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Full Length Research Paper

Effect of calcium nitrate application on the structural behavour of okra (cv.Kirikou) fibre reinforced epoxy composite

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ABSTRACT: This experimental research was carried out to determine the effect of pre-harvest fibre treatment, on the structural behaviour of the epoxy composite. The okra (cv Kirikou) plants, from where the fibre, used for the production of the composite was obtained, was pre-harvest treated with calcium nitrate $Ca(NO_3)_2$. Five treatments, which were (0 mg/l, 100 mg/l, 200 mg/l, 300 mg/l and 400 mg /l) $Ca(NO_3)_2$, were used in this research. The 0 mg/l $Ca(NO_3)_2$ was taken as the control experiment. The Okra stem were cut at 60 days after planting, and fibre extracted through retting method. The epoxy composite samples were produced by partial replacement of the matrix with 20% (by weight), of the processed okra fibre. The composites production and testing were conducted according to the ASTM International recommended procedures. Results obtained from the laboratory tests showed that pre-harvest treatment had significant ($p \leq 0.05$) effect on the mechanical behaviour (tensile strength and flexural strength) of the composite samples. Tensile strength of the composite increased linearly, with increment in the treatment concentration. The untreated okra fibre reinforced epoxy composite recorded tensile strength of 34.73 MPa, while the okra fibre treated with 400 mg/l $Ca(NO_3)_2$ had a tensile strength of 57.87 MPa. Likewise, the untreated okra fibre reinforced epoxy composite had the lowest flexural strength (126 MPa), when compared the flexural strength of composite samples reinforced with treated okra fibre. These results showed the relevance of pre-harvest fibre treatment, on the mechanical properties of composites. This research will be useful in composite production and utilization, and had opened a new front in plant fibre production and management.

Keywords: Calcium nitrate, flexural, okra, pre-harvest, tensile

INTRODUCTION

Composite consists of two or more constituents, which are combined in a specific ratio, so that the composite (product) will be distinct but identifiable. The composite constituents must be able to improve most of the engineering properties of the products produced; therefore, composite often compensates for the mechanical fragility of an individual constituent (Verma *et al*., 2012). Composite are mainly produced with resins such as epoxy, phenolic, polyester, polyimide, polyurethane, silicone etc.; and reinforcement such as synthetic or natural fibres (or fillers). The composite product is usually described by the type of resin and reinforcement materials used (Sudheer *et al*., 2015; Potluri *et al*., 2017). According to You *et al*. (2015) and Sudheer *et al*., (2015), reinforcement size, volume and placement orientation, greatly influenced the mechanical properties of composite material produced. According to Reddy *et al*. (2011), the mechanical properties of Epoxy composites filled with betel nut short fiber at 10% volume exhibited better mechanical properties, when compared with the other reinforcement volume.

Recently, there is growing demand for plant fibres, because of their light weight, low cost, biodegradability, availability, environmental friendly, non-toxicity to human beings and fairly suitable engineering properties; when compared with synthetic fibers (Baley, 2002; Thakur *et al*., 2014). According to Chen et al. (2005), plant-based materials reinforced composites are widely used in aircraft components, such as; truss members, rotor blade, spars, etc. Maleque *et al* (2007) reported that the tensile and flexural properties of woven banana (*Musa* spp) fibre reinforced epoxy composite were better when compared with the pure epoxy sample; while the flexural strength increased from 45 MPa to 75 MPa with the addition of the woven banana fibre. Furthermore, John *et al.* (2010) observed that carbonized bagasse filler in composite production significantly increased its tensile strength, abrasion resistance, and hardness properties. Likewise, Mir *et al.* (2012) reported an increment in the tensile properties of coir fibre reinforced composite, when compared with unreinforced composite. Girisha (2012) studied the tensile properties of natural fibres (sisal, ridge gourd and coconut leaf sheath) reinforced epoxy-hybrid composites, and observed an improvement in the tensile properties of the composites produced, as the fibres volume increased from 5% to 25%. The engineering properties of sisal fibre reinforced epoxy composite were investigated by Algarraja *et al.* (2014), and observed that the incorporation of sisal (*Agave sisalana*) fibre into the composite greatly improved the mechanical properties of the composite. Apart from the low cost and other numerous advantages of the plant fibres, the utilization of plant fibres in composites will minimize agro waste disposal problem, and encourages waste-to-wealth policies.

For the past decades, several researchers had worked on the utilization of okra (*Abelmoschus esculentus* L.) parts in composites production. Reddy and Reddy (2016) studied the tensile properties of okra fibre reinforced polymer composite, and reported that the tensile strength of the composite increased linearly (from 23 MPa to 51.15 MPa), as the okra fibre volume increased from 0% to 20%. According to Rambabu *et al*. (2018), the mechanical properties of epoxy resin composite was improved by the incorporation of okra fibre. Okra fibre (chemically treated with 0.125 M NaOH solution) recorded a higher tensile strength and modulus (64.41 MPa and 946.44 MPa), when compared with pure polyester sample; likewise, the specific tensile strength and modulus was higher (34.31% and 39.84% respectively), when compared with pure polyester sample (Srinivasababu *et al.,* 2009).

Okra, which belongs to the *Malvaceae* family, is widely cultivated in many Africa to Asia, Southern European and America countries. The pods and seeds harvested from okra plant have a lot of nutritional, medicinal and industrial applications (Bryant *et al*., 1988; Zaharuddin *et al*., 2014). In 2018, about 10 million tons okra pods were produced globally, and Nigeria was the second leading country, with a total production of 2.04 million tons of okra pods (FAOSTAT, 2018). A total world production of 9.64 million metric tonnes was in 2017, with India accounting for 6 million metric tonnes, about 63% (FAOSTAT, 2019). According to the Food Agriculture Organization (FAO) data, Nigeria followed India with 2 million metric tons production in 2017 (20.83% of total production), harvested from 1.48 million hectares of land. Some okra varieties are; *kirikou, everluck, greenie, blondy, burgundy, clemson spineless, cow horn*, etc. These different okra varieties differ both in their morphology and cellular structures. According to Pramono *et al*. (2015) and Reddy and Reddy (2016), the engineering properties of the plant fibres are dependent on the plant variety, climatic conditions, maturity stage of the plant, and the composite production method.

Information on the effect of farming method on the tensile properties of plants' fibres, and the composites produced from them is lacking. Hence, this study evaluated the effect of pre-harvest calcium chloride treatment, on the tensile properties of okra (cv. Kirikou) fibre reinforced epoxy composite. The results obtained from the study will be useful in the construction and automobile industries.

MATERIALS AND METHODS

Materials

Epoxy resin and hardener

The epoxy (LY556), and the hardener (HY951) 951, used for this study were procured from a local chemical shop at Onitsha, Anambra State, Nigeria.

Okra plant

The okra (cv. Kirikou) seeds, used for this study were collected from the Department of Agricultural and Bio-environmental Engineering Technology, Delta State Polytechnic, Ozoro, Nigeria. Then the seeds were cultivated and maintained at the research centre of Delta State Polytechnic, Ozoro, Nigeria. Kirirkou okra is a local okra variety, widely cultivated in Nigeria (according to information obtained from local farmers), due to its early maturity age and high pods yield

Experimental design

Randomized complete block design (RCBD) was used for the study. The design consisted of 5 treatments; which were one control (distil water), and four (4) concentrations (100 mg/l, 200 mg/l, 300 mg/l and 400 mg/l) of calcium nitrate ($Ca(NO_3)_2$). Each treatment was had three replications. The treatments were applied on the okra plants twice weekly, starting from the three weeks after planting (WAP), until they were harvested for the laboratory tests. All other variables, such as; irrigation, diseases and pests control methods, weeding rainfall, etc., were the same in all the treatments. The okra seeds were planted at a spacing of 30 cm x 40 cm, in the experimental plots, which grossly measured 3 m x 4 m. During the treatment application, each plant was sprayed with a knapsack sprayer, until the plant body was completely soaked with the solution.

Okra stems harvesting

The okra stems were harvested at the peak maturity stage, that is, at 60 days after planting. All the harvested stems were inspected to remove of disease and pest infested stem. This is because; these diseases and pests infected stems will not produce high quality fibre.

Okra fibre extraction and preparation

The fibre was extracted from the okra stem using the retting method. The stems were harvest in green stage, before they were subjected the extraction process. During the process, the okra stems were totally immersed inside muddy water for a period of 10 days, at ambient environmental condition. Then on the 10^{th} day, the fibre was extracted from the stem, and washed thoroughly with clean water to remove all the pulp from the fibre. The fresh fibre was sun-dried for 5 days at ambient environmental conditions (Temperature ~ 29±5°C; Relative Humidity ~ 87±5%). sample.

Composite production

The composite was produced by using the hand-lay-up technique. The matrix to be used for the composite production was made by mixing the epoxy and hardener together, at a ratio of 8:2 (percentage by weight). The composite was produced by partial replacement of the matrix with 20% (by weight) of the dried okra fibre During the composite production, the matrix was poured into an already prepared (oiled) standard steel mould (200 mm × 150 mm × 5 mm), the okra fibre was laid unidirectional on top of the matrix, and then the remaining matrix was poured in. The casted mould was rolled over with a heavy weight, and kept under a 25 Kg load for 24 hours to minimize porosity (by expelling any entrapped air) in the composite board to be produced. After 24 hours, the composite were de-moulded, and was cured in the open air for another 48 hours, before it was taken to the laboratory for mechanical testing.

The composite samples were coded according to the pre-harvest treatment applied to the okra fibre used, and shown in the format below.

T1 = composite produced with untreated okra fibre;
T2 = composite produced with 100 mg/l $Ca(NO_3)_2$ pre-harvest treated okra fibre
T3 = composite produced with 200 mg/l $Ca(NO_3)_2$ pre-harvest treated okra fibre
T4 = composite produced with 300 mg/l $Ca(NO_3)_2$ pre-harvest treated okra fibre
T5 = composite produced with 400 mg/l $Ca(NO_3)_2$ pre-harvest treated okra fibre

Tensile test

The Universal Testing Machine (UTM) (Testometric model,), equipped with a 500 N loading cell and micro-processor, was used to determine the tensile properties of the composite produced. The tensile properties of the composite, were tested according to ASTM D638 (2014) recommended procedures (Oghenerukevwe and Hilary Uguru, 2018). During the test, the sample is fixed to the machine as shown in (Figure 1), and the machine will pull it slowing at the speed of 1 mm/min, until the sample snapped. At the end of each test, the tensile parameters of the sample are calculated electronically by the microprocessor of the UTM, and displayed on the screen attached to the machine. Four samples were tested from each experimental group.

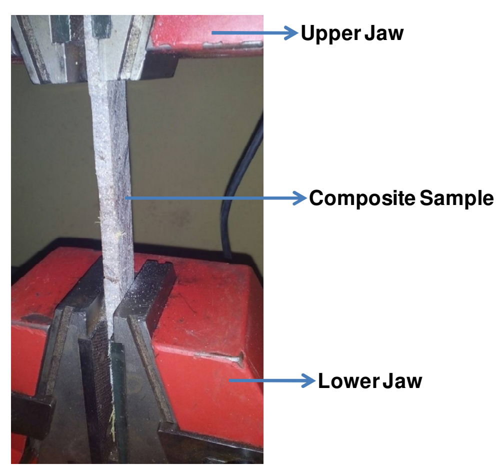

Figure 1: A composite sample undergoing tensile strength test.

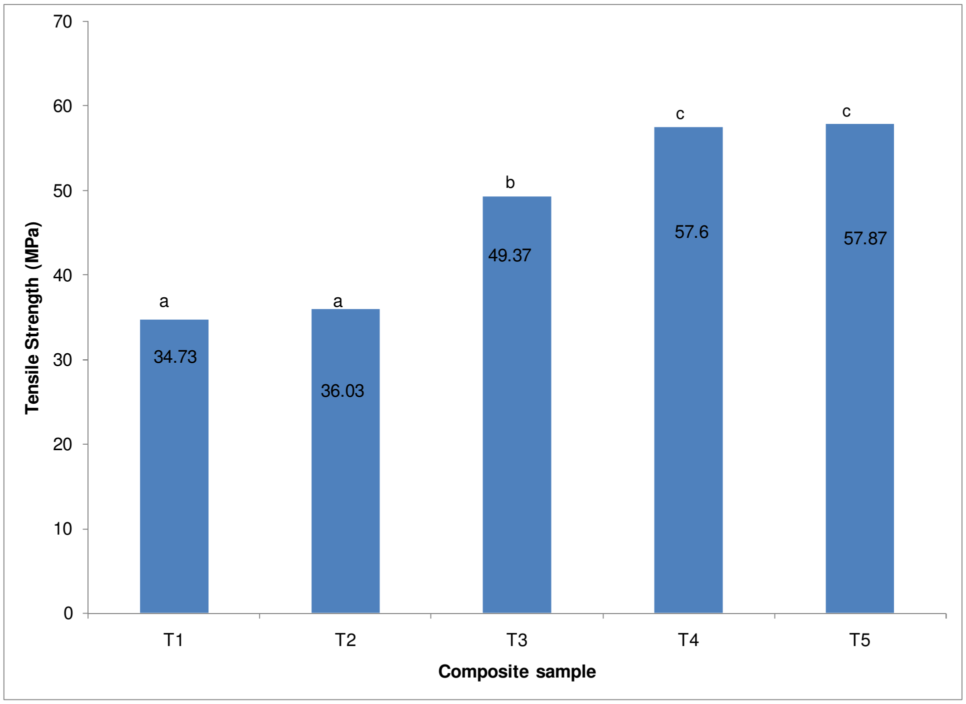

Figure 2: Tensile strength of pre-harvest treated okra fibre.
Bars with the same common letters means that they are not significant different at (P ≤0.05).

Flexural Test

The flexural strength of the composite was determined using the ASTM D790M-81 recommended procedures. The test was carried using the three-point loading method the Universal Testing Machine (UTM). At the end of each test, the flexural strength of the sample was calculated automatically by the machine and results displayed on the screen.

Four samples were tested from each experimental group. All the laboratory tests were carried out at ambient room temperature.

Statistical analysis

Results gotten from this study will be subjected to Analysis of variance (ANOVA), using the SPSS software (version 20.0). Furthermore, the mean values were separated using Duncan's multiple range tests at 95% confidence level.

RESULTS AND DISCUSSION

Tensile strength

The study results revealed that pre-harvest treatment of the okra fibre, significantly ($p \leq 0.05$) affected the tensile strength of the composite produced with it. As shown in (Figure 2), although the treatment applied had significant effect on the tensile strength of the composite, composite T4 and T5 did not exhibit any significant difference. Likewise, there was no significant difference between the untreated okra fibre and the fibre pre-harvest treated with 100 mg/l ($Ca(NO_3)_2$. Results displayed in (Figure 2) showed that T1 composite recorded the lowest tensile strength (34.73 MPa), while T5 composite had the highest tensile strength (57.87 MPa). This study had revealed that pre-harvest treatment of okra plant significantly increased the tensile strength of the composite produced from its fibre. Increment in the tensile strength of the composite, could be attributed to the increased in calcium and cellulose content of the okra fibre. According to Li *et al*. (2012) pre-harvest application of calcium solution greatly increased the mechanical strength of plants' tissues, as it greatly increased their breaking strength of herbaceous peony (*Paeonia Lactiflora* Pall) stems. In addition, Peter (2005) stated that calcium is a key plant nutrient, which played a vital role in the mechanical properties of cell wall and membranes. While Muengkaew *et al*. (2018) reported that calcium has the ability of improving fruit's tissues and mesocarp cells. Likewise, Mwaikambo (2009) reported that 0.26% increment in the cellulose content of jute fibres, led to 11% increase in the tensile strength of the composite produced with them. Apart from calcium, which has being experimentally confirmed of increasing plant tissues firmness (Hocking *et al*., 2016); the nitrate content of the $Ca(NO_3)_2$ has the ability of increasing the okra tissues

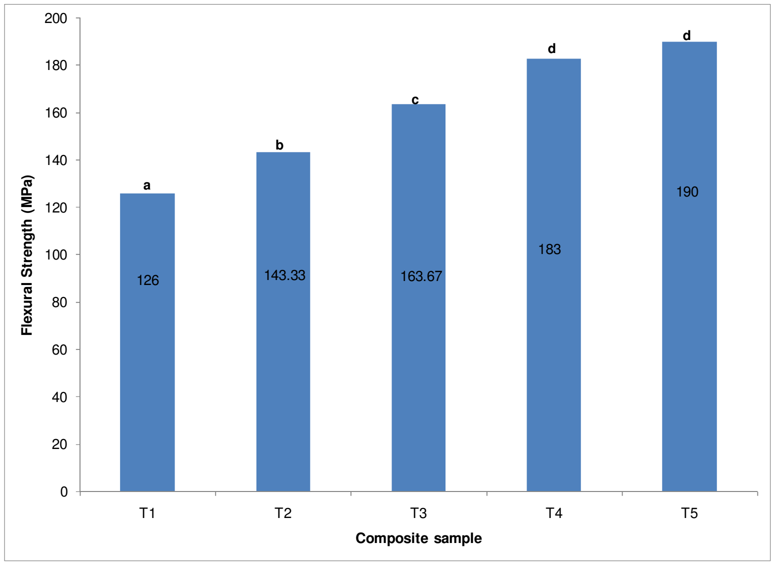

Figure 3: Flexural strength of pre-harvest treated okra fibre.
Bars with the same common letters means that they are not significant different at (P ≤0.05).

growth and development. This study has open a new niche in composite production, as more focus is new required in the pre-harvest treatment of the plant fibres, instead of the post-harvest treatment, where a lot of researches have been done. In addition, the pre-harvest treatment will improve crop production, because calcium nitrate is a potential fertilizer, which will increase crop yields.

Flexural strength

The study presented in (Figure 3) showed that pre-harvest treatment of the okra fibre had significant (p ≤0.05) effect on the flexural strength of the composite produced from it. As presented in (Figure 3), T1 composite had the lowest flexural strength (126 MPa); while T5 composite recorded the highest flexural strength (190 MPa). It was observed in the study that the flexural strength of the composite linearly increased as the pre-harvest treatment concentration increases. The regression analysis (Equation 1) showed a high correlation value (r = 0.989). This further confirmed is a strong relationship between the pre-harvest fibre treatment, and the flexural strength of the composite produced from it. This study has revealed the significant of pre-harvest treatment on the mechanical properties of plant's fibres. The improvement in the structural behaviours of the treated okra fibre reinforced composite, can be attributed to the higher calcium and cellulose content of the fibre. Edafeadhe and Uguru (2020) observed that calcium nitrate, significantly increased the calcium and cellulose content of pre-harvest treated okra fibre. According to Peters (2005) and Arabloo *et al.* (2017), pre-harvest calcium treatment increased the plant's tissues rigidity; thereby, improving their strength others mechanical properties. These results have opened a new research area for plant fibres treatment in composite production. This is because plant fibres are gaining rapid popularity in composite production, due to their numerous advantages, but their (plant fibres) production is conflicting with food security.

$$y = 16.76x + 110.9;\ R^2 = 0.979 \quad (r = 0.989) \qquad (1)$$

Where y = flexural strength
x = treatment option
R^2 = coefficient of determination
r = correlation

Conclusion

In this research, some structural parameters (tensile strength and flexural strength) of calcium nitrate treated okra fibre reinforced epoxy composite were evaluated. The okra plants were pre-harvest treated with calcium nitrate, at five concentrations (0 mg/l, 100 mg/l, 200 mg/l, 300 mg/l and 400 mg /l). The okra plants were harvested at 60 days after planting, and the fibre extracted using the retting method. Composite samples were produced by partial replacement of the matrix with 20% (by weight) of the fibre, using the hand-lay-up technique. Tensile and flexural testing of the composite samples, were done according to ASTM International recommended procedures.

Results obtained from the tensile and flexural tests indicated that, pre-harvest treatment of the fibre significantly increased the tensile strength and flexural strength of the composite. The composite produced with the untreated (0 mg/l $Ca(NO_3)_2$) fibre had the lowest flexural strength (126 MPa); while the composite produced with fibre treated with 400 mg/l $Ca(NO_3)_2$ had the highest flexural strength (190 MPa). Likewise, the study showed that the composite reinforced with untreated fibre had the lowest tensile strength (34.73 MPa); while the composite reinforced with okra fibre treated with 400 mg/l $Ca(NO_3)_2$ recorded the highest tensile strength (57.87 MPa). These results showed the importance of pre-harvest fibre treatment in composite production, since the composite produced can be used in structural applications.

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