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# MECHANICAL PROPERTIES AND MICROSTRUCTURE OF EPOXY, HORN, ALKALINE TREATED/UNTREATED COCONUT SHELL PARTICULATES HYBRID COMPOSITE

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## ABSTRACT

The mechanical properties (Tensile and Flexural Strengths) of sheep horn, treated and untreated coconut shell particles reinforced epoxy composite were investigated in this work. The composite was formulated using design expert software, with weight fraction of epoxy resin varied from 90 to 100%, while that of each of the sheep horn and coconut shell particles varied from 0 to 5 % weight, resulting in the composite of single and hybrid reinforcements. The results obtained showed that tensile and flexural strengths of the hybrid composite were superior to those of the individual fiber reinforced composite. Maximum tensile strength and flexural strength of 36.52 MPa and 67.93 MPa respectively, representing 74.3%, and 35.6% improvement, compared to the tensile strength and flexural strength of the control sample were obtained with the hybrid composite sample containing a blend of 5% wt. sheep horn and 3% wt. treated coconut shell particles. The microstructure analysis revealed the enhanced interfacial adhesion between the matrix and the reinforcement of the composite samples containing alkaline treated coconut shell particles. Hence, alkaline treatment is a good natural fiber's surface modification technique to improve adhesion between the fibers and the matrix.

**Keywords**: Composite, Polymer, Synthetic, Natural, Alkaline

## INTRODUCTION

The growing need for lightweight materials with excellent mechanical properties for structural applications has made it a necessity for the manufacturing industries to recurrently search for enhanced properties and cost effective new materials. Polymers are lightweight materials, but they are rarely used for structural applications because of their lower mechanical properties. However, modern technology showed that polymers could be designed to exhibit some of the important mechanical properties required in a good number of structural applications such as in construction, aerospace, military, electronics, and automobile applications among others, by reinforcing them with other materials to form composite material, while maintaining their light weight (Osokoya, 2017; Adah *et al*., 2024).

Composite materials are multiphase materials system, consisting of a distinct constituent, known as the reinforcement, distributed in a continuous phase, known as the matrix. The use of non-reinforced polymers as structural materials is limited because of their lower mechanical properties. The uniqueness of composite materials is that the engineering properties which are required in the end product can be achieved by a careful selection of the matrix and the reinforcing phases. Composite materials are currently recognized as a class of materials with outstanding performance because they combine the best characteristics and minimizes the effects of deficiencies of each of the constituents, thus, resulting in a material with properties not achievable using each of the constituents individually (Bodunrin *et al.,* 2015; Nithyanandhan *et al*., 2017).

Traditionally, polymeric materials are reinforced with synthetic fibers such as glass, aramid and carbon fibers to enhance or modify their properties (Osokoya, 2017). However, there are several drawbacks of using synthetic fibers in composites development. These include toxicity, high cost, limited availability, abrasion on the processing equipment, and non-biodegradability (Gupta *et al*., 2015; Onuoha *et al*., 2017). A significant goal of natural fibers application is the enhancement of polymer's mechanical properties at a reduced cost. However, lignocellulosic and other natural fibers have some demerits, such as higher water absorption, lower thermal stability, and lower mechanical properties, compared to their synthetic fibers counterparts (Senthilkumar *et al*., 2022). To overcome this challenge, hybrid natural fibers (two or more different natural fibers) are incorporated into a single matrix (Palta *et al*., 2018; Abdulrahim *et al.,* 2021). As a consequence of this, synergistic effect may be obtained in the composite, leading to enhanced mechanical properties. Advantages of natural fibers reinforced composites includes biodegradability, reduced dependence on non-renewable material resources, environmental pollution control and reduced greenhouse emission (Keya *et al*., 2019). Thus, the use of these fibers satisfies both economic and environmental benefits.

Several works have been reported on the suitability of various bio-wastes as reinforcement in polymeric composites development. Obiukwu *et al*., (2016), investigated the properties of coconut shell powder reinforced high-density polyethylene (HDPE) composite and reported that the hardness of the composite increased with increase in coconut shell powder content, while the impact strength and ductility decreased. Setty *et al*., (2020), developed vinyl ester based composites reinforced with raw and alkaline treated *limonia acidissima* shell powder, and reported that the properties of the composites filled with alkaline treated filler were superior to those of untreated filler. Reddy and Dhoria (2018), studied the effect of alkaline treatment on the mechanical properties of kenaf fiber reinforced polyester composites. It was reported that the composites made of alkaline treated fibers have good mechanical properties compared to those made of untreated fibers, due to the improvement of fiber-matrix compatibility and interfacial adhesion. Similar results were obtained by Fiore *et al*., (2015), and Setty *et al*., (2020). Andezai *et al*., (2020), investigated the mechanical properties of coconut shell powder filled epoxy resin composites. They reported that the modulus of elasticity and hardness of the composite increased with increasing percentage weight of coconut shell particles, while the tensile strength, percentage elongation and impact strength of the composite decreased. Similar results were obtained by Akindapo *et al*., (2014), Kumar *et al*., (2018), Agunsoye, *et al*., (2012), Sarki *et al*., (2011), and Obiukwu *et al*., (2016). Mohankumararadhya *et al*., (2020), conducted a study on the development and characterisation of polymeric composites with coconut shell, walnut shell and wood apple shell as hybrid reinforcements. The fabricated hybrid composites showed better properties compared to individual bio-waste particles reinforced composite under both tensile and flexural test. The same observation was reported by Somashekhar *et al.,* (2018), who employed coconut shell powder and tamarind shell powder as hybrid reinforcement. Abdulrahim *et al*., (2021), conducted an experiment on hybridization of polyester/banana stem fiber and cow horn particulate composite for possible production of military helmet. They reported that the values of the impact energy, hardness, and strength obtained were within the range for the production of military helmet.

Amongst the reviewed previous works, coconut shell particles have been widely utilised with a considerable improvements in the hardness and wear resistance properties, but reduced impact and tensile strengths of the composites (Akindapo *et al*., 2014, Kumar *et al*., 2018, and Obiukwu *et al*., 2016). Over the last few years, structural biological materials such as bones, mollusk shells, and hooves have attracted increasing attention from materials researchers. However, very few literatures exist on the use of animal horn's sheath as filler in polymeric composites. To the best of author's knowledge, no work has been done on the effects of sheep horn and coconut shell particles as hybrid reinforcement on the mechanical properties and microstructure of epoxy based composite. Against this background, the present work was carried out to investigate the mechanical properties and microstructure of sheep horn and alkaline treated/untreated coconut shell particles as hybrid reinforcement in epoxy based composite.

## MATERIALS AND METHODS

### Materials

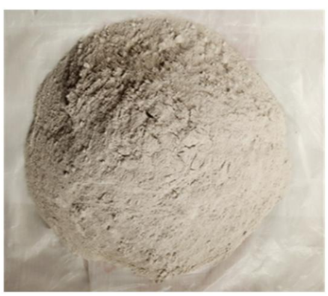
Epoxy resin and amino based hardener, procured from Tony chemical store in Ojota, Lagos, Nigeria, were utilized as matrix material in this study. While sheep horn and coconut shell that were employed as hybrid reinforcement material, as well as the sodium hydroxide and distilled water used for surface treatment of the coconut shell particles were sourced in Ilorin, Kwara State, Nigeria.

### Methods

***Preparation of reinforcing materials***

Sheep horns and coconut shells were cleaned with water to remove foreign materials and sun-dried for 8 weeks with average daily temperature of 28$^{o}$C and relative humidity of 22%, after which they were manually broken into chips using sledge hammer, and milled into microparticles using hammer mill. After the alkaline treatment, the treated and untreated coconut shell particles were subjected to further milling for 72 hours to obtain coconut shell nanoparticles, using ball milling machine with ball to powder weight ratio of 10:1, in line with the work of Bello *et al*., (2015). Particle size analysis of the treated and untreated coconut shell nanoparticles produced was carried out using Zetasizer (serial number: MAL1084260).

a



b

Figure 1: (a) sheep horn particles (b) coconut shell particles

***Alkaline treatment of the coconut shell particles***

Alkaline treatment of the coconut shell particles was necessary because plant based natural fibers are less compatible with the polymer matrix and thus require surface modification. This was done by soaking the coconut shell particles in NaOH solution of 5% concentration for 60 minutes at room temperature (Arthanarieswaran *et al*., 2015; Rajkumar *et al*., 2016; Herlina-Sari *et al*., 2018; Samaei *et al*., 2020). After which the coconut shell particles were sieved and rinsed repeatedly in distilled water until a neutral pH value was obtained. The treated coconut shell particles were then, dried at room temperature for 48 hours, and followed by oven drying at 60$^{o}$C for 8 hours (Kumar *et al*., 2016).

***Production of the composites***

Sheep horn microparticles and coconut shell nanoparticles of 43 µm and 50 nm average particle sizes respectively, were utilized as hybrid reinforcement. Blending of coconut shell nanoparticles with sheep horn microparticles as hybrid reinforcement was adopted due to high propensity of nanoparticles to agglomerate. Randomised mixture formulation of the matrix and the two reinforcement phases was done using design expert software (version 12.0.12.0). Weight fraction of epoxy resin was varied from 90 to 100% weight, while that of each of the sheep horn and coconut shell particles was varied from 0 to 5% weight. During the production of each formulation of the composites, calculated amount of the hybrid reinforcing particles were thoroughly blended to obtain homogenous mixture, using Rico electric blender (serial number: MG1701), rotating at 1500 rpm. The blend was then gradually added to a weighed amount of epoxy resin while stirring it manually to avoid clustering, using a glass rod until homogenous mixture was obtained, and followed by motorized stirring to ensure homogenous dispersion of the reinforcement in the epoxy resin. Then, measured amount of hardener equivalent to ratio 2:1 (resin:hardener) by weight was added to the mixture of epoxy resin and reinforcement, then stirred manually for about 5 minutes using a glass rod, followed by motorized stirring for about 10 minutes at 300 rpm, after which the mixture was poured steadily into the mould and left in the mould for 24 hours to set and harden at room temperature. Prior to the casting of the composites, the mould cavities were neatly coated with masking tape to facilitate easy removal of the composite samples after curing. Same process was repeated for the production of all the composite formulations.

**Mechanical properties of the composite**

***Tensile strength test***

Tensile strength test was conducted on a computerised testometric materials testing machine (machine number: 0500-10080), and the test samples satisfied ASTM D638 standards. Dumbbell shape sample of dimension 160 mm x 12 mm x 5 mm, with a gauge length of 50 mm was mounted between the two arms of the machine, one end of the machine is fixed and load is continuously applied to the other end. The test was carried out using a load cell of 5 kN and test speed of 5 mm/minute. At a certain load, the sample breaks, and the results are displayed digitally. For each composite formulation, three samples were tested and the average values were recorded.

**a**

**b**

Figure 2: (a) Tensile test samples (b) Tensile test set-up

***Flexural strength test***

The flexural strength test was conducted on a computerised testometric materials testing machine (machine number: 0500-10080), and the test samples satisfied ASTM D790 standards. The cross-head speed and the dimension of the test samples were 5 mm/minute and 160 mm x 20 mm x 5 mm respectively, while the load cell utilised was 5 kN. At a certain load, the sample breaks, and the results are displayed digitally. For each composite formulation, three samples were tested and the average values were recorded.

**a**

**b**


Figure 3: (a) Flexural test samples (b) Flexural test set-up

**Microstructure analysis**

Selected samples of the composite were subjected to microstructure examinations through their magnified images in order to assess the distribution of the reinforcement within the matrix, using amscope optical microscope.

**RESULTS AND DISCUSSION**

**Tensile strength of the composite**

The variation in tensile strength with percentage weight addition of the single and hybrid reinforcements into the epoxy matrix are illustrated in Figures 4a and 4b respectively.

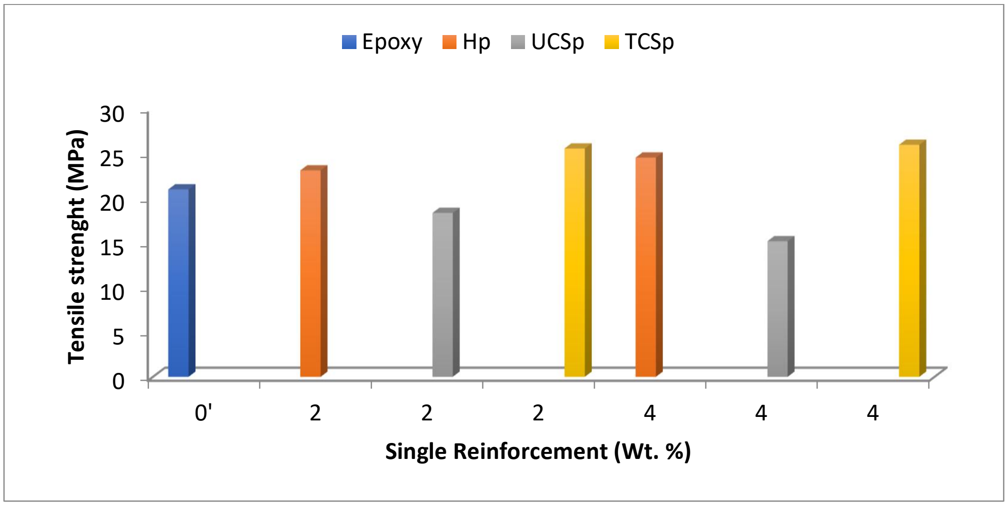



Figure 4a: Tensile strength of horn, untreated and treated coconut shell particles reinforced epoxy composite

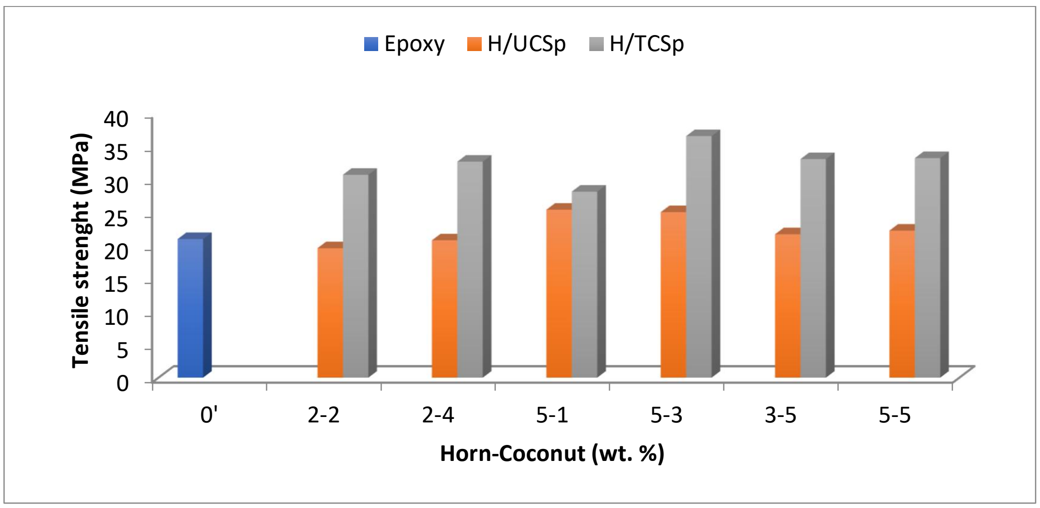



Figure 4b: Tensile strength of hybrid horn and untreated/treated coconut shell particles reinforced epoxy composite

It was observed from the results obtained (Figure 4a), that composite samples with single reinforcement; 2% wt. sheep horn microparticles, 2% wt. treated coconut shell nanoparticles, 4% wt. sheep horn microparticles and 4% wt. treated coconut shell nanoparticles, exhibited improved tensile strengths compared to the control sample (100% epoxy). Tensile strength of 20.95 MPa was obtained for the control sample, while tensile strengths of 23.08 MPa and 25.53 MPa, which is 10.2% and 21.9% higher than the tensile strength of the control sample, were recorded with the addition of 2% wt. sheep horn microparticles, and 2% wt. treated coconut shell nanoparticles respectively. Incorporation of 4% wt. sheep horn microparticles, and 4% wt. treated coconut shell nanoparticles into the epoxy matrix further increased the tensile strength to 24.51 MPa and 25.94 MPa respectively, which is 17.0% and 23.8% higher than the tensile strength of the control sample. The interaction between the horn particles and the epoxy matrix might have resulted in molecular entanglement, leading to extensive crosslinks within the composite structure. Also, higher tensile strengths obtained for the treated coconut shell nanoparticles reinforced composite samples, compared to the sheep horn microparticles reinforced composite samples may be linked to the finer particles size of the treated coconut shell. However, tensile strengths of the single reinforced composites containing 2% wt., and 4% wt. untreated coconut shell nanoparticles declined to 18.32 MPa and 15.16 MPa respectively, which is 12.6% and 27.6% lower than the tensile strength of the control sample. Fiore *et al*., 2014, and Wang *et al*., 2019, reported that cellulose content of natural fibers play a key role in their mechanical properties. Hence, it could be inferred that the NaOH treatment of the coconut shell particles enhanced its cellulose content, leading to improved tensile strength of the resulting composites. Similar observation was reported by Setty *et al*., (2020). From the results obtained for the hybrid composite samples (Figure 4b), synergistic effect was suspected with the interaction of the two reinforcing particles as the hybrid composite sample containing a blend of 2% wt. sheep horn microparticles and 2% wt. treated coconut shell nanoparticles, possessed superior tensile strength (30.67 MPa) compared to the composite samples with 4% wt. sheep horn microparticles (24.51 MPa), and 4% wt. treated coconut shell nanoparticles (25.94 MPa), having single reinforcement of equal weight fraction as that of the hybrid composite sample. It could also be seen that all categories of hybrid composites containing NaOH treated coconut shell nanoparticles exhibited superior tensile strength compared to those with untreated coconut shell nanoparticles.

The enhanced tensile strength could be attributed to strong interfacial adhesion/bonding between epoxy matrix and the reinforcement, which might possibly, improved the stress distribution within the composite. Maximum tensile strength of 36.52 MPa was obtained with hybrid composite sample containing a blend of 5% wt. sheep horn microparticles and 3% wt. treated coconut shell nanoparticles. This amount to 74.3% improvement, compared to the tensile strength of the control sample. However, the tensile strength of hybrid composite samples incorporating 5% wt. treated coconut shell nanoparticles fell a little below the maximum tensile strength obtained. This suggests that at higher reinforcement loading, there is high tendency of nanoparticles clustering/agglomeration, which may results in increasing voids formation, and consequently impede the efficiency of stress transfer within the composites.

**Flexural strength of the composites**

The effect of addition of different weight fractions of the single and hybrid reinforcements into epoxy matrix on the flexural strength of the composite are illustrated in Figures 5a and 5b respectively.

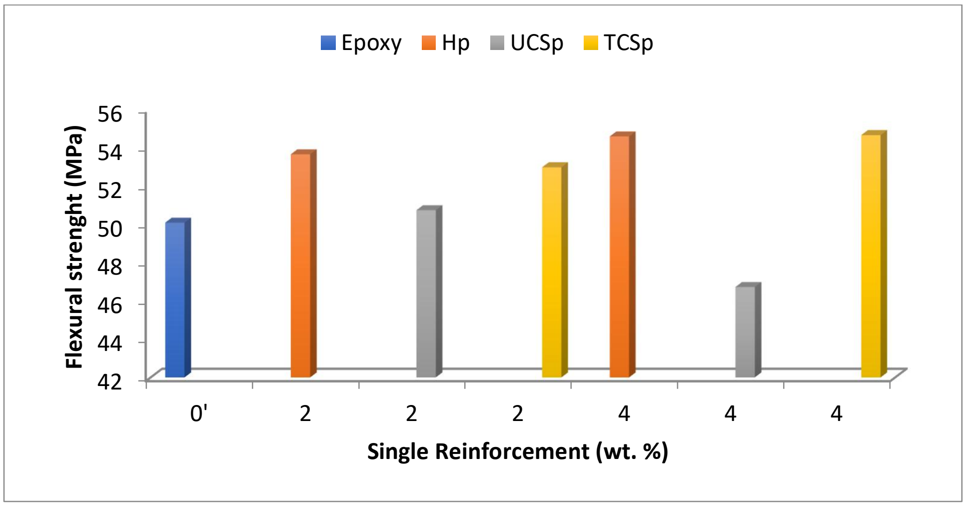



Figure 5a: Flexural strength of horn, untreated and treated coconut shell particles reinforced epoxy composites

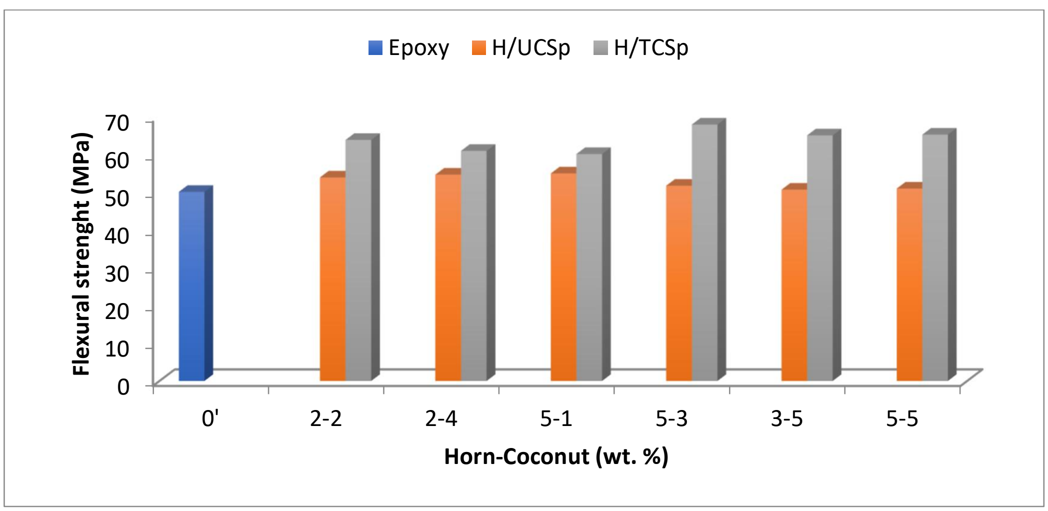
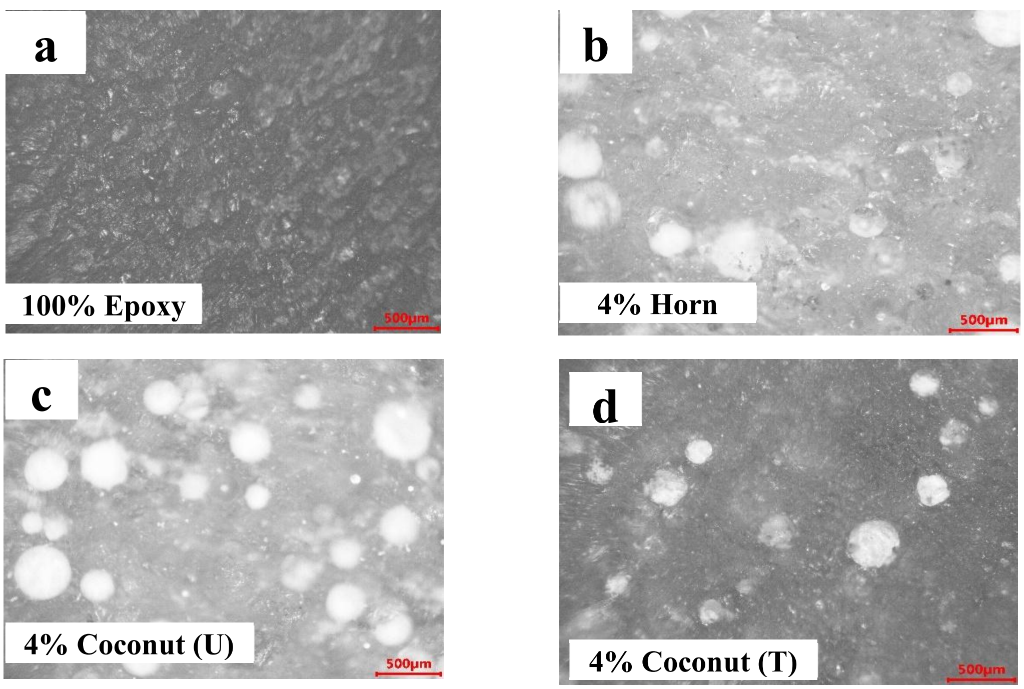



Figure 5b: Flexural strength of hybrid horn and untreated/treated coconut shell particles reinforced epoxy composites

From the results obtained (Figure 5a), composite samples with single reinforcement; 2% wt. sheep horn microparticles, 2% wt. treated coconut shell nanoparticles, 4% wt. sheep horn microparticles, and 4% wt. treated coconut shell nanoparticles, were found to possess improved flexural strengths, compared to the control sample (100% epoxy). Flexural strength of 50.08 MPa was obtained for the control sample, while flexural strengths of 53.65 MPa and 52.97 MPa, which is 7.1% and 5.8% higher than the flexural strength of the control sample, were respectively recorded with the addition of 2% wt. sheep horn microparticles, and 2% wt. treated coconut shell nanoparticles into the epoxy matrix. Addition of 4% wt. sheep horn microparticles, and 4% wt. treated coconut shell nanoparticles, increased the flexural strength of the composite to 54.58 MPa and 54.65 MPa respectively, which is 9.0% and 9.1% higher than the flexural strength of the control sample. Also, a slight increase in flexural strength (50.73 MPa) was noted in the single reinforced composite containing 2% wt. untreated coconut shell nanoparticles, which represent 1.3% increase in the flexural strength of the control sample. However, flexural strength of the single reinforced composite containing 4% wt. untreated coconut shell nanoparticles decreased to 46.72 MPa, which is 6.7% lower than the flexural strength of the control sample. This could possibly due to incompatibility and poor interfacial bonding between the untreated coconut shell nanoparticles and the epoxy matrix, leading to high void content in the composite. Similar observation was reported by Mansour *et al*., (2011). Moreover, by blending sheep horn microparticles and coconut shell nanoparticles (Figure 5b) in the hybrid composite sample containing a blend of 2% wt. sheep horn microparticles and 2% wt. treated coconut shell nanoparticles, enhanced flexural strength (63.85 MPa) was obtained compared to the composite samples with 4% wt. sheep horn microparticles (54.58 MPa), and 4% wt. treated coconut shell nanoparticles (54.65 MPa), containing single reinforcement of equal weight fraction as that of the hybrid composite sample. All categories of hybrid composites containing NaOH treated coconut shell nanoparticles exhibited superior flexural strength compared to those with untreated coconut shell nanoparticles. Hence, NaOH treatment is a good natural fiber's surface modification technique to improve adhesion between the fibers and the matrix, thus, enhancing stress transfer (Moh'd-Nazarudin *et al*., 2013; Kumar *et al*., 2016; Reddy and Dhoria, 2018; Onwumere *et al*., 2019; Setty *et al*., 2020). Optimum flexural strength of 67.93 MPa was obtained with the hybrid composite sample containing a blend of 5% wt. sheep horn microparticles and 3% wt. treated coconut shell nanoparticles, which represents 35.6% improvement in flexural strength, compared to the flexural strength of the control sample. The observed decrease in flexural strength of the hybrid composite samples incorporating 5% wt. treated coconut shell nanoparticles may be due to clustering/agglomeration at higher concentration of the nanoparticles, resulting in increased void content (micro-pores), which reduces the stress transfer efficiency of the composites.

**Microstructure of the composite**

Figure 6a showed the optical micrograph of the control sample (100% epoxy), while Figures 6b, 6c, and 6d showed the optical micrographs of the composite containing single reinforcement, and Figures 6e, 6f, 6g, and 6h showed the optical micrographs of the hybrid composites. Homogeneous microstructure was observed in the micrograph of the control sample (Figure 6a). In Figure 6c, the reinforcement was seen to be poorly distributed, which implies poor interfacial interaction between the matrix and the untreated coconut shell particles. Whereas, fairly good interfacial interaction between matrix and reinforcement was observed in Figure 6d. The spherical whitish regions in Figure 6b represent agglomeration of the reinforcement, that is, segregation and clustering of the reinforcing particles, which is more pronounced in the Figure 6c, confirming uneven dispersion of the reinforcement. However, the microstructure of hybrid composite samples in Figures 6f and 6h, containing treated coconut shell particles, revealed less agglomeration and good interfacial adhesion between the matrix and the reinforcement. Thus, resulting in effective stress transfer believed to be responsible for the superior mechanical properties recorded for these composites.



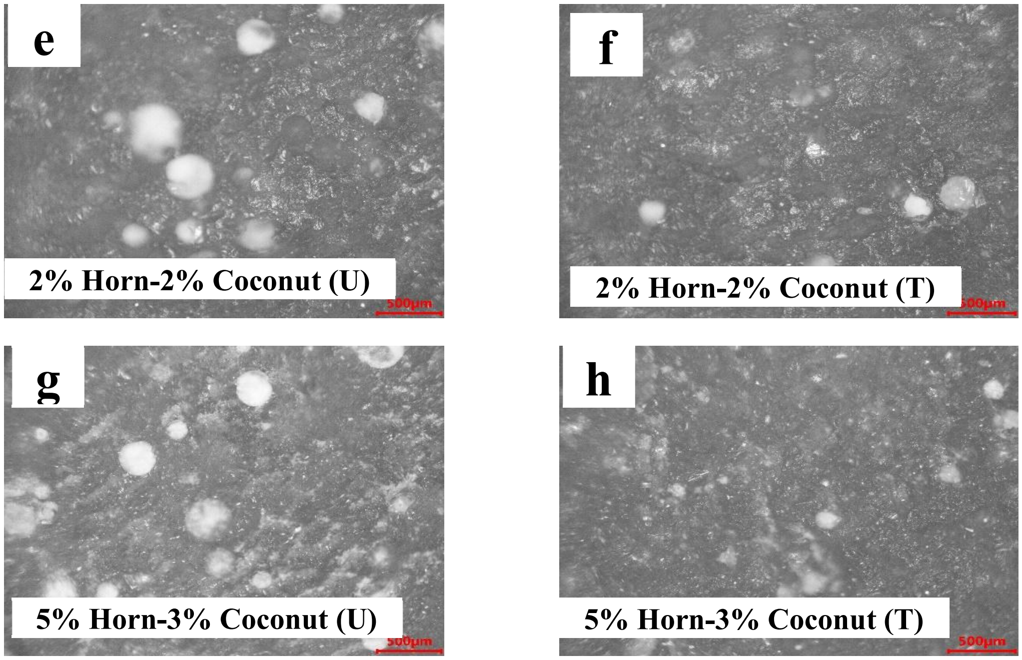



Figure 6: (a) Optical micrograph of pure epoxy, (b - d) Optical micrographs of the composite containing single reinforcement, (e - h) Optical micrographs of the hybrid composites

**CONCLUSION**

Mechanical properties and microstructure of hybrid sheep horn and alkaline treated/untreated coconut shell particles reinforced epoxy composite were investigated in this work. The results showed that the hybrid composite had superior properties compared to individual fiber reinforced composites. Maximum tensile strength and flexural strength of 36.52 MPa and 67.93 MPa respectively, representing 74.3%, and 35.6% improvement, compared to the tensile strength and flexural strength of the control sample, were obtained with the hybrid composite sample containing a blend of 5% wt. sheep horn and 3% wt. treated coconut shell particles. The microstructure analysis revealed the enhanced interfacial adhesion between the matrix and the reinforcement of the composite samples containing NaOH treated coconut shell particles, thus, enhancing stress transfer within the composite.

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