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Characterisation of *Ovis Aries* Horn and *Cocos nucifera* Shell Particles for Hybridisation in Polymeric Composites

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Abstract

The suitability of ovis aries (sheep) horn and Cocos nucifera (coconut) shell particles as hybrid reinforcement in polymeric composites was investigated in this work. Ovis aries horn and Cocos nucifera shell particles were prepared and characterised using TGA, XRF, FTIR, XRD, and SEM analyses. Both samples exhibited appreciable thermal stability as revealed by the TGA curves. XRF analysis featured varied proportions of similar elemental and oxides constituents known to possess good reinforcement characteristics, while SEM micrographs revealed that NaOH treatment of Cocos nucifera shell particles had desirable effect on the fibres surface. XRD patterns of the NaOH treated Cocos nucifera shell particles featured minor peak around $2\theta = 35^{o}$, suggesting a little alteration in crystallinity. FTIR analysis established the presence of amine groups and disulphide bond in the ovis aries horn, and potentially reactive carbonyl, carboxyl, and hydroxyl groups in the Cocos nucifera shell. Removal of waxy materials, part of lignin and hemicellulose, and reduction in hydroxyl groups of Cocos nucifera shell particles after NaOH treatment were confirmed by FTIR analysis. Hence, the potential of hybridising ovis aries horn and Cocos nucifera shell particles as reinforcement in polymeric composites development is high.

Keywords: *Hybridisation, natural material, polymer, reinforcement, synthetic.*

1.0 Introduction

Composite material plays a prominent role in the development of lightweight and high-performance engineering products applicable in automobile and aerospace industries. The term "composite material" broadly refers to a multiphase materials system, consisting of a distinct constituent, known as the reinforcement, distributed in a continuous phase, known as the matrix. The uniqueness of composite materials is that the engineering properties which are required in the final product can be achieved by a careful selection of matrix and reinforcing phases (Akaluzia *et al*., 2020).

Conventionally, polymers are reinforced with synthetic fibres such as glass and carbon fibres. Several polymer-based composites with synthetic reinforcements have been developed and used in construction, aerospace, military, electronics, and automobile applications among others. However, considerable drawbacks have been recorded in the use of synthetic fibres in polymeric composites manufacturing. These include toxicity, high cost, limited availability, abrasion on the processing equipment, and non-biodegradability (Gupta *et al*., 2015; Onuoha *et al*., 2017). The growing environmental concerns and the declining availability of synthetic reinforcements have necessitated their substitution with natural reinforcements, either in fibres or particulate form (Adah *et al*., 2024). Advantages of natural fibres in composites manufacturing includes biodegradability, less dependence on non-renewable material resources, environmental pollution control and reduced greenhouse emission (Hashim *et al*., 2012; Keya *et al*., 2019). However, polymers reinforced with natural fibres exhibits inferior mechanical properties compared to the polymers reinforced with synthetic fibres. To address this challenge, two or more natural fibres are introduced into a single matrix, forming hybrid reinforcement (Palta *et al*., 2018; Abdulrahim *et al*., 2021).

A good number of research works have been reported on the suitability of various bio-waste materials as reinforcement in polymeric composites for different applications. Naik *et al*. (2021), carried out experimental investigation on the tribological properties of orange peel particulate-epoxy composite. They reported that the wear behaviour of the neat epoxy was significantly enhanced with the incorporation of orange peel particulate as reinforcing material. Akindapo *et al*. (2014), studied the morphology and mechanical properties of coconut shell reinforced epoxy resin composite. They confirmed the suitability of coconut shell fibre as a reinforcing material in polymer matrix composites. Akinlabi *et al*. (2015), explored the use of coconut shell as an additive in natural rubber compounding. It was reported that carbonised

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coconut shell is much better as reinforcement than uncarbonised coconut shell in polymeric composite. Andezai *et al*. (2020), investigated the mechanical properties of coconut shell powder-epoxy resin composite. They reported that the elastic modulus and hardness of the composite increased with increasing proportion of coconut shell particles in the formulation, while the tensile strength, percentage elongation and impact strength of the composite decreased with increasing proportion of coconut shell particles. 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 composite with coconut shell, walnut shell and wood apple shell as hybrid reinforcements. The produced hybrid composites displayed better properties compared to individual bio-waste particles reinforced composite under both tensile strength and flexural strength tests. Same observation was reported by Somashekhar *et al.* (2018), who employed coconut shell and tamarind shell particles as hybrid reinforcement. Abdulrahim *et al*. (2021), produced a composite material with hybridisation of polyester/banana stem fibre and cow horn particulate for possible production of military helmet. The results obtained for the mechanical properties of the composite are within the range for the production of military helmet. Reddy and Dhoria (2018), investigated the influence of alkaline treatment of kenaf fibres on the mechanical properties of polyester-kenaf fibres composite. It was reported that the composite made of alkaline treated kenaf fibres exhibited enhanced mechanical properties when compared to those made of untreated kenaf fibres. This was attributed to the improvement of fibre-matrix compatibility and interfacial adhesion, due to the alkaline treatment of kenaf fibres. Similar results were obtained by Fiore *et al*. (2015), and Setty *et al*. (2020). Amongst the reviewed previous works, *cocos nucifera* (coconut) shell particles have been widely utilised with considerable improvements in the hardness and wear resistance properties, but reduced impact and tensile strengths of the composites (Akindapo *et al*., 2014; Obiukwu *et al*., 2016; Kumar *et al*., 2018).

In the last few years, structural biological materials such as bones, mollusk shells, and hooves have attracted special attention of the researchers in materials science and engineering. However, the employment of animal horn keratin sheath as reinforcing material in the development of polymeric composite is rare. Particularly, *ovis aries* (sheep) horn is very strong and highly resilient. Thus, it may serve as a good structural material suitable as reinforcement in the manufacture of polymer based composite material (Johnson *et al*., 2016). Chemically, keratin, which forms the major structural constituent of horns, is highly stable and insoluble in water, acids and most of the organic solvents (Mishra *et al*., 2010). It is expected that synergistic effects, leading to enhanced properties will be obtained by incorporating o*vis aries* horn and *cocos nucifera* shell particles as hybrid reinforcement in the manufacture of polymer-based composite material. Therefore, the aim of this study is to characterise o*vis aries* horn and *cocos nucifera* shell particles for possible hybridisation in the production of polymeric composite material for structural applications.

2.0 Materials and Methods

2.1 Materials Preparation

O*vis aries* horns were collected from slaughter house in Ilorin, Kwara State of Nigeria, cleaned with water to remove foreign materials and sun-dried for 8 weeks. The horns were manually broken into chips, and milled into particles using sledge hammer and a locally fabricated hammer mill respectively. While dried *cocos nucifera* shell were sourced at its dump site around mandate market, Ilorin, Kwara State, Nigeria. After proper removal of the accompany coir, the shell was cleaned with water to remove other foreign materials, sun-dried for 8 weeks, and milled into particles using a locally fabricated hammer mill. Three (3) separate portions of the *cocos nucifera* shell particles were subjected to chemical treatment at room temperature for different periods of 30, 60 and 90 minutes, by soaking in NaOH solution of 5% concentration (Rajkumar *et al*., 2016; Herlina-Sari *et al*., 2018; Samaei *et al*., 2020). After NaOH treatment, the *cocos nucifera* shell particles were sieved out, using a common fine mesh straining fabric, and rinsed in distilled water until a neutral pH value was obtained. Each portion of the *cocos nucifera* shell particles was dried at room temperature for 48 hours, and oven dried using DHG model oven (Serial number: 9053A) at 60^{o}C for 8 hours (Kumar *et al*., 2016).

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Plate 1: (a) *Ovisaries* horn (b) *Ovisaries* horn chips (c) *Ovisaries* horn particles

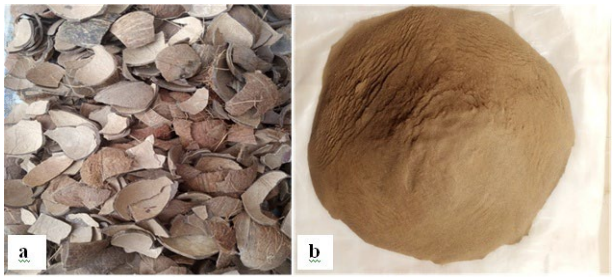

Plate 2: (a) *Cocos nucifera* shell chips (b) *Cocos nucifera* shell particles

2.1.1 Thermo-Gravimetric Analysis (TGA)

Thermal stability of *ovis aries* horn and *cocos nucifera* shell particles were determined using thermogravimetric analyser (Perkin Elmer TGA-4000). Temperature was increased at a rate of 10^{o}C/minute from room temperature to 1000^{o}C. The analysis was carried out under a controlled environment of nitrogen gas.

2.1.2 X-Ray Fluorescence Analysis (XRF)

The X-ray fluorescence (XRF) analysis was conducted on the *ovis aries* horn and *cocos nucifera* shell particles. Each sample, prepared into pellets was placed on the sample holder and the ray point was positioned over it. The ray button was pressed to start taking data. The data were collected in triplicates, and the average was automatically taken to determine the percentage chemical composition in oxide and elemental form.

2.1.3 Fourier Transform Infra-Red Spectroscopy Analysis (FTIR)

FTIR spectroscopy analysis was carried out on the *ovis aries* horn, treated and untreated *cocos nucifera* shell particles, using KBr (Potassium Bromide) pellet technique. 10 mg of each sample was homogenised with KBr, prior to being compacted in a stainless-steel die with hydraulic press at 1.2 psi to obtain transparent pellet. The FTIR spectra of the samples were recorded in the range of 400 to 4000 cm^{-1}, using Shimadzu FTIR-8400s spectrometer. The recorded spectra were interpreted to determine the chemical structure, molecular bond, and the functional group of each sample as the basis of the spectrum type.

2.1.4 X-Ray Diffraction Analysis (XRD)

The XRD analysis of the treated and untreated *cocos nucifera* shell particles were conducted using an Empyrean X-ray diffractometer DY 674 (2010) with 40mA, 45VA, and 240mm, tube current, voltage rating, and goniometer radius, respectively. Each sample was analysed using the reflection-transmission spinner stage and Theta-Theta settings scanning range of 4 to 75 degrees with a two-theta step of 0.02626 at 8.67700 seconds per step. The intensity of the diffracted X-rays is recorded continuously and automatically on a chart, the appropriate theta (θ) and delta (d) values were then obtained as the sample and detector rotate through their respective angles in line with the description of Madakson *et al*. (2012) and Kolawole *et al*. (2017).

2.1.5 Scanning Electron Microscopy (SEM) Analysis

Morphology of the treated and untreated *cocos nucifera* shell particles were studied using scanning electron microscope (Phenom Prox Model) operated at 15 kV. This was carried out to detect the possible alterations in the structure of the *cocos nucifera* shell particles as a consequence of NaOH treatment.

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3.0 Results and Discussion

3.1 Thermo-Gravimetric Analysis (TGA)

The thermal degradation patterns of *ovis aries* horn and *cocos nucifera* shell particles are illustrated in Figures 1a and 1b (TG and DTG).

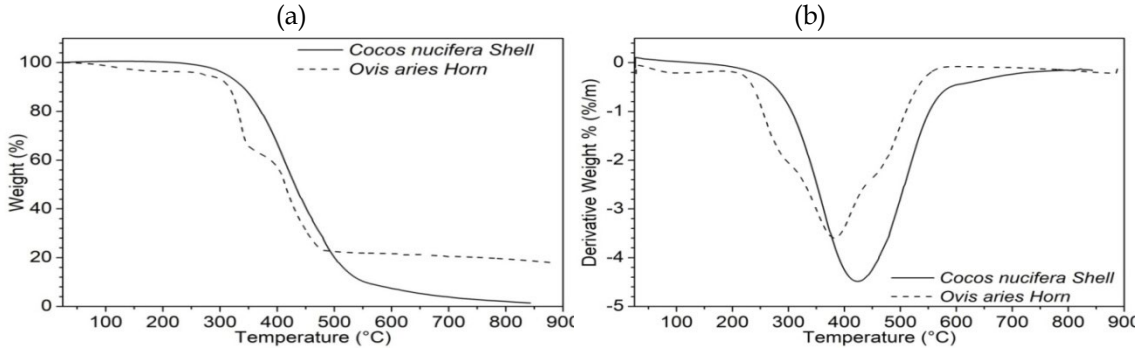

Figure 1: (a) Thermo-gravimetry (TG) curves (b) Derivative thermo-gravimetry (DTG) curves of ovis aries horn and cocos nucifera shell particles

The TG curve indicated that *cocos nucifera* shell exhibited appreciable thermal stability up to a temperature close to 300^{o}C. This may be attributed to absence of moisture in the sample as a consequence of thorough drying and its high thermal stability. The *cocos nucifera* shell had the first phase of weight loss at approximately 300 - 550^{o}C. Here, the weight loss was remarkably accelerated, and this was attributed to the degradation of hemicellulose and cellulose mainly, and part of lignin. The degradation pattern was found to be in good agreement with those reported in literature for different cellulosic biomasses (Protic *et al*., 2018; Reza *et al*., 2020; Ahmed *et al*., 2021). The second phase of weight loss in *cocos nucifera* shell began at approximately 550^{o}C, indicating gradual degradation of lignin into a carbon rich residual solid. As indicated in the TG curve of *ovis aries* horn, initial weight loss was observed at temperature between approximately 100 - 300^{o}C, due to evaporation of residual moisture and loss of the light volatiles. Thermal degradation and the major weight loss took place in two stages. The first stage which occurred between approximately 300 - 375^{o}C, is attributed to denaturation of keratin (Prochon *et al*., 2012), while the second stage which took place between approximately 375 - 475^{o}C resulted in the total destruction of keratin. Also, the DTG curve of *ovis aries* horn is characterised by multi-stage degradation events, indicating that the decomposition products are released in steps. DTG curves of both samples peaked around 400^{o}C, indicating that 50% weight loss in both samples occurred around this temperature. Thus, the two bio-waste samples possessed appreciable thermal stability for hybridisation in polymeric composites.

3.2 X-Ray Fluorescence Analysis (XRF)

Results of XRF analysis showed that both *ovis aries* horn and *cocos nucifera* shell samples have similar major elemental constituents, but in varied proportions (Table 1). The elemental constituents obtained is consistent with the findings of Buddhachat *et al*. (2016), who identified aluminium (Al), silicon (Si), phosphorus (P), sulphur (S), chlorine (Cl), potassium (K), calcium (Ca), titanium (Ti), manganese (Mn), iron (Fe), and zinc (Zn), in ten different horns species, with the major constituents being chlorine (Cl), sulphur (S), and silicon (Si). However, higher percentage of oxygen (O) was detected for both *ovis aries* horn and *cocos nucifera* shell samples in this work, which may be attributed to environmental contamination.

Table 1: Elemental Composition (weight %) of the Particles

Elements	O	Mg	Al	Si	S	Cl	K	Ca	Fe	Ru	Rh	Others
Horn	46.8	3.7	4.6	4.7	21.2	9.3	4.7	2.5	-	-	-	2.5
Coconut	36.3	14.0	5.8	10.3	-	2.8	11.0	2.2	3.3	6.3	2.8	5.2

3.3 FTIR Spectroscopic Analysis of Cocos Nucifera Shell Particles

Structural changes on the treated *cocos nucifera* shell particles are illustrated in Figure 2. The nature of the chemical bond and the functional groups present are shown by the characteristic peaks.

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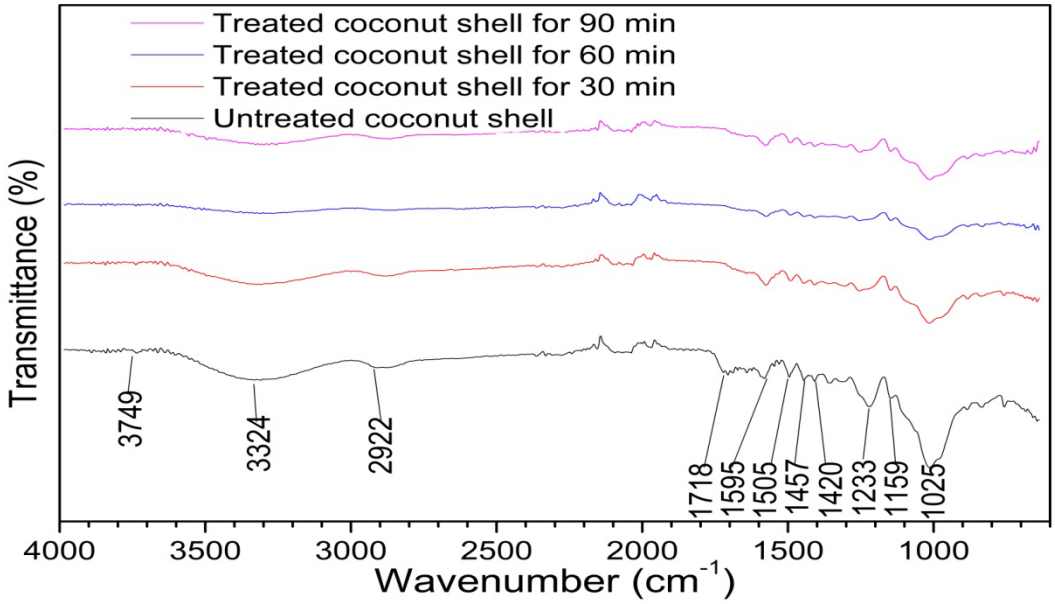

Figure 2: FTIR spectra of treated and untreated Cocos nucifera shell particles

In FTIR spectroscopy, infrared radiation in the range of 4000 to 400 cm-1 is used to irradiate a material sample. On interaction of the radiation with a material sample, the material absorbs and converts the energy to vibrations of the chemical bonds that join the atoms (Mishra *et al*., 2010). Different species in the molecules vibrate and rotate, producing bands at particular frequencies (measured in cm-1). The absorption regions for different molecules are generally known, then, a spectrum produced can be compared to the known values to identify the functional groups in a material. Because different material is a unique combination of atoms, no two compounds produce the same infrared spectrum. Hence, in the spectra of the untreated *cocos nucifera* shell particles, the band occurring at 2922 cm-1 is associated with aromatic C-H stretching vibration in lignin, evidencing the presence of cellulose and hemicellulose. Whereas, the pronounced band around 3324 cm-1is due to extensive hydrogen bonding of cellulose (Kumar *et al*., 2016; Zhuang *et al*., 2020; Salim *et al*., 2021). The intensity of bands at 3324 cm-1 and 2922 cm-1, almost disappeared in the sample treated for 60 minutes. Disappearance of the band at 3324 cm-1 indicates removal of waxy materials, and reduction in the number of hydroxyl groups. While the disappearance of the band at 2922 cm-1 is probably confirming the removal of lignin and hemicellulose (Yan *et al*., 2015; Kumar *et al*., 2016; Zhuang *et al*., 2020). Also, the narrow peak at 3749 cm-1 which reduces after NaOH treatment, is assigned to stretching vibration of hydroxyl group, O-H. The peak at 1718 cm-1 is assigned to carbonyl (C=O) stretching vibration (Nanda *et al*., 2013; Dias-Junior *et al*., 2018; Bosenbecker *et al*., 2019; Song *et al*., 2019). It was observed that this peak disappeared in the treated samples in as short as 30 minutes treatment period, suggesting that lignin and hemicellulose were removed (Tran *et al*., 2013; Kumar *et al*., 2016). The peak at 1595 cm-1 relating to aromatic stretching vibration of C=O, almost disappeared in the treated samples due to the removal of lignin. The peak at 1505 cm-1 is assigned to C=C stretching vibration in the aromatic ring, which appears disappearing after NaOH treatment, suggesting the removal of lignin (Nanda *et al*., 2013; Dias-Junior *et al*., 2018; Song *et al*., 2019; Kundu *et al*., 2021).

The peak at 1420 cm-1 which decreased after NaOH treatment, is C-C stretching vibration relating to lignin (Cunha-Pereira *et al*., 2020). Likewise, the C-H deformation peak at 1457 cm-1 appears disappearing due to removal of lignin. The peak around 1233 cm-1 is associated with the C-O stretching vibration of esters, ethers, and phenolic groups, attributed to the presence of lignin (Ariawan *et al*., 2015; Kumar *et al*., 2016). The reduction observed in the intensity of this peak after NaOH treatment signifies the removal of lignin. In the spectra of 90 minutes treated sample, a weak reappearance of the bands around 3324 cm-1 and 2922 cm-1 were observed. This may be attributed to dissolution and subsequent re-deposition of waxy substances, lignin, and hemicellulose, at higher or increased treatment period. Hence, 60 minutes treatment period is more effective in modifying the fibre's surface of *cocos nucifera* shell particles in 5% NaOH solution.

3.4 FTIR Spectroscopic Analysis of Ovis Aries Horn Particles

The FTIR spectra of *ovis aries* horn particles as illustrated in Figure 3, showed characteristic peaks of amides I, II, and III.

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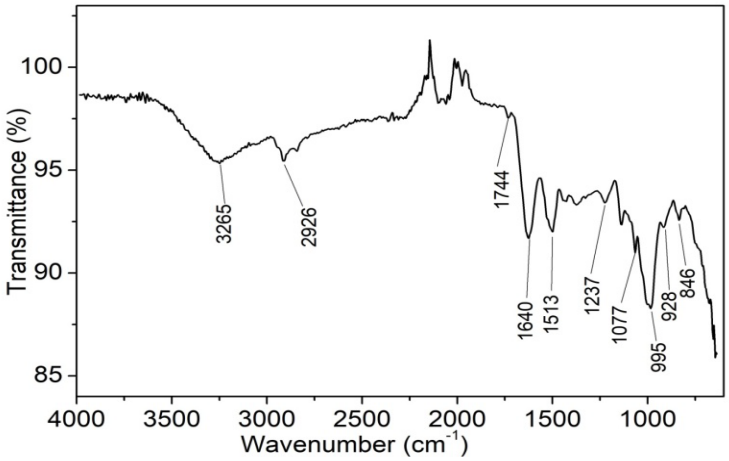

Figure 3: FTIR spectra of ovisaries horn particles

The narrow band observed at 1640 cm^{-1} is present due to stretching vibration of C=O. This band corresponding to secondary structure of protein is identified as amide I. The band at 1513 cm^{-1} identified as amide II related to out-plane bending vibration of N-H, while the amide III band observed at 1237 cm^{-1} is assigned to the C-N stretching with N-H bending vibrations (Mujeeb and Zafar, 2017; Alashwal *et al*., 2019; Wang *et al*., 2021). The broad band at 3265 cm^{-1} is assigned to the peptide bonds, -CO-NH- (Lim *et al*., 2017). Peaks at 1077 cm^{-1} and 846 cm^{-1} are due to symmetric stretching vibration of disulphide bond, S=O (Wang *et al*., 2021). The peak around 2926 cm^{-1} is attributable to C-H stretching vibration in cellulose (Lim *et al*., 2017; Galiwango *et al*., 2021). The peak at 928 cm^{-1} is related to C-C stretching vibration in carbohydrates, believed to be more specific to glucose (Mujeeb and Zafar, 2017). The peak at 1744 cm^{-1} is attributed to C=O stretching vibrations of esters groups, while the peak reflected at 995 cm^{-1} is attributable to bending vibrations of C-H group (Krishma and Patel, 2020).

3.5 X-Ray Diffraction (XRD) Analysis of Cocos Nucifera Shell Particles

The XRD patterns of the untreated and treated *cocos nucifera* shell particles are illustrated in Figure 4.

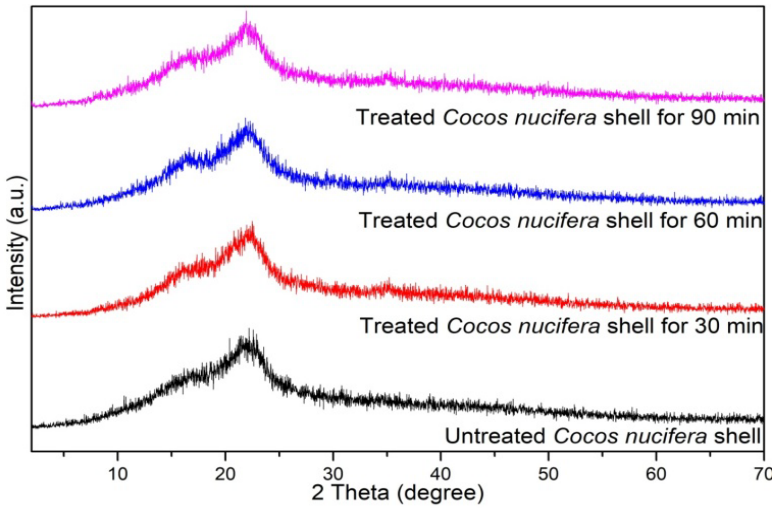

Figure 4: XRD patterns of treated and untreated cocos nucifera shell particles

The XRD patterns obtained for all samples showed peaks at $2\theta = 16.5^{o}$ and $2\theta = 22^{o}$, which are typical of amorphous materials (Tran *et al*., 2013; Lim *et al*., 2017; Bosenbecker *et al*., 2019). Alteration in crystallinity caused by the NaOH treatment was very light as there was no significant change in the XRD pattern of the samples. However, a minor peak was noticed around $2\theta = 35^{o}$, which may be attributed to the partial removal of lignin and hemicellulose, thus, suggesting a little alteration in crystallinity (Liyanage and Pieres, 2015). Likewise, the peak at 16.5^{o} was a bit more pronounced for the sample treated for 60 minutes, which is consistent with what was observed in the FTIR spectroscopic analysis, indicating that 60 minutes treatment period is more effective.

3.6 Scanning Electron Microscopy (SEM) Analysis of *Cocos Nucifera* Shell Particles

Figure 5 showed the SEM micrographs of treated and untreated *cocos nucifera* shell particles.

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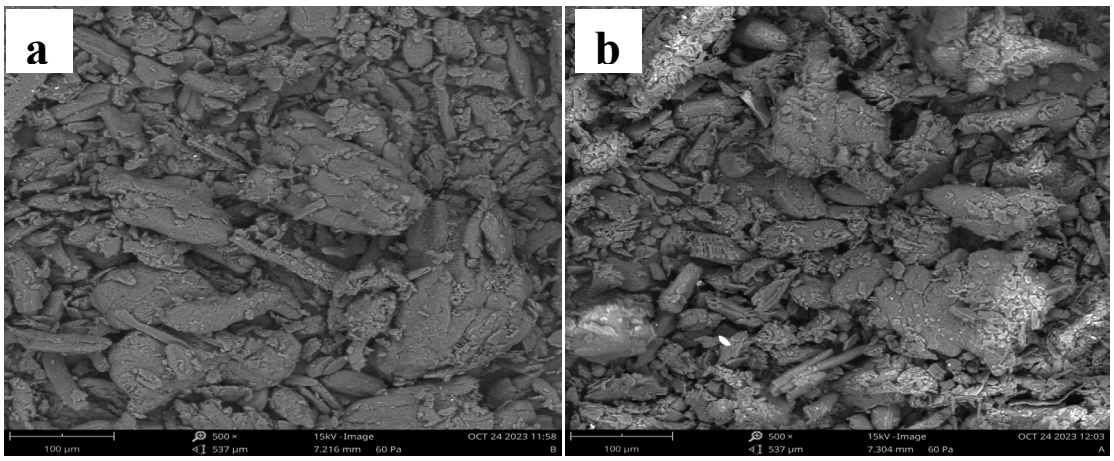

Figure 5: SEM micrographs of Cocos nucifera shell particles (a) treated (b) untreated

Difference between the morphology of the treated and untreated *cocos nucifera* shell particles was obviously revealed by the SEM micrographs. Presence of wax and other impurities can be seen in Figure 5b. The surface became clean after NaOH treatment (Figure 5a), indicating that the wax and other non-cellulosic components were significantly removed. This observation had also been reported for similar cellulosic biomass byFiore *et al*.(2015). It was also observed that the surface of the treated *cocos nucifera* shell particles appeared rougher than the untreated sample. This is desirable since the rough surface of the fibres will enhance interfacial bonding between the fibres and the polymer matrix, leading to improved mechanical properties of the composite.

4.0 Conclusions

The results obtained in this study showed that *ovis aries* horn and *cocos nucifera* shell particles have similar major elemental constituents in varied proportions, and both exhibited appreciable thermal stability, suggesting their suitability as hybrid reinforcement in polymeric composite development.The amine groups, and disulphide bond found in the *ovis aries* horn are responsible for its high chemical stability, and the presence of carbonyl, carboxyl, and hydroxyl groups in *cocos nucifera* shell suggests their suitability as hybrid reinforcement in polymeric composites. NaOH treatment of *cocos nucifera* shell particles had desirable effect on the fibres surface. Hybridization of *ovis aries* horn and NaOH treated *cocos nucifera* shell particles in polymer matrix could yield a composite material with enhanced mechanical properties.

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