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Effects of Alkali Concentration and Temperature on the Imbibition Properties of Okro Stem *Habiscus esculentun* Fibre

Abubakar Ahmed Hamidu, Mayen Nelson Ekott
Chemistry Department, Federal university, Dutse, Jigawa State, Nigeria

Original Research Article

***Corresponding author**
Abubakar Ahmed Hamidu

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Abstract: Okro fiber was obtained from okro stem by soaking in ammonium oxalate for 20minutes.The effect of alkali treatment (mercerization) on 10grammes of each sample was investigated. Various concentration of the NaOH (Sodium hydroxide) ranging from 0% to 22% was prepared. The temperature of the treatment was varied from 25 to 40^{o}C .Water imbibitions studies was also carried out on the fiber samples by varying the temperature and concentration of the fiber samples obtained. Result of treatment of the fiber with ammonium oxalate indicated a whitish lustrous material. The mercerization effects on the fiber indicated an increase in mass of the fiber from an initial weight of 0.4g in 1.0M to 0.8g in 4.0M at 25^{o}C .On the other hand, there was a substantial swelling noticed in the case of the fiber sample treated with 1.0M (0.6g) to 0.9g (4.0) at 40^{o}C.For imbibitions studies, the water uptake recorded the best value with fiber treated with 1.0M showing a maximum imbibitions value at 25^{o}C. The overall results of the study indicated that treatment of fiber with chemical enhanced some crucial properties of the fiber and has inhibited substantially the amount of water absorbed by fibrous materials. Industries involved in the exploration of natural fibers as potential fibers may benefit immensely from this investigation as this may add to the list of fiber utilized for fiber production due to its abundance.

Keywords: Water absorption, Chemical treatments, Hydrophobic Nature, Hibiscus esculentum, Fibers, Mercerization.

INTRODUCTION

Man in his ingenuity has discovered the use and application of fibers since the prehistoric period. Perhaps some of the earliest evidence of Man's innovative tendencies was in the discoveries of wool and dyed flax fibers found in prehistoric caves. Since then there has been increase in the application of fibers and fiber related products.

This scaled up increased intensified research in the application of fibers as reinforcements for polymer related materials. Although some of the earliest polymers produced were sourced from synthetic means, a little proportion of the products produced was from natural means.

Conventional synthetic polymers reinforcement fibers can be produced with engineered properties to suit particular applications, resistant to moisture, posses' mechanical properties that are suitable for application as fiber reinforcement. Despite the mentioned properties, synthetic fibers are faced with sustainability and environmental issues.

Natural fiber on the other hand is largely sustainable and able to resolve the environmental issues posed by their synthetic counterparts and therefore present a better option for reinforcement materials. Many researchers have been geared towards harnessing natural fiber for improvement of material properties as reinforcement materials and some of these works include [1-9].

Many factors will have to be taken into consideration in the design of natural fiber composites. One of such factors included water uptakes which affects the degradation behavior of composites exposed to the environmental conditions of humidity sunlight and microorganisms. The other factor which is largely responsible for the stability of materials in the environment is poor resistance of fiber to absorption and can result in the reduction of the desirable properties of mechanical and dimensional stability of composites and other materials produced from them. This therefore necessitated extensive study of the water absorption behavior of the fiber sample with a view to estimate the consequences of water absorbed by fibers will have but also to establish the durability of natural fibers aged under water.

In view of the above, the objective of this work is to investigate the use of chemical as a means of improving the properties of natural fiber for improved application with a view to;

- Study the effects on the water imbibitions property on the fiber.
- Study the alkali treatment effects on the water absorption of fiber.

MATERIAL AND METHODS

Materials

The Hibiscus esculentum stem was collected fresh from a farmland and identified by a botanist in the Department of forestry of the Modibbo Adama University of Technology YOLA. The chemicals used in this experiment which included sodium hydroxide, ammonium oxalate were supplied by BDH (British Drug House)

Sample Preparation

The plant sample of Okro plant collected (Hibiscus esculentum) were mechanically cleaned, dried under the shade and stored in polyethene bags until needed for further analysis.

METHODS

Retting

The methods described by [10] were adopted for this treatment. This involved the treatment of a weighed mass of 10g of the fiber in a 200ml of 15% ammonium oxalate ($C_2H_{10}N_2O_5$).This was properly rinsed in an overflowing tape water for 5minutes and dried in an air oven at 60^0C for one hour.

Mercerization

This was carried out according to the methods of [12, 13] as modified by [11]. This treatment involved treatment of the fabrics with 1.0M-4.0M solution of sodium hydroxide at 25^0C and at 40^0C. This was carried out in an ice bath followed by occasional turning with glass rod to ensure proper mixing.

Water Imbibitions Studies

The method of described by [14] as modified by [15] was adopted for this analysis. Water absorption studies were carried out on the treated and untreated fiber samples. Distilled water with a $_p$H was used for this study. The specimens were dried in a hot air oven at 60^0C for 24hours. The humidity chamber was set up to 100% humidity using the distilled water sample. The specimens used in this study were unbundled from bundles of single fibre bond together. The fibre samples were then placed in the humidity chamber after weighing in a balance. These were brought out of the chamber after 24hours and excess water carefully mobbed with a filter paper. Finally the fibres treated at different concentrations were measured and the water imbibitions at various chemical concentrations by weight difference.

The water absorption was measured by the mathematical relation below:

$$\text{Water absorption} = \frac{Ma - Md}{Ma} \times 100\%$$

Where

M_d = mass of dried samples

M_a = mass of sample after exposing to water.

RESULTS AND DSCUSSIONS

Table-1: Effect of Mecerization at 25^oC

Concentration (M)	Weight before treatment (g)	Weight after treatment (g)
1.0	1.0	0.4
2.0	1.0	0.6
3.0	1.0	0.7
4.0	1.0	0.8

Table-1 represents the mercerization effects study carried out against the fibres obtained from the *Habiscus esculentum* plant species. From the table it could be observed that there was a corresponding increase in the weight of samples as the concentration of the of sodium hydroxide solution.

Table-2 also represents the mercerization of the fiber sample at a much higher temperature of 40^oC.From the table, it was also observed that there was also a gradual increase in the weight of the sample as the concentration of sodium hydroxide was from 1.0M to 4.0M.

Table-2: Effects of Mercerization at 40^0C

Concentration(M)	Weight before treatment (g)	Weight after treatment (g)
1.0	1.0	0.5
2.0	1.0	0.6
3.0	1.0	0.7
4.0	1.0	0.9

Equally observed was that the weight obtained for a 4.0M sample at 25°C, which is slightly more than the weight of sample mercerized at 40°C.

From the above observations with respect to Table-1 & 2, the following implication could result;

The implication of these observations could be attributed to the fact that the main reinforcement material in plants is cellulose, which is made up of amorphous and crystalline regions. The amorphous providing the flexibility, whereas the crystalline portions provide the dimensional region for material stiffness. The lignin provides the cementing needed to bind natural fiber together effectively protecting them from mechanical damage.

Fibers will absorption moisture or shrink depending on the depending on the contents of their cell walls. This in turn, is affected by the type of chemical modification and the temperature of treatment during the period of modification.

Higher temperature of mercerization results in the absorption of more moisture by extending the three dimensional lignocelluloses network and a consequent increase in weight of the material subjected to such modifications. At a slightly lower temperature, the increase may not be as pronounced as in the earlier case. This may have contributed to the lower weight of the fiber sample treated at 25°C.This corroborates an earlier observation by [16], in which studies pointed out that cell modification by chemical treatments plays a role in material property of the modified product.

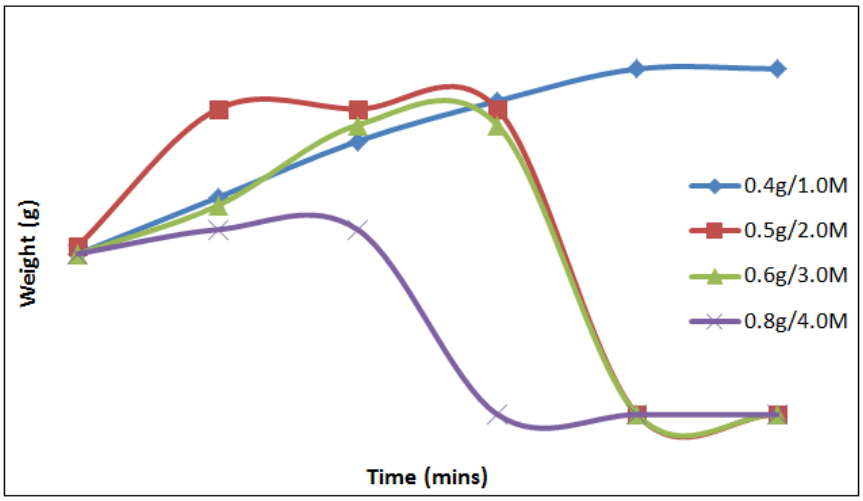

Fig-1: Water Imbibitions of Fibers at Various Solvent Concentrations at 25^{0}C

Figure-1 shows the water inhibition study of the modified cellulose fiber at 25^{0}C.

From the figure, it was observed that the modified fiber sample with the weight of 0.8g/4.0M showed the lowest imbibitions values and the highest imbibitions values was seen in the case of the sample subjected to the chemical treatment of 0.4g/1M.However, an extended imbibitions values was seen in the case of the case of 0.4g/ 1M.

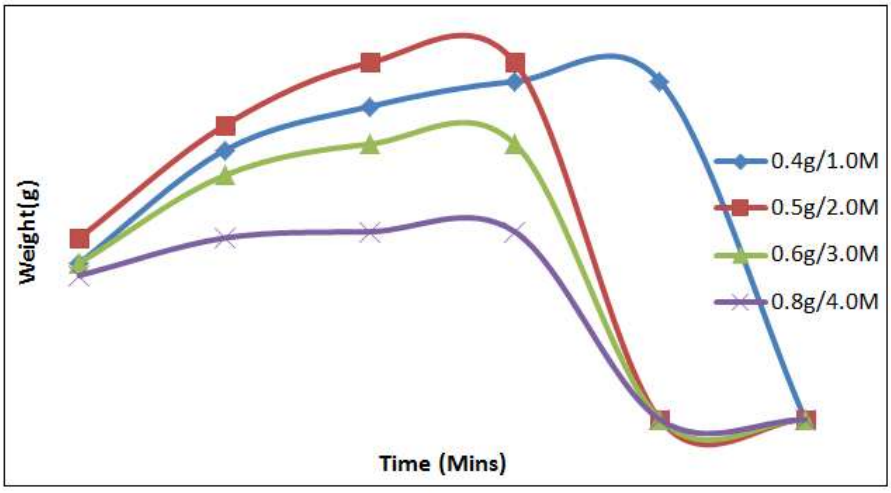

Fig-2: Water Imbibition of Fibers at Various Solvent Concentrations at 40^{0}C

The imbibitions studies carried on the fibers chemically modified at 40^0C is shown on Figure-2. From the figure, it was observed that the highest imbibitions values was seen in the case of the sample modified in 0.5g in 2.0M and the lowest in the case of the sample treated with 0.8g in 4.0M.

For samples treated with 0.4g in 1.0M, there was a steady increase in weight as the temperature was increased as the weight increased from an initial weight of 2.5g at a 5min to a weight of 4.5g after 15mins. This value increased from 4.5g to 5g between the times of 15mins to 20mins.This value was however steadily maintained within the time of 25mins to 30mins.

For sample treated with 0.5g in 2.0M, there was a steady increase in weight in 5mins until the time of 15mins at a weight of 5.9g after which equilibrium water absorption was reached.

For samples weighing 0.6g treated in 3.0M sodium hydroxide solution, a steady increased in weight from 2.3g to a steady weight 3.7g at 15mins.This low imbibitions value was maintained through 20 to 30mins.

Lastly, for the sample weighing 0.8g treated with 4M sodium hydroxide solution. There was an increase in weight of sample as the time of treatment was increased from 5 to 10mins.This was however maintained from 10mins through 30mins.

Comparing Figure-1 & 2, in both scenarios, there appears to be an extended length of time on samples treated with 0.4g in 2M of up to 25min under both temperature conditions of 25^oC and 40^oC.The values obtained for all other conditions presents better result owing to the reasons attributed to chemical modifications.

By and large, the implications of these observations are that, chemically modified fibers experienced an improved surface area owing to the exposure of functional groups occasioned by the chemical treatments. The alkali treatment results in the partial removal of waxes and therefore an increase in surface area for interactions by other substances such as water.

The treatment of fibers with alkali results in the removal of hydrophobic substances waxes and other substances for instance size to eventuating the result of water absorption.

In line with the statement above and observed in the results of the undergone experiments at the varied concentration of 1M to 4M, the treatment results in the removal of hydrophobic substances. In the light of the aforementioned, the five samples treated with lower concentration observed at both temperature of 25^0C and 40^0C showed better swelling and consequently imbibitions properties compared to the fiber samples treated with more concentrated samples of caustic soda. This may not be unconnected to the fact that hemicelluloses as a constituent of the fibrous plays a critical role in the sorption properties of fibers. As seen in the case of the results of this investigation, there is a case of observed corroboration with similar studies conducted by [17] in which studies indicated that the treatment of cellulose with more concentrated solutions of alkali may partly dissolve the hemicelluloses and may affect the absorption of solution by potentially useful fibers.

Temperature also plays a distinct role in the sorption properties of a polymer system. At low temperature, there are two distinct layers of amorphous and crystalline region. This may have contributed to the initially restricted swelling of the fibers samples at lower temperature. This may have accounted for the better swelling property at lower concentration of 1M of 4g of fiber sample as compared to other samples. This also corroborates the results obtained by studies carried out by [18].

Comparing this to the results obtained for sample treated with the same concentration but at a higher temperature, the treated fibers in this case presented better imbibitions properties partly due to the effect of chemical treatment on elevated temperature. This may have resulted in some modifications in the amorphous as well as the crystalline regions thereby transforming or causing alterations in the crystalline segments large enough to cause monumental changes in the overall properties and behavior of potential polymer fibers. This is also in line with the observations of [19].

In terms of weight to volume ratio, both cases presented better results in the samples of 4g in 1M solution probably due to the fact that polymer solutions may also be characterized in terms of their solution properties. In good and less diluted solutions, the polymers appear swollen and occupy a large volume. Under this condition, intermolecular forces between solvents and fiber dominate over intermolecular interaction. The reverse is the case in higher concentration of the alkali used. This observation corroborates the results obtained in similar studies carried out by [20].

CONCUSION

From the results of the investigations carried out on Habiscus esculentum fiber, the following conclusions could be made:

- For effective treatments of the fiber lower concentration of the alkali sodium hydroxide is needed.
- The fiber samples mildly treatment at lower temperature showed better swelling properties. Therefore in terms of the fiber treatment an optimized temperature of not more than 40^0C was arrived at.

- For effective interaction of the fiber samples with an efficient concentration of the sodium hydroxide light weight of the fibers is used.

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