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# The effect of moisture imbibition on cellulosic bast fibres as industrial raw materials

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The moisture imbibition on four *Cellulosic bast* fibres, Roselle (*Hibiscus Sabdariffa*), Okra (*Hibiscus esculentus*), Baobab (*Adansonia digitata*) and Kenaf (*Hibiscus Cannabinus*), were investigated. The fibres were chemically purified by retting, scouring, bleaching and mercerizing. Thereafter, moisture sorption method was adopted. The moisture imbibition ranking of the fibres is in the order. The materials were treated with 10, 15, 20 and 25% NaOH. The moisture imbibition for the treated materials were as follows: Roselle 20 > 25 > 10 > 15%; Okra 20 > 15 > 25 > 10%; Baobab 15 > 20 > 25 > 10% and Kenaf 20 > 15 > 10 > 25%. These results suggest that the aforementioned fibres are viable industrial raw materials and have been a best alternative to inorganic/mineral - based reinforcing fibres.

Key words: Moisture imbibition, bast fibres, moisture sorption.

# INTRODUCTION

Some natural fibres from plants have not been characterrized to evaluate their potential as industrial raw materials. For Cellulosic bast fibres to be useful as textile raw materials, the fibres should have the potential for use as reinforcing fillers in thermoplastics (Francois and Donovan, 1992). In addition, it should have some secon-dary properties like moisture regain and moisture absorp-tion as a desirable character to the end product (Harry, 1995).

Absorbency or moisture regain has been defined as the percentage of moisture a bone-dry fibre will absorb from the air under standard conditions of temperature and pressure at 25°C and 65% relative humidity (Sadov and Korchangin, 1978). Previous studies (Ajayi et al., 2000), have shown that the presence of hydroxyl groups and amorphous areas in the cellulosic structure gives the necessary properties of moisture absorption and dyeability.

The products of natural fibres are now rapidly becoming one of the fastest-growing additives for thermoplastics as indicated by the increased use of natural fibres as reinforcements (Cowie, 1991). The most important sources of cellulose are cotton fibre; cotton linters (very short

fibres adhering to the cotton seed after removal of the long fibres). Also listed in the category are hard and soft wood, hemp, flax, straw and bagasse. Cotton fibre is widely used for textile materials, while its properties can be improved considerably by chemical treatment. For example, removal of fat and wax impurities from the fibre surface, this as a result makes the cotton capable of absorbing water readily (Billmeyer, 1984). Similarly, absorbent cotton is also made for medical uses. Thus, cellulose treated with mineral acids removes mineral matter and make it practically a non-conductor of electricity. Outer coating of flexible electrical wires is made of such cotton. Mercerization with 10 - 25% aqueous caustic soda under tension, reorient the fibre, the ribbon like fibre of cotton in a yarn or fabric undergo swelling and become almost cylindrical assuming a silky luster. In this method of treatment, the tensile strength of fibres increase by about 25% (Sharma, 2006)

The primary advantages of natural fibres have been recognized, viz, low densities, low cost, and highest tilling levels possible. In addition they have low energy consumption, and indeed wide variety of fibres is locally available.

The main disadvantage of using natural fibres in thermoplastics is the high moisture absorption of the fibres and composites (Heckert, 1953). The moisture absorbed by the composite and the corresponding dimensional

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changes can be reduced dramatically if the fibres are thoroughly encapsulated in the plastic and there is good adhesion between the fibre and the matrix (Bhatnagar, 2004). If necessary, moisture absorption of the fibres can be significantly reduced by the acetylating of the hydroxyl groups present in the fibre (Rowell et al., 2000). Moreover, selecting applications where the high moisture absorption is not a major drawback can minimize the disadvantage of the high moisture absorption of the composite. For example, polyamide and its composite absorb water, but their applications are such that this deficiency is not of prime importance (Webber, 1993).

On the whole, cellulosic bast fibres are often used in the form of a blend in order to improve or modify properties such as moisture absorption (Attah, 1995). Bast fibres were extracted from the plants Kenaf (*H. cannabinus*), Okro (*H. esculentus*), Baobab (*A. digitata*) and Roselle (*H. sabdariffa*). Filament length longer than 1 m is common. These filaments consist of discrete individuals' fibres, generally 2 - 6 mm long, which are themselves composites of predominantly cellulose, lignins and hemi -celluloses.

The aforementioned fibres were chosen for the study because it is now the fiber crops grown locally and commercially in Yola, Adamawa State and Nigeria. Retting involves the use of ammonium oxalate (15% solution) in the dissolution of binding material. The boiling of cellulosic fibres with 15% solution of ammonium oxalate for 30 min at 100°C which results in dissolution of non-cellulosic materials which originally binds the bundles together.

Hydrogen peroxide is useful for bleaching cellulose materials, because the oxidative damage to the material is usually minimal. In the present investigation, hydrogen peroxide (5%) was used. It has been suggested that the bleaching action is as follows (Trotman, 1970):

$$H_2O_2 \xrightarrow{\text{NaOH}} H^+ + HO_2^{-}$$

$$HO_2 \xrightarrow{\text{OH}^+} OH^- + [O]$$

$$[O] + I \xrightarrow{\text{IO}^-} IO$$

Where I = the impurities

In an alkaline condition, the hydroxyl ions (OH<sup>-</sup>) produced by the perhydroxyl ion  $HO_2^-$ , hydrolyses the hydrogen ions (H<sup>+</sup>), thereby promoting the liberation of more perhydroxyl ions.

 $H^+(aq) + OH^-(aq) \rightarrow H_2O(I)$ 

Care must be taken in the production of perhydroxyl ions, since it may decompose into hydroxyl ions and atomic oxygen. The atomic oxygen is responsible for bleaching. If atomic oxygen is produced in excess, this may dissociate to molecular oxygen.

$$[O] + [O] \longrightarrow O_2(g)$$

Oxidation of cellulose as a result of this reaction may lead to depolarization of cellulose with a consequent reduction in tensile strength.

Scouring is the process that removes all undesirable impurities, which include pectins, waxes, gums fats and oils. During this treatment, natural impurities and adventitious dirt were removed by saponification. The scoured sample appeared soft, smooth, slightly thicker and brighter. It has been established (Sadov and Korchangin, 1978) that during alkali treatment, fatty acids are converted to soap which helps to emulsify other wax-like substances. Caustic soda hydrolyses protein and the molecules are broken along peptide links with the formation of alkali soluble amino acids. Similarly, pectins are hydrolyzed, gradually destroyed or decomposed to form methyl alcohol and glacturonic acid (Sadov and Korchangin, 1978). Pentoses are hydrolysed to pentosans, which in turn contain aldehyde groups. This together with the size starch decomposition impacts a reducing property to the boiling liquor.

Mercerization involves the treatment of fibre with 10 - 25% solutions of Sodium hydroxide for 20 min at 5°C.

In this study, the moisture imbibition of four natural bast fibres as industrial raw materials for plastic composites was investigated. The effects of moisture absorption and equilibrium moisture loss on cellulosic bast fibres were determined.

# MATERIALS AND METHODS

# Materials

# Sample collection

The samples were collected in Yola Town of Adamawa State, Nigeria. The fibres were removed mechanically and shade dried for 24 h. The vegetable fibres whole stalks were air -dried and the blast fibres were hand separated to 99% purity. The apparatus used is Mettler Toledo measuring balance model AB 204UK. The chemicals used were of Analar Standard

Cellulosic fibre samples of Kenaf, (*H. cannabinus*) Okro (*H. esculentus*), Baobab, (*A. digitata*) and Roselle, Hibiscus (*Sabdariffa*) were processed, by the retting scouring, bleaching and mercerizing procedures described by (Ajayi et al., 2000; Eromosele et al., 1999).

# Retting

Raw fibre (2 g) was weighed and immersed in 15% solution of Ammonium Oxalate (200 ml). In order to prevent oxidation, it was ensured that the fibres were not exposed to air during the treatment. The materials were rinsed in fast-flowing tap water for 5 min, followed by drying in an oven at  $50^{\circ}$ C for 20 min.

#### Scouring

The bast fibre (2 g) was completely immersed in 2% solution of Sodium hydroxide and heated at  $100^{\circ}$ C for 45 min. Care was taken to ensure that materials were not exposed to air to prevent oxidation. The materials were rinsed severally in an over - flowing tap water, followed by drying in an oven at 50°C for 20 min.



Figure 1. Effects of mercerization on moisture inbibition of *Hisbicus* esculentus (okro)



The materials were boiled in the liquor for 45 min while pH was maintained between 10 - 10.8.

The materials were rinsed in tap water for 10 min, neutralized with 5% glacial acetic acid and dried at room temperature.

#### Mercerization

The experiment was carried out in an ice bath followed by occasional turning with a glass rod to ensure an even treatment. The samples were rinsed in tap water, for 5 min and neutralized with 5% acetic acid.

#### Determination of fibre density

The determination of the density of all the fibre samples was carried out using standard method as described in a standard hand book (TAPPI, 1980). The specimens were conditioned for 24 h at 65% relative humidity and 25°C before carrying the density test. 2 g the samples were accurately weight out for each fibre type. Each weight was immersed in toluene, in a calberated glass tube, and the value of toluene displaced determined. It was as equally to the volume of fibre immersed. Knowing the mass of fibre, the density was calculated from:

Density = Mass/Volume

Toluene was used because it was not absorbed into the fibre structure

#### Determination of moisture imbibition of fibres

A known quantity of the sample (1 g) was weighed and socked in distilled water (100 ml) for 20 min. The excess water was carefully



Figure 2. Effects of mecerization on moisture inbibition of *Hibiscus cannabinus* (Kenaf)

mopped with filter paper and the weight of the wet sample was taken. Subsequently, the weights after 30 min time interval under laboratory condition were also recorded until a constant weight was obtained and the mass of moisture loss against time was determined.

### **RESULTS AND DISCUSSION**

The results of the various analyses are summarized in Table 1 and presented for the individual materials in Figures 1 - 4. There was a significant increase in moisture uptake of the fibres with various concentration of NaOH. The result was consistent with varying amount of NaOH used in the treatment of the fibre and the time taken. Materials treated with 20% NaOH concentration were the most compact, followed by materials treated with 15, 25, and 10% NaOH. The 25% concentration was the exception otherwise, the rigidity of fibre increased as the concentration increased. This might be due to the fibre shrinkage below 50 min of dehydration. The moisture content of H. esculentus at 10 and 15% NaOH concentration was greater than materials treated with 25 and 20% concentrations of NaOH. Between 50 to 175 min, the rate of imbibition of samples treated with 10% NaOH was greater than 25 and 10% NaOH concentration. The rate of moisture desorption of fibres after 175 min was such that 10% NaOH and 25% NaOH treated samples occurred at the same rate, but did at a faster rate for the samples treated with 15% NaOH and 20% of NaOH concentrations respectively.

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Table 1. Moisture inbibition values of the various fibres.

	Kenaf (hibiscus cannabinus)				Okro (Hibiscus esculentus)				Baobab ( Adansonia digitata)				Roselle (Hibiscus Sabdariffa)			
NaOH% / Properties	10	15	20	25	10	15	20	25	10	15	20	25	10	15	20	25
Moisture absorption (g)	2.62	2.58	2.67	2.56	4.06	4.05	2.64	2.96	2.79	3.02	2.93	2.89	2.12	2.05	2.25	2.18
Equilibrium Moisture Loss(g)	1.22	1.39	1.60	1.49	1.15	1.13	1.04	1.27	1.47	1.76	1.44	1.42	1.54	1.54	1.63	1.49
Density (g / cm- <sup>3</sup> )	1.36	1.31	1.28	1.20	1.25	1.23	1.20	1.19	1.40	1.29	1.27	1.19	1.45	1.41	1.38	1.32



Time (mins)

Figure 3. Effects of mercerization on moisture inbibition of Adansonia digitata



Figure 4. Effects of mercerization on moisture inbibition of Hibiscus subdariffa

Notably, at 350 min, equilibrium was reached for materials treated with 10% NaOH, this Sample also had the highest moisture uptake. The effect could be due to the removal of surface materials opening up the fibre with more scooped individual cells (Ajayi et al., 2000) The removal of surface impurities on plant fibres is advantageous for fibre-matrix adhesion as it facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to chemicals such as resins and dyes (Eromosele et al., 1999)

The effect of mercerization on moisture imbibition and desorption characteristic of *H. cannabinus* (Kenaf) fibre are shown in Figure 2. Between 30 and 50 min, the fibre treated with 20% NaOH had the highest moisture uptake, followed by that treated with 15% NaOH then 10% NaOH and the lowest moisture uptake was observed for samples treated with 25% NaOH.

Before desorption times of 50 to 110 min the rate of moisture uptake was highest for samples treated with 20% NaOH then 15, 10 and 25% NaOH in that order. The desorption curve Figure 2 also followed the same trend. At 300 to 350 min, equilibrium desorption of moisture was attained for Kenaf. The highest moisture uptake was observed for the fibre treated with 20% NaOH concentration, which can be due to the high content of semi-crystalline and amorphous materials, such as Hemi-Cellulose and Lignin and the presence of the hydroxyl groups on the fibre surface and in the amorphous region. This result agrees with those reported by Das and Maiti (1999).

However, the fibre treated with 10% NaOH had low moisture uptake compared to the others, and exhibited

least activity, because of its crystallinity thus rendering fewer hydroxyl groups available for reactions with interacting chemicals.

Most of the fibres absorbed moisture that was over 200% of their mass. The samples treated with 15% NaOH absorbed the highest moisture while the least was found for samples treated with 10% NaOH. Samples treated with 25% also ranked among the non-absorbent fibres. This could perhaps be due to inherent shrinkage resulting from of this treatment (Gordon, 1967).

The desorption of moisture of these samples also show similar trends with the sample mercerized with 15% NaOH showing the highest moisture retention and that treated with 25% NaOH showing the least moisture retention.

The moisture imbibitions of samples treated with 20% NaOH were highest while those of 15% were least. In terms of moisture absorption the samples may be ranked as follows: 20 > 25 > 10 > 15%. The inconsistent trend may be attributed to the process variable and inherent fibre properties.

The fibres showed varying moisture absorption and relaxation properties, as shown in Table 1 ranging from 2.05 - 4.006%. Okro treated with 10% NaOH gave the highest absorption value 4.06%, Kenaf treated with 20% NaOH had 2.67%, Baobab treated with 15% NaOH had 3.02% and Roselle treated with 20% NaOH had the value of 2.25% indicating higher levels of moisture absorption properties, which compared favourably with the 2.69% reported for the water sorption capacities of cotton fiber, kenafcore fiber, and mossf iber (Seca and Cavaleiro, 1998).

The relaxation, moisture loss equilibrium derived from the plots of Figures 1 - 4 ranges between 1.04 - 1.76%. The low values of the moisture kinetic relaxation recorded in this study for the four cellulosic bast fibres compare favorable with 1.05% reported for the water absorption of kenaf - filled propylene composite (Azzini and Salgado, 1993).

The densities of the fibres were higher than that of water, Fibre density is believed to be closely related to the mechanical properties and moisture absorption (Eromosele et al., 1999). Since the average density of the local vegetable fibres falls between 1.19 - 1.5 gcm<sup>-3</sup>. It gives a good indication that they will be suitable as reinforcing fillers in plastics.

# Conclusion

The effects of moisture Imbibition absorption are attributed to the presence of hydroxyl groups and amorphous areas in the fibre structure. Typically, chemically purified fibres are hydrophillic, resulting in high capacity to sorb moisture. The natural bast fibres as common raw materials find uses as industrial reinforced fillers for thermplastic and automotive materials. These fibres have excellent specific properties and have the potential of being outstanding reinforcing fillers in plastic industries and are viable alternative to inorganic/ mineral based reinforcing fibres as long as the right processing conditions are used. Furthermore they can be used in applications where the higher water absorption is not critical.

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