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Development of Egg Shell Powder Solution as Ecofriendly Reagent: for Chemical Treatment of Natural Fibers for Polymer Composites Production

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Abstract

Problem associated with natural fibres have to be overcome before using them in composites reinforcement. The most serious concerned with natural fibres is its hydrophilic nature. Also most of the reagent's used for the treatment of natural fibers are: expensive, hazardous and reduce the strength of the fibers after treatment. The development of ecofriendly reagent for the treatment of natural fibers using eggshell powder solution is the thrust of this present investigation. The natural fiber used in this work is banana peduncle fibers. The fibers were pretreated with (NaOH) and treated with eggshell (ES) powder solution. The microstructure, tensile strength and water absorption were determined. Maximum strength of the pretreated fiber (99.844N/mm^2) was achieved at concentration of 0.25M NaOH for 1hr. ES treatment drastically reduced water absorption to 2.6% which is an improvement of 91.6% when compared to that of the raw fiber (35.83%) and was not harsh and hazardous. Based on the results obtained in this study, it is recommended that ES treatment at 20%ES in H_2O can be used to treat naturals fibers for composites production.

1. Introduction

The interest in using natural fibers in composite has increased in recent years due to their light weight, non-abrasive, non- combustible, non- toxic, low cost and biodegradable properties. However, lack of good interfacial adhesion, low melting point and poor resistance to moisture absorption, makes the use of natural fiber reinforced composite less attractive[1-2].

Problem associated with natural fibres have to be overcome before using them in composites reinforcement. The most serious weakness with natural fibres is its hydrophilic nature, which causes the fibre to swell and ultimately degradation takes place through attack by fungi[3-6]. Natural fibres are hydrophilic, they are derived from lignocellulose, which contain strongly polarized hydroxyl groups[7-8]. The major limitations of using these fibres as reinforcement in matrices includes: poor interfacial adhesion between polar-hydrophilic fibre and non polar-hydrophilic matrix; difficulty in mixing because of poor wetting of the fibre with the mixture; and the high moisture absorbance of the fibres. To reduce the moisture absorption, the fibre has to be changed chemically and physically through fiber pretreatment [8-14]. Most of the reagents used for the treatment of natural fibers are: expensive, hazardous and reduced the strength of the fibers after treatment. Therefore, there is urgent need to develop a reagent that will be ecofriendly and cheap. Hence, this present work is looking into the possibility of using eggshell powder solutions as chemical treatment for natural fibers.

2. Materials and Method

2.1. *Materials*

The materials used in this research are: banana peduncle fibre obtained from Nsukka, Nigeria, they were free of dirt and organic matter(see Figure 1a), sodium hydroxide, water and eggshell(see Figure 1b).

Figure 1a: Photograph of Banana peduncle and their fibers

Figure 1b: Photograph of the Eggshell before and after grinding

2*.2 Method*

The pre-treatment of the fibers was done by the mercerization process. The fibers with measured average cross-sectional area of 0.6mm^2 were cut into gauge length of 150mm. Six different concentrations (from 0.25M to 2.0M at equal interval) were prepared. The samples of the 150mm fibers were immersed in these NaOH solutions at room temperature. At time interval of 30mins from 0 to 3hrs, samples were taken from the different beakers washed with dilute acetic acid, rinsed with distilled water and sun dried differently. The percentage of eggshell powder used for the treatment ranged from 5 to 25v/v% were thoroughly dissolved in the corresponding volume of water, the mixed blend was poured into five (5) plastic buckets. The microstructure of the surface morphology of the eggshell powder and treated fibers were determined using a JOEL JSM5900LV Scanning Electron Microscope equipped with an Oxford INCA^{TM}Energy Dispersive Spectroscopy system. For the water absorption; fibers, were soaked in water for twenty-four hours, removed, cleaned and weighed immediately and recorded as soaked weight. The percentage water absorption was calculated using the calculation:

$$\text{Percentage of water absorption} = \frac{[wet\ weight - dry\ weight]}{dry\ weight}\ x\ 100$$

The tensile test was determined in accordance with ASTMD638-10 standard test method for tensile properties. Cross-head speed of 5 mm/min was used. The XRD patterns of the samples were recorded by PANalytical X-PERT PRO diffractometer. The crystallinity indices (CI) were calculated according to the Segal empirical method.

3. Results and Discussion

The microstructure of the eggshell (ES) powder reveals that the size and shape of the powder vary; however, they consist of porous irregular shape powder. The EDS of the ES particles reveals that the particles contain Ca, Si, O, C(see Figure 2a). These elements confirm that, the ES powder consists of calcium carbonate in the form of calcite ($CaCO_3$). Tilleyite($Ca_5 Si_2 O_7 (C O_3)_2$) etc. The eggshell powder have a similar compounds with other regents used in treatment of natural fibers [3-5].

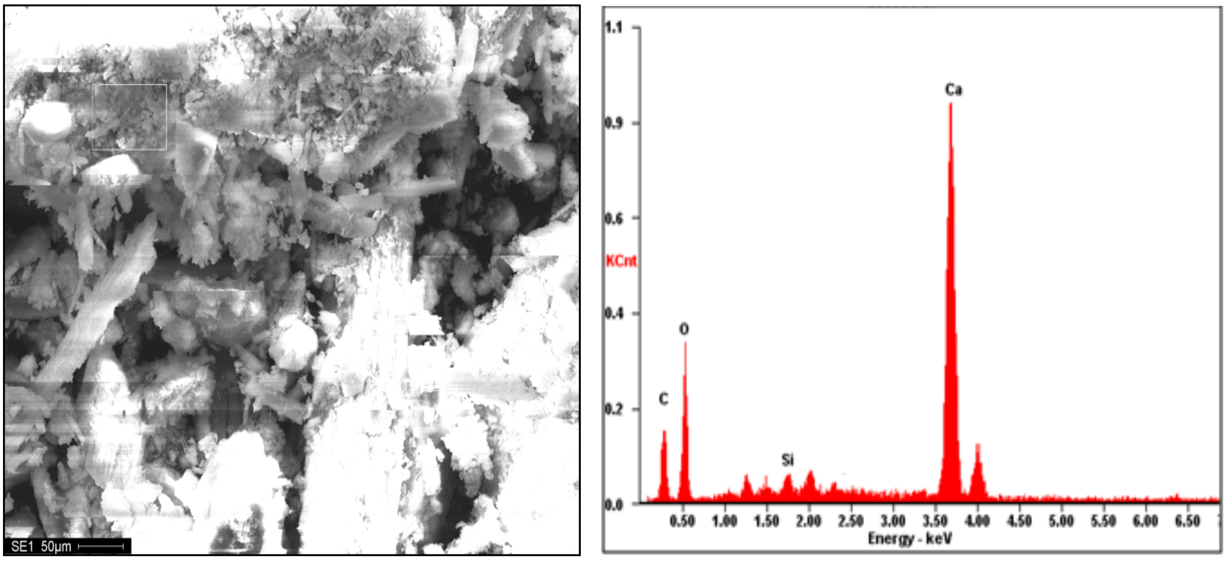

Figure 2a: SEM/EDS of ES powder.

The chemistry of the eggshell was determined by dissolving the ES in water. When water was added to ES powder, calcium salts underwent displacement reaction and this reaction results in a basic solution that was observed in this experiment, then the calcium salts may partially dissolve and release $Ca^{2+,}$ HCO_3^-, CO_3^{2-} and OH^- ions through the following reaction:

$$2CaCO_3 + H_2O = Ca^{2+} + HCO_3^- + OH^- + CO_3^+$$

These ions may be adsorbed onto the surfaces of the eggshell particles and form a negative charge. Similar phenomena have also been reported by other researchers with calcite[13-14]. The eggshell powder addition to water was not increased above 25%eggshell powder, it was observed that beyond 25% eggshell powder in water, the slurry was thick and it was difficult to agitate the bath very well. The results of the tensile strength of the pretreated and treated fibers were showed in Figures 2b-2c. From the results it can be observed that sodium hydroxide (mercerization) pretreatment of the fibers has serious effect on the chemical composition and molecular orientation of the cellulose crystallites due to the removal substances like lignin and hemicelluloses. That reduced the tensile strength of the fiber to 99.844N/mm^2 against 108.906N/mm^2 of the untreated fiber This maximum strength of the pretreated fiber (99.844N/mm^2) is achieved at a NaOH concentration of 0.25M for 1hr. Also recorded here is a positive reduction of water absorption for the untreated fibers. The optimized tensile properties for the pretreatment were obtained at 0.25MNaOH concentration at 1hr (see Figure 2b)

Eggshell powder treatment on the mercerized fibers as showed in Figure 2c. From Figure 2c there is appreciable and significant improvements on the mechanical and physical properties of the fibers. This treatment improved the strength of the fiber to 146.329N/mm^2, well above that of the mercerized fiber (99.844N/mm^2) and untreated fiber (108.906 N/mm^2). The key limitation of high water absorbance of plant fiber is hereby drastically reduced to 3.5% which is an improvement of 91.6% when compared to that of the raw fiber (35.83%). These improvements could be attributed to the $Ca^{2+,}$ HCO_3^-, CO_3^{2-} and OH^- ions formed by ES powder solution and condensation reaction with the fiber hydroxyl groups to form strong and stable covalent bonds. The hydrocarbon chains provided by this process restrain the swelling of the fiber by creating a strong cross-linked network, thus improving on the strength and making the resultant structure less permeable to water [11]. This study shows that a concentration of 20% ES powder in H_2O on the mercerized fibers produced the best results. This particular investigation reveals that eggshell powder treatment is not as harsh as acetylation, sulfate and permangate treatments previously used by others authors [12-18] rather it initiates intense cross-linking and strong bond formation.

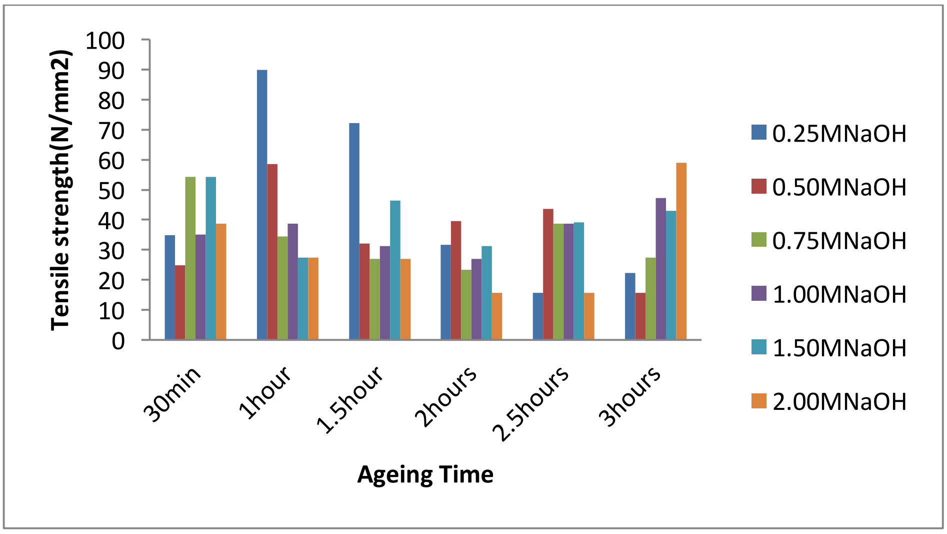

Figure 2b: Variation of Tensile strength with immersion time for the pretreatment

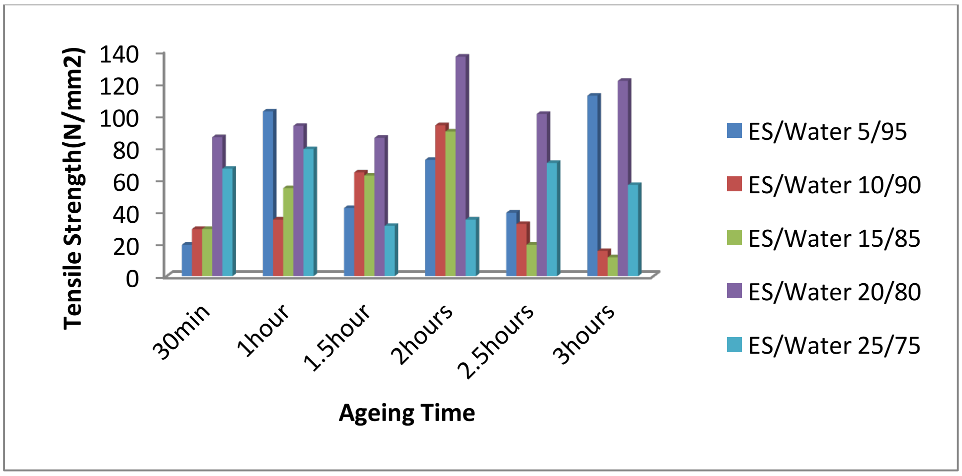

Figure 2c: Variation of Tensile strength with immersion time for the treatment

The SEM analysis is shown in Figures 3a-3b. From the SEM analysis of the banana peduncle fibers it can be observed that the fibers are not roundish but longitudinal in shape and continuous form which confirms that the fibres can be used for continuous fiber fabrication. But the treated fibers are clean, straight and longitudinal. The raw fiber was covered with a layer of substances (see Figure 3a).

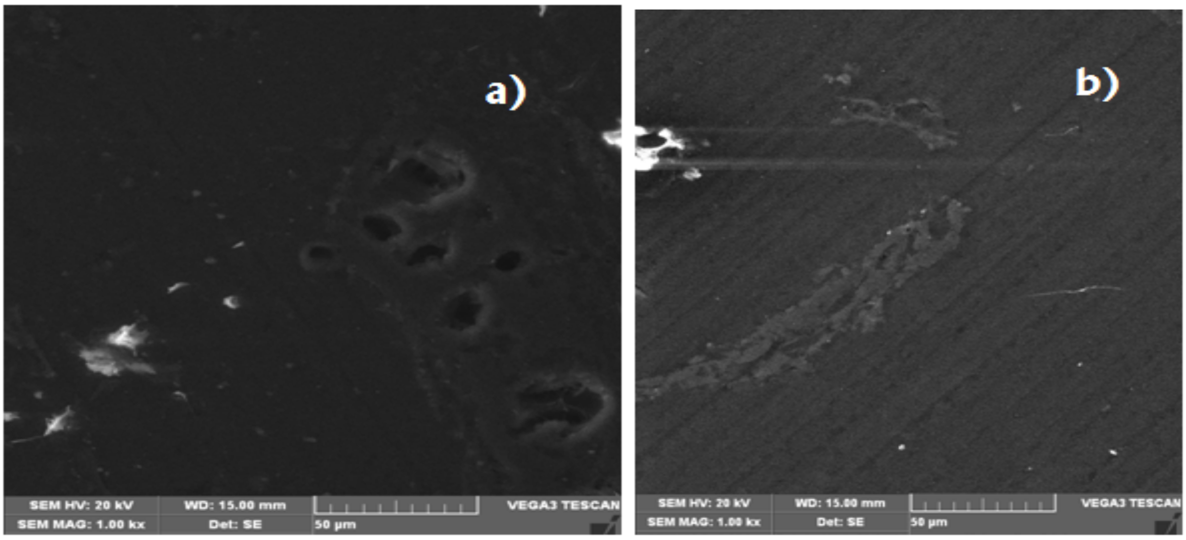

Figure 3: SEM of the a) untreated and b) ES treated banana peduncle fibers at the optimized condition.

After ES treatment, it can be seen that the treatment has removed the surface debris from the fiber (see Figure 3b), most of the lignin and pectin are removed resulting in a rougher surface. This will provides better interlocking between matrix and fiber. The fiber surface morphology plays a vital role in case of composite materials. External features of particles such as contours, defects, damage and surface layer were not seen in the SEM.

The x-ray diffraction patterns of the untreated and treated fibers scanned from 0-90°(2θ) showed several peaks due to the different constituents (Figures 3c-3d). The major diffraction peaks were: 20.5, 30.4, 45.6, 64.9, 72.1 and 83.4 ^{o} and their inter-planar distance are: 9.98, 3.57, 3.34, 3.23 1.23 and 2.12Å. The fibers exhibited typical cellulose I pattern and some lower peaks of pectin and lignin (see Figures 3c-3d). It can be clearly observed that in the treated fibers, the diffraction peaks appear in the pattern corresponding to phase with good crystalline nature. Maximum value of the intensity is clearly observed in the fiber treated. The diffraction peaks for the raw fiber increased to about 18%. This improvement contributed to enhancing the tensile strength of the fiber.

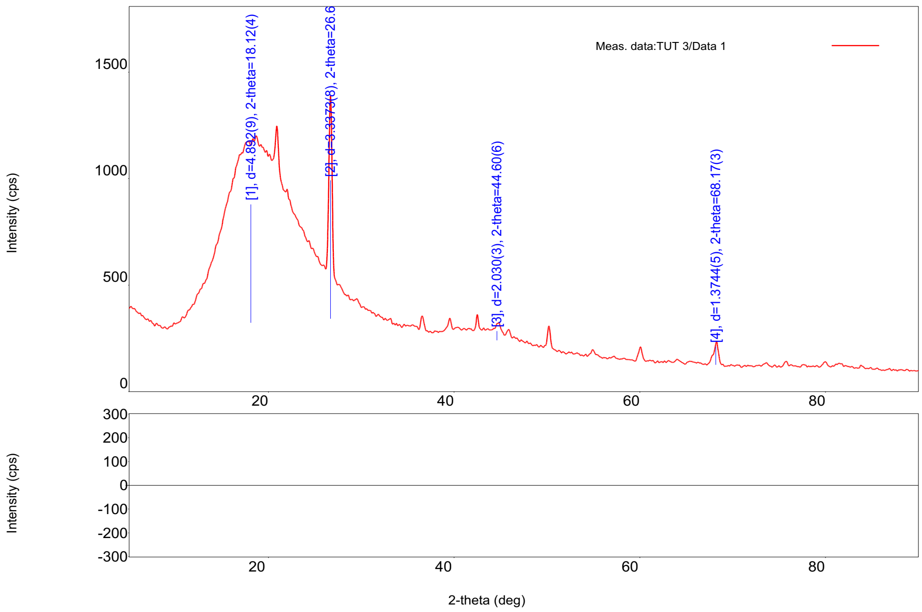

Figure 3c: The XRD pattern of the untreated banana peduncle fibers

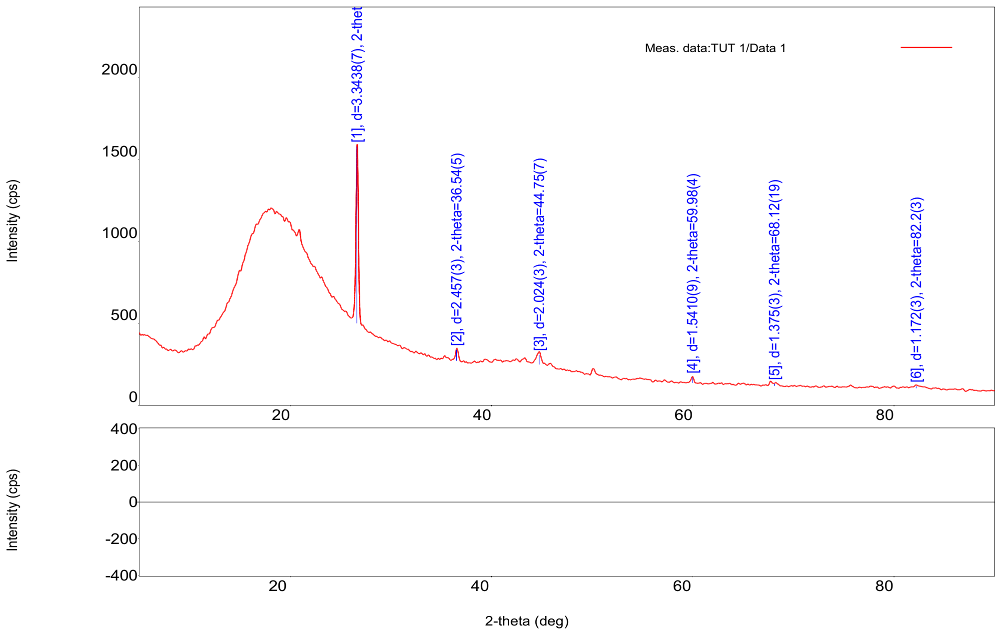

Figure 3d: The XRD pattern of the treated banana peduncle fibers

Conclusions

The effect of Eggshell powder solution treatment on the tensile properties and water absorption of banana peduncle fibers for composite production has been investigated as a function of concentration and ageing time. Based on the results and discussion above; the following conclusions can be made: This maximum strength of the pretreated fiber (99.844N/mm^2) is achieved at concentration of 0.25M NaOH for 1hr. ES treatment is not as harsh like acetylation, sulfate and permangate treatments. This study shows that a concentration of 20% ES powder in H_2O on the mercerized fibers produces the best results with a reduction of water absorbance by 2.5% (35.83%).Based on the results obtained in this study, it is recommended that ES treatment of 20% in H_2O can be used to treat fibers for composites production.

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