# Unsaturated Polyester Resin Reinforced With Chemically Modified Natural Fibre

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**Abstract:** The effects of surface treatment on some mechanical properties of sisal fibre reinforced unsaturated polyester resin has been investigated. The sisal fibre was extracted by manually beating the sisal leaves with a smooth edged stick followed by chemical modification of the fibres using alkaline treatment method with NaOH at different concentrations and time at a constant temperature of 65 °C. The surface morphology and characteristics of the treated and untreated sisal fibre samples was studied using Scanning Electron Microscope (SEM). Polymer matrix composite (PMC) using treated and untreated sisal fibre as reinforcement and unsaturated polyester resin as matrix material was prepared. The composite laminates was cut into different sizes and shapes for mechanical testing. Universal testing machine (UTM) was used to test the flexural and tensile test, while Charpy Impact testing machine was used for the impact test. From the results of the mechanical tests, it was observed that, concentrations of 10% NaOH at (2 & 5 hours), 6 % NaOH at 5 hours and 2 % NaOH at 5 hours show great improvement of the mechanical performance for tensile, flexural and impact test respectively than the other various concentrations of NaOH at different time intervals. This shows that, the extent of surface modification depends on the concentration of NaOH solution and time of treatment. The changed fibre surface properties observed from SEM images of the treated sisal fibres were responsible for better adhesion.

Keywords: Sisal, Alkali, SEM, Unsaturated polyester, Composite,

# I. Introduction

In the past years, growing environmental pollution has called for the use of natural materials for different applications (Espert *et al.*, 2004). Government environmental policies have been implemented to force industries like the automotive, packaging and construction to search for environmentally friendly or biodegradable materials to substitute the traditional non-biodegradable composites (Chandra and Rustgi, 1998). The new environmental regulations and uncertainty about petroleum and timber resources have triggered much interest in developing composite materials from natural fibres. This interest in the natural fibres has resulted in a large number of modifications to bring it to the level or even superior to synthetic fibres. Due to such tremendous changes in the quality of natural fibres, they are fast emerging as a reinforcing material in composites (Girisha *et al.*, 2012).

Recently natural fibre reinforced polymer composites have experienced a significant growth in the composites industry. They are characterized by easy processability, good dimensional stability and adequate mechanical performance. The improved material performance primarily varies with the fibre matrix bond strength and choice of suitable processing parameters (Joseph *et al.*, 1999; Oksman *et al.*, 2003 Mohanty and Nayak, 2007). The quality of the fibre reinforced composites depends considerably on the interface, because only a well formed interface allows stress transfer from the matrix to the fibre (Wollerdorfer and Bader, 1998).

All Plant fibres are composed of cellulose and include bast, leaf, or hard fibres. Bast (flax, hemp, jute) and hard fibres (sisal, coir) are commonly used in composites and are available throughout the world. They may also represent an economic interest for the agricultural sector (Bledzki and Gassan, 1999; John and Thomas, 2008). The various advantages of natural fibres over man-made glass and carbon fibres are low cost, low density, comparable specific tensile properties, nonabrasive to the equipments, non-irritation to the skin, reduced energy consumption, less health risk, renewability, recyclability and biodegradability (Malkapuram *et al.*, 2008). These composites materials are suitable for aerospace, leisure, construction, sport, packaging and most especially in the automotive industries (Wambua *et al.*, 2003; Malkapuram *et al.*, 2008).

Among the various natural fibres, sisal fibre is one of the most commercially available useful natural fibres. Sisal fibre is a hard fibre extracted from the leaves of the sisal plant *Agave sisalana*. The sisal fibre contains cellulose, lignin, hemi-cellulose, waxes and ash depending on the origin and age of the fibre. Currently sisal is mainly used as ropes for the marine and agricultural industries. Other applications of sisal fibre include twines, cords, upholstery, padding and mat making, fishing nets, and fancy articles such as purses. Sisal fibre possesses a moderately high specific strength and stiffness, and can be used as a reinforcing material in polymeric resin matrices like polypropylene to make useful structural composite materials (Joseph *et al.*, 1999;

Mukhopadhyay and Srikanta, 2008). Sisal fibre originated from Mexico and is now mainly cultivated in East Africa, Brazil, Haiti, India and Indonesia (Nilsson, 1975; Mattoso *et al.*, 1997). It is second to manila in durability and strength (Weindling, 1947). It is one of the most extensively cultivated hard fibre in the world (Mukherjee and Satyanarayana, 1984).

Although these biodegradable or green composites are not as strong as the traditional glass fibre reinforce polymer, but the moderate mechanical properties are suitable for applications in non-durable consumer products and packaging materials, etc. Moreover, the hollow tubular structure of natural fibres reduces their bulk density. Therefore, composites made from them are expected to be lightweight. The main drawback of natural fibres is that their hydrophilic property lowers compatibility with the hydrophobic polymeric matrix during composite fabrications. As a result, the poor fibre-matrix adhesion engenders low mechanical properties.

Among the various chemical treatment tried, alkaline treatment is one of the least expensive and environment-friendly ways to improve the mechanical and interfacial properties of the natural fibres because it does not need any toxic organic chemicals (Pimeta *et al.*, 2008; Duhovic *et al.*, 2009). Alkaline (NaOH) treatment of natural fibres is one of the well known chemical treatments to increase the cellulose content through removing the hemicelluloses and the lignin (Jahn *et al.*, 2002). The introduction of polymer coatings on fibre surfaces helps to separate fibres from each other, eliminating the hydrogen bonding that holds them together. This approach also induces bond formation between the fibres and the matrix resulting in improved composite properties.

Incorporation of sisal fibre into thermosetting plastics has been reported by various workers (Paramasivam & Abdulkalam 1974; Pavithran *et al.*, 1987, 1988; Joseph *et al.*, 1996). Paramasivam & Abdulkalam (1974) have investigated the feasibility of developing polymer based composites using sisal fibres due to the low cost of production of composites and amenability of these fibres to winding, laminating and other fabrication processes. Satyanarayana *et al.*, (1984) have studied the mechanical properties of chopped sisal fibre – polyester composites. Chopped sisal fibre-polyester composites were prepared by the compression molding technique. It was found that the specific modulus of the composite was 1.90 compared with 2.71 for glass fibre reinforced plastics, while the specific strength was of the same order as that of polyester resins (34 - 41 MPa). The impact strength was 30 Jm<sup>-2</sup>, which is three times higher than that of polyester and 30% less than glass fibre reinforced plastics. Acceleration testing revealed little change in initial modulus, and reductions of 5% in ultimate tensile strength, 16% in flexural strength and 5.4% in water absorption.

Gupta *et al.*, (1998), have studied the nature of interfacial adhesion between chemically modified sisal fibre and polyester resin in composites.

It is apparent from open literature on polyester composites reinforced with treated sisal fibres that the ideal processing conditions to achieve best mechanical properties has not been established. Thus the aim of this work is to find the ideal concentration of NaOH and treatment time for the best mechanical properties.

#### II. Materials And Methods

Materials used include sisal leaves (obtained from Kaduna, Nigeria); NaOH, unsaturated polyester resin and hardener.

#### **Sisal Fibres Extraction**

The dried sisal leaves were crushed and beaten manually with a smooth edged stick until the fibres. After the extraction, the fibres were washed thoroughly in plenty of distilled water to remove surplus wastes and air-dried following the method adopted by Mukherjee and Satyanarayana 1984 and Chand *et al.*, 1988.

#### Morphology study

Scanning electron microscopic (SEM) photographs of untreated and alkali (NaOH) treated sisal fibres were obtained with a Phenom ProX SEM at room temperature.

#### Alkali treatment of sisal fibres

20 grams each of the extracted sisal fibres was soaked in 2, 6 and 10 % NaOH solution for 2, 3 and 5 hours each at a temperature of  $65^{\circ}$ C under constant stirring. The fibres were then rinsed with distilled water followed by neutralization in 2 % acetic acid solution to remove the residual NaOH solution. A final rinse in distilled water till the fibres was neutral to litmus paper and then dried in open air for 4 to 5 days. With this the moisture content in the fibres were completely removed. The treated sisal fibres were labelled and packed in air tight polyethylene bags to prevent dust and dirts from coming in contact with them.



Figure 1a: Sisal plant

1b: Treated and untreated sisal fibres

#### **Fabrication of Composites**

The unsaturated polyester resin was mixed with the methyl ethyl ketone peroxide (catalyst) and cobalt accelerator. The sisal fibres were cut to size, each weighing 0.3 g. Composites laminates were obtained by impregnating 0.3 g of each of the treated and untreated sisal fibre samples with polyester matrix in a mould using the hand lay-up method at room temperature to obtain 0.65 fibre volume fraction.

### **Tensile and Flexural Testing of Composites**

Tensile tests and 3-point flexural tests was conducted with a Universal testing machine (model 4202). Tensile tests were performed at a strain rate of 10 mm per min and guage length of 140 mm according to ASTM D638-06. Flexural testing was also carried out in accordance with ASTM D-790-08. A 3-point loading system was employed to determine the flexural strength of the various composites fabricated. The dimensions of the specimens in each case were 140 mm  $\times$  25 mm  $\times$  4 mm. The tensile strength at break was calculated from the equation below:

Tensile strength = 
$$\frac{Breaking force (N)}{Original Crossectional Area (m^2)}$$

The flexural strength (F.S.) and flexural modulus of the composite specimens were determined using the following equations.

Flexural Strength = 
$$\frac{3PL}{2bt^2}$$
  
Flexural Modulus =  $\frac{PL^3}{4bt^3w}$ 

Where, L is the span length of the sample. P is the load applied; b and t are the width and thickness of the specimen respectively, w is the deflection.

Tensile strength indicates the ability of a composite material to withstand forces that pull it apart as well as the capacity of the material to stretch prior to failure.

# Impact Strength Test

Charpy impact test was conducted according to ASTM E 23. The dimension of the specimens was 100 mm× 15mm× 4 mm. At least three replicate specimens were tested and the results were presented as an average of tested specimens.

# III. Results And Discussion

The natural fibre reinforced composite of sisal fibres was developed and tested for mechanical properties. This was further compared with the plain unsaturated polyester matrix. The discussions of the results are presented below.

#### Morphological Assessment

The treated sisal fibres were observed under SEM for assessing the changes in the fibres. Untreated and treated fibre surface morphologies are shown in Plates A, B & C.









Plate C (10 % NaOH treated sisal fibre)

The SEM photomicrograph of the surface of untreated sisal fibres in (plate A), present a network structure in which the fibrils are bound together and wrapped with some cementing substances. These substances are the residual pectin, lignin, hemicelluloses, e.t.c. From plate B (6% NaOH) and C (10 % NaOH), the micrographs clearly show that the alkali treatment removed the cementing substances. The removal of pectin, oils, waxes and other extractives from the sisal fibre is responsible for producing the rough surface topography which offers better fibre/matrix interface mechanical adhesion and improved mechanical properties. After the alkali treatment, fatty deposits were removed which reveals empty cavities on the fibre surface. Also, it can be observed that in some part of plate B, there is an increase in fibrillation which seems to appear as cracks on the fibre surface.

#### **Tensile Properties**

The mechanical property of the composite relies on the type of fibre and matrix used. This further depends on the quantity of the fibre content, the increase in fibre content leads to improved tensile properties. The tensile test results of the treated and untreated sizal fibres composites are shown graphically in Figure 2:



USP - plain unsaturated polyester matrix (control) USF - untreated sisal fibre composite



Figure 2: Tensile strength determination at varying NaOH concentrations

USP - plain unsaturated polyester matrix (control)

USF - untreated sisal fibre composite

# Figure 3: Tensile modulus determination at varying NaOH concentrations

It was observed that the tensile properties of the sisal fibre reinforced composites improved with the alkali treated sisal fibres. From figure 2, maximum tensile strength of 7.0 Mpa was found for 10% NaOH at 5 hours, followed by 10% NaOH at 2 hours and 6% NaOH at 5 hours with tensile strength of 5.5 and 5.3 Mpa respectively. The plain unsaturated polyester (control) has the lowest tensile strength of 2.0 Mpa while the untreated sisal fibre composite has tensile strength of 3.0 Mpa. It may be observed that 2 % NaOH treatment results in 60 % increase in tensile strength over the samples reinforced with untreated fibres, while the 6 % NaOH treated sample showed a further 10 % increase in tensile strength. 10 % NaOH treatment for 5 hours resulted in an average tensile strength increase of 28 % relative to the 6 % NaOH treated samples. Further increase in NaOH concentration and time of treatment beyond 5 hours led to significant decrease in tensile strength, thus establishing that 10 % NaOH concentration and 5 hours of treatment is the ideal condition for maximum tensile strength. It is probable that beyond this point, cleavage of cellulose molecules of the sisal fibre sets in. For the tensile modulus (figure 3), it is evident from the graph above that there was increase in the tensile modulus of all the treated sisal fibres reinforced polyester composites. A maximum tensile modulus of 55 Mpa was observed at 10 % NaOH at 5 hours, followed by 10 % NaOH at 3 hours and 2 % NaOH at 5 hours with tensile modulus of 54 and 50 respectively. The plain unsaturated polyester has the lowest tensile modulus of 18 Mpa.

It is evidenced from the graph that the concentration and time greatly affected the tensile properties and performance of sisal fibre reinforced composites. Therefore this indicates that the surface treatment of the fibres significantly increased the adhesion characteristics and thereby improving mechanical properties. In general, the mechanical performance of a fibre composite basically depends on the strength and toughness of the matrix, and efficiency of interfacial stress transfer. Similar improvement or increase in the tensile properties of alkali treated natural fibre reinforced composites were reported by Rout *et al.*, (1999), Ray *et al.*, (2001), Lai *et al.*, (2008).

#### **Flexural Properties**

The flexural properties of sisal fibre reinforced composites are shown above in figure 4 and 5, according to the results, the introduction of sisal fibres significantly improved the flexural strength compared to the plain unsaturated polyester matrix.



USP – plain unsaturated polyester matrix (control) USF – untreated sisal fibre composite





USP – plain unsaturated polyester matrix (control) USF – untreated sisal fibre composite

Figure 5: Flexural Modulus of Composites at different concentrations of NaOH and time.

From the graphs, the maximum results were observed at 125 Mpa and 11 Gpa of 10 % NaOH at 5 hours for both the flexural strength and modulus respectively. This was due to improving the bonding between the fibre and the matrix. It may be observed that, the trends of the flexural properties are similar with the trends of tensile strength and modulus. Similar results were obtained by (Jacob et al. 2004) in case of sisal/oil palm

hybrid fibre reinforced natural rubber composites. Bledzki and Gassan (1999) and Cao et al. (2006) who also made similar observations for natural fibre composites explained that the alkali treatment of natural fibre resulted in higher modulus values due to increased cross linking and formation of a strong fibre/matrix interface. They explained further that the fibres in the untreated fibres were packed together but got split after the treatments. This fibrillation breaks the untreated fibre bundle down into smaller ones by dissolution of the hemicelluloses. The fibrillation increases the effective surface area available for contact with the matrix, and hence the interfacial adhesion was improved.

#### Impact Test

On a general note, the impact strength of the unsaturated polyester was improved by about 50 % when reinforced with the sisal fibres as shown in the figure 6. It is also observed that there is an increase in the impact strength of the composites reinforced with the treated fibres as compared with the untreated fibres, showing the positive effect of the alkali treatment on impact strength of composite samples.



USP – plain unsaturated polyester matrix (control) USF – untreated sisal fibre composite

# Figure 6: Effects of Alkali treatment on impact strength of composite at different concentration of NaOH and time

From figure 6, it was observed that the impact strength of sisal fibre reinforced composite has a maximum value of 1.35j at 10% NaOH 5 hours, followed by 10% NaOH at 3 hours, 2% NaOH at 5 hours and 6% NaOH at 3 hours of 1.0j and 0.8j respectively. The plain unsaturated matrix has the lowest impact value of 0.4j. This result shows significant improvement on the impact strength of the surface treated sisal fibre reinforced composite compared to the untreated samples.

#### IV. Conclusion

It was observed that the surface of the sisal fibres before treatment was filled with hemicelluloses, waxes, lignin, pectin, impurities e.t.c. covered up by cementing materials. However, NaOH solution treatment at various percentage concentrations (2%, 6%, and 10%), and at different timing (2 hours, 3 hours and 5 hours) at  $65 \, ^{\circ}C$  temperature carried out on the sisal fibres, removed the adhesives and impurities constituents in the fibres according to the degree of modification. The results of the investigation of the effect of surface treatment on the mechanical properties of sisal fibre reinforced composites revealed that the chemical treatment actually enhanced the mechanical properties; the observed enhancement was due to the stronger bond that exists between the treated fibre and the polymer matrix. The treatment removed the lignin and hemicelluloses which acts as obstructions being a matrix in the natural fibres.

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