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Experimental study on the effects of surface treatment reagents on tensile properties of Banana fiber reinforced polyester composites

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Abstract

The interaction of the matrix and fiber at the interface between them plays an important role in the mechanical properties of a composite. Poor adhesion across the interface due to its hydrophilic nature results in weak bond and poor mechanical properties and is taken to be a major limitation. This study focuses on the effects of various surface treatment reagents in improving the tensile properties of the banana pseudo stem reinforced polyester composites. An unsaturated polyester composites reinforced with fibers well above 200mm and laid at 0^o to the loading direction were produced using hand lay-up method. The result shows that the tensile strength and tensile modulus of unreinforced polyester resin was 31.26±0.02MPa and 973.47±0.9MPa respectively while the tensile strength of the untreated fibers was 141.46±1.9MPa and 0.4MNaOH mercerized fibers gave the highest single fiber tensile strength of 84.32±1.2MPa at 1hour among the NaOH concentrations. The tensile strength of acetic acid and sodium sulphite post-treated mercerized fibers reinforced composites was 97.02±2.5MPa and 108.30±0.6MPa respectively, while silane solution gave the highest improvement in tensile strength of the composite of 142.61±2.2MPa with a tensile modulus of 3,798.89±5.8MPa. It was concluded that acetic acid, sodium sulphite are good surface treatment reagents but less effective to 3-aminopropyltriethoxysilane (APTES) silane solution for the improvement of the tensile properties of banana pseudo-stem fiber reinforced polyester composites for industrial applications.

1. Introduction

The interest in natural fiber reinforced polymer composites is rapidly growing both in terms of their industrial applications and fundamental research [1-3]. Natural fibers are readily available, easy to process, renewable, low density, and low cost and possess adequate mechanical properties thus becoming an attractive ecological alternative to glass, carbon and other synthetic fibers for use in composite manufacturing. They are mostly used to produce non-structural parts for the automotive industry such as car wheel covers, car bumpers, upholsteries, doors panels etc. and also possess very good acoustic and thermal insulation properties [4]. Various types of natural fiber yarns are commercially available, such as cotton, jute, flax, sisal, kenaf, pineapple, ramie, bamboo, and banana. They are rich source of lignocellulosic fibers, and are more often applied as reinforcements for polymer composites [5]. Even though natural fibers have several advantages, the lignocellulosic fibers are hydrophilic and absorb moisture which limits its use in engineering applications. Kabir et al. [6] reported that there is a large amount of hydrogen bonds (hydroxyl groups -OH) present between the macromolecules in the plant fiber cell wall and that it becomes multiplied when moisture from the atmosphere comes in contact with the fiber, due to bond breakdown and formation of new hydroxyl groups with the water molecules.

This incompatibility of fibers with the polymer matrix leads to low fiber-matrix interfacial bond strength and poor wetting of the fibers by the matrix resin which usually results in poor mechanical properties of the composites. However, surface modifications by physical methods or chemical methods tend to reduce these drawbacks, [7]. The electric discharge such as corona and cold plasma treatments are the most common of the physical methods in which surface oxidation activation of the fibers occur, but this method is comparatively expensive. Chemical modifications such as alkali treatment, acetylation treatment, isocyanate treatment, silane coupling agents, and grafting agents have been shown to improve fiber matrix adhesion [8].

Silanes have reactive alkyl groups that bond chemically to form adhesives and have been found to reduce the number of hydroxyl groups in the fiber matrix interface thereby effectively modifying the natural fiber-polymer matrix interface and promote adhesion and stabilize the composite. [9]. Acetic acid treatment of Napier grass fiber strands was carried out by Venkata et al. [10] and reported effective changes in the surface topography of the fiber strands and their crystallographic structure and removal of surface impurities and increase in the crystallinity index of the fibers. There are scanty data on use of sodium sulphite for surface treatment of natural fibers. On the other hand, alkali treatment increases surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface.

Banana has long been considered a food and fruit, but it is also abundant source of fibers. Banana fibers can be sourced from the stem (trunk) or from the bunch (peduncle), both are agro-waste materials readily available in every part of the world. Banana trees produce fruit once year, and are then cut down, such that the trunk is an abundant agro-waste product [11]. There are various ways of extracting banana fibers from pseudo-stem, this include mechanical methods, chemical methods and microbiological methods. Mechanical method of extraction results in incomplete removal of vegetable matter from the fibers Kulkarni et-al. [12]. Pothan et al. [13] adopted the chemical extraction method and found out several drawbacks. Also, Ganan et al. [14] adopted the fiber extraction method by retting, and reported that retting is a long-term process which affects the fiber quality significantly however it is the easiest way. A method of bioextraction has been developed by (NIIST-Thiruvananthapuram) - (CSIR-India) recently, which involves use of enzymes in the interaction of the fibers, it is very efficient and produces clean strong banana fibers in 2-3 days in a bioreactor Arya et al. [15].

Several studies have been reported in the field of polymer matrix composite using banana fiber as the reinforcement, [16-18]. Deepa et al. [16] studied the structure, morphology and thermal characteristics of banana nanofibers. They used steam explosion method for the extraction of banana nanofibers and it was reported that the developed nanofibers exhibited enhanced thermal properties over the untreated fibers. Musanif and Thomas [17] showed that alkali treatment of banana fiber improved the mechanical properties of both the epoxy/vinyl ester and hybrid composites. Joseph et al. [18] modified banana fiber using silane treatment, acetylation, cyanoethylation, latex treatment and NaOH (alkali), which improved the interfacial bonding with phenol formaldehyde (PF) resin.

Kaur et al. [19] studied the performance of bamboo fibre modified with different concentrations of sodium hydroxide at different processing conditions, in combination with potassium hydroxide and chlorine containing agent sodium chlorite solution and reported that lignin content analysis of the extracted fibre bundles indicated a remarkable reduction in lignin content and removal of short elementary fibres from their surfaces after these treatments. Aigbodion et al.[20] worked on the development of egg shell powder solution as ecofriendly reagent; for chemical treatment of natural fibers for polymer composites production and reported that maximum strength of the pretreated fiber was achieved at concentration of 0.25M NaOH for 1hr and an egg shell treatment drastically reduced water absorption to 2.6% which is an improvement when compared to that of the raw fiber. Zin et al. [21] reported a slight reduction in fibre diameter with increasing NaOH concentration as observed with a digital image analyzer in their work on the effects of alkali treatment on the mechanical and chemical properties of banana fibre and adhesion to epoxy resin. The study futher reported that 6% NaOH treatment with a two-hour immersion time gave the highest tensile strength and interfacial shear stress.

William and Patrick [22] worked on two different chemical treatments designed to promote the interfacial bonding between banana fibers and an LDPE matrix: peroxide treatment and permanganate treatment and reported that the peroxide treatment enhanced the tensile properties and permanganate treatment having an inconclusive effect.

The aim of this study is to know the effects of using sodium sulphite and acetic acid, alongside with sodium hydroxide (NaOH) and silane solution (Aminopropyltriethoxysilane (APTES)) as surface modifiers/ coupling agents on the tensile properties of banana pseudo-stem fiber reinforced polyester composites prior to applications.

2. Materials and methods

2.1 Materials

The Banana pseudo-stem (BPSF) fibers were obtained as an agro-waste material from a farm in Nsukka, Nigeria. The surface treatment reagents used include acetic acid, sodium sulphite, sodium hydroxide and silane solution (3-aminopropyltriethoxysilane (APTES) 98% Sigma Aldrich) and citric acid. The unsaturated polyester resin, cobalt naphthenate and methyl ethyl ketone peroxide were supplied by a vendor.

2.2 Methods

2.2.1 *Extraction of banana fiber*

Banana pseudo-stem fibers was extracted via water retting method. A banana trunk (Plate1a) was cut into length above 500mm and separated with hand into smaller sizes (Plate1b) and was completely immersed in a water tank for 14 days. Water penetrates into the central stalk portion, by breaking the outermost layer, and thus provoking an increased absorption of moisture and the development of pectinolytic enzymes [23]. This activity dissolves the lignin and hemi-celluloses from the fibers. The separated fibers were washed severally and dried by spreading them under sun for 4days (Plate1c)

Plate 1: (a) Banana plant (b) separated banana stem (c) extracted banana fibers

2.2.2 *Fiber surface treatment*

(i) Alkali treatment

Varying concentrations of sodium hydroxide solution were prepared in 250 ml of distilled water, 0.2M, 0.4M, 0.6M, 0.8M and 0.10M. The banana fibers were soaked in the prepared NaOH solution for 1 hour at room temperature with intermittent stirring to remove soluble cellulose, hemi-cellulose, pectin and lignin. After soaking, the fibers were washed severally with distilled water to remove any alkali solution sticking on their surface and then neutralized with diluted 5%(w/v) of citric acid and rewashed severally with water. Finally, the fibers were dried in open air/sun for hours and further dried in the oven at 60^{o}C for 24 hours.

(ii) Post alkali surface treatments

Sodium sulphite (Na_2SO_3), 3-aminopropyl-triethoxy silane (APTES) and acetic acid were used to treat the mercerized (NaOH) treated fibers. Some quantity of the fibers was soaked in 6% (w/v) of Sodium sulphite for 1 hour at room temperature, silane treatment was carried out with APTES. First, the required amount (v%) of silane solution was prepared, by mixing silane coupling agents with an ethanol/water mixture and allowed to stand for 1 hour, where the ratio of fiber to solvent was maintained at 1:40. Then, the fibers were dipped in this solution and stirred intermittently for 1 h at room temperature. The fibers were brought out washed thoroughly with distilled water and dried in open air and then in an oven, at 60^{o} for 24hours. Similar process was repeated using 5% (v/v) of acetic acid in water. All the treated fibers were thoroughly washed with distilled water and dried in open air/sun for hours and in an oven at 60°C for 24hours. The fibers were cut to about 200mm and matted with fevicol adhesive.

2.2.3 *Composite preparation*

Hand lay-up technique was used to obtain the composite plates of the size; 300 mm x 300 mm × 5mm (Plate2a), the matrix was prepared by mixing unsaturated polyester (UP) resin with 1% the accelerator and 1.5% of catalyst and vigorously stirred. The volume fraction of the fibers used was calculated using mass fraction relation model (equation 1), [24]

$$V_f = \frac{(M_f/\rho_f)}{(M_f/\rho_f) + (1 - M_f)\rho_m} \quad \text{………} (1)$$

Where M_f = mass fraction of the fiber used, ρ_m = density of the matrix, ρ_f = density of the fiber

2.2.4 *Mechanical testing*

(i) Single fiber tensile strength

Single fiber tensile strength (SFTS) was carried out on the fibers treated with various concentrations of sodium hydroxide in line with earlier work [25]. The fibers were separated into single strand by hand and fibers of about 0.1- 0.4 mm diameter were selected using a hand-held optical microscope and a scale rule. The two ends of the fiber were attached to a piece of thick board paper measuring 25 mm x 100 mm with provision of 50 mm gauge length properly secured with fevicol adhesive. The single fiber strand tensile test was done using a Testometric model Universal Testing Machine according to ASTM D3379 at 10 mm/min load speed and an approach speed of 1mm/min and within a stress range of 0-500 MPa. The treated fiber with the best tensile strength was selected for further treatment with other reagents and for composite lamination.

(ii) Composite tensile strength

The composite tensile specimens were tested as per ASTM D3039 using computerized Testometric model Universal Testing Machine at a crosshead speed of 10 mm/min and strain rate of 2mm/min. The test was conducted at room temperature at a relative humidity of about 60%. The specimen dimensions were 200mm × 20mm × 5mm (Plate2b). Three specimens were tested to failure for each sample group.

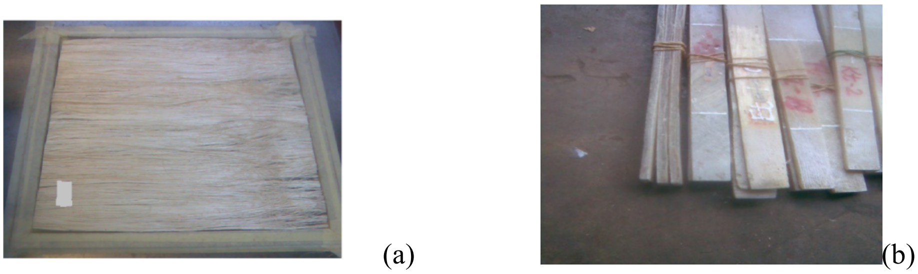

(a) (b)

Plate 2: (a) matted banana fiber in a mould (b) tensile specimen

2.2.5 Microstructural Analysis

The scanning electron microscope (SEM) JEOL-JSM-6480LV was used to identify the surface morphology of the banana fibers. The samples were cleaned thoroughly, air-dried and coated with 100Å thick platinum JEOL sputter ion coater and with gold to increase surface conductivity, and observed under SEM at 20kV.

Results and discussion

3.1 Micrograph Analysis by scanning electron microscopy

Figures 1(a, b, c & d) shows the micrographs for the untreated and treated banana fibers with NaOH, APTES silane and $NaSO_3$ respectively. The micrograph of the untreated fiber (Figure 1a) shows lumens covering the cellulose, while all the treated fibers show some degree of roughness of the fibers, and collapse of fiber lumens, with $NaSO_3$ indicating the least roughness (Figure 1d). Fibrillation and surface roughness are evident in the mercerized banana fiber (Figure 1b).

These roughing is an indication of lignin wax, oil and impurities removal from the fiber surface. This increase in the surface roughness may enhance the interfacial bonding between the fibers and the polymer and the possibility of load transfer between the fibers and polymer increased as well, which is consistent with the results obtained by Rodríguez eta-al. [26].

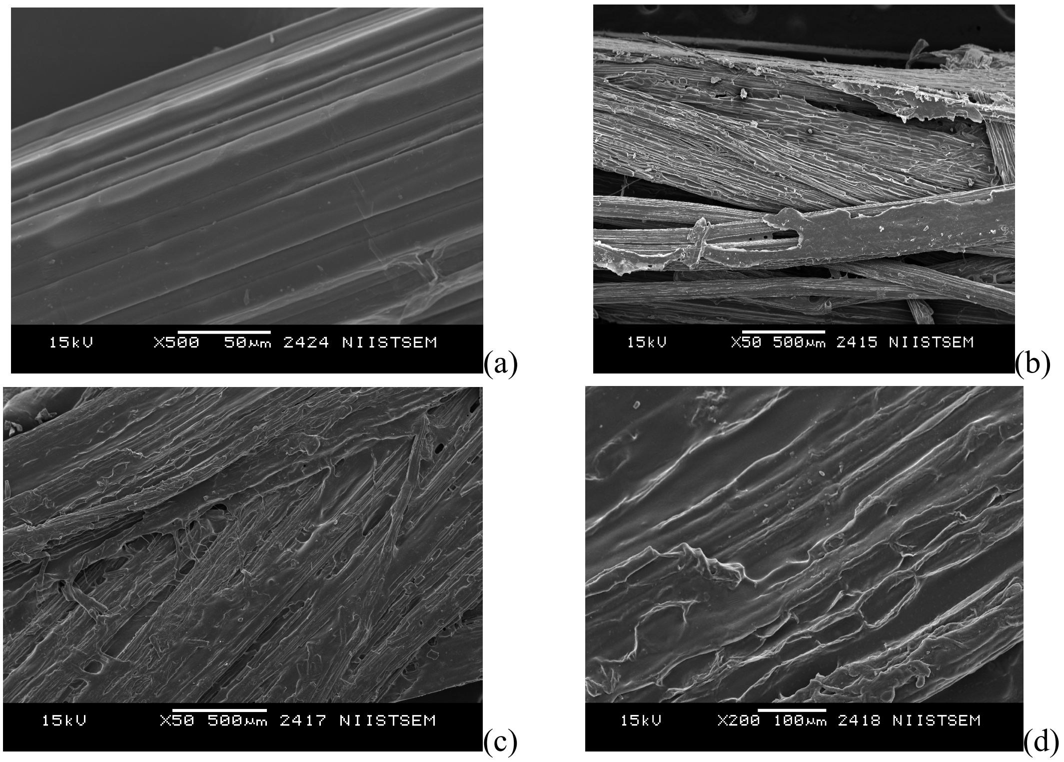


Figure 1: SEM micrographs of BPSF surface treatments with (a) untreated BPSF (b) 0.4MNaOH (c) APTES (d) $NaSO_3$

3.2 Single fiber tensile strength.

The results of the single fiber tensile strength, tensile modulus and percentage elongation of the banana pseudo stem fibers treated with various concentration of NaOH are shown in Figures 2-4 respectively. The results indicated that pretreatment of the banana fibers with NaOH has serious effect on the fiber tensile properties due to the removal of the lignin and hemicellulose (Figure 1) which promotes its affinity to water in-take. This may be the reason why the untreated fibers have a higher single fiber tensile strength of 141.46±1.9MPa and tensile modulus of 2090.67±0.5MPa than the best-mercerized fibers (0.4M NaOH) with a tensile strength of 84.32±1.2MPa and tensile modulus of 1948.26±1.3MPa.

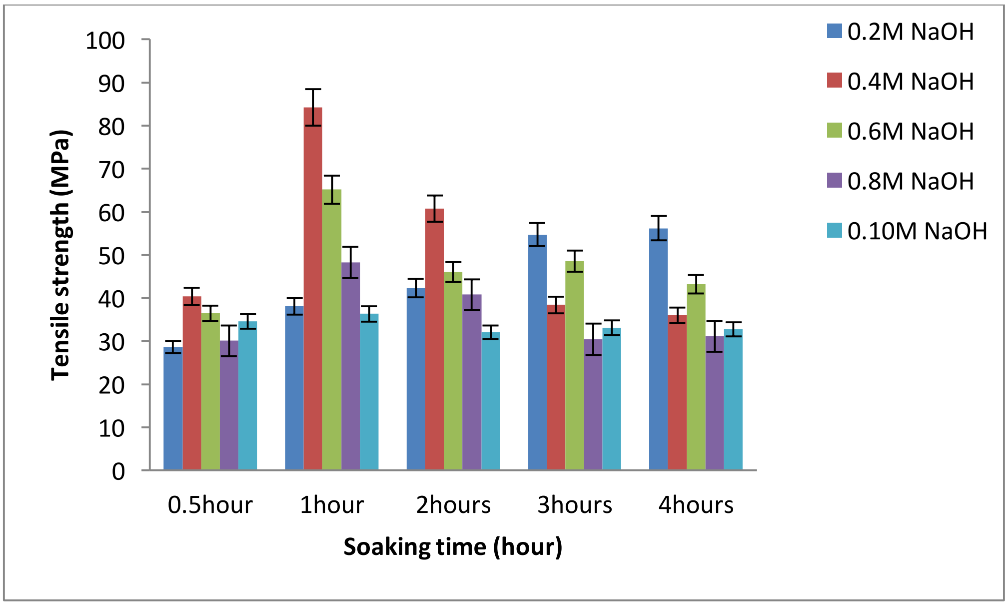
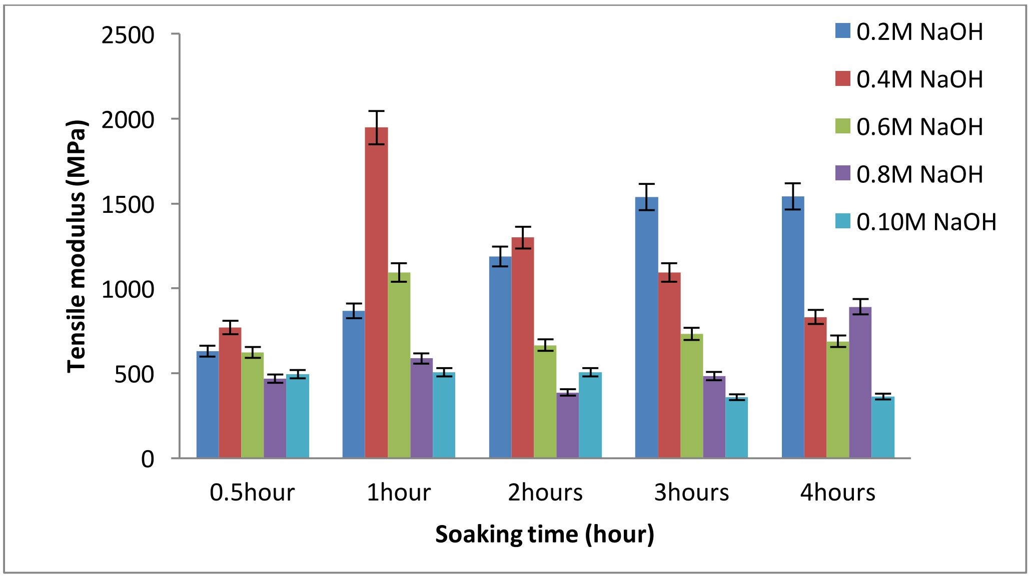


Figure.2: Variation of NaOH concentration and soaking time on single fiber tensile strength



Fig**ure.3:** Variation of NaOH concentration and soaking time on the single fiber tensile modulus

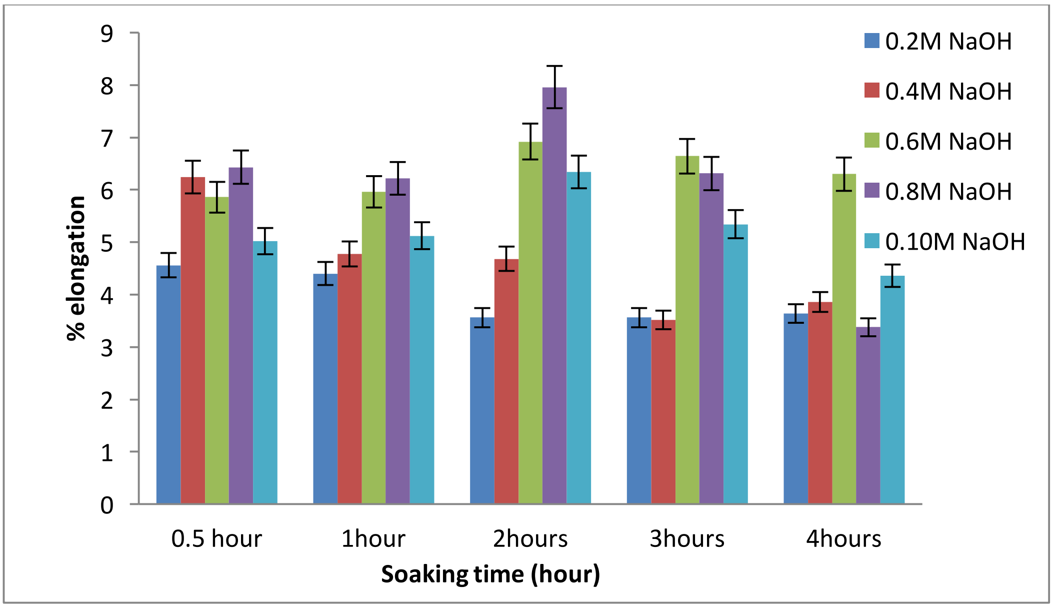


Figure.4: Variation of NaOH concentration and soaking time on the % elongation

It was also observed that the tensile properties of the 0.4MNaOH concentrations increased from 40.32±1.8MPa and 769.47±0.03MPa for tensile strength and tensile modulus respectively at 0.5 hour, to its highest values at 1 hour. However, the tensile strength and modulus decreased with subsequent increase in the soaking time from its peak at 1 hour to 36.04 ±2.8MPa and 830.06±2.1MPa at 4 hours of soaking time, meaning that the optimal soaking time for the optimal 0.4M NaOH concentration is 1 hour.

All the other concentrations of NaOH concentrations almost showed the same trend as the 0.4M NaOH, except for 0.2M NaOH, which showed increase in tensile properties as the soaking time increased, reaching its maximum tensile strength and tensile modulus of 56.17±1.3MPa and 1543.13±1.8MPa respectively at four hours. Surface treatment increases surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface and ionizes the hydroxyl to alkoxide and remove lignin, hemicelluloses as given in equation 2, [27]:

Fiber-OH + NaOH — Fiber-O-Na+ + H2O .... (2)

Naveen et al [28] reported that the fibers tend to undergo delignification after 1 hour which leads to decrease in the strength, however this was not true for low concentration of NaOH such that 0.2M NaOH which improved in strength after 1 hour soaking time. Figure 4 denotes the percentage elongation of the single fibers tested. It was observed that high percentage elongations were observed at 2 hours with 0.8M NaOH and 0.6M NaOH treated fibers. Also it was recorded that there is a positive percentage (%) elongation for all the treated fibers within 2 hours of soaking time, after which the % elongation of the lower NaOH concentration decreased.

3.3 Composite tensile properties

Results of APTES silane, sodium sulphite and acetic acid treatments on the 0.4MNaOH mercerized banana fibers reinforced composites are as shown in Figures 5-7. From Figure 5 and Figure 6, it can be seen that there are appreciable and significant improvements on the tensile properties due fiber reinforcement compared to unreinforced polyester. The improvement was further enhanced with the Sodium sulphite and acetic acid treated fibers but more pronounced with APTES silane solution treated fibers. The $NaSO_3$ and acetic acid treated fibers gave a composite tensile strength of 108.30MPa and 97.02MPa respectively which was a little above the mercerized fiber composites, however these are all lower than the untreated. The fiber treatment with APTES silane shoots up the strength of the composite to 142.61±2.2MPa, well above that of the mercerized fiber reinforced composites with 76.34MPa and untreated fiber composite with 101.72MPa. The high tensile properties of the silane treatment fibers composites were attributed to the fact that silanes treatment reduces the number of hydroxyl groups such that the hydrolysable group formed silanols which react further with the fibers to form a well stable covalent bond that is more compatible with the hydrophobic polymer resins [29]. Allan et al. [30] earlier reported that the hydrocarbon chains formed create a strong cross-linked network that restrains the swelling of the fiber thereby taking in less water and thus improving the strength. Figure 7 also indicates that the silane solution tends to retain the original ductility of the fiber which is evident in its good percentage elongation compared to the untreated, an indication that it modifies the fiber chemistry and reduces its hardness hence improving the ductility of the composite.

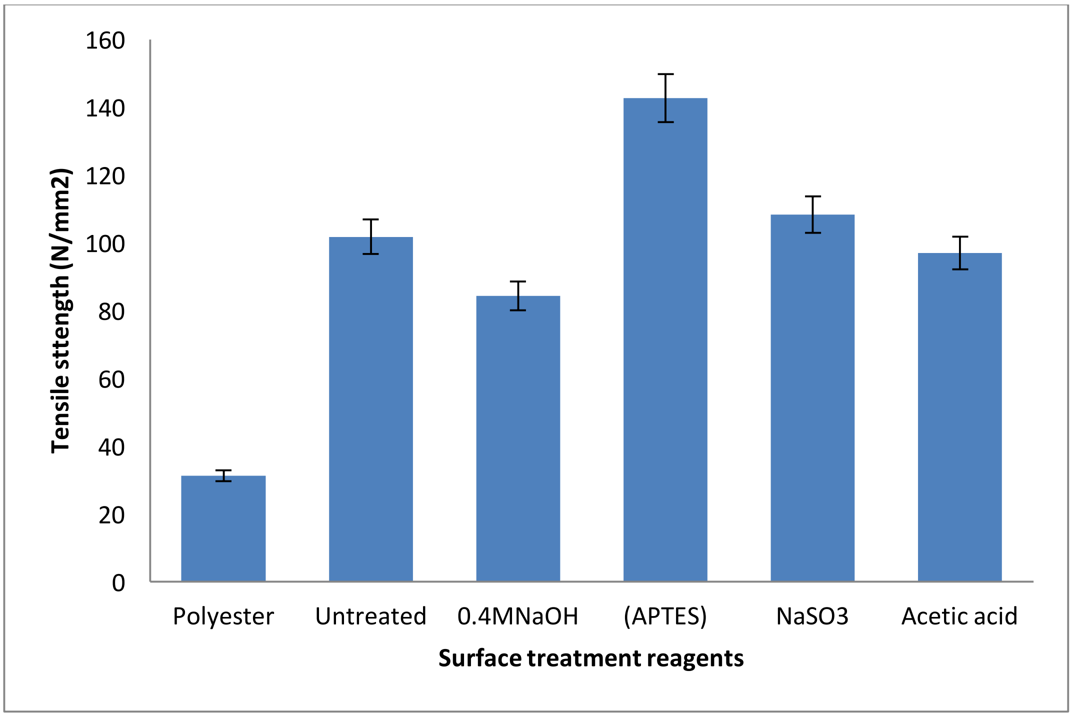


Figure 5: Variation of surface treatment reagents on tensile strength of the composite

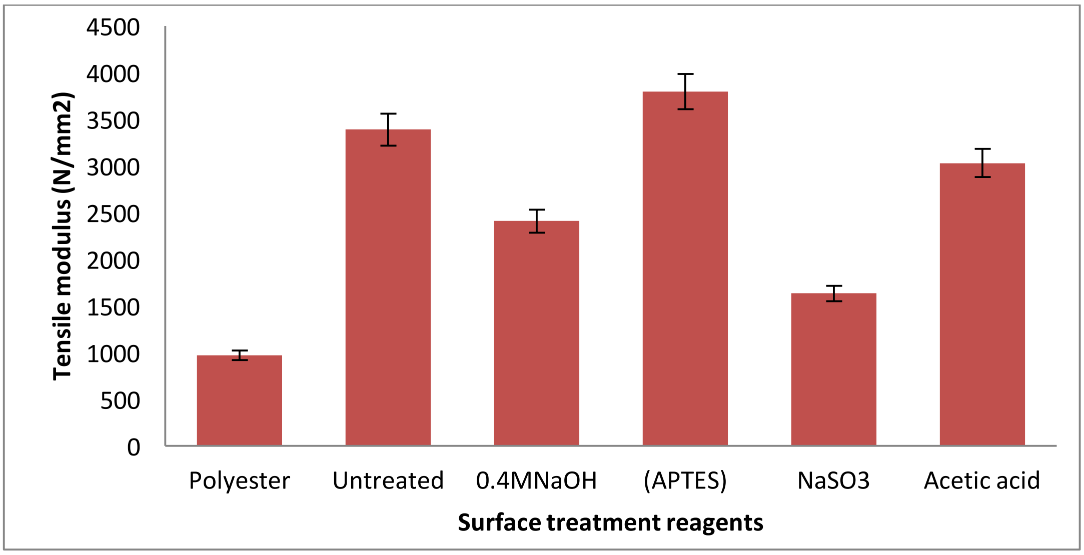


Figure 6: Variation of surface treatment reagents on tensile modulus of the composite

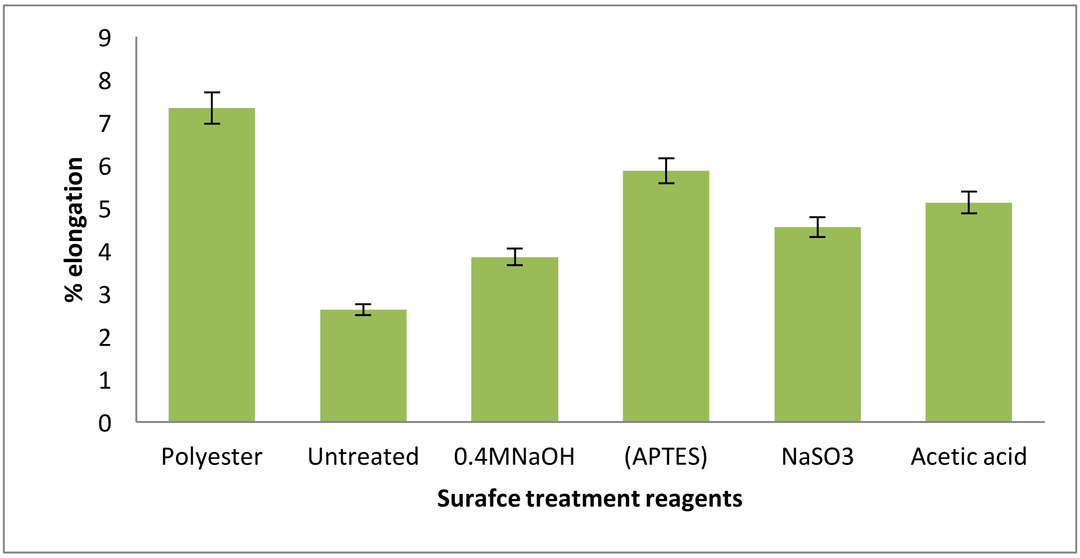


Figure 7: Variation of surface treatment reagents on % elongation of the composite

Conclusion

A study on the effects of surface treatment reagents on tensile properties of banana pseudo stem fiber reinforced polyester composites was carried out. The result shows that mercerization reduced the tensile strength of the untreated fibers, while further treatment on the mercerized fibers with APTES silane solution and sodium sulphite enhanced the tensile properties of the reinforced composites. Concentration of NaOH above 0.4M reduces the tensile strength and tensile modulus of the single fiber strand while concentration below 0.4M is effective but takes longer soaking time to improve the tensile properties of the fiber, for this reason 0.4M NaOH was observed the best concentration for soaking banana fibers. A soaking time of 1hour is also found to be optimum for most of the NaOH concentrations. The chemical reagents used in this study; acetic acid, sodium sulphite are good surface treatment reagents but less effective to 3-Aminopropyltriethoxysilane (APTES) silane solution for the improvement of the tensile properties of banana pseudo fibers reinforced polyester composites for industrial applications.

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