International Journal of Physical Sciences Vol. 4 (11), pp. 698-704, November, 2009
Available online at http://www.academicjournals.org/ijps
ISSN 1992 - 1950 © 2009 Academic Journals

Full Length Research Paper

The effect of mercerization media on the physical properties of local plant bast fibres

U. U. Modibbo, B. A. Aliyu and I. I. Nkafamiya*

Department of Chemistry, Federal University of Technology, P. M. B. 2076 Yola, Adamawa State, Nigeria.

Accepted 6 August, 2009

The effect of using different mercerization media on some mechanical properties of local plant bast fibres, Roselle (*Hibiscus sabdariffa*), kenaf (*Hibiscus cannabinus*), okra (*Hibiscus esculentus*), Baobab (*Adansonia digitata*) respectively, were generally investigated. The fibres from these sources were purified using the retting method which involved heating with 15% ammonium oxalate followed by scouring using 2% sodium hydroxide (NaOH) solution and finally bleaching with 5% hydrogen peroxide (H_2O_2). All the fibres were subsequently mercerized at a temperature of 5°C for 20 min using 10, 15, 20, and 25% NaOH solutions in turn. The parameters used to evaluate their potential as raw materials for textile making under different treatments (different NaOH concentrations) include extension at break, tenacity, specific work of rupture and density. Results show that the variation in the magnitude of extension at break is in the following order: for Roselle we have 25 > 15 > 20 >10%, for Kenaf 10 > 20 > 15 and 25%, for Okra 25 > 15 > 10 > 20% and for Baobab 20 > 15 > 25 > 10%. Tenacity values for the fibres show the following variation in order of magnitude: for Roselle 25 > 20 > 10 > 15%, for Kenaf 10 > 20 > 15 > 25%, for Okra 10 > 20 > 15 > 25%, and Baobab 10 > 20 > 15 > 25%. While specific work of rupture shows the following order: 25 > 10 > 20 > 15% for Roselle, 10 > 20 > 15 > 25%, for Kenaf, 10 > 15 > 20 > 25% for Okra, and 15 > 10 > 20.25% for Baobab. For the Density (g/cm^3), the variations are in the following order: For Roselle 10 > 15 > 20 > 25%, for Kenaf 10 > 15 > 20 > 25%, and for Okra and Baobab 10 > 15 > 20 > 25, 10 > 15 > 20 > 25 respectively. From the forgoing, we can say that mercerization condition exert significant effect on the properties of the fibres studied and this may be traced to the structural features of these fibres. Thus, Baobab appears to have high percentage extension at break compared to the other fibres. In all cases investigated, different ranges of values are associated with the parameters involved, thus extension at break range between 2.0 - 7.6 mm, tenacity 0.21 - 1.0 kgf, specific work of rupture 0.01 - 0.28 kgf while density range between 1.19 - 1.45 g/dm^3 for the materials. The relative proportions of amorphous (higher in Baobab) to crystalline regions are presumed to be the major determinant in the variation of these properties.

Key words: Bast fibre, mercerizations, tenacity, work of rupture.

INTRODUCTION

The production of natural fibres continues to be a major agricultural activity world-wide. The textile and paper industries are the primary converters of fibres into the numerous products needed in our modern society (Raymond and Herman, 1988). Fibres and fibrous products should possess some primary properties for acceptance as suitable raw materials. These properties include length to diameter ratio (aspectratio), tenacity (strength) and flexibility. Acceptable extensibility for processing, cohesion and uniformity of properties is also important (Bhatnagar, 2004). The textile fibre products identification act 1960 covered among it list that classified fibres into a comfort fibre, safety fibre and industrial fibres. Fibres used for making undergarments and garments are generally known as comfort fibres (Sharma 2006). Also, fibres used for making carpets, seat covers, curtains are known as safety fibres.

Industrial fibres are used as reinforcing agents an as such they possess high modulus strength, thermal stability, toughness and durability. They are used for making rigid and flexible tubes, pipes, tyre cords and composites, construction of boats, cars and planes (Bhatnagar, 2004).

*Corresponding author. E-mail: iliyankafamiya@yahoo.com.

It has become necessary to consider affordable, sustainable alternative local raw materials of plant bast fibres for industrial growth. This is because; recent studies have reported that, natural fibres from plants, animals and minerals, account for more than half (about 65%) the fibres produced annually in the world (Khurmi and Sedha, 2005). Out of this percentage, a larger portion (46%) is cotton. The other 35% of annually produced fibre is from animal fibres (wool and silk) 5%, and artificial fibres (cellulose and synthetic fibres) provide 30% of the world's production (Alexander et al., 2002).

Cotton, the most widely used natural fibre, is processed into numerous apparel, home furnishings and industrial products. Other types of natural fibres include: flax, a strong, silky fibre from the stems of flax plants, used in making clothing, napkins and other linen products: Hemp, jute and sisal are coarse fibres used in cords, ropes and rough fabrics (Khusk, 1993).

Animal fibres include fur and hair, wool; the hair sheared from sheep and certain other animals is popular in clothing and home furnishings. Rough surfaces on wool fibres give bulk and warms to wool clothing and blankets. Silk is the strongest natural fibre and manufacturers unwind silk filaments from silkworm cocoons and make silk yarn for clothing and household fabrics (Saha and Sarkar, 1990)

In another study (Ajayi et al., 2000), studied the physical properties of natural cellulose fibres and found that these properties are inextricably linked to their gross morphological features. The same worker also reported that, the bast fibres cells are embedded n a continuous inter-molecular matrix. The molecules are oriented and arranged parallel along the major axis of the fibre to yield high strength and anisotropy.

In an experiment with other fibre mercerization (Eichhorn et al., 2001), studied the effectiveness of fabrics chemical and physical properties observed that, the fibrous transformation from cellulose I to cellulose II occurs during mercerization, which consists of a swelling of the initial fibres in alkali, followed by recrystallization during subsequent washing. In cellulose I, the chains within the unit cell are in parallel conformation (Vawdrey and Stirling, 1992; Ahmad and Islam, 1995).

In activities related to the chemical bio-processing of the fibres, the samples retted with ammonium oxalate solutions have effected the separation of fibre bundles from their non-cellulosic components which have accounted for about 25% of the fibre layers and these include pectin, hemi cellulose, lignin, fats and waxes. Ammonium oxalate, easily decomposes during retting according to the equation (Ajayi and Elder, 1995):

$$(NH_4)_2C_2O_4 + 2H_2O \longrightarrow 2(NH_4)OH + H_2C_2O_4$$

Generally, finished products from macerated fabrics have very good impact strength, and this can be seen from the following treatment. The cellulosic samples are treated with 10 - 25% NaOH solutions at low temperature of 5°C for 20 min. It swells up due to both physical and chemical changes, collectively known as mercerization. The extent of hemi cellulose removal depends upon concentration of the dissolved matter in the liquor, time of digestion, temperature and pH of the digesting liquid. The process of mercerization involves partial destruction of inter-molecular bonds, penetration of NaOH in the swollen amorphous regions of the cellulose where they are held by hydrogen bonding and where sodium cellulose and sodium alcoholates are also formed. A large part of residual hemi cellulose is removed and rupture of some C-C bonds occurs which reduces the lengths of the chain as reported by Sharma, 2006.

$$R_{cell} - OH + NaOH \longrightarrow R_{cell}\left[-OH.\ NaOH\right]$$

Alkali cellulose

$$R_{cell} - OH + NaOH \longrightarrow R_{cell}\left[-ONa.\ H_2O\right]$$

Retting involves the use of ammonium oxalate (15% solution) in the dissolution of binding materials. 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.

Bleaching

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

$$H_2O_2 \xrightarrow{NaOH} H^+ + HO^-_2$$

$$HO_2 \longrightarrow OH^- + [O]$$

$$[O] + I \longrightarrow IO$$

Where; I = is the impurities.

In an alkaline condition, the hydroxyl ions (OH^-) produced by the perhydroxyl ion HO^-_2, hydrolyses the hydrogen ions (H^+), thereby promoting the liberation of more perhydroxyl ions.

$$H^+(aq) + OH^-(aq) \longrightarrow H_2O\ (l)$$

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

This involves the treatment of fibre with 10 - 25% solutions of Sodium hydroxide for 20 min at 5°C. Therefore, to have some indications of the suitability of local bast fibres for textile making, it is necessary to characterize the cellulose in terms of relevant parameters, such as tenacity, extension at break, fibre density and work of rupture, as recommended by the Textile Fibre Products Identification Act. This study presents the results of these investigations and suggests how the local plant fibres varieties grown in Nigeria can be used for textile making.

MATERIALS AND METHODS

Materials

The materials were collected in Yola Town of Adamawa State, Nigeria. The materials fibres (Roselle, Kenaf, Okra and Baobab) were removed mechanically and shade dry for 24 h. These fibres were processed by the retting, scouring, bleaching and mercerizing procedures described by Eromosele et al. 1999 and Ajayi et al. 2000.

METHODS

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 treatment. The materials were rinsed in over-flowing tap water for five minutes, followed by drying in oven at 50°C for 20 min.

Scouring: The lignin fibre (2 g) was completely immersed in 2% solution of sodium hydroxide and heated at 100°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 over flowing tap water, followed by drying in oven at 50°C for 20 min.

Bleaching: The materials were boiled in 5% H_2O_2 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 cellulose fibres were mercerized with 10 - 25% NaOH aqueous solution at 5°C for 20 min, while the fibres were allowed to shrink. 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 handbook of TAPPI (1980). The specimens were conditioned for 24 h at 65% relative humidity and 25°C before carring the density test. 2 g of samples were accurately weight out for each fibre type. Each weight was immersed in toluene in a calibrated glass tube, and the value of toluene displaced was determined. It was equal to the volume of fibre immersed. The density of the fibre was calculated from the formula:

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}}$$

Note: Toluene was used because it was not absorbed into fibre structure.

Determination of tensile properties

Four samples of equal length (5 cm) and equal weight (0.03 g) were prepared for tensile testing. The gauge length was set at 2.5 mm, corresponding to the distance between the spacers on the prepared fibre sample. A tensiometric tensile tester model 220D Equiptex LTD, UK was used. The length of the fibre specimen was tightly secured in the spacers to prevent samples from slipping off during the tensile testing. Here, there is no danger of samples slippage and false elongation measurement. The speed of the machine was adjusted to 5 mm/min and the digital recorders of load and elongation were adjusted to zero. The tensile tester was actuated and the cross-head traversed upward so that the sample was stretched until breakage. At this point, the cross-head stopped, and the load and the correspond elongation at the breaking point were read directly from the digital recorder. The broken sample was unscrewed, and removed. The cross-head was returned to the starting position, for the next test. This was carried out for four samples, and the average values for both the load, and elongation were obtained. All tests were carried out in a standard atmosphere maintained at temperature of 20°C and relative humidity of 67%.

RESULTS AND DISCUSSION

Table 1 shows the summary results of the various analysis

Table 1. Tensile value of the various fibres.

	Roselle (*Hibiscus sabdariffa*)				Kenaf (*Hisbiscus cannabinus*)				Okra (*Hisbiscus esculentus*)				Baobab (*Adansonia digitata*)			
NaOH properties	10	15	20	25	10	15	20	25	10	15	20	25	10	15	20	25
Gauge length/mm	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Extension at break	2.0	3.4	2.7	3.5	3.5	2.5	3.4	2.5	3.6	4.1	3.5	4.3	5.3	7.0	7.6	6.0
Tenacity (Kgf)	0.4	0.3	0.59	0.66	0.21	0.15	0.18	0.14	0.38	0.23	0.34	0.19	1.0	0.63	0.66	0.6
Specific work of rupture (Kgf)	0.16	0.05	0.08	0.28	0.06	0.025	0.05	0.01	0.14	0.1	0.09	0.04	0.2	0.21	0.05	0.19
Density (g/cm^3)	1.45	1.41	1.38	1.32	1.36	1.31	1.28	1.20	1.25	1.23	1.20	1.19	1.40	1.29	1.27	1.19

for individual materials as represented in Figures 1 - 4. The load – elongation behaviour of fibres *Hibiscus sabariffa* mercerized with various concentrations of sodium hydroxide is shown in Figure 1. The mercerization effect on natural fibres is of particular importance in the sense that the fabric gets stronger and more elastic after mercerization (Sadov and Korchangin, 1978). From the Figure 1, it can be seen that mercerized fiber at 10% NaOH solution showed linear variation between breaking load and elongation (0.05 kgf against 0.0 or ~1.0 mm). Beyond the breaking load of 0.05 Kgf, the fiber deformation cease to be linear and this point is known as yield point or elastic limit. With further addition of load (0.11kgf) to the fiber, a plateau (or slow marching) effect is being observed. The ultimate tensile stress and breaking load of 0.4 kgf is observed. This ultimate breaking load can be attributed to both, the oxidation of cellulose and chain rupture, because of the mercerization effect (Premamoy, 2002). Similarly, fiber mercerized with 20% NaOH, had yield point at 0.1 kgf with corresponding I.5 mm breaking elongation. The point of rupture of the fibre specimen occurred at 0.6 kgf breaking load to a corresponding breaking elongation of 2.6 mm. The fibre specimen mercerized with 25% NaOH solution shows a very small change in breaking load with addition of load (0.001 Kgf), and formed another yield point at 0.31 kgf and corresponding breaking elongation of 2.6 mm. With increasing addition of load, the point of rupture of the specimen occurred at 0.66 kgf with corresponding 3.3 mm breaking elongation. This is consistent with the result reported by Ajayi et al. (2000).

Figure 2: The breaking load and breaking elongation, curves for *Hibiscus cannabinus* fibre mercerized with various concentrations (10 - 25%) of NaOH solution are shown in Figure 2. The breaking elongation of a sample mercerized with 10% NaOH increases sharply initially up to 1.5 mm suggesting linear relationship between breaking elongation and breaking load (Hook's law obeyed). The point 1.5 mm, where the curve deviates from the straight line, is known as elastic point. This is in accordance with the result reported by (Shukla and Sharma, 1987). The breaking elongation also increases from 1.5 to 2 mm with increase in stress from 0.05 to 0.2 kgf. The point corresponding to 2 mm is called the yield point. It may be noted that if load (kgf) from the specimen is removed, then the decrease in the length of the fibre specimen from 0 to 2 mm will not disappear. But it will remain as a permanent set. An upward curve is observed from 0.12 to 0.16 kgf which indicates that the specimen regains some strength, and higher values of stresses are required. The fibre mercerized with 10% NaOH solution showed a terminal stage by rapid extension of the specimen, which finally leads to rupture from 2 to 2.5 mm breaking elongation. The result correlates well with mechanical tests reported by Khurmi and Sedha (2005).

Figure 3: Shows load - elongation behaviour of fibres (Okra) mercerized with various concentrations of 10 - 25% NaOH solutions. This figure, showed proportional increase in the breaking load with increase breaking elongation up to elastic point (1.5 mm). The fibres mercerized at 10 and 20% with separate treatment of NaOH solutions show similar patterns of physical and mechanical behaviours while fibres treated using 15 and 25% NaOH solutions have obviously showed similar trend of tensile characteristics. All the fibres showed characteristic of fibres elongation in (mm) more quickly than the stress in (kgf). They reach another yield point precisely at 3 mm. the samples mercerized at 10 and 20% NaOH solutions showed upward curve which indicated that the fibres regain some strength and higher values of breaking loads 0.35 and 0.38 kgf. The fibres 15 and 25% NaOH solutions showed downward curve with breaking elongation which increases from 4 to 4.25 mm.

The load-elongation behaviour of *Adansonia digitata* mercerized with 10 - 25% NaOH solutions is indicated in Figure 4. The fibres at different alkali treatment 10, 15 and 25% NaOH solutions showed intersection with proportional load-

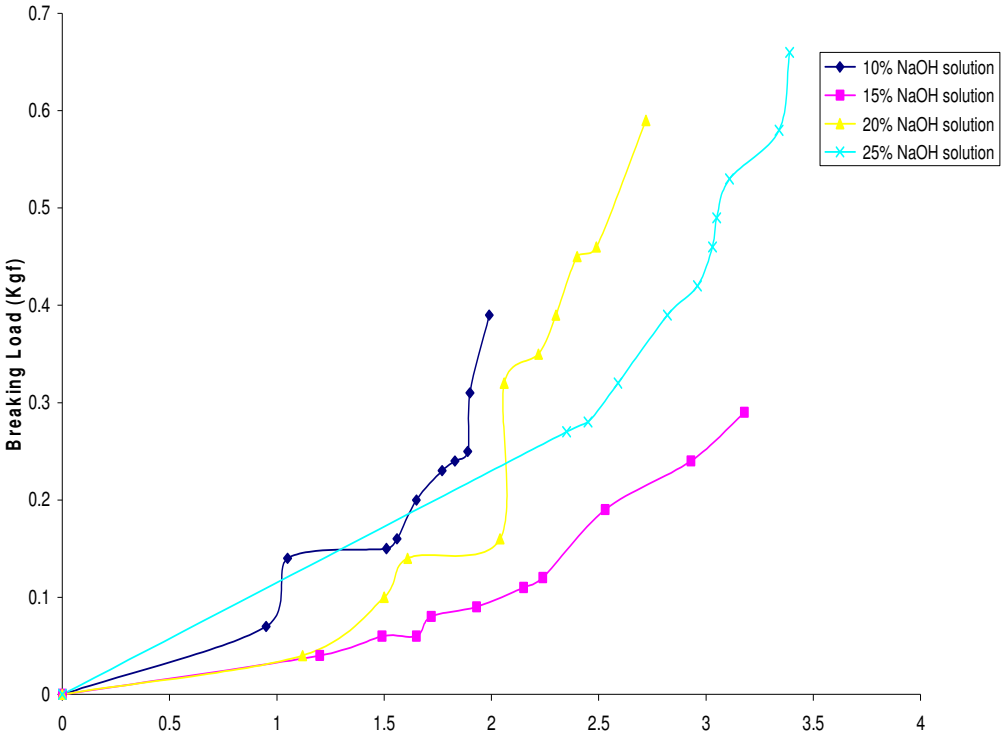

Figure 1. Effects of concentration of sodium hydroxide on the load-elongation curves of *Hibiscus subdarrifa.*

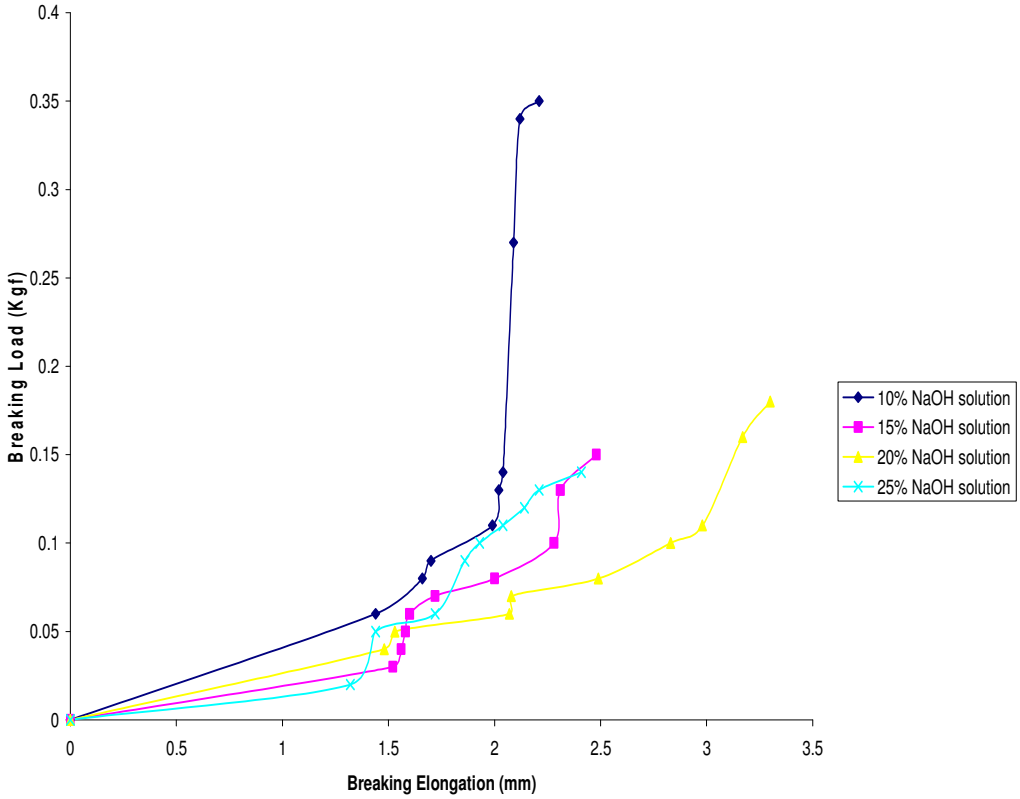

Figure 2. Effects of concentration of sodium hydroxide on the load-elongation curves of *Hibiscus subdarrifa.*

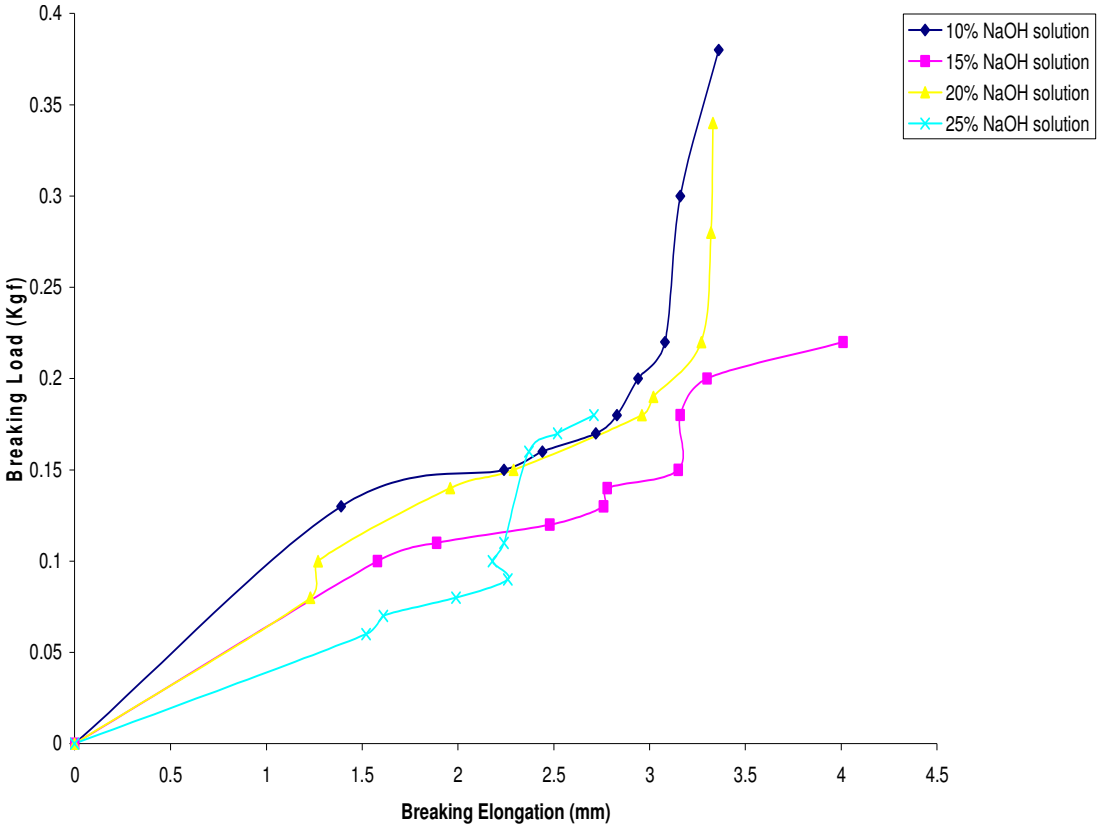

Figure 3. Effects of concentration of sodium hydroxide on the load-elongation curves of *Hibiscus esculentus.*

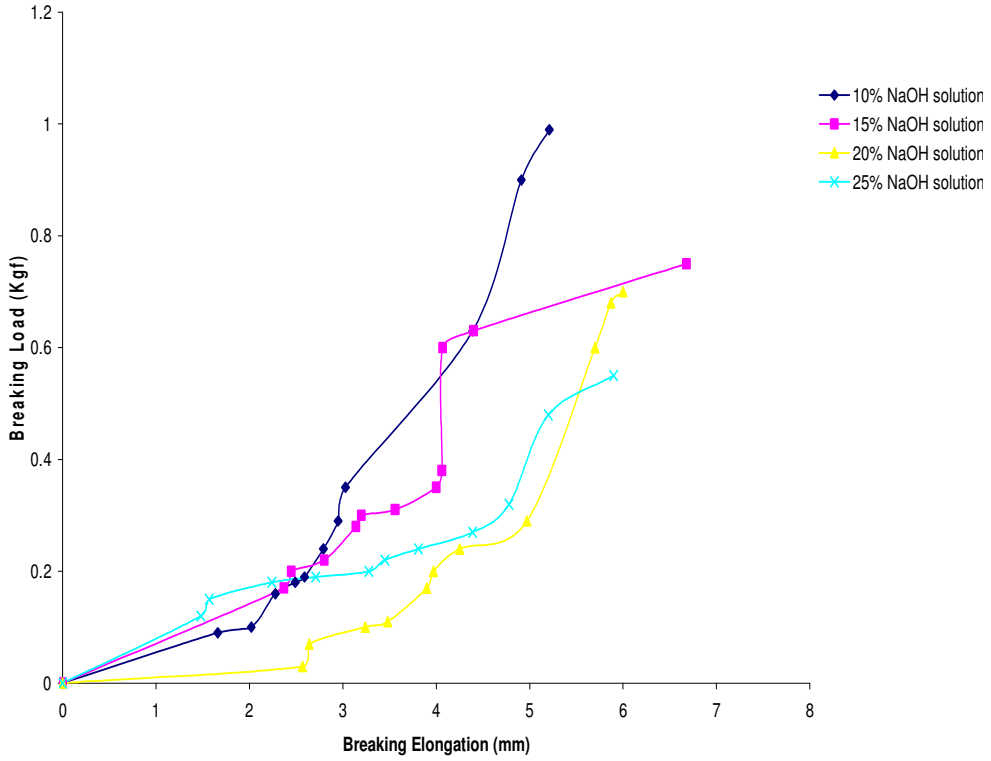

Figure 4. Effects of concentration of sodium hydroxide on the load-elongation curves of *Adansoria digitata.*

elongation at 0.2 kgf and 2.5 mm extension respectively. It is clear that the sample mercerized at 10% NaOH displayed the highest breaking load (1 kgf) and breaking elongation (5 mm), which makes the fibre the stiffest with the least damping energy. The fibres processed under the same condition but at different concentrations e.g. 15, 20 and 25% NaOH solutions displayed breaking loads varying from 0.75, 0.67kgf and the least breaking load at 0.6 kgf but the highest elongation at break among them is the fibre treated with 20% NaOH which had value of 7 mm. Generally, elongation is a property of amorphous areas. Thus, the amorphous area in this fibre may be proportionally higher than the highly ordered crystalline region (Morton and Hearle, 1975).

Result for specific work of rupture (Table 1) showed that Roselle had it maximum value at 25% NaOH solution. While Kenaf and Okra had their maximum value at 10% NaOH each and Baobab had its own maximum value at 15%. For density, all the samples attained their maximum values at 10% NaOH.

Conclusion

The effects of mercerization media on the physical properties of local plant bast fibres have been observed. Fibrous materials such as cellulose bast fibres, have extensively been used as insulators because of their high mechanical strengths, flexibility, durability with extreme fineness and easy processing. They, however, have low dielectric strengths and absorb moisture. Mercerization process was however an important unit operation in this study, the medium (NaOH), has reoriented the fibre and consequently improved its crystallinity and strengths. In this study it was observed that fibre mercerized in 10% NaOH showed the best properties for fibre samples of *H. cannabinus, H. esculentus,* and *A. digitata*, while 25% NaOH medium was the best for *Hibiscus sabdariffa* in increasing its tenacity. The result therefore showed that, NaOH medium of 10% gave the best result. The final mercerized fibre could be used as good fibrous filler and composite. They are added to impact valuable service properties such as high strengths, heat resistance, low shrinkage, during solidification and lower the cost of the article formed. The fibre also reduces combustibility of wares made from the plastic and often increases water resistance and improves the appearance.

REFERENCES

Ajayi JO, Bello KA, Yusuf SD (2000). The Influence of Retting on the Physical Propertues of Bast Fibres. J. Chem. Soc. Niger. 25: 112-115.

Ajayi JO, Elder HM (1995). “Effect of Finishing Treatment on Fabric Friction” ASTM. J. Testing Eval. 23: 55.

Ahmad Z, Islam SH (1995). The Root Characterizatics and Pattern of Dry Matter Accumulation of Kenaf and Mesta as Influenced by the Alluvial and Red Soils of Bangladesh. Inter. J. Trop. Agric. 13(1-4): 177-183.

Alexander B, Ibon A, Jurgen S, Thomas L, Bernhard W, Artemis S, Ilja S, Hans-Heinrich L (2002). Surface Characterization of Flax, Hemp and Cellulose Fibers; Surface Properties and the Water Uptake Behaviour.

Bhatnagar MS (2004). Chemistry and Technology of Polymers. S. Chand and Company Ltd. Ram Nagar, New Delhi-110055.

Eromosele IC, Ajayi JO, Njaprim KG, Modibbo UU (1999). Characterization of Cellulosic Fibre. J. App. Poly. Sci. 73: 2057.

Eichhorn SJ, Baillie CA, Zafeipouls N, Nwaikambo LY, Ansell MP, Duresne A, Enteristle KM, Herrera-Franco PJ, Escamilla GC, Groom I, Huglies M, Hill C, Rials TG, Wild PM (2001). Current International Research into Cellulosic Fibres and Composites. J. Mater. Sci. 36: 2107-2131.

Khusk MT (1993). The Structure and Utilization of Kenaf (*Hisbiscus cannabinus L).* Oil Seeds. Sarhad Agric. 9(2): 145-148.

Khurmi RS, Sedha RS (2005). Textbook of Material Science. S. Chand and Company Ltd. Ram Niger, New Delhi-110055.

Morton WE, Hearle JWS (1975). Physical Properties of Textile Fibres and Textile Institutes (2nd Edition). Manchester, USA. pp. 27-288.

Premamoy G (2002). Polymer Science and Technology. Tata McGraw-Hill Publishing Company Ltd., New Delhi pp. 4-15.

Raymond BS, Herman FM (1988). Application of Polymers, Plenum Pres, New-York 233 Spring Street, New York, 10013: 5-151.

Sadov F, Korchangin M (1978). Chemical Technology of Fibrous Material. Mir Publishings, Moscow pp. 419-428.

Saha M, Sarkar AK (1990). Structural Studies on Pectic Acid Isolation from Unretted Mesta (*Hisbiscus cannabinus)* fibre. Indian. Chem. Section B. Organic Chemistry Including Med. Chem. 29(2): 188-190.

Sharma BK (2006). Textbook of Industrial Chemistry, Including Engineering. Goel Publishing House, St. No.2 Darya Ganji, Delhe pp. 928-939.

Shukla JS, Sharma GK (1987). Evaluation of the Properties of Bast Fibres with Potential Use as Raw Materials. J. Polym. Sci. Part A Polymer. Chem. 25: 595.

TAPPI (1980). Standard and Suggested Mehtods. Technical Association of Pulp and Paper Industries New York. 2(1): 14-19.

Trotman W (1970). Deying and Chemical Technology of Textile Fibre. Hillman London pp. 318-325.

Vawdrey LL, Stirling GR (1992). Reaction of kenaf and Roselle, Grown in the Burdekin River Irrigation Area to Root-Knot nematodes. Austr. Plant Pathol. 21(1): 8-12.