymeric matrices has recently attracted a growing interest, as shown by the large number of articles and reviews published in the past decade<sup>[2][5-8]</sup>. The main driving force of this trends resides in the potential of these fibres as serious substitutes to conventional glass fibres in many applications, including composites for the automotive and building sectors<sup>[9]</sup>. The volume and numbers of application of composite materials has grown steadily, penetrating and conquering new markets ranging from the everyday products to engineering construction materials<sup>[10]</sup>. The advantage of composite material over conventional materials reside their higher specific strength, stiffness and fatigue characteristics which makes structural design and material design to be more versatile<sup>[1]</sup>. Compared with conventional inorganic fillers such as glass fibres and carbon fibres, natural fibres provides wide advantages including abundance resulting in low cost, biodegradability, flexibility during processing, minimal health hazards, low density, desirable fibre aspect ratio, moderate tensile and flexural

modulus. Incorporating the tough and light-weight natural fibres into polymer (thermoplastic and thermoset) matrices produces composites with a high specific stiffness and strength<sup>[11]</sup>.

Although, NFRPCs is limited by their hydrophilic character, which arises from the presence of hydroxyl groups in the cellulose structure<sup>[2]</sup>. The usual polar fibres have low compatibility with non-polar polymer matrices, especially hydrocarbon matrices such as polypropylene and polyethylene<sup>[3]</sup>. The notable difference in the surface properties is inadequate adhesion between the fibre and the matrix<sup>[10]</sup>. Thus, the efficiency of the load transfer across the fibre to matrix is poor and results in composite materials display low strength properties<sup>[12]</sup>. To eliminate these shortcomings, natural fibres are modified by chemically treating the fibres, which is considered to be the best surface modification<sup>[13]</sup>.

Sugarcane is the world largest crop which is mainly grown for sugar production in more than 100 countries<sup>[14,15]</sup>. Sugarcane is mainly composed of about 12% of sucrose, 15% fibre and 70% sugar<sup>[16]</sup>. Bagasse is the remaining dry fibre after sugar-containing juice in the sugarcane is extracted from the stalk<sup>[17]</sup>. A good number of researches has been carried on sugarcane bagasse including the use of sugarcane bagasse as activated carbon for clean-ups and water treatment. Chemical treatment of sugarcane bagasse has attracted more attention in Nigeria because of its high availability leading to environmental pollution. In other to fill the gaps in the published researches, this research was focused on treating less than 3 mm bagasse particles with sodium hydroxide, acetic acid and grafting of methacrylic acid monomer, fabrication of epoxy resin filled with treated bagasse and finally investigating and relating the mechanical and biodegradability properties of resultant composites.

## II. MATERIALS AND METHODS

### Materials

All materials used in this research was sourced and procured in Nigeria. The epoxy resin and hardener was purchase form AG Scientific Company, Kaduna, Kaduna state while the sugarcane was purchased in a commercial sugarcane farm in Pateka along Express, Kaduna, Kaduna state.

### Methods

#### Pretreatment of Sugarcane Fibre

The collected Bagasse was washed and boiled in water for 30 minutes to remove sugar. The bagasse was further dried under the sun until complete dryness for 7 days followed by pulverizing and sieving. The yielded particle size was between 0.6-2.35 mm in length. Pre-treatment was done to remove any impurities or foreign particles present on the surface of the fibre that might affect the treatment process.

#### Fibre Modification

The pretreated bagasse was divided into four portions (10 grams each), one was left as Untreated Bagasse (UTB) while the other three were treated with sodium hydroxide, acetic acid and grafted with methacrylic acid monomer using 1:50 mass to volume ratio and designating each treated bagasse as SHTB, ACTB, and MAAGB respectively. The choice of chemical concentration were based on literature reviews<sup>[2][8][18][19]</sup>.

#### Sodium Hydroxide Treatment

The pre-treated fibre was subjected to mercerization by soaking 10 gram of sugarcane fibre in 500 ml of 2% (w/v) sodium hydroxide solution for an hour at 25±1°C. The alkaline treated fibre was then thoroughly rinsed with distilled water to remove sodium hydroxide on the surface of the fibre and the pH was measured to ensure the fibre is neutral (pH 6-7). Then, the sodium hydroxide treated sugarcane fibre was dried in oven for 24 hours at 60°C.

#### Acetic Acid Treatment

The pretreated bagasse fibre (10 g) were soaked in 500 ml of 2 % acetic acid (v/v) at room temperature (25±1°C) for an hour. Then, the acid treated fibre was thoroughly rinsed with distilled water to remove acetic acid on the surface of the fibre and the pH were measured to ensure the fibre is neutral (pH 6-7). Then, the acetic acid treated sugarcane fibre was oven dried at 60°C.

#### Grafting of Monomer on Bagasse

Grafting of methacrylic acid monomer (MAA) on bagasse is a stepwise process that involve extraction of cellulose from the bagasse followed by redox reaction and finally reaction with the intended methacrylic acid.

**Cellulose Extraction:** Bagasse (10 g) was soaked in 500 ml of 2 % (w/v) NaOH for 30 minutes. The treated fibre was sieved and washed with distilled water before treatment with 500 ml of 17.5% (w/v) NaOH at 50 °C

for an hour. The treated fibre was then repeatedly washed with distilled water, sieved and dried in the oven at a temperature of 60 °C to constant weight.

**Bleaching:** Cellulose extracted bagasse were further treated with 500 ml of 2% (v/v) acetic acid in 2 % (w/v) Sodium chloride. The bleached bagasse was repeatedly rinsed with distilled water to remove excess reagents and then dried at a temperature of 70 °C to constant weight.

**Treatment with Potassium Permanganate (KMnO<sub>4</sub>):** The bleached fibre (3.75 g) was treated with 100 ml of 0.03 M concentration of KMnO<sub>4</sub> for 15 minutes at 70 °C in water bath. The fibre was washed with distilled water and squeezed in between two filter papers.

**Graft Polymerization:** The permanganate-treated sample was put in reaction bottle containing Methacrylic acid monomer (MAA) (100% weight of fibre), 100 ml of citric acid (0.03 M) was further added followed by stirring and heated in water bath at 70 °C for two hours. After two hours, the mixture was sieved and the residue was repeatedly washed with cold water, then hot water to remove homopolymer of MAA and ungrafted monomer. Then the fibre was dried at room temperature and labelled Methacrylic Acid Grafted on Bagasse (MAAGB).

### Composite Fabrication

The bagasse fibre-epoxy resin fibre was fabricated using hand lay-up technique. This method is an easy to use and low cost technique. Epoxy resin and hardener was gently mixed in 2 to 1 ratio before the addition of bagasse and poured in the mold. The composite was left to cure for four hours at room temperature.

### Mechanical Testing of the Composites

#### Tensile Strength Tests

Tensile property test was carried out in accordance with ASTM D638-06 using Testometric Universal testing machine (FS 50AT) using a load cell of 50KN. Running at a cross head speed of 10 mm/min. The tensile test, the specimen elongation and the resistance of the specimen was recorded until the rupture of the specimen occurred.

#### Flexural Strength Test

Flexural strength is the combination of tensile strength and compressive strength. The 3-point tests were carried out using Universal testing machine in accordance with ASTM D790. Each sample rest on two supports and loaded by means of a loading nose midway between the supports. The samples were deflected until rupture occurs in the outer surface of the samples or until a maximum strain of 5 % is reached.

**Impact Strength Test:** Avery Denison Impact Testing Machine was used to conduct the impact test in accordance with ASTM D 256.

#### Water Absorption Test

Samples of each composite grade was oven dried before weighing in accordance with ASTM D5229M-12. The initial weight of the composites were recorded. The samples were placed in water maintained at room temperature (25 °C) and for a time interval of 24 hours. The samples were then removed from water and weighed. The weight measurement will be taken periodically for 30 days.

The percentage water absorption was calculated using the below formula:

$$\% W = \frac{Wt - Wo}{Wo} \times 100$$

Where: Wt = final weight and Wo = initial weight.

#### Biodegradation Testing

The biodegradation of the composites were carried in three condition; Weather, Soil burial and Compost condition.

#### Degradation by Weather

Weather testing of composites is the controlled polymer degradation and polymer coating degradation under lab or natural conditions. Samples were weighed and then exposed to the natural weather for up to 30 days. The samples was weighed after 10days for a month to determine the weight loss using the below formula.

$$\% D = \frac{Dt - Do}{Do} \times 100$$

Where: Dt = weight before degradation, Do = weight after degradation.

### Soil Burial Degradation

Soil burial is a traditional and standard method for degradation because of its similarity to actual conditions of waste disposal. Biodegradability of the samples was studied by weight loss over time in a soil environment. Samples were weighed and then buried in the soil at a depth of 5 cm for 30 days. The soil moisture was maintained to keep the microorganisms active.

### Degradation by Compost

Compost degradation was carried out by following the ASTM D5338. The biodegradation of the specimen was checked after 10, 20, and 30 days. Exactly 5g of treated and untreated composites were placed in 250ml Pyrex bottle containing compost. For the survival of microorganisms of the compost O<sub>2</sub>, temperature and water are required. Water was poured in the bottles in every two days to keep microorganisms active.

## III. RESULTS AND DISCUSSION

### Tensile Properties

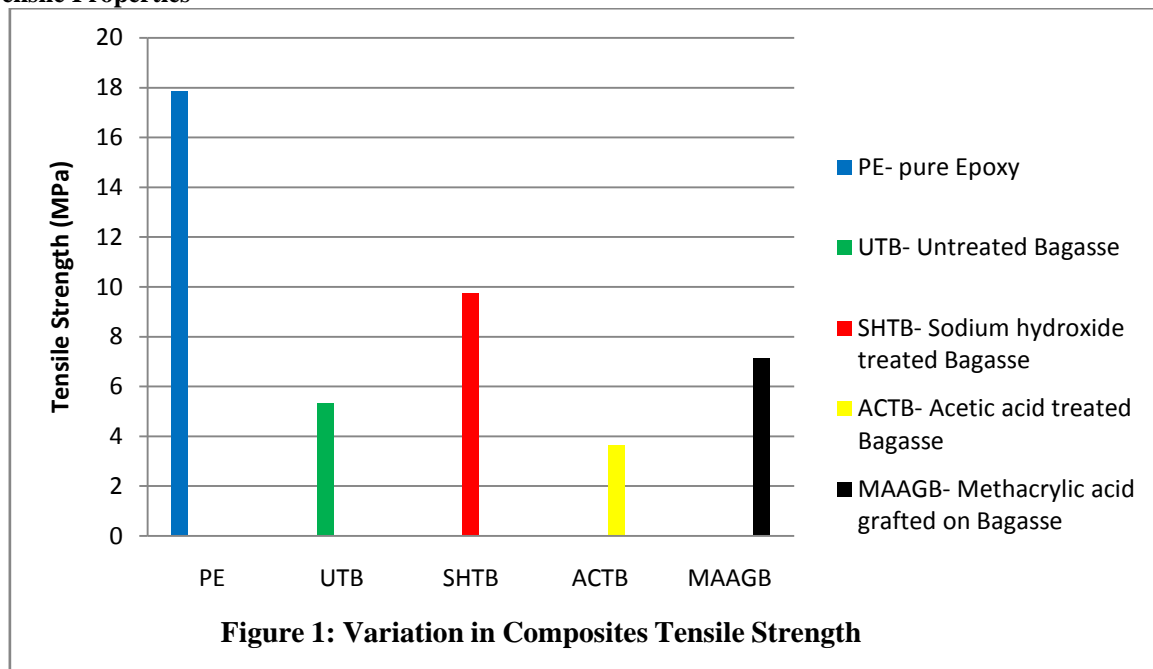
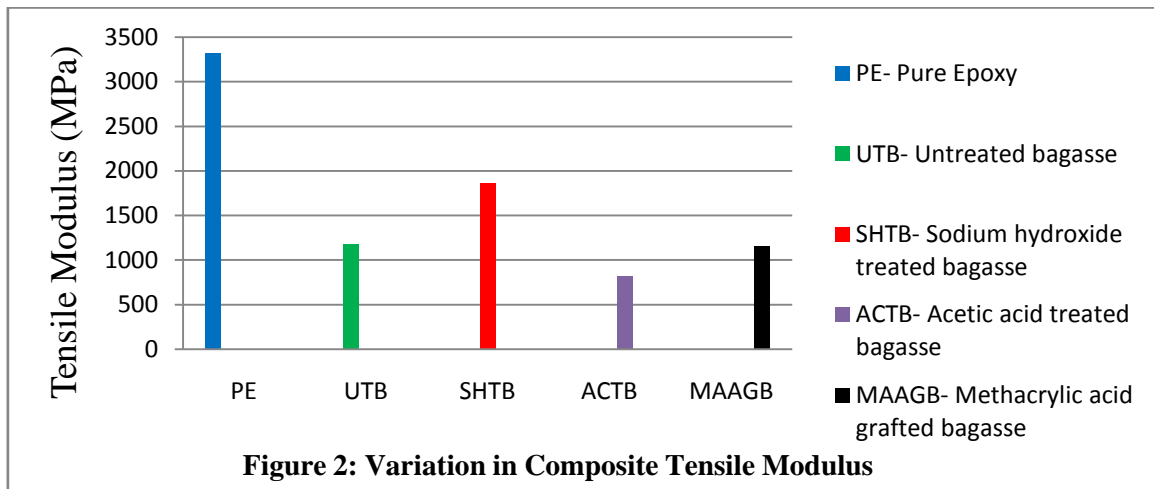


Figure 1 above represents the various tensile strength of epoxy resin and sugarcane bagasse; Pure Epoxy resin (PE), Untreated bagasse (UTB), Sodium hydroxide treated Bagasse (SHTB), Acetic acid treated bagasse (ACTB) and Methacrylic acid grafted on bagasse (MAAGB). The chart shows that PE has the highest tensile strength. It is observed that no enhancement in tensile properties of epoxy resin by reinforcement of bagasse fibre. The reason is most likely because bagasse fibre has much lower tensile strength compared to epoxy resin. The strength of SHTB decreased by 54% compared to pure epoxy strength. This indicates that treating of bagasse with Sodium hydroxide successfully modified the fibre which led to improvement in tensile strength. One of the known effects of NaOH treatment is that 'OH' group which gives cellulose fibres their hydrophilic nature has been reduced significantly so that there is more compatibility of fibre and epoxy. This will enhance proper transfer of load from matrix to fibre, leading to improved tensile strength. ACTB showed the least tensile strength which indicates that treatment of bagasse with acetic acid led to reduction in strength compared to untreated bagasse. It is probable that some hydrolysis (deplomerization) of the cellulose molecule of the bagasse must have taken place, leading to a reduction in strength of bagasse fibres. Grafting of methacrylic acid monomer on bagasse improved strength of the fibre compared to untreated fibre. Methacrylic acid polymer grafted onto the bagasse surface has made bagasse surface more compatible with epoxy matrix, leading to better load transfer between the matrix and the fibres; hence, the improved tensile strength when compared to the ungrafted fibre. Therefore, treatment of bagasse with sodium hydroxide and grafting with

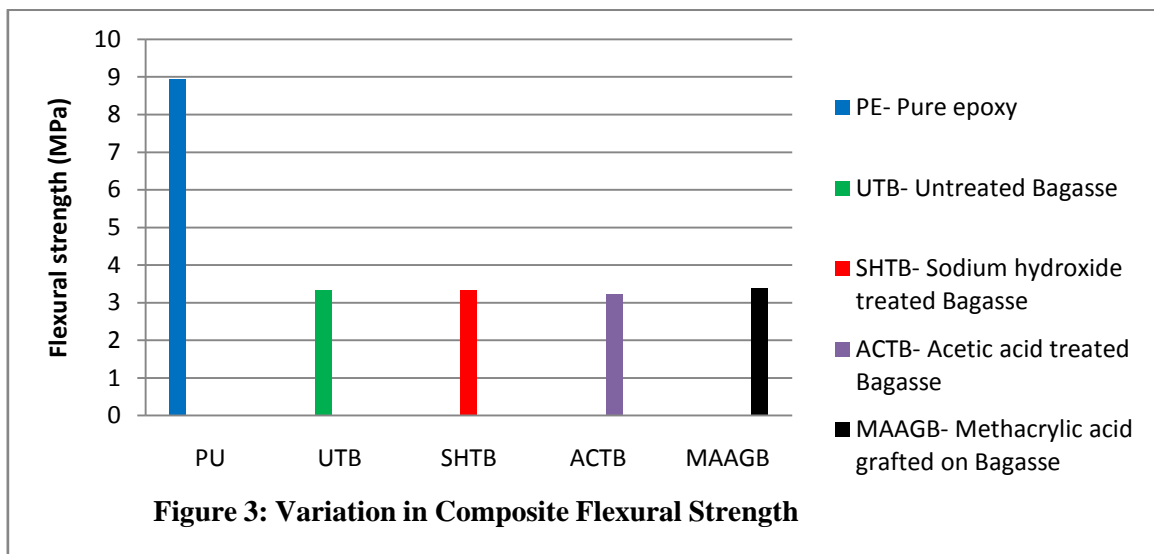
methacrylic acid monomer increases the strength of the composite than treated composite. Although filling Bagasse fibre in epoxy resin has reduced epoxy resin tensile strength.



The result in figure 2 shows that PE has the highest load holding capacity before expansion of the composite. In comparison of treated bagasse to the untreated bagasse, SHTB has a higher modulus than the untreated and other chemically treated fibres. Alkali treatment using NaOH has been known to be very effective for surface modification of natural fibres due to removed 'OH' groups which improve the interaction between the fibre and the matrix. Thus the superior tensile modulus of SHTB composite is probably as a result of this effect. MAAGB shows a slight decrease of tensile modulus compared to UTB. It is probable that MAAGB shows improved modulus if the concentration and time is varied. From the fabricated composite consisting of bagasse, bagasse treated with sodium hydroxide has the highest ability to withstand stress before elongating while bagasse treated with acetic acid shows the least ability.

**Flexural Strength**

Flexural strength is the ability of a material to hold stress applied perpendicular to its axis before bending. The result in figure 3 shows that PE has high flexural stress holding capacity before bending compared to other composites. Since Untreated bagasse (UTB) and treated bagasse (SHTB, ACTB and MAAGB) have practically the same average flexural strength it means that treatment with Sodium hydroxide and with MAAGB shows no significant difference in flexural strength against bending. These results show that chemical treatment of bagasse has little improvement against bending property of the fibre. Therefore the fibre reinforced composites are highly susceptible to bending upon application of little force. The reason for this is not immediately apparent.



Flexural modulus is the ratio of stress to strain in bending deformation. It determines the tendency of a material to resist bending. Figure 4 shows that PE has the highest resistance to bending. ACTB shows a higher resistivity to bending compared to untreated bagasse which has the least resistivity to bending. SHTB also shows an increase flexural modulus while MAAGB shows an improvement to resist bending compared to UTB and SHTB. Thus, it can be deduce that chemically treating bagasse led to improvement in composite resistivity to bending.

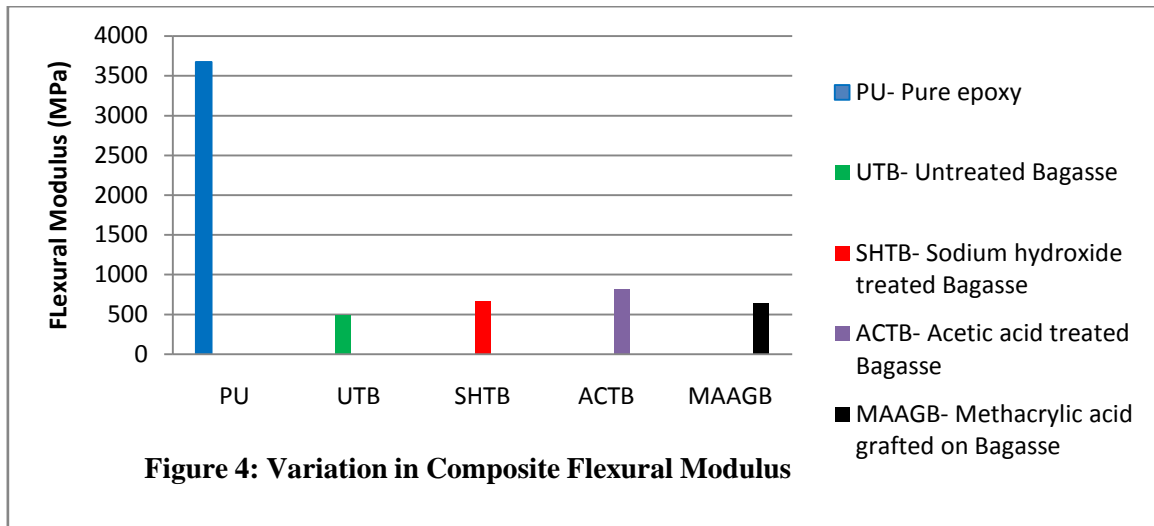


Figure 4: Variation in Composite Flexural Modulus

**Elongation**

Elongation is the process of increase in length of material as a result of stress applied on the material. The result in figure 5 shows that MAAGB has the highest elongation indicating it can experience higher strain compare to other composite. SHTB shows greater elongation compared to UTB because of the removal of Hemicellulose and Lignin, which are stiffening materials. PE has greater elongation than UTB but not more than MAAGB, indicating that the grafting of MAA polymer on the fibre significantly enhances its elongation and also that the composite that it is incorporated into. ACTB shows the lowest elongation, which can be explain that modification of bagasse with acetic acid makes the fibre to be more rigid compared to UTB, SHTB and MAAGB. It can be deduce that MAAGB can elongate better than other composite.

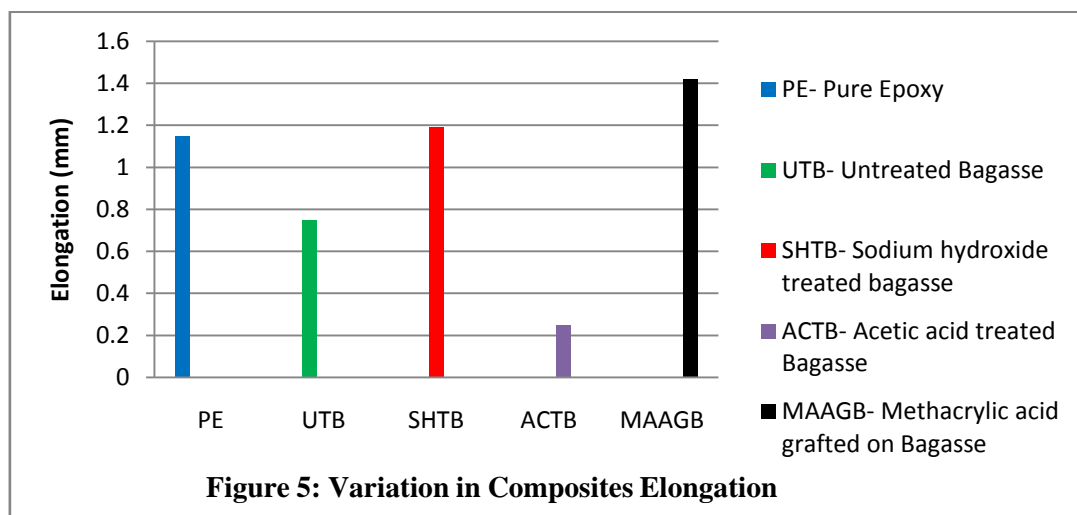
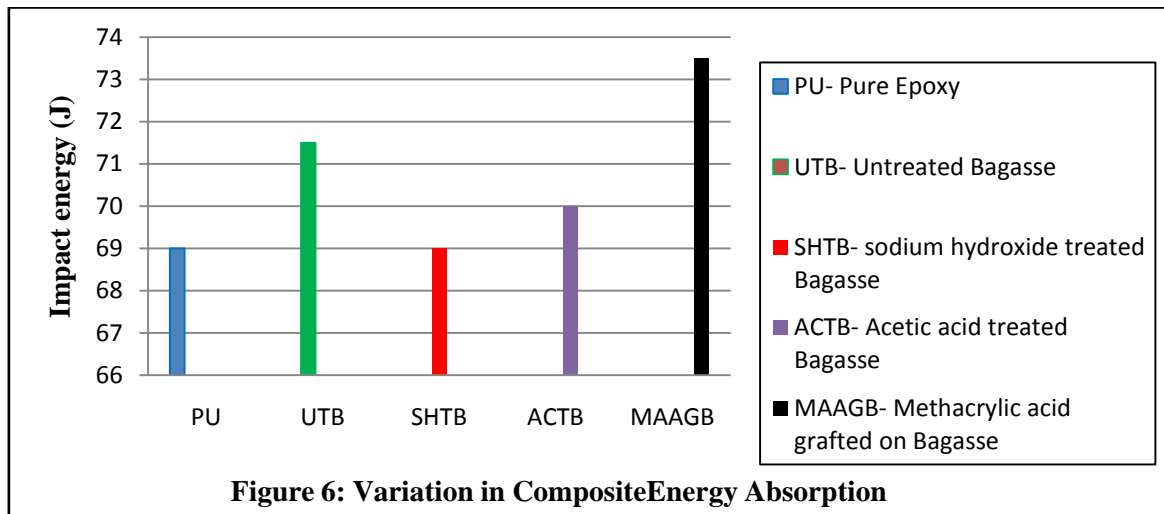


Figure 5: Variation in Composites Elongation

**Impact Properties**

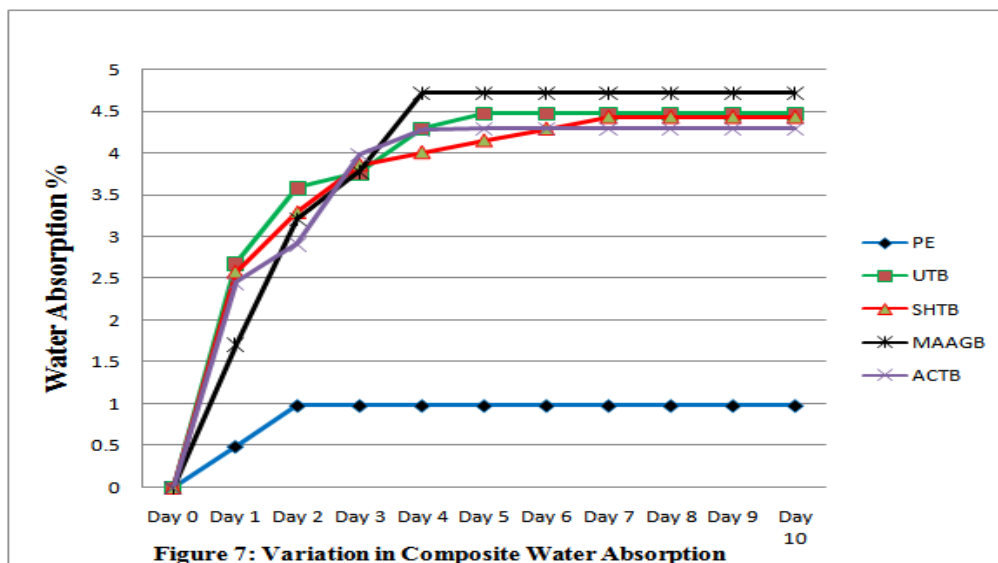
Impact test measures the energy required to break the samples under standard condition. This test determines the amount of energy the composites can absorb during fracture which is related to the composite toughness. From figure 6 the result of the test shows that grafting of methacrylic acid monomer (MAA) on bagasse improve the amount of energy the composite can absorb. SHTB shows a slight decrease in energy

absorbed compared to MAAGB and ACTB. Although ACTB shows increase in energy absorb than untreated fibre (UTB), SHTB and PE.



**Water Absorption**

Fabricated bagasse reinforced epoxy samples (5 g) were immersed in water. Figure 7 implies that PE has the lowest water absorptivity which is due to lack of bagasse in the sample. MAAGB show the higher absorptivity than UTB, which explains that grafting of MAA on bagasse increase its water absorptivity than the untreated fibre. ACTB and UTB showed the lowest absorptivity amongst the treated samples, which shows that treating bagasse with acetic acid and NaOH will reduce its water absorptivity by the same value. As it is well known the ‘OH’ group in cellulose fibre such as bagasse is responsible for its good water absorption capacity.



**Biodegradability**

The samples were subjected to three conditions for thirty days; Weather, Soil burial and Compost.

**Degradation by Weather**

It worth noting that samples subjected to weather condition shows no weight loss due to absence of micro-organisms.

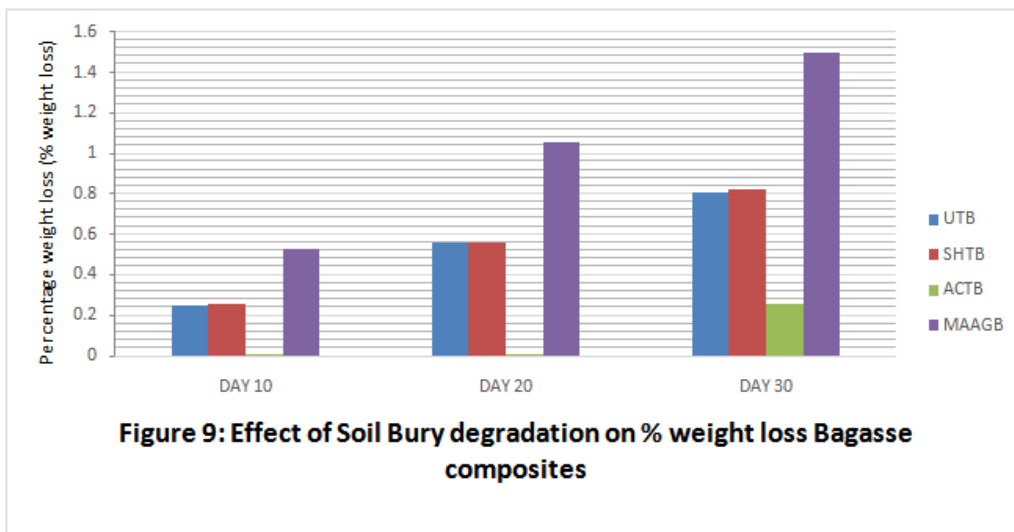
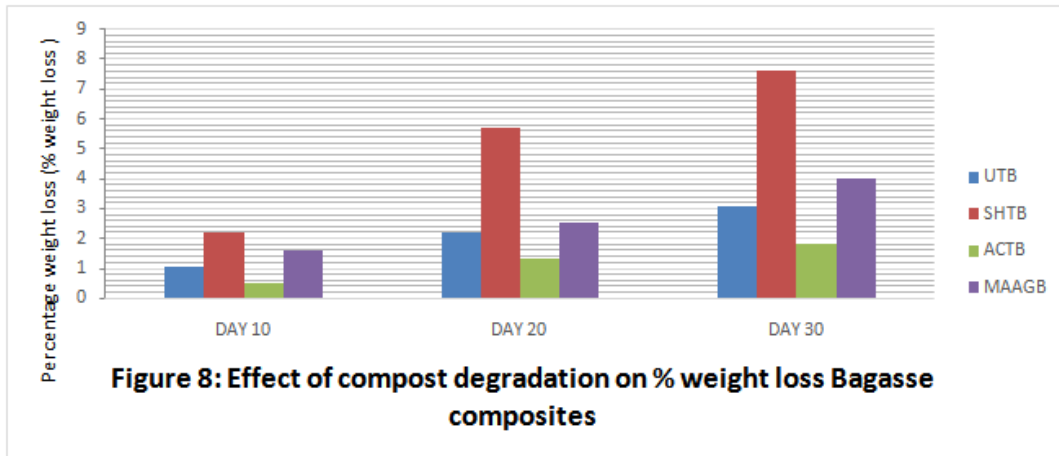
**Degradation by Compost**

Figure 8 shows the variation of percentage weight loss as a function of time for bagasse fibre reinforced epoxy resin of both treated and untreated fabricated composites subjected to Compost degradation. It

is observed that that Bagasse treated with Sodium hydroxide has more tendencies to decay than the other treated fibres and untreated which indicated that treating the fibre with Sodium hydroxide will hasten degradation process of bagasse composites. The zero loss in weight of pure epoxy is due to the absence of fibre in pure epoxy resin which indicates that no degradation occurs over 30 days of biodegradation process.

**Degradation by Soil Burial**

Figure 9 shows the percentage weight loss over time when the samples were subjected to soil burial which is natural decomposition condition of materials. It is observed that grafting of bagasse with MAA improve biodegradability process compared to untreated and treated bagasse composite. Comparing soil burial, weather and compost conditions it is observed that samples subjected to compost condition has the highest degradation by percentage weight loss. This can be attributed to high micro-organisms activity on the samples.



**IV. CONCLUSION**

From the present research study, it is concluded that bagasse is a good agro-waste to be used as epoxy filler because of its good mechanical properties which can be improved by chemically treating the bagasse. The aim of introducing a composite material that has better strength was not achieved but better flexibility, moderate water absorption, high energy absorption and a greater degree of degradation was achieved. It was also established that;



- Treating bagasse fibre with Sodium hydroxide (NaOH), Acetic acid and Grafting of methacrylic acid (MAA) enhanced strong bond between treated fibre and epoxy resin polymer.
- The result showed that composites treated with Sodium Hydroxide (SHTB) has the highest strength compared to other treatment and greatest degradation in compost condition.
- Sample treated by grafting Methacrylic acid on the bagasse fibre (MAAGB) has longer elongation, higher water absorption, higher energy absorption and greatest degradation in Soil burial condition while
- Acetic Acid Treated Bagasse (ACTB) shows the lowest strength, lowest flexibility resistance and slowest degradation in soil burial and compost condition.

The results obtained implies that Grafting of methacrylic acid on sugarcane has the highest effect on improving the adhesion between bagasse fibre and epoxy resin but since filling epoxy resin with bagasse fibre did not improve tensile strength of the composites then particulate bagasse can be as filler that induce considerable degradation of the composites.

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**Table 1: Tensile, tensile modulus, Flexural, Flexural Modulus, and Impact test result for untreated and treated composite.**

| S/N | composites | Tensile strength (MPa) | Tensile modulus (MPa) | Flexural strength (MPa) | Flexural modulus (MPa) | Elongation (mm) | Impact (J) |
|-----|------------|------------------------|-----------------------|-------------------------|------------------------|-----------------|------------|
| 1   | PE         | 17.86                  | 3317                  | 8.94                    | 3673                   | 1.15            | 69         |
| 2   | UTB        | 5.33                   | 1181                  | 3.34                    | 487                    | 0.64            | 71         |
| 3   | SHTB       | 9.73                   | 820                   | 3.34                    | 673                    | 1.18            | 69         |
| 4   | ACTB       | 3.64                   | 1863                  | 3.22                    | 812                    | 0.20            | 70         |
| 5   | MAAGB      | 7.15                   | 1157                  | 3.39                    | 643                    | 1.40            | 73.5       |

**Table 2: Effect of Chemical treatment on Composite water Absorption**

|       | Day 1 (%) | Day 2(%) | Day 3(%) | Day 4(%) | Day 5(%) | Day 6(%) | Day 7(%) | Day 8(%) | Day 9 (%) | Day 10 (%) |
|-------|-----------|----------|----------|----------|----------|----------|----------|----------|-----------|------------|
| PE    | 0.5       | 0.98     | 0.98     | 0.98     | 0.98     | 0.98     | 0.98     | 0.98     | 0.98      | 0.98       |
| UTB   | 2.60      | 3.55     | 3.70     | 4.35     | 4.50     | 4.50     | 4.50     | 4.50     | 4.50      | 4.50       |
| SHTB  | 2.60      | 3.25     | 3.75     | 4.00     | 4.10     | 4.19     | 4.29     | 4.29     | 4.29      | 4.29       |
| ACTB  | 2.45      | 2.90     | 4.00     | 4.29     | 4.29     | 4.29     | 4.29     | 4.29     | 4.29      | 4.29       |
| MAAGB | 1.60      | 3.25     | 3.75     | 4.72     | 4.72     | 4.72     | 4.72     | 4.72     | 4.72      | 4.72       |

**Table 3: Effect of Chemical treatment on Composite Compost condition Weight loss**

| S/N | Composite Name | Day 10 (% weight loss) | Day 20 (% weight loss) | Day 30 (% weight loss) |
|-----|----------------|------------------------|------------------------|------------------------|
| 1   | PE             | 0.00                   | 0.00                   | 0.00                   |
| 2   | UTB            | 1.02                   | 2.22                   | 3.10                   |
| 3   | SHTB           | 2.22                   | 5.73                   | 7.63                   |
| 4   | ACTB           | 0.50                   | 1.33                   | 1.83                   |
| 5   | MAAGB          | 1.80                   | 2.50                   | 4.00                   |

**Table 4: Effect of Chemical treatment on Composite Soil Burial Weight loss**

| S/N | Composite Name | Day 10 (% weight loss) | Day 20 (% weight loss) | Day 30 (% weight loss) |
|-----|----------------|------------------------|------------------------|------------------------|
| 1   | PE             | 0.00                   | 0.00                   | 0.00                   |
| 2   | UTB            | 0.25                   | 0.56                   | 0.81                   |
| 3   | SHTB           | 0.26                   | 0.56                   | 0.82                   |
| 4   | ACTB           | 0.00                   | 0.00                   | 0.26                   |
| 5   | MAAGB          | 0.53                   | 1.06                   | 1.50                   |

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