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Effects of Chemical Surface Treatment on Mechanical Properties of Sisal Fiber Unsaturated Polyester Reinforced Composites

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ABSTRACT
Sisal fiber was extracted from Sisal leaves by enzymatic retting method. A portion of the fiber was subjected to alkali, benzoyl chloride and silane treatments. Sisal fiber composites were fabricated using unsaturated polyester resin, by hand lay-up technique using both the treated and untreated fiber. Tensile and flexural tests were conducted and evaluated on the composites. The morphology of the materials was studied using scanning electron microscopy (SEM). The fibre chemical modification improves its adhesion to the matrix as well as the mechanical properties of the composites.

Keywords: Scanning Electron Microscopy, Sisal fiber, Tensile test, Unsaturated polyester resin

INTRODUCTION

Over the past few decades, it has been found that natural polymers have replaced many of the conventional metals/materials in various applications. This is due to the advantages offered by natural polymers over conventional materials. The properties of many natural polymers include ease of processing and productivity (Saheb and Jog, 1999). The properties of the polymers are usually modified using fillers and fibers to suit the high strength/high modulus requirements. These modifications usually lead to the development of natural fiber composites. Natural fibers are low cost materials, with low density and high specific properties; they are biodegradable and non-abrasive (Saheb and Jog, 1999).

Fibers are materials that continuously discrete elongated pieces, similar to that of thread, they can be spun into filaments, thread or rope, can be used as component of composites, can be matted into sheet-like products such as paper or felt. Fibers are broadly classified as natural or synthetic. Natural fibers can originate vegetable (plant), animal or minerals. The plant fibers are composed of cellulose as the main component and also lignin, as such they are termed ligno cellulose with the exception of cotton. Animal fibers generally comprise of proteins such as collagen, keratin and fibroin. Examples of fibers from animal sources include Silk, Sinew, Wool, Catgut and Mohair among others. Many synthetic fibers are based on wood (Rayon), or petroleum such as Dacron, Nylon and many others (David, 2005).

Composites have become an integral part of our daily life and can be found everywhere. In the olden days, bricks made up of straw and mud are very good examples of composites. Composites in form of wood, teeth, bones, muscle tissue also have their importance in nature. Also environmental awareness, increasing concern with green house effect and bio-degradation has urged so many industries to look forward to sustainable materials with least impact on the existing surroundings. As such, natural fiber reinforced composites seem to be a good alternative due to their eco-friendly nature and renewability. This has made them attractive materials for their application in diverse fields (Rakesh, 2011). Properties of the composites are governed by the properties of the fiber, aspect ratio of the fiber, thermal stability of the fiber and fiber-matrix interface. The surface adhesion between the fiber and the polymer and, also the cohesion in the fiber material plays vital roles in transmitting stress from the matrix to the fiber and thus, contributes towards the performance of the composite. In the case of thermoplastic composites, the thermal stability of the fiber is very important because the process involves the use of high temperature, hence dispersion of the fiber in thermoplastic composites must be considered (Saheb and Jog, 1999). In this paper, Sisal fibre was used as reinforcing fibre and unsaturated polyester resin as the matrix. The effect of chemical treatment on the mechanical properties of the Sisal fibre reinforced unsaturated polyester composites were investigated.

MATERIAL AND METHODS

Materials:

Sisal plant was obtained from Gurin-gawa of kumbotso local government, Kano state. Sodium hydroxide (NaOH) Analar Co, Benzoyl Chloride (C_6H_5OCl) and phenyltriethoxysilane ($C_6H_5Si(C_2H_5O)_2$) coupling agents (Merck Co Ltd). Surface morphologies of the sisal fiber and the composites were observed using Scanning Electron Microscope (Phenom ProX super) at Ahmadu Bello University, Zaria, Kaduna State, Nigeria. The tensile and flexural tests were conducted using Ultimate testing machine (UTM), Shimadzu (MODEL AG-1).

Fibre Surface treatments

Sisal fibre was obtained from the dried leaves of the Sisal plant by enzymatic retting method for seven days according to the method reported by (Favaro *et al*., 2010). The fibre was obtained by gently squashing the leaves with hand followed by scraping and carding with soft nylon brush. Distilled water was used to wash up the surplus wastes on the fiber such as chlorophyll, leaf juices, adhesive and other solid materials. The clean extracted fibre that was obtained was dried under shade for two days, after which combing was done to give fine Sisal fibre strands.

Alkali treatment

The fibre was treated with 5% NaOH solution for 1hour. It was then rinsed with distilled water. The fibre was washed with dilute hydrochloric acid (HCl) solution to neutralize the alkali. The fibers were then rinsed with distilled water until the fibers were alkali free. The washed fibers were dried under shadow at room temperature (Velmurugan, 2012).

Benzoyl treatment

Portion of the mercerized fiber was suspended in 10% NaOH and benzoyl chloride (C_6H_5OCl) solution for 15mins, after which the fiber was removed and soaked in ethanol for 1 hour to remove excess benzoyl chloride. It was finally rinsed with distilled water and dried in an oven at 80^0 C according to the method reported by Joseph, (2000) and Kalia, (2011).

Silane treatment

A solution of 1% phenyltriethoxysilane in acetone was prepared. Acetone was used in preference to water to promote hydrolysis to take place in presence of moisture on the surface of the fiber rather than with the carrier. The solution was maintained at pH 4 by adding acetic acid, stirred for 10mins. Portion of the dried alkali pretreated fibers were soaked in the solution for 1hour. The Fibers were removed from the solution and dried in hot air oven at 60 ^{0}C until they were dried (Uma, 2012).

Composite fabrication

The composite was fabricated by using a 16cm hand lay-up mold. A matrix comprising of 100ml unsaturated polyester cured with 1.6% cobalt naphthenate and 2.7% methyl ethyl ketone peroxide (MEKP) was poured on 1.0g of the fiber whose length is 11.1cm in the mold. Curing took 15mins at room temperature before removal of the composite (Abdullah and Ahmad, 2012).

Mechanical property tests

The tensile and flexural tests were conducted on the samples (150mm × 25mm × 4mm) according to ASTM-D 636-03 and ASTM-D 790-97A standard respectively. An Ultimate Testing Machine (UTM) and a Shimadzu instruments (MODEL AG-1) for the two tests respectively. The Shimazu instrument was operated with cross head speed 0f 20mm/min in each case and also a support of 51mm in carrying out the tensile test at room temperature.

FT-IR Spectra

FTIR spectra of the untreated and the treated fibre composites were obtained using a Cary 630 FTIR machine of Agilant Technology, in accordance with FT-IR Attenuated total reflection mode Technique (Favaro, 2010).

Scanning Electron Microcopy (SEM)

The samples (430 x 624µm) length were gold plated by sputtering technique and observed under the Scanning electron microscope at different magnifications. The composite fractured surface analyses were performed after immersing the material in liquid nitrogen for 10 minutes (Favaro, 2010).

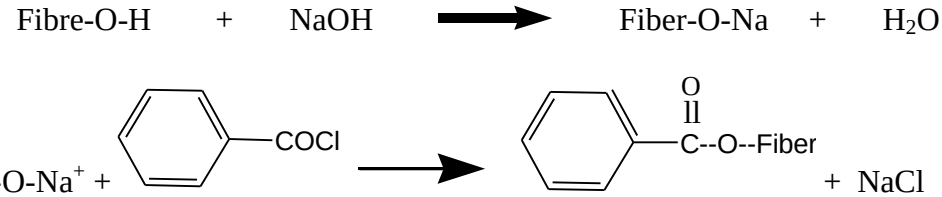
RESULTS AND DISCUSSION

Fibre Surface treatments

Treatment of fibre with some reagents usually changes the surface characteristics of the fibre. Sisal fibre was modified using Alkali (NaOH), benzoyl Chloride and Phenyltriethoxysilane. Mercerization of the fibre activates the O-H groups on the cellulose of the fibre and further replaced by Na^+ of the alkali, resulting in the modification of the fibre (Velmurugan, 2012). The process changes the chemical behaviour of natural fibre. The effect of alkali on cellulose fibre is a swelling reaction, during which the natural crystalline structure of cellulose relaxes. Type of alkali and its concentration influence the degree of swelling, the method has lasting effect on the mechanical behaviour of natural fibres especially on their strength and stiffness (Hashim, 2012), as shown in the reaction.

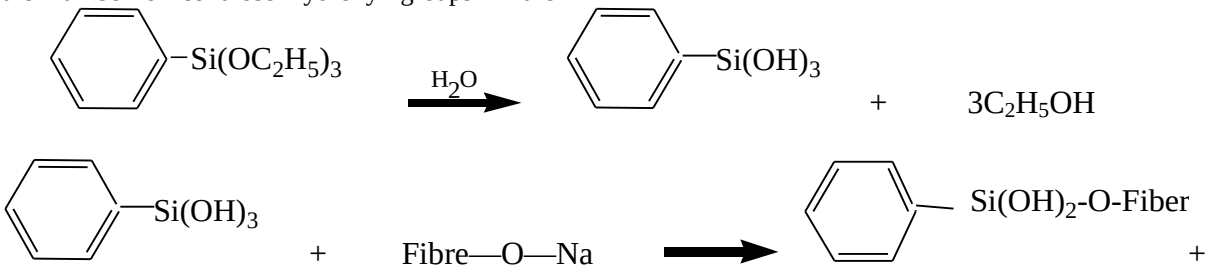
Scheme 1: Reaction between cellulosic fibre and alkali (Rakesh *et al*, 2011).

Inclusion of benzoyl group on to the fibre occur during the treatment, it decreases the hydrophilic nature of the treated fibre and improves interaction of the fibre with the hydrophobic polymer. It improves fibre-matrix adhesion resulting in increased strength of the composite, reduces water absorption and improves thermal stability (Chitta, 2010).



Scheme 2: Reaction between mercerized fibre and benzoyl chloride (Joseph, (2000) and Kalia, (2011))

Silane used as a coupling agent allows natural fibres to adhere to polymer matrix. It may reduce the number of cellulose hydroxyl groups in the fibre matrix, and can also be hydrolysed into silanol when it reacts with moisture on the fibre (Abdullah and Ahmad, 2012).



Scheme 3: Reaction between NaOH pre-treated fibre with silane coupling agent (Abdullah and Ahmad, 2012).

FT-IR Analysis

The FT-IR analysis was conducted to ascertain whether reaction has taken place between the cellulosic fibre and benzoyl chloride, and also phenyl triethoxy silane by revealing the optical film photomicrograph results. The functional groups present are shown in representative FT-IR spectra for untreated and alkali treated sisal fibre (Fig. 1a and 1b) below.

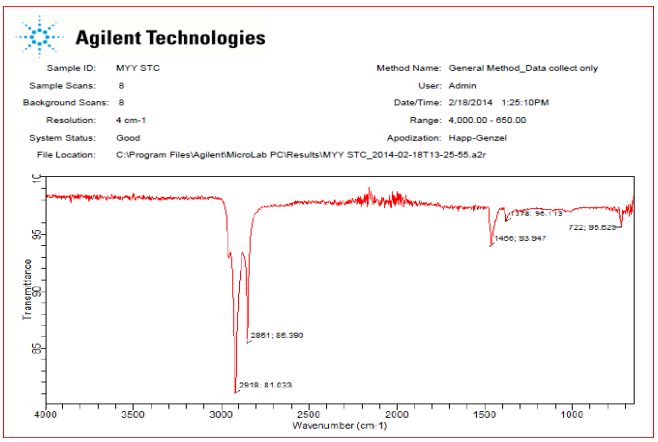


Fig. 1a: FTIR of Untreated Sisal Fibre

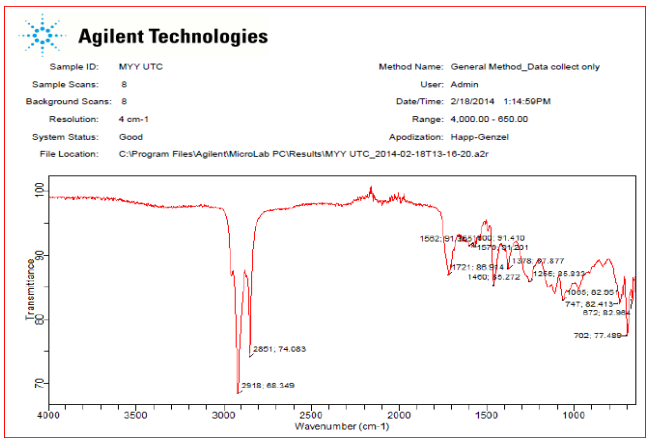


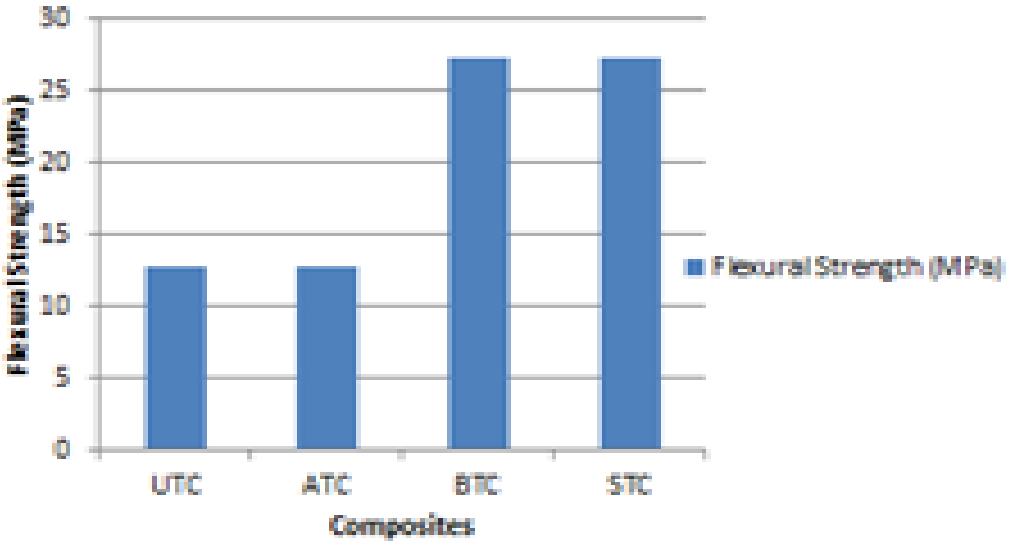
Fig 1b: FT-IR of Alkali Treated Sisal Fibre

The untreated fibre shows peak at 1255cm^{-1} corresponding to C-O stretching of primary alcohol. Important modifications were observed at peaks 1182cm^{-1} indicating C-O for stretching of primary alcohol in the alkali treated fibre, reduction in the peak compared to that of untreated fibre, indicating the removal of lignin and hemi cellulose from the fibre. Also the peak 1460cm^{-1} indicated C-C stretching in aromatic ring and also 1719cm^{-1} for C=O stretching of the benzoyl carbonyl group in the benzoylated fibre. The peak at 1488cm^{-1} is attributed to the stretching of C-C in the aromatic ring of the phenyl triethoxy silane used on the fibre. The results obtained are in accordance with the work of Rakesh *et al.* (2011) and Uma *et al.* (2012).

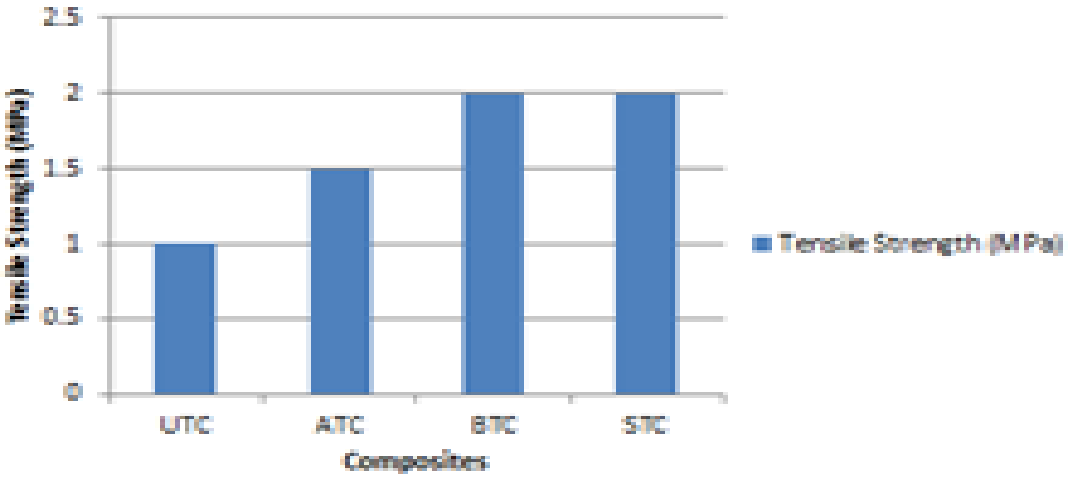
Mechanical properties of the composites

The tensile and flexural strengths of the sisal fibre composites are shown in Fig. 2(a and b). The tensile strength of the treated fibre composites is higher than the untreated composites. Fig. 2a showed that the silane treated composite demonstrated the highest tensile and flexural strength values while, the untreated fiber composites showed the least values. Meanwhile, the trend of the tensile strengths of the composites is similar to the flexural strength.

The untreated sisal fiber showed the least tensile and flexural properties. This might be due to weak compatibility between the fiber and the matrix because hemicellulose, lignin and pectin are present as revealed by SEM analyses.



a



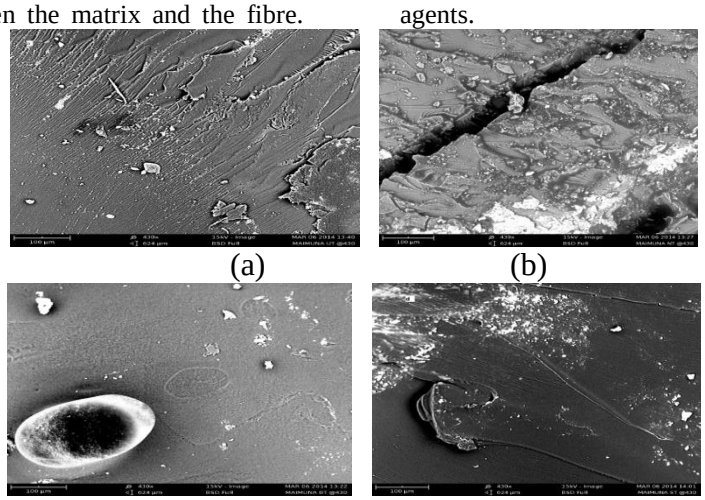
b

Fig. 2(a and b): Effects of Chemical Treatment on the Flexural and Tensile Strengths of the Composites

According to the figures the flexural and tensile properties changes to higher values in the alkali treated fibre composites, this could be attributed to the fact that mercerization process has increases the fibre-matrix interaction due to removal of some portion of lignin, hemicellulose and cellulose, thereby destructing the structure of the fibre filaments and also resulting in increased surface area available for contact with the matrix (Thimothy, 2007, Velmurugan 2012 and Wang, 2004). Silane and benzoyl treated fibre composites had the highest tensile and flexural strength. Silane used as a coupling agent allowed the natural fibres to adhere to the polymer matrix, it reduces the number of cellulose hydroxyl groups in the fibre, allowing its hydrolysis into silanol when it reacted with moisture on the fibre. The silanol formed is capable of condensing with adjacent silanol group (Si-O-Si), to form a cross-linked network of covalent bonds between the matrix and the fibre. Hence the fibre can adhere to the matrix and improve interfacial strength and benzoylation on the fibre leading to high fibre matrix interaction as well as increased mechanical properties (Chitta, 2010 and Uma, 2012). This explanation is supported by the SEM observation in Fig. 3 which shows fibre fibrillation in the fractured surface of the composites.

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopic analysis examined the surface morphology of the treated and untreated Sisal fiber. The impurities at the surface of the fibre play vital roles in fiber-matrix adhesion as they facilitate both mechanical interaction and the bonding reaction. Fig. 3 shows the SEM photomicrographs of fractured Sisal fiber composites before and after modification with NaOH, benzoyl chloride and silane coupling agents.



(a) (b)

(c) **(d)**

Fig.3. SEM Photomicrographs of the composites. (a) Untreated fiber (b) NaOH treated fibre (c) Benzoylated fibre (d) Silane treated fibre

Fig. 3 (a) shows the presence of wax, oil and surface impurities which provide a protective layer to the surface of the sisal fibres. Also surface roughness of the lignocelluloses of sisal fiber was observed indicating presence of lignin. Fig (3b) showed a rougher surface compared to Fig. 3a and this is more visible compared to the untreated Sisal fiber. The rough surface topography in the mercerized fiber was due to removal of hemicelluloses, lignin and amorphous waxy layer in Fig (3c), the surface of the benzoylated Sisal fibre becomes more smoother compared to that of untreated fibre and alkali treated fibre. These could be due to the fact that benzoylation increased fibre-matrix interaction resulting in better adhesion. Similar morphology was observed in Fig (3d) in the silane treated fibre. This could be due to the cross-linked network brought about by covalent bonding formed between the matrix and Silane treated fibre which promotes fibre-matrix interaction as well as increased interfacial strength. The treatments used provide clean surfaces on the fibers which result in direct bonding between the cellulose and the matrix. Comparing the photomicrographs of the treated and untreated Sisal fibers, it can be clearly seen that the morphology of the treated Sisal fiber resulted in separation of the micro-fibrillar of the structure due to delignification. The treatment also increased the effective surface area by fibrillation, thereby facilitating interfacial fiber-matrix adhesion. This result is in accordance with the work of Abdullah and Ahmad (2012), Rakesh *et al.* (2011) and Suradi *et al.* (2009) on SEM analyses of alkalized, benzoyl and silylated modified natural fibres.

The result obtained in this research will give the possibility of evaluating the composites for use in many domestic and industrial purposes. This will give room for a new market that produces more improved products and also prevent wastage of some important materials in our environment.

CONCLUSION

Alkali, benzoyl and silane treatments of Sisal fiber have modified the Sisal fiber surface and increased the tensile and flexural properties of the composites. The highest tensile and flexural properties were observed in benzoyl and silane treated fibre composites with values of 2MPa and 27.2MPa respectively compared to the untreated fiber composites which had values of 1MPa and 26.7MPa respectively. FT-IR analysis of the fiber showed inclusion of benzoyl and silane groups on the fiber as supported by the SEM photomicrograph observations.

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