

Physical and Mechanical Properties of Alkali Modified Nigerian Sisal Fibres

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ABSTRACT: The effects of chemical treatment on the surface characteristics of sisal fibres and their physical/mechanical properties have 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 intervals and at a constant temperature of 65 oC. The surface morphology and physical and mechanical characteristics of the treated and untreated sisal fibre samples was studied. The Scanning Electron Microscope (SEM) micrographs revealed the rough surface topography of the sisal fibres caused by alkali modification which increased with increased concentration. Furthermore it was observed that fibre tenacity increases with increase in NaOH concentration. However, beyond 20 % NaOH concentration, decrease in tenacity is observed. Fibre breaking extension increases with increase in NaOH concentration. Moisture regain decreases with increase in NaOH concentration.

Keywords: Sisal fibre, SEM, Tenacity, Moisture regain, Sodium Hydroxide

1 Introduction

Natural fibres are prospective reinforcing materials and their use until now has been more traditional than technical. Due to the relatively high cost of synthetic fibres such as, glass, plastic, carbon and Kevlar that are being used in fibre reinforced composites, and the health hazards of asbestos fibres, it has become necessary to explore natural fibres. Natural fibres are produced from renewable resources that are biodegradable and relatively inexpensive when compared to the traditionally used synthetic fibres. Natural fibres like banana, sisal, hemp and flax, jute, and coconut have attracted

scientists and technologists for applications in consumer goods, low-cost housing and other civil structures “[1]”, “[2]”.

However, the presence of surface impurities and the large amount of hydroxyl groups makes plant fibres less attractive for reinforcement of polymeric materials. The attractive features of these fibres are light weight, non-toxicity, friendly processing and absorption of CO₂ during their growth “[3]”. With the increase of environmental protection consciousness, natural fibres as a group of environmental friendly reinforcements are in considerable demand in the production composites “[4]”.

Sisal fibre is a hard fibre extracted from the leaves of sisal plant (*Agave sisalana*). Though native to tropical and subtropical North and South America, sisal plants are widely grown in tropical countries of Africa, the West Indies and the Far East. Though sisal fibre is one of the most widely used natural fibres, a large quantity of this economic and renewable resource is still under-utilized.

Sisal fibre is a lignocellulosic in nature; the presence of a large amount of hydroxyl groups is responsible for the hydrophilic nature of sisal fibre. Several fibre surface treatment methods have been studied to improve the adhesion properties between sisal fibres and the surrounding matrix. The chemical modification of natural fibres improves the adhesion of fibre-matrix interfaces in composites, which in turn improves the mechanical properties of the composites. In certain cases it was found to bring about changes in the morphology, chemical groups and hydrophilicity of the fibres “[5]”.



Figure 1: Sisal plant Figure 2: Sisal fibres

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 “[6]”, “[7]”. Chemical modification has been known to enhance interface between the hydrophilic sisal fibre and hydrophobic polymer matrices when both are combined to produce fibre reinforced composites. Both mechanical and moisture absorption resistance properties can be improved. Alkali treatment of natural fibres is the common method to produce high-quality fibres. Alkali treatment can remove natural and artificial constituents such as hemicelluloses, lignin, pectin, wax and oil covering materials “[8]”. In addition, alkali treatment reduces the level of fibre aggregation which leads to fibre fibrillation, i.e. breaking down the fibre bundle into

smaller fibrils. This increases the fibre aspect ratio caused by reduced fibre diameter and produces a rough surface topography. Several workers have performed work on alkali treatment “[9]”, “[10]” and reported that alkalization leads to increase in the amount of amorphous cellulose at the cost of crystalline cellulose and the removal of hydrogen bonding in the network structure. The important modification occurring here is the removal of hydrogen bonding in the network structure.

“Mwaikambo and Ansell [11] carried out the alkali treatment of sisal fibres and analyzed the changes with respect to diameter and internal structure, such as cellulose content, crystallinity index, and micro-fibril angle. Alkalization was found to change the internal structure of sisal fibres which exhibited approximately same specific stiffness as that of steel. The crystalline nature of the treated fibres was also found to increase due to alkali treatment. Their results indicated that the structure of sisal fibre can be chemically modified to attain properties that will make the fibre useful as a replacement for synthetic fibres.”

It is apparent from open literature on alkali treatment of sisal fibres that the ideal processing conditions to achieve best mechanical properties and improved hydrophobic nature of sisal fibres has not been established. Thus the aim of this work is to find the ideal concentration of NaOH and treatment time for best fibre tenacity and improved hydrophobic character of sisal fibre.

2 Materials and Methods

2.1 Materials

Materials used include sisal leaves (obtained from Kaduna, Nigeria); NaOH, acetic acid and basic laboratory glass wares.

2.2 Method

2.2.1 Extraction of the Sisal Fibres

The sisal leaves were crushed and beaten manually with a smooth edged stick to get the fibres. After

the extraction, the fibres were washed thoroughly in plenty of distilled water to remove surplus wastes such as chlorophyll, leaf juices and adhesive solids (hemicelluloses) and air-dried following the method adopted in northern Nigeria by local rope makers.

2.2.2 Preparation of 2, 6, 10 & 20 %

NaOH

2, 6, 10 & 20 % NaOH solutions were prepared separately by weighing out 20, 60, 100 and 200g respectively of sodium hydroxide pellets and dissolving each in separated 1000 cm³ of distilled water in volumetric flasks,

under constant stirring to obtain a homogeneous solution.

2.2.3 Surface Treatment of Fibres

20 grams of the extracted sisal fibres were weighed and soaked in 2, 6, 10 and 20 % NaOH solutions for 2, 3 and 5 hours each at a temperature of 65 °C under constant stirring. The fibres absorbed the NaOH solution; the NaOH solution penetrates and reacts with the surface of the sisal fibres to remove the impurities and cementing substances. 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. The treated sisal fibres were labeled and packed in air tight bags to prevent dust and dirt from coming in contact with them.



Plate 1: treated and untreated sisal fibres

2.2.4 Morphology Study

Scanning electron microscopic (SEM) photomicrographs of untreated and alkali

(NaOH) treated sisal fibres were obtained with a Phenom Pro X SEM at room temperature



Plate 2: Scanning Electron Microscope

2.2.4 Moisture Regain Test

The sisal fibres were weighed and dried in an oven at a temperature of 103⁰C for 30 minutes, followed by cooling for 30 minutes and then weighed again. This step was repeated until the weight was constant. The

moisture regain was determined using the formula:

$$\text{Moisture Regain (R)} = \frac{M}{1 - [M/100]}$$

Where:

$$M = \text{Moisture content} = \frac{w_0 - w_1}{w_0} \times 100 (\%)$$

w₀ = weight of fibre before drying

w₁ = Weight of fibre after drying

2.2.4 Tensile Test

The tensile properties of the treated and untreated sisal fibres were determined according to ASTM D3822. The following tensile properties were determined using the Instron tensile tester; tenacity and breaking extension.

3 Results and Discussion

In this experimental work, the sisal fibres were treated with NaOH at different concentrations and time intervals.

3.1 Alkali Modification of Sisal Fibre

After the alkaline treatment process of sisal fibres, initial optical microscopic examination of the fibres showed increase in the fibre diameter. It is probable that the sisal fibre cell walls absorb the NaOH solution, thus enabling the relative large sodium ions to penetrate the crystalline regions; this resulted in fibre shrinkage and subsequent decrease in fibre length. The effect of alkali on sisal fibres is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. Native cellulose shows a monocyclic crystalline lattice of cellulose I, which can be changed into different polymeric forms through chemical treatments. Increase in the concentration of alkali will influence will the degree of swelling and hence the degree of lattice formation into cellulose II “[12]”. “Studies [13] have shown that Na⁺ has got a favorable diameter, able to widen the smallest pores in between the lattice planes and penetrates into them. Consequently, NaOH treatment results to higher degree of swelling”.

After alkali modification, it was also observed that there was colour change from the original white colour of the untreated fibre to a creamy colour. The creamy colour shade deepens with severity of treatment, i.e. as time of treatment and concentration of NaOH increased, the cream colour deepens. This can be attributed to the fact that the NaOH solution has acted on the sisal fibre altering its internal structure and morphology which reflected in the change of colour. Another observation was on the handle of the fibre, the alkali treated sisal fibres were softer and more extensible depending on the concentration of NaOH while the untreated sisal fibres were hard and not extensible. This may be attributed to the fact that alkali treatment removed the residual cementing constituents (lignin, pectin, hemicelluloses and waxy substances), causing the fibre to be soft and more extensible.

3.2 Fibre Surface Characteristics (SEM Analysis)

For an indepth study of the surface morphology, scanning electron

microscopic studies of some of the sisal fibres were carried out. The SEM micrographs obtained are displayed on plates A to D, all exposed to 5 hours caustic soda treatment.

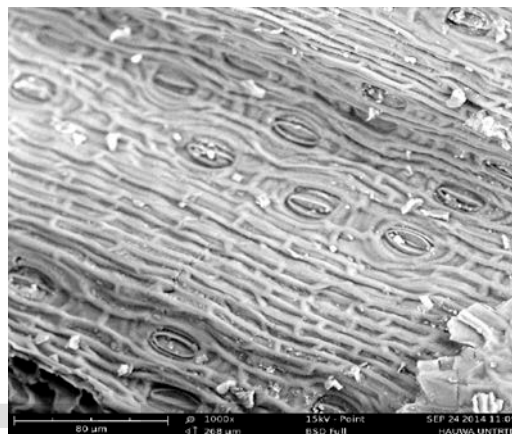


Plate A: Untreated sisal fibres

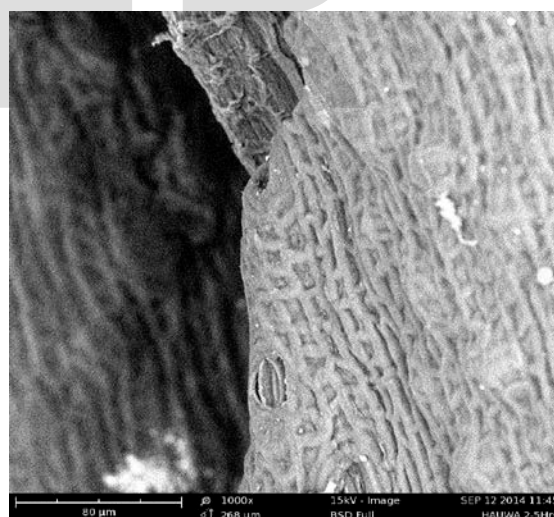


Plate B: Treated sisal fibres 2% NaOH at 5 hours

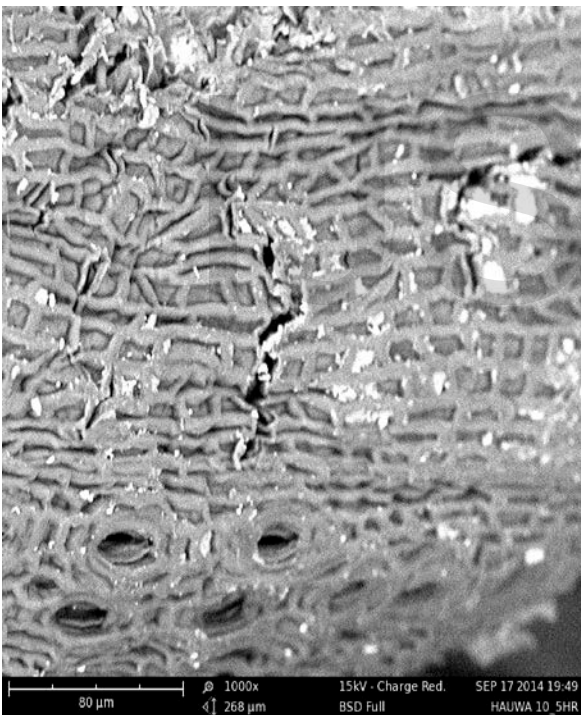
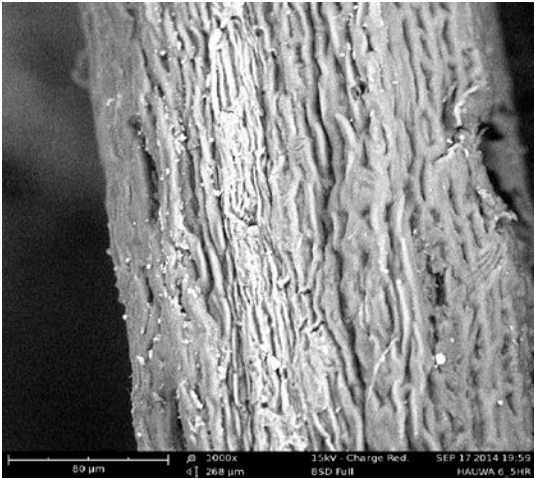


Plate D: Treated sisal fibres 10% NaOH at 5 hours

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, hemi

cellul

Plate C: Treated sisal fibres 6% NaOH at 5 hours

oses, etc. From plate B to plate D, 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. The rough surface is made up of pits, which are circular holes of about 1 to 5 μm^2 in diameter. Also, it can be observed that in some part of plate D, there is an increase in fibrillation which seems to appear as cracks on the fibre surface.

In general pits are hidden on the surface of untreated fibres due to superficial layer of extractives, oils and waxes. However, with layer removal by alkali treatment the pits are revealed. The presence of pits and globular marks on the surface of treated sisal fibres is important for the increase in effective surface area available for contact with the matrix. This also increases the surface roughness and consequently

improves the mechanical bonding with the polymer matrix.

3.3 Moisture Regain

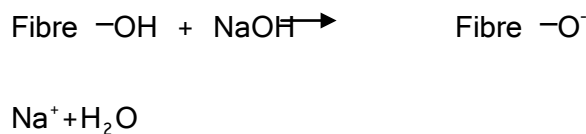
The effect of varying the concentration of alkali on moisture content was determined for the treated and untreated fibre samples. Ten were carried out for each sample and the mean values obtained.

Table 1 Average Tenacity, Breaking extension and Moisture regain of Sisal

NaOH concentration (%)	Tenacity (cN/Tenax)	Breaking extension (%)	Moisture content (%)
0 (untreated)	12.22	6.11	8.58
2	15.15	7.16	8.29
6	16.23	8.21	7.29
10	16.11	9.27	7.49
20	15.12	10.82	6.81

Table 1 shows that as concentration of NaOH increases, there is an almost corresponding decrease in moisture regain, with the untreated samples showing the highest moisture regain value. This may be due to the fact that as NaOH concentration increases, OH groups in the cellulose chain are replaced by the sodium ion (Na^+) thereby

reducing the site for water molecules to anchor on the cellulose chain.



3.4 Determination of Tensile Properties

3.4.1 Tenacity

Table 1 gives result of tenacity at different concentrations. NaOH treated fibres showed increase in tenacity compared to the unmodified ones; the higher the alkali concentration, the higher the fibre tenacities up to 10 % concentration of NaOH. This may be due to the increase in strength caused by the treatment which improves the adhesive characteristics of fibre surface by removal waxy substances from the fibre surface thereby producing a rough surface topography. In addition, alkali modification leads to fibre fibrillation, i.e. braking down of fibre bundles into smaller fibrils and consequently increases in the fibre tenacity. Fibrillation increases the proportion of effective surface area available for contact with adjacent fibrils. However at 20 % NaOH concentration, a drop in tenacity is observed; this is likely a result of cellulose

chain cleavage due to high concentration of the NaOH.

In addition the NaOH treatment of the sisal fibres led to the breaking down of the fibre bundle into small fibres. This increased the effective surface area available for wetting by the matrix resin, also increasing the fibre aspect ratio which was caused by reduce fibre diameter producing a rough surface topography which offers better fibre/ matrix interface adhesion and increase in mechanical properties.

3.4.2 Breaking extension

Table 1 shows that as NaOH concentration increases, there is a corresponding increase in extension of the sisal fibre i.e. modified sisal fibres have higher breaking extension than the untreated ones. This is expected since untreated fibres have high content of lignin, hemicelluloses, etc. which stiffen the sisal fibre. On removal of these stiffening substances, the fibre becomes more extensible. Furthermore, as concentration of the NaOH increases, the quantity of these substances removed increases correspondingly. Increase in concentration of NaOH beyond 20% is likely to result in cleavage of the cellulose chains, hence the NaOH concentration did not exceed 20%.

Nigeria for allowing the use of the equipment to carry out SEM test.

4 Conclusions

It was observed that the surface of the sisal fibres before treatment was filled with hemicelluloses, waxes, lignin, pectin, impurities e.t.c. i.e. covered up by cementing materials. However, NaOH solution treatment at various percentage concentrations (2%, 6%, 10% and 20%), and at different timing (2 hours, 3 hours and 5 hours) at 65 °C temperature carried out on the sisal fibres, removed the adhesives and impurities constituents in the fibres according to the degree of modification thereby leading to improvement in tenacity and extension at break of the fibre. However at NaOH concentrations of 20%, a decrease in tenacity sets in, due probable to fibre cellulose chain damage.

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