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Original Research Article

Investigation of Functional Properties Changing in Different Chemical Treatments of Various Cellulosic Fibers Using FTIR

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Abstract: The effects of combined scouring-bleaching and reactive dyeing were investigated by characterizing the functional groups changed of cotton, betel nut, banana and jute fibers using caustic soda, Hydrogen peroxide and reactive dyestuffs. FTIR ATR spectroscopy provided a fast and semi-quantitative assessment of the removal of pectin, lignin, Hemicelluloses, oil, waxes etc on those fibers surface by comparing the changes in intensity of the carbonyl peak induced by Hydrogen peroxide and caustic soda treatments well as bond changing in reactive dyeing around 4000 cm^{-1}. Above all fibers are not reacting identically during changing impurities and covalent bond forming between cellulose and reactive dyes.

Keywords: Combined scouring-bleaching, FTIR ATR spectroscopy, Caustic soda, Hydrogen peroxide and Reactive dyestuffs

INTRODUCTION

Cellulose fibers are fibers made with ether or esters of cellulose, which can be obtained from the bark, wood or leaves of plants or from a plant-based material. Besides cellulose, these fibers are compound of hemicelluloses and lignin, and different percentages of these components are responsible for different mechanical properties observed. But, in this study four vegetables cellulosic natural fibers (cotton, betel nut, banana and jute) are chemically characterized by FTIR.

Cotton Betel nut Banana Jute

Fig-1: Vegetables cellulosic natural fibers

Cotton fiber is mainly composed of cellulose (94%) with some non cellulosic components surrounding the cellulose core. These non-cellulosic components are waxes (0.5-1%), pectin (0.1-0.2%), Oil (0.5-1%) and protein (1.5%) and are mainly found in the cuticle layer and the primary wall which are the outermost layers of the cotton fiber [1-3]. Jute composed of cellulose (65.2%), Hemi-cellulose

(22.2%), Lignin (12.5%), Water Soluble matter (1.5%), Fat and Wax (0.6%) [4-5].The betel nut husk is the fibrous part of the fruit, which equals to approximately 60-80% of total volume and weight of betel nut. The husk part is the outer cover of the areca fruit [6-7]. The anatomy of betel nut husk can be divided into 3 zones. The outer layer is covered with cuticle, the middle layer is where the fiber is enclosed, and the hard and stony inner layer is the nut part [8]. Betel Nut fiber is mainly composed of cellulose (53.20%), Hemicelluloses (32.98%), (Lignin7.20%), Fat and wax (0.64%), Ash (1.05%) and other materials (3.12%) [8]. Chemical compositions of Banana fiber are cellulose (31-35%), hemicelluloses (14-17%) and lignin (15-16%) [9-11]. Combined Scouring and Bleaching process of the raw cotton, Jute, Betel Nut and banana fibers removes these impurities and natural color for better dyeing. Combined scouring and bleaching remove waxes, pectin, natural pigments and other impurities from the natural cellulosic fibers by treating the fiber in hot sodium hydroxide solution [12]. The impurities removed from those fibers have been characterized. In most cases, they isolate there moved impurities to identify which impurities are removed from the raw [13]. However, not many methods have been reported to characterize the combined scoured and bleached fabric for remaining waxes and other impurities. Few years ago, waxes, lignin and pectin left on the fibers were detected with some color forming reagent [13]. These methods are only qualitative and not exact. We would introduce very simple and fast method to measure the impurities left on fibers by FT-IR attenuated total reflectance (ATR) spectroscopy. This is a surface sensitive technique which has penetration depth of a few micrometers [14]. Since the waxes and other impurities of cellulose are located in outermost layer of these natural cellulosic fibers, this technique would be very useful to characterize small amount of these impurities. The change of the impurities constituents (namely cellulose, hemicelluloses, lignin, protein, Pectin, oil & wax etc) of these natural cellulosic fibers were tested by FTIR as well as these combined scoured bleached fibers dyed with reactive dyes to investigate further chemical bond forming [15]. L. Yusriah and S.M. Sapuan explored the potential of betel nut husk fiber as reinforcing material in polymer composites [16]. F. Carrillo and X. Colom investigated structural FTIR analysis and thermal characterization of cellulose originated of lyocell and viscose fibers [17]. Noureddine Abidia and Luis Cabrales experimented the changes in the cell wall and cellulose content of developing cotton fibers investigated by FTIR spectroscopy [18]. Barbara Hinterstoisser and Margaretha A kerholm determined effect of fiber orientation in dynamic FTIR study on native cellulose [19]. Wei-ming Wang and Zai-sheng Cai reckoned the changes in composition, structure, and properties of jute fibers after chemical treatments [15]. Juan I. Mora and Vera A. Alvarez showed extraction of cellulose and preparation of nano-cellulose from sisal fibers [20]. Anna Carlmark and Eva Malmstro investigated atom transfer radical polymerization from cellulose fibers at ambient temperature [21]. The present paper focuses chemical behavior of different sources cellulose, such as cotton, jute, banana and Betel nut fibers, by presenting a comparative analysis with the combined scouring & bleaching and reactive dyeing, using Fourier transform infrared spectroscopy (FTIR).

MATERIALS AND METHODS

Materials

CIS Cotton fibers were obtained from Silver line Spinning Ltd and Bangladeshi betel Nut, banana & white-B jute were extracted and collected from different sources. Combined scouring-bleaching carried out for all fibers by treating @ of Fino wet OSR (Detergent): 1 gm/l, Caustic soda: 5 gm/l (Dosing at 60^0C for 10 min), Hydrogen Peroxide: 3 gm/l, Albafluid C (Anti-creasing agent): 1 gm/l, Arboquest 340 (Sequestering agent): 0.5 gm/l, Time: 40 min, Temperature: 90^0C. Neutralization (Acetic acid: 1 gm/l Time: 10 min Temperature: 60^0C). Reactive dyeing is carried out @ of GSS (Leveling agent): 0.12 gm/l, Arboquest 340 (Sequester): 1 gm/l, Albafluid C (Anti-creasing): 1 gm/l,Glauber salt: 35 gm/l, Reactive Turquoise Blue H2GP: 3%, Sodium carbonate: 15 gm/l (at50^0C for 20 min.),Time: 30 min, Temperature: 60^0C. Cold wash (Acetic acid: 1.5 gm/L, Time: 15 min, Temperature: 40^0C). Hot wash (Jintex /Detergent: 1 gm/l, Time: 20 min. Temperature: 90^0C). Cold wash (Time: 10 min. Temperature: 40^0C).

Methods

Three samples pallet were prepared for each of four fibers (raw fiber sample pallet, combined scouring-bleaching sample pallet and reactive dyed sample pallet). Thermo Nicolet Nexus FTIR spectrophotometer equipped with liquid nitrogen cooled MCT-A detector was used. Transmission mode spectra (32 scans, 4 cm^{-1} resolution) were measured with KBr pellets of powdered fibers. FT-IR ATR spectra (216 scans, 4 cm^{-1} resolution) were collected with a MIRacle, single reflection horizontal ATR accessory (PIKE instruments) having a diamond ATR crystal fixed at incident angle of 45^0. A 10 mm×10 mm piece of each fiber pallet were mounted on top of ATR crystal and pressed gently by a pre-mounted sample clamp. ATR effect and atmospheric contributions from carbon dioxide and water vapor were corrected by the Omnic software.

RESULT AND DISCUSSIONS

Infrared transmittance peaks (cm^{-1}) comparison of untreated cotton, combined scoured-bleached cotton and reactive dyed cotton

In case of cotton fibers, The FT-IR transmission spectra could tell the differences of the raw fibers and combined scoured- bleached fibers because it measured most of the chemical compositions of fibers. Waxes, oil, protein, pectin etc of cotton fibers components are located on the rim of cotton fibers. FT-IR ATR spectroscopy which is a surface sensitive technique can measure the presence of waxes and pectin semi-quantitatively. Fig. 2 shows the FT-IR ATR spectra of the raw cotton, combined scoured- bleached cotton and Reactive dyed cotton. Following peaks prove the presence of residual waxes (2903.096 cm^{-1}) in the combined scoured- bleached cotton and new bond formation after reactive dyeing (2324.927 cm^{-1}).

Table 1: FTIR Report analysis of Cotton Fiber

Bond type	Raw cotton (C-1001)	Combined scoured-bleached cotton(C-1002)	Reactive dyed cotton (C-1003)
–OH stretching	3368.505	3433.871	3417.476
C–H vibration	2904.626	2903.096	2906.016
C=C stretching	1638.608	1639.947	1638.934
C–C stretching	-	1059.924-1101.759	1115.552
C≡ C/C ≡N	-	-	2324.927

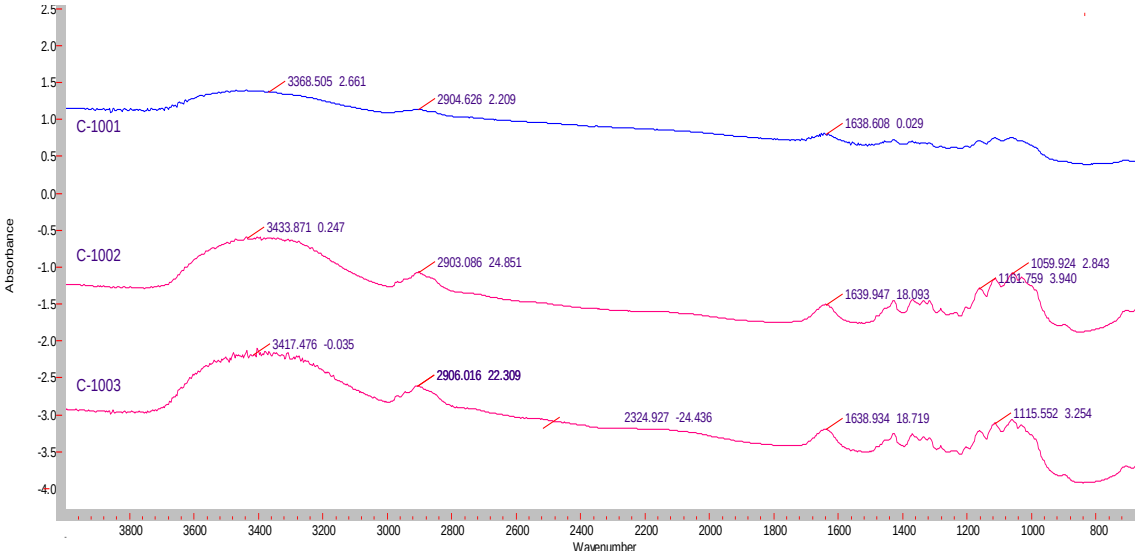

Fig-2: FTIR Graphical Representation of Cotton Fiber

Infrared transmittance peaks (cm^{-1}) comparison of untreated jute, combined scoured-bleached jute and reactive dyed jute

Hemicelluloses, Lignin, pectin etc of jute fibers components are both surface and inter-fibril space of the jute fibers. Fig.3 shows the FT-IR ATR spectra of the raw jute, combined scoured- bleached jute and Reactive dyed jute. Following peaks verify the presence of hemicelluloses (1724.587cm^{-1}) in the fresh jute and Lignin deceases gradually (1637.756 cm^{1}, 1643.771 cm^{-1}, 1606.269 cm^{-1}) and natural pigment bond breaking after Combined scouring-bleaching.

Table1: FTIR Report analysis of Jute Fiber

Bond Type	Raw Jute (BT 1)	Combined scoured-Bleached Jute(BT 2)	Reactive dyed Jute (BT 3)
–OH stretching	3380.663	3381.072	3412.847
C–H vibration	2918.214	2903.527	2911.662
C=O stretching	1724.587	-	-
C=C stretching	1637.756	1643.771	1606.269
C≡ C/C ≡N	-	2144.979	-
C–O	1060.190	1059.934	1061.669

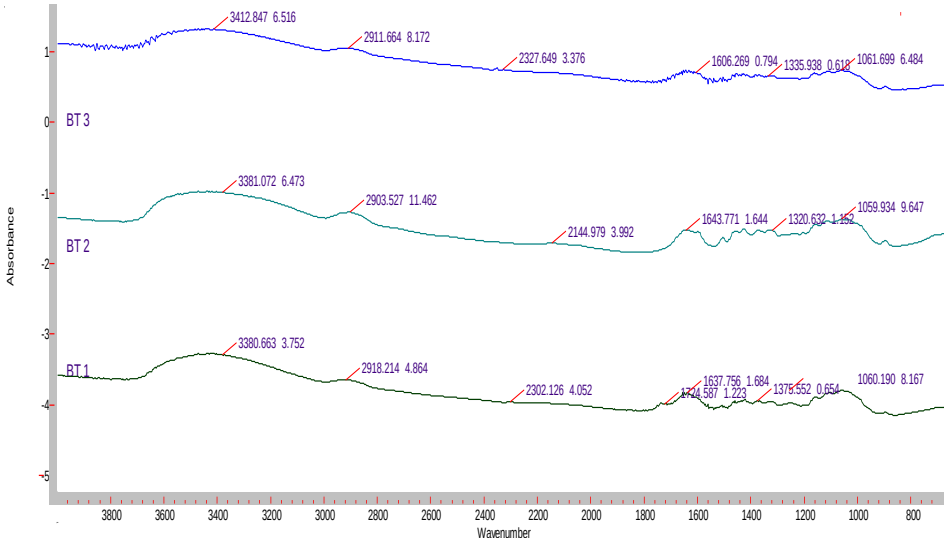

Fig-3: FTIR Graphical Representation of Jute Fiber

Infrared transmittance peaks (cm^{-1}) comparison of untreated betel nut, combined scoured-bleached betel nut and reactive dyed betel nut

Fig.4 shows the FT-IR ATR spectra of the fresh Betel Nut, combined scoured- bleached Betel Nut and Reactive dyed Betel Nut. Following peaks argue the presence of residual lignin & Hemicelluloses and natural pigment bond breaking after Combined scouring-bleaching but negligible bond formation after reactive dyeing.

Table 3: FTIR Report analysis of Betel Nut Fiber

Bond type	Untreated betel nut (Betel 1)	Combined scoured-bleached betel nut (Betel 2)	Reactive dyed betel nut (Betel 3)
–OH stretching	3469.613	3413.897	3486
C–H vibration	897.684	1463.530	1459.386
C=C stretching	1638.868	-	1637.217
C–H stretching	2918.744	-	-
–OH Bending	1376.723	-	-
C≡ C/C ≡N	2170.501/2360.327	2360.741	

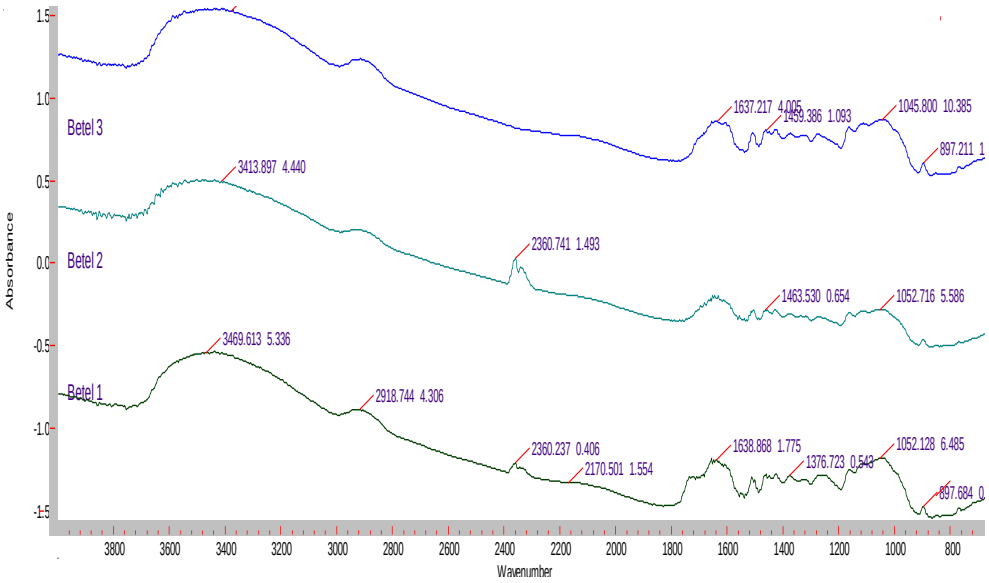

Fig-4: FTIR Graphical Representation of Betel Nut Fiber

Infrared transmittance peaks (cm^{-1}) comparison of untreated banana, combined scoured-bleached banana and reactive dyed banana

Fig.5 shows the FT-IR ATR spectra of the fresh Banana, combined scoured- bleached Betel Nut and Reactive dyed Banana. Following peaks evidence the presence of residual lignin & Hemicelluloses and natural pigment bond breaking after Combined scouring-bleaching. Significant changed is visualized in –OH stretching (3572.031 cm^{-1}, 3411 cm^{-1}, 408 3375.471 cm^{-1}).

Table 4: FTIR Report analysis of Banana fiber

Bond type	Raw banana (BN1)	Combined scoured-bleached banana(BN2)	Reactive dyed banana (BN3)
–OH stretching	3572.031	3411.408	3375.471
C=C stretching	898.858	897.513	897.688
N-H stretching	-	2915.203	2910.178
C≡ C/C ≡N	2333.323	2140.482	2150.917
C=N	1643.277	1639.702	1643.471
C–O	1112.268	-	-

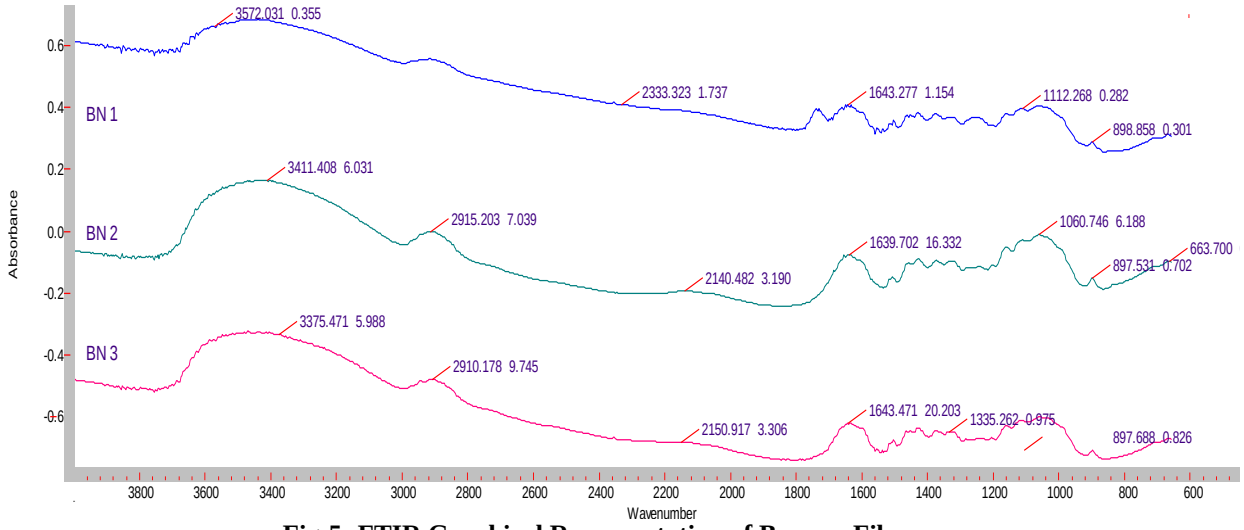

Fig-5: FTIR Graphical Representation of Banana Fiber

CONCLUSION

FTIR ATR spectroscopy test has been reported that cotton, jute, banana and betel nut fibers contain different amount of cellulose and various impurities which are not identically sensitive under hot caustic and hydrogen per oxide treatment as well as covalent bonding in reactive dyeing. It is proved that 100% wax cannot be removed from cotton through caustic treatment and residual lignin must be present in banana, jute and betel nut after combined scouring- bleaching. It is also graphically represented that cotton formed covalent bond strongly but less strong in jute, banana and betel nut consecutively after reactive dyeing. It is suggested that reactive dye is not fairly suitable for betel nut, if it is required deep shade. It is strongly recommended that lignin from banana, jute and betel nut can be removed, if their cellulose totally disintegrated into micro fibril.

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