



Chemical Analysis of Okra Bast Fiber (*Abelmoschus esculentus*) and Its Physico-chemical Properties

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ABSTRACT

The chemical composition of okra bast fiber (OBF) has been isolated and isolated values are 67.5 % α -cellulose, 15.4 % hemicellulose, 7.1 % lignin, 3.4 % pectic matter, 3.9 % fatty and waxy matter and 2.7 % aqueous extract. So, the main constituents of OBF are α -cellulose, hemicellulose and lignin. The bleached OBF were dyed with Direct Green 27 and Acid Orange 52. The color fastness of OBF on exposure to sunlight have also been studied. It is seen that color fastness of raw fiber is higher than bleached fiber. The degradation of OBF with the action of sunlight was studied by tensile strength. Bleached fiber shown lesser tensile strength loss than raw fiber. Infrared spectra (IR) of OBF were measured for different exposure time under sunlight. The OBF contains higher α -cellulose and has good characteristics like high molecular weight thus it gives high tensile strength, more dye ability and better color fastness properties.

Keywords: OBF; Chemical composition; Oxidative degradation; Viscosity average molecular weight; Tensile strength; Infrared spectroscopy; Colorfastness.

INTRODUCTION

In earlier days, natural fiber served a crucial role to mitigate the everyday needs in a wide range of uses. But in recent years, the rival synthetic products are dominating over the uses of natural fiber. Due to the low cost, high service and expected qualities of the synthetic products, the defects of natural fiber have become prominent [1-2]. But the synthetics are not biodegradable, they are causing serious pollution problems. Thus for the proper utilization of these biodegradable natural fibers, scientists are now trying to find out the ways of alternative uses. As for example fiber glass, insulating material are

used in the replacement of plastics. Jute, a major source of natural fiber has some drawback such as low tensile strength, poor color fastness and high production cost [3-5]. On the other hand, OBF possess better characteristics as compared with other natural fiber specially jute fiber and it can be obtained from the discard stem of okra plant and it grows all over Bangladesh.

In the present investigation effort has been exerted to isolate the constituents of okra bast fiber and also to study the physico-chemical properties of the fiber.

EXPERIMENTAL

Isolation of compositions

The okra bast was collected from Jhenaidah regions in Bangladesh. These were taken for investigation as it possesses excellent quality of fiber. It was then retted into water for 1 month where the fiber was separated from cementing and gummy materials. To remove dirty substances, OBF was scoured in a solution containing 5 mg Na_2CO_3 and 5 g detergent per liter of water at 70°C in a large beaker. The fiber-liquor ratio was maintained 1:50. Then the fiber was washed, dried and stored in desiccator. The main constituents of okra bast fiber were isolated according to TAPPI Standard and Abou-Zeid et. al [6-7].

The raw OBF was immersed in a benzene-alcohol mixture (2:1 by volume) for 10 hours maintaining the fiber liquor ratio 1:100. The fiber was washed with fresh benzene-alcohol mixture and finally with alcohol. The loss in weight gives the amount of fatty and waxy matters present in OBF.

To determine the amount of pectic matter the dewaxed fiber was heated with 0.5% ammonium oxalate solution at $70-80^\circ\text{C}$ for 72 hours (fiber liquor ratio 1:100) in a heating mantle. The fiber was filtered, washed thoroughly with hot distilled water.

The dewaxed and depectinised fibers were dried at 105°C and treated with 72% H_2SO_4 in the ratio of 1 g fiber per 15 ml of solution with frequently stirring at ordinary temperature. The mixture was allowed to stand for 2 hours and then diluted to 3% acid concentration. After refluxing the mixture for 4 hours, it was allowed to stand for whole night and filtered through a sintered glass funnel and then washed thoroughly with hot distilled water. The constant weight of the residue gives the amount of lignin content in OBF and this fiber is called delignified fiber.

The dewaxed and depectinised fiber was heated with 0.7% NaClO_2 solution

buffered at pH 4 at $90-95^\circ\text{C}$ for 90 minutes maintaining the fiber liquor ratio 1:50. To reduce chlorite action the fiber was treated with 2% sodium metabisulphite solution for 15 minutes and then washed thoroughly with distilled water. The fiber treated with sodium chlorite is called the chlorite holocellulose and also bleached fiber.

The dried chlorite holocellulose was treated with 24% KOH solution for 4 hours with occasional stirring in the fiber liquor ratio of 1:100. By this treatment hemicellulose goes into solution and α -cellulose is separated by filtration, washed thoroughly with 2% acetic acid solution and finally with distilled water. The amount of α -cellulose thus obtained was deducted from the weight of holocellulose taken gives the amount of hemicellulose.

Methods

Bleached OBF was dyed with Direct Green 27 and Acid Orange 52 in a dyeing bath at 60°C for 50 minutes, pH of dye bath was adjusted at 4. In order to measure dyeability as well as percentage of dye absorption of the bleached fiber was examined in a Spectronic 20 [8].

Percentage of dye absorption =

$$\frac{D_o - D_e}{D_o} \times 100$$

Where D_o and D_e are the original and exhausted dye bath concentration, respectively.

The raw, bleached and dyed OBFs were attached on a flat board separately, and placed on the roof of a building for exposure in open air under the sun for 300 hours without any protection from weather. Care was taken so that the effect of weathering would be confined to the surface of the fiber. After every 50 hours, the fibers were collected from the board for experimental purpose.

Measurements

Certain amount of cellulose was dissolved in phosphoric acid at $55\pm 2^\circ\text{C}$ in a 250 ml conical flask with continuous stirring. When the solution becomes clear, it is suddenly cooled to 30°C in a cold water bath. Finally the solution was filtered on a buchner funnel. The Prepared solution was taken quickly in a viscometer upto the mark. The viscometer then placed in a thermostatically controlled water bath at $30\pm 0.2^\circ\text{C}$. To attain the temperature, viscometer was allowed for five minutes in water bath. Then viscosity is determined by measuring the flow time of cellulose solution. The viscosity of phosphoric acid was also measured at $30\pm 0.2^\circ\text{C}$. The relative viscosity of the cellulose in phosphoric acid solution obtained by following formula [9].

$$\eta_r = \frac{\eta_{\text{solution}}}{\eta_{\text{solvent}}}$$

Then the specific viscosity of the solution is

$$\eta_{\text{sp}} = \eta_r - 1$$

Intrinsic viscosity $[\eta]$ is then obtained by plotting η_{sp}/C against C and extra plotting to zero concentration, where C is the concentration in g/100 ml of the solution.

From the measured intrinsic viscosity the molecular weight of α -cellulose calculated by the equation: $[\eta] = KM^a$,

Where the values of k and a are 2.77×10^{-4} and 0.60 respectively.

Torsee's Schopper type-05-100 tensile tester was used for measuring the tensile strength of OBF. Tensile strength of each specimen was measured according to International Standard [10].

The colorfastness of raw, bleached and dyed OBFs was assessed with Grey Scale. In this investigation the specimens were exposed in open air under sunlight for 300 hrs and after every 50 hrs the fastness was assessed by comparing the change in

color of the specimen with that of the standard [11].

IR spectra of virgin and treated OBF (exposed in sunlight) were recorded with FTIR-500 spectrometer (JAIST, Japan) with a 2 cm^{-1} resolution and 20 scans using KBr pellet technique. The dried fiber samples were crushed to a size finer than 20 meshes before pelleting with KBr. The test KBr pellet contained about 1% powdered fiber.

RESULTS AND DISCUSSION

Okra bast, a multicellular fiber was analyzed and the estimated average chemical compositions of OBF (*Abelmoschus esculentus* variety) are 67.5 % α -cellulose, 15.4 % hemicellulose, 7.1 % lignin, 3.4 % pectic matter, 3.9 % fatty and waxy matter and 2.7 % aqueous extract. It is clear that the main constituents of OBF are α -cellulose, hemicellulose and lignin and the rest are very minor in proportion, so render a little influence to the structure of OBF. Therefore, the structure of α -cellulose, hemicellulose and lignin and the mode of combinations that exist in between themselves are dominating the structure of OBF.

Determination of molecular weight of okra bast fiber

The physico-chemical properties of macromolecule depend as well as vary with molecular weight. Mechanical properties increase and reach a limiting value with molecular weight. The intrinsic viscosity of raw fiber, bleached fiber and cellulose were 2.7, 2.32 and 2.18 and respective molecular weights were 1.517×10^5 , 1.303×10^5 and 1.225×10^5 . It was seen that, the molecular weight of raw OBF is greater than bleached fiber and α -cellulose. It is due to the bleaching action of sodium chlorite. During bleaching lignin is removed and breakdown of cellulosic chain occurred by oxidizing of cellulose. So molecular weight decreases with the decrease of cell length of fiber. α -cellulose is pure cellulose produced after removing of all constituents. Thus, cell

length became smaller and molecular weight is reducing in proportion [12].

Dyeing behavior of Okra bast fiber

Dye absorption of a fiber depends on the availability of total external surface of the fiber pores and cavities in a fixed amount of fiber and the attraction forces between the fiber and dye ions. Figure 1 shows the dye exhaustion of direct dye and acid dye by bleached fiber. It was observed that dye absorption of bleached OBF increase with the increase of dye concentration upto a certain value then it decreases. The total available pores or cavities in fixed amount of fiber for dye absorption is constant, the dye absorption capacity is limited. As a result, manifesting a decrease in percent absorption of the dye corresponding to an increase of initial dye concentration. On the other hand, the

presence of more dye ions in dye bath hinder the absorption of dye by the fiber whereas rare ions favour it. It is to be mention that with the increase of dye concentration, the absolute quantity of the absorbed dye increases too, while the relative quantity diminishes [13].

From the Figure 2 it is observed that absorption of dye increases with the increase of electrolyte concentration in the dye bath upto saturation absorption and after this, no change in absorption occurs on further addition of electrolyte. The electrolyte (Na_2SO_4) dissociates into Na^+ and SO_4^- ions in aqueous solution of dyes. The positively charged Na^+ ions migrate towards the negatively charged fiber surface and thus, by reducing the negatively charged potential between the similar charged fiber surface and colored dye anions that improve dye ability.

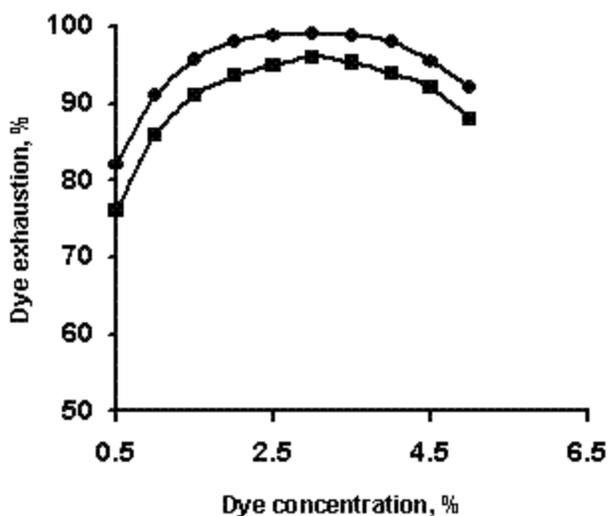


Figure 1: Plots of dye exhaustion vs dye concentration for bleached okra bast fiber; (○) Direct green 27; (◻) Acid orange 52.

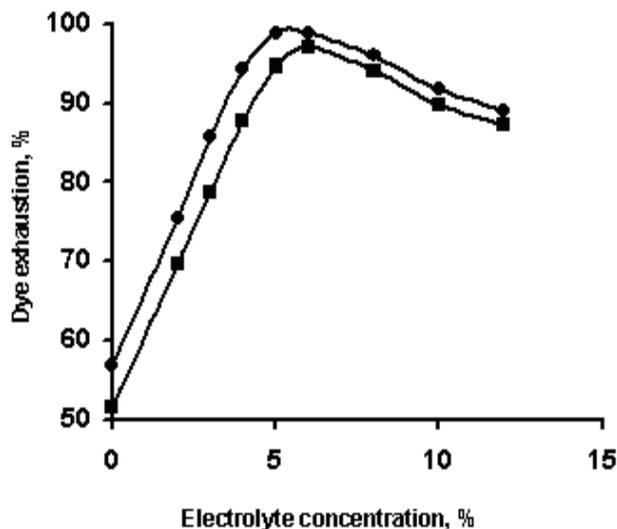


Figure 2: Plots of dye exhaustion vs electrolyte concentration for bleached okra bast fiber; (●) Direct green 27; (◻) Acid orange 52.

From the Figure 3, it is observed that the absorption of dye by bleached OBF increases with the increase of temperature and become maximum at 70°C for Direct Green 27 and 80°C for Acid Orange 52. The increase of dye diffusion co-efficient at higher temperature is first of all caused by greater kinetic energy acquired by the dye molecules and by the increase of frequency amplitude of thermal oscillations in the separate sections of the substrate molecular chains, which provokes an increase in the

number of the pores in the fiber structure and their diameter. A reduction in the affinity of the dye for the fiber at an increase of the temperature is also considerable importance. At very high temperature the dye absorption was decreases. The possible explanation is that the temperature above from equilibrium absorption the rate of the backward reaction becomes so much larger than that of forward reaction so the bond between dye-fiber are broken and absorption becomes fall down.

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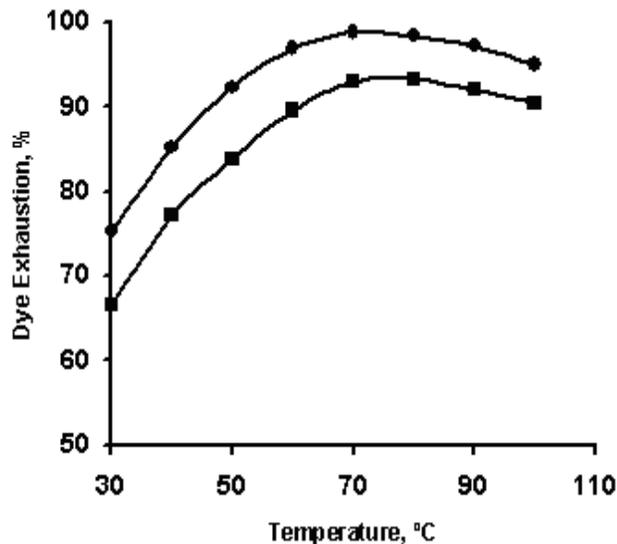


Figure 3: Plots of dye exhaustion vs bath temperature for bleached okra bast fiber; (○) Direct green 27; (□) Acid orange 52.

Color fastness of Okra bast fiber on sunlight

From the Table 1 it is observed that the color fadeness of raw OBF is higher than bleached fiber. The presence of greater amount of lignin in raw OBF may have a possible enhancing action of decoloration compared to bleached OBF. Yellowing of okra fiber is caused by lignin, which on the

action of light (UV) undergoes photochemical degradation with some loss of methoxyl groups leading to the formation of ortho-diphenols and ultimately to orthoquinones in the sequence of reactions. The formation of quinones is claimed to be the main cause of yellowing of OBF [14]. As the bleached fiber contain relatively smaller quantity of lignin, the yellowing of bleached OBF become slow and lesser.

Table 1: Color fastness and change in color of raw and bleached okra bast fiber on exposure to sunlight in air.

Exposure period (hours)	Fastness grade and color of raw fiber	Fastness grade and color of bleached fiber
00	5 (Dull white)	5 (white)
50	4-5 (Yellowish white)	4 (Slight yellowish)
100	4 (Light yellow)	4 (Slight yellowish)
150	3-4 (Light pale yellow)	4 (Slight yellowish)
200	3 (Pale yellow)	3-4 (Slight yellowish)
250	2-3 (Dull yellow)	3 (Slight yellowish)
300	2-3 (Dull yellow)	3 (Slight yellowish)

Effect of sunlight on the strength of Okra bast fiber

The strength loss of raw and bleached OBF was 2.11 and 1.72% respectively. But at the end of 300 hours exposure, the strength loss of raw fiber is 31.07% which is considerably greater than

that of bleached fiber i.e.21.9%. This happens due to the presence of more lignin content in raw fiber than bleached fiber. Lignin having phenolic and alcoholic hydroxyl groups, which is highly sensitive to the action of sunlight. The reaction involved in photochemical degradation of:

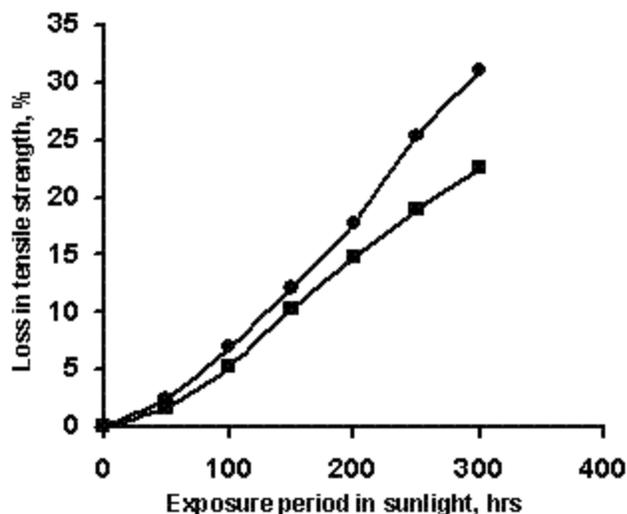


Figure 4: Plots of loss in tensile strength vs exposure period of okra bast fiber on exposure to sunlight in air; (□) raw fiber; (○) bleached fiber.

OBF mainly oxidative in nature and prolonged exposure to light, the constituent α -cellulose chain are gradually attacked and ultimately breakdown into smaller fragments, as a result, breaking strength of OBF is decreased. In polymeric materials, usually strength is contributed by crystalline region. But in OBF, strength is not only contributed by crystalline region but also in the amorphous region, because lignin acts as cementing material and forms incrustants with hemicellulose. On exposure to light in presence of oxygen this incrustants are broken down due to involvement of lignin in the reactions and hence breaking strength is reduced.

IR spectra and oxidative degradation mechanism of Okra bast fiber

The oxidative degradation of cellulose has been reported to proceed by a free radical chain reactive mechanism to form carbonyl (C=O) group via a reaction involving hydroperoxide group formation [15-16].

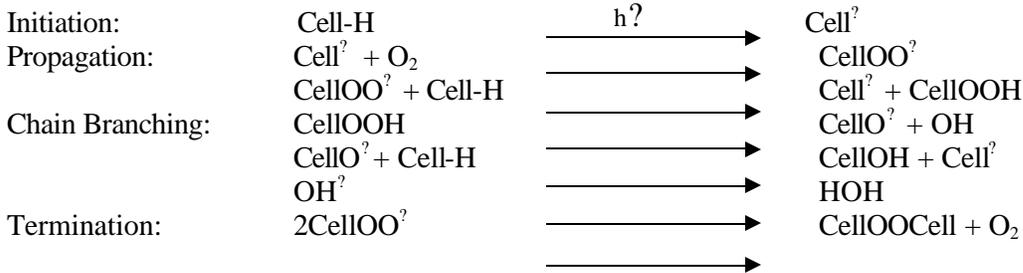
The comparative IR spectrum of OBFs are shown in Figure 5. From the figure it is clear that an additional peak at

the wave number 1735 cm^{-1} appears and become higher at long period of exposure in sunlight while the virgin okra fiber does not has any. So, it can conclude that, by the action of sunlight the cellulose molecule of okra fiber degraded and a new addition product obtain by the formation of C=O or COOH radicals.

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The oxidation reaction can be envisaged as proceeding in three stages i.e. initiation, propagation and production of degradation products. The formation of alkyl radical by the abstraction of hydrogen occurs first. The reaction of this alkyl radical with oxygen leads to the production of peroxy radical which propagates the chain reaction by intramolecular H-abstraction from cellulose chain. The abstraction reaction produced hydroperoxide, which is responsible for the autocatalytic nature of the oxidation. The hydroperoxide formation could split homolytically to give an alkoxy radical and a hydroxy radical. A β -scission could then result in cleavage of the glycosidic bond and alkoxy radical on the neighbouring glucose unit. This postulated mechanism of oxidative bond scission is shown in Scheme 1.

The possible mechanism may as follows:



Scheme 1: Chain scission reaction in okra bast fiber

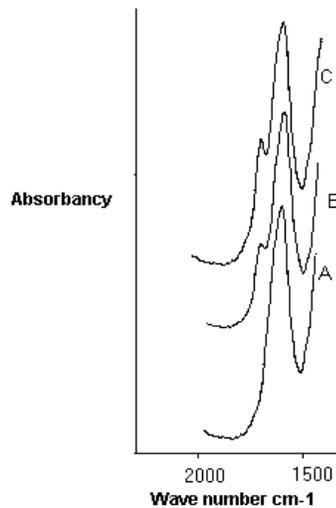


Figure 5: IR Spectrum of okra bast fiber (partially)

- A. Virgin okra bast fiber
- B. Okra bast fiber after exposing for 50 hours
- C. Okra bast fiber after exposing for 300 hours

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CONCLUSION

The okra bast fiber possesses an excellent quantity of cellulose, hence it can be used as cellulosic raw materials in cellulose based industries. It also contains low percentage of lignin, which is responsible for yellowing and photochemical degradation. It is a high molecular weighted compound. So, it has some developed properties like color fastness, tensile strength etc. The dye exhaustion property of this fiber is also better. In Philippine OBF is used as a textile

fiber. But in Bangladesh we reject this bast as wastages, which can be great source of textile fiber. The plantation of okra is not much expensive and we can collect this bast from the discarded after vegetable extraction. If we can collect and properly use the okra bast by isolating fiber from it then a good prospect must be awaited for our country.

ACKNOWLEDGEMENT

The authors are grateful to Professor M. Terano, Department of Chemical Materials Science, Japan Advanced Institute of

Science and Technology, Hokuriku, Japan for his cooperation to measure the samples using IR, which were used in this investigation.

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