Infra-Red Studies OfDagger, Lady's Finger And Betel Nut Fibres

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ABSTRACT :

Inthis article, the crystallinity of Dagger, Lady's finger, Betel nut fibres are studied from the assigned bands in Infra-red (IR) absorption spectroscopy. With a double beam infra-red spectrometer (Perkin Elmer, model 1600 series FTIR), the infra-red absorption spectrograms for the fibrous samples were recorded. It is seen that all the natural fibres of present investigation are semicrystalline in nature and this nature of crystallinity does not affect much due to heat treatment at lower temperature.

KEYWORDS -Crystallinity, annealed sample, Infra-red (IR) absorption spectroscopy, assigned bands

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INTRODUCTION

Infra-red (IR) absorption spectroscopy has served as a powerful tool in the study of the basic structure and chemical properties of molecules. The analysis of IR absorption spectra is greatly facilitated by the fact that certain grouping in the molecules gives rise to characteristics group frequencies which in most cases are not strongly affected by neighbouring atoms. Surface and fine structural characteristics of cotton fibre were investigated by Bora and Talukdar with the help of Infra-red spectroscopy and found that, the structural behaviour with setup of chemical bonding of the cellulosic cotton fibre remain unaltered in caustic and thermal treatments [5]. Structural properties of cellulose materials were investigated by several workers.Nadiger et.al. studied the structural and chemical properties of natural Indian silks with the help of Infra-red spectroscopy.Baruah at.el.studies these properties of Muga, Eri and Pat fibres [6]. For estimation of lignin in jute fibre, B.N.Bandyopadhyay et.al. used the IR spectroscopic method. Rowen and Plyler describe the effects of deuteration, oxidation and hydrogen bonding of the Infra-red spectrum of cellulose. Mann and Marrinan have carried out detailed studies of deuterium exchange with cellulose and shown that the exchange takes place first in the amorphous regions. The crystalline regions are affected only after considerable time. In all the forms of cellulose, there is no free O-H stretching frequency in the crystalline region and all the hydroxyl groups are hydrogen bonded. For detailed information on the structural properties of cellulosic materials, several workers, have used the potassium bromide disc technique to record the Infra-red spectra. The Infra-red absorption spectrum of cellulose shown many bands that are poorly resolved or of lower intensity. But second derivative Infra-red spectra may provide more detailed on structural information. Pandey studied the derivative Infra-red spectra of cotton and ramie cellulose. Talukdar et.al. studied the functional groups present in some plant fibres and the effect of chemicals and temperature on the behaviour of these groups with the help of Infra-red spectroscopic method. [1], [2], [3],[4].

Infra-red technique provides a spectrum containing large number of absorption bands from which a wealth of information can be derived about the structure of an organic compound. The absorption of infrared radiation can be expressed either in terms of wavelength or in wave number. Mostly infra-red spectra of organic compounds are plotted as percentage transmittance against wave number.

In terms of wave number, the ordinary infra-red region covers 4000 cm^{-1} to 667 cm^{-1}, and in terms of wavelength it is from 2.5 micron to 15 micron. The region below 1500 cm.-1 is called fingerprint region. This region is important for comparing the identity of the two compounds and also for the detection of certain functional groups.[6], [7], [8], [9].

SAMPLE PREPARATION

For recording of infra-red absorption spectrograms, the fibre samples were finely cut into particle size and then passed through a 100-mesh sieve. About 1 mg. of these samples were thoroughly mixed with 100 mg KBr and ground manually for about 10 minutes in an Egat Morter. The mixture was then pressed on the anvil of Die and pellet was prepared under high pressure in carver hydraulic press to be used for infra-red spectroscopic study. [9], [10].

With a double beam infra-red spectrometer (Perkin Elmer, model 1600 series FTIR), the infra-red absorption spectrograms for the fibrous samples were recorded in the wave number region 4000 cm^{-1} to 500 cm^{-1} using NaCl prism.

The essential components of the spectrometer are source of infra-red radiation, monochromator, detector, and recorder. The optical path and the principal components of the spectrometers are shown in fig.3.8. Here two beams of infra-red radiation emanating from the same source are allowed to pass through the sample and a reference material respectively. When the energy of the radiation transmitted by the reference and the sample cell is the same, the detector does not produce any signal. If the sample absorbs radiation, there will be inequality in the two transmitted beams and a pulsating electrical signal is produced. The frequency of the signal is equal to the frequency of the beam slitting by the selector mirror. After amplification and rectification of the signal, it is used to move the attenuator which cuts down the amount of transmitted radiation from the reference beam until the energy balance between the reference and sample beam is restored. The reduction in the reference beam necessary for the energy balance is directly proportional to the absorption of radiation by the sample. By synchronizing the attenuator with the pen of the recorder, a pen-trace of the absorption spectrum of the sample on the paper-chart can be obtained by rotating the Littrow mirror, changes in the frequency of radiation reaching the detector is obtained and thereby the sample absorption changes. In this way, the whole spectrum of the radiation can be scanned.

OBSERVATION AND RESULTS

The infrared spectrogram of different plant fibres such as Dagger, Lady's Finger and Betel Nut fibre are shown in figures 1.1 to 1.3. The spectra of all the three fibres show similar absorption peaks along with similar wave numbers. The important infrared spectroscopic bands in wave number and their assignment are given in table 1.1.

The IR spectra of Normal and heated (annealed from 473 K) samples of Dagger and Lady's Finger and Betel Nut reveal the following facts:

In the 3600-3130 cm^{-1} OH stretching region, the Lady's Finger fibre shows a band of lower intensity than the Dagger fibres. As water absorption increases the height of the OH band to a certain extent, thus the water absorption of the Lady's Finger fibre is the lower than the other fibres. The area of these broad bands decreases for heated samples. This may occur due to dehydration of the fibres.

The band at about 2940 cm^{-1} is due to the C-H stretching vibration and is found to be similar for Dagger and Lady's Finger. This remains same for unheated and heated samples.

The presence of ester carbonyl group of the three fibres are observed in the region 1760 cm^{-1}. For the C=O stretching the peak arises at 1730 cm^{-1} which observed for all three fibres.

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The other major band observed at 1650 cm^{-1} range for all the fibres is due to adsorbed water. The height of this band peak decreases for annealed and quenched samples. This result indicates that the hygroscopy of plant fibres is significantly tempered under thermal treatments.

For all the three fibres the peak arises at the region for from 1600 cm^{-1} to 1430 cm^{-1} which are due to presence of lignin in all of the fibres. In the lignin spectrum the specific absorbance peak at 1506 cm^{-1} is attributed to the skeletal vibrations of aromatic ring and the peak at 1471 cm^{-1} is attributed to C-H asymmetric deformation.

The next important band occurs at 1430 cm^{-1} and is caused by CH_2 symmetrical bending and this band is related with the crystallinity.

In the region 1430 cm^{-1} to 1022 cm^{-1}, there is a series of sharp band which are due to various C-H, OH and C-O stretching and bending motion. In this region the Dagger fibre shows some distinct peaks and Lady's Finger fibre shows very weak peaks.

The band at 900 cm^{-1} which has been attributed to the CO group frequency. Dagger shows sharp band in this region.

The peak at the region 1430 cm^{-1} considered to associated with the crystalline structure, whereas those at 1370 cm^{-1} are associated with amorphous structure.

It has also been observed from the figure that the crystalline peak height is large than the amorphous peak height for the Lady's Finger fibre and slightly more for the Dagger fibre which indicate that the crystallinity (%) is more than 50% for both the fibres. The slightly more intense crystalline peak than the amorphous peak indicate that the crystallinity percent is slightly more than 50%. From the figure it also observed that the difference of crystalline peak height and amorphous peak height for Lady's Finger fibre is more than that of Dagger fibre, which indicate that the crystallinity percent is more for Lady's Finger fibre than that of Dagger fibre.

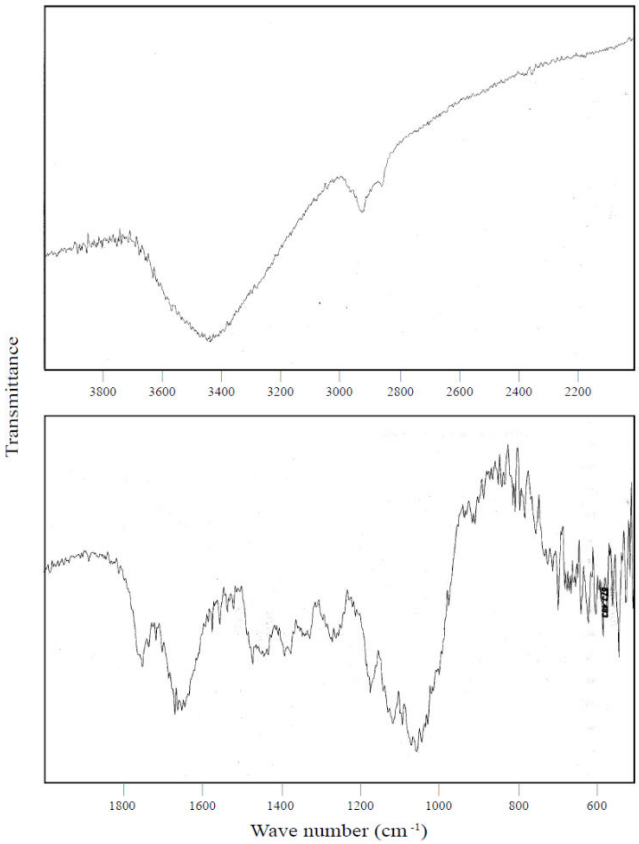

Figure 1.1(a) - Infrared spectrogram of dagger fibre

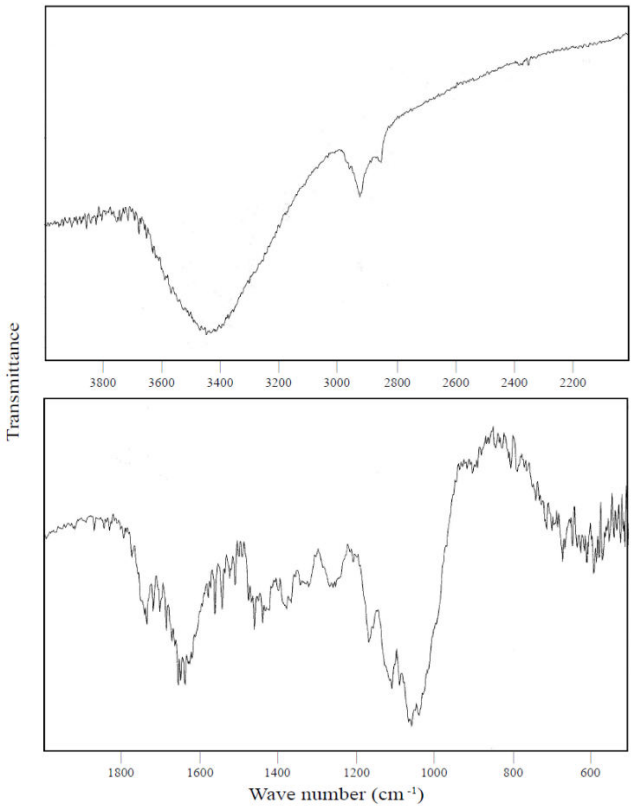

Figure 1.1(b) - Infrared spectrogram of annealed dagger fibre

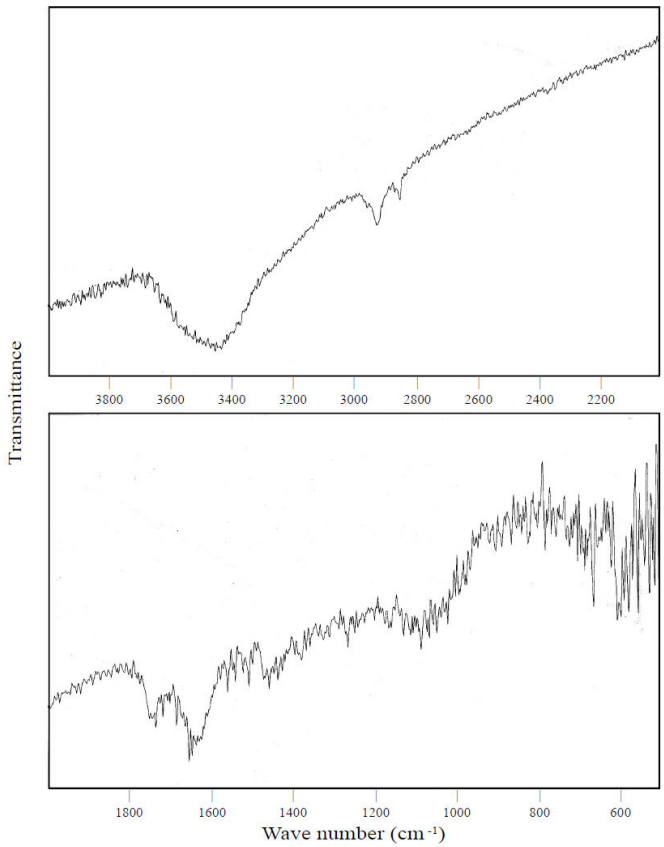

Figure 1.2 (a) -Infrared spectrogram of lady's finger fibre

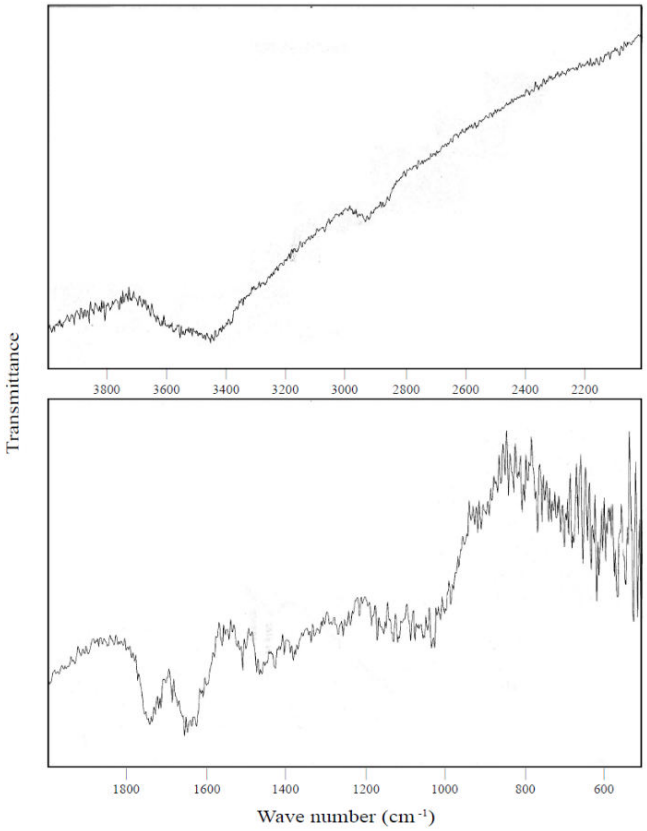

Figure 1.2(b) - Infrared spectrogram of annealed lady's finger fibre

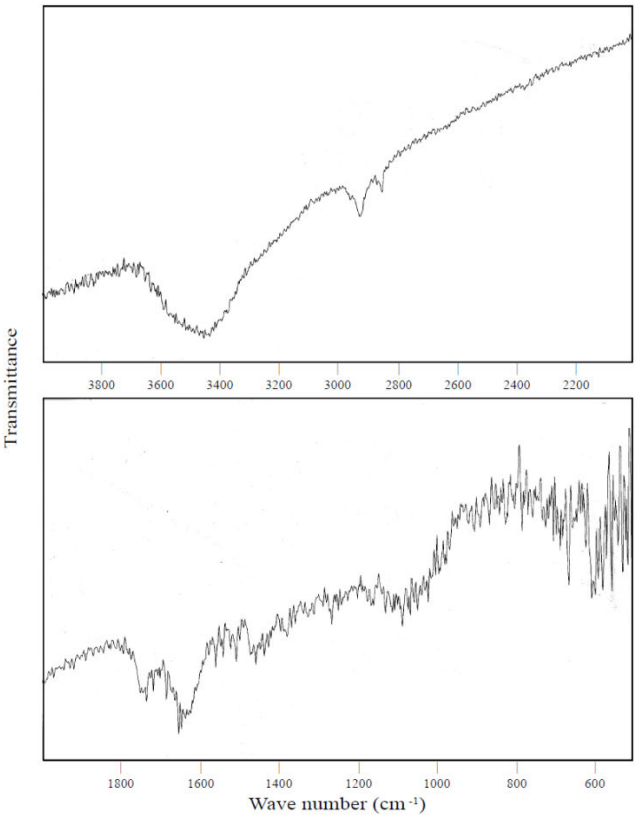

Figure 1.3 (a) -Infrared spectrogram of betel nut fibre

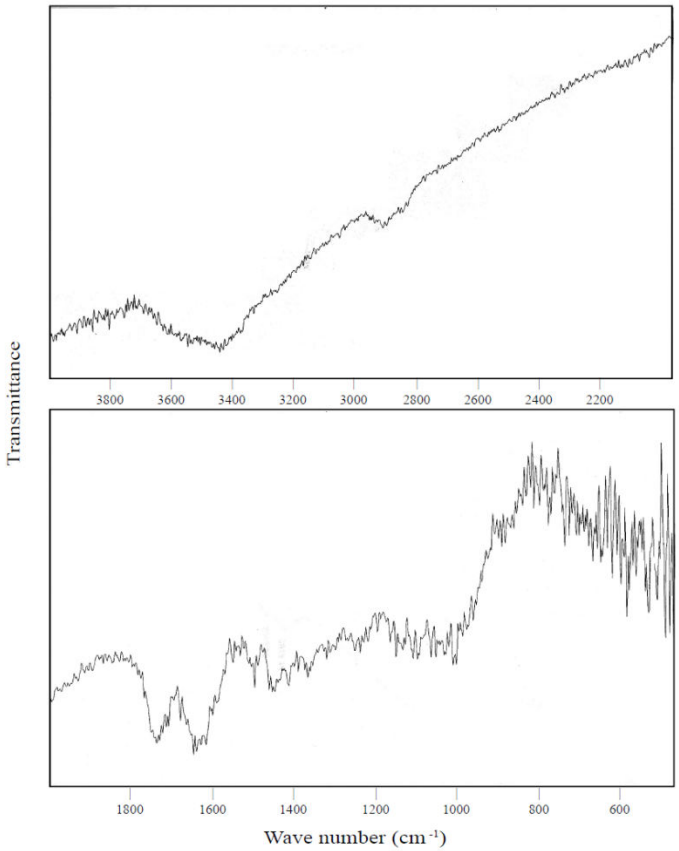

Figure 1.3 (b) - Infrared spectrogram of annealed betel nut fibre

Table 1.1: Assignment of functional groups of IR Spectra of the fibres:

Approximate position of Band Maximum in wave number			Assignment of observed bands
Dagger	Betel Nut	Lady's Finger	
3600	3700	3650	Free OH and bonded
3130	3100	3130	O-H stretching
2937	2950	2950	C-H stretching
2151	2150	-	C=C stretching
1738	1740	1750	C=O stretching
1659	1660	1650	Absorbed H_2O
1500	1500	1500	COO – ion stretching
-	1475	1475	CH_2 symmetric bonding
1430	1440	1425	
1371	1375	1360	C-H deformation
1161	1167	-	C-O-C stretching
1105	1100	1100	
1057	1050	-	OH bonding
906	900	900	C-O stretching
-	850	825	
700	-	740	
-	675	650	O-H deformation
600	600	600	
550	550	550	

The plant fibres Dagger, Betel Nut and Lady's Finger are dark white in colour. The fibres, when degummed, become slightly soft and bright due to removal of the gums embedded in the amorphous region of the fibres. when heated, the fibre samples pass through a sequence of colour change from white to yellow to reddish brown at 250 ºC and then to almost black at about 280 ºC.
From these investigations, it is seen that all the natural fibres of present investigation are semicrystalline in nature and this nature of crystallinity does not affect much due to heat treatment at lower temperature. However, crystallinity is not observed above around 573 K. It is inferred that the degree of crystallinity has changed due to heating.
The variation in crystallinity and corresponding physio-chemical properties may be due to the water molecules absorbed in amorphous regions and in the crystalline part of the hygroscopic fibres. Crystallinity of all these fibres increase slightly due to heat treatment.

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