



Crystallographic Behavior Of Dagger, Lady's Finger And Betel Nut Fibres Observed From X-Ray Diffraction

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ABSTRACT

In this article we studied the crystallinity of Dagger, Lady's finger, Betel nut fibres as observed by X-ray diffraction method. The degree of crystallinity percent (DC%) of raw and degummed Dagger, Lady's Finger and Betel Nut fibres are measured and calculated from X-ray diffractograms. The identical diffractograms of the samples indicate that all these three plant fibres possess similar crystallographic characteristics.

KEYWORDS - crystallinity, degumming, X-ray diffractogram, annealed, quenched

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207

I. INTRODUCTION

The crystallinity of fibre has a very important role in describing the thermophysical properties. Reports have been found about the studies on the crystallinity in plant fibres. Some worker studied the crystallinity of silk and plant fibres by X-ray diffraction method.[1]

Guinier, Kratky and other coworkers developed a method for the determination of the size, shape and distribution of the crystallites from the study of the intensity distribution of the continuous X-ray scattering at very low angles near the incident beam. By using the method Heyn estimated the average diameter of the crystallites of raw and chemically treated plant fibres. Herzog and Jancke shows that cellulose crystalline structure of ramie, jute, cotton etc are same.[4]

Many studies have been made on the degumming trial of some natural fibres[2],[5]. Comprehensive studies on plant fibres such as Dagger, Lady's finger, Betel nut which are readily available in North-East India are not adequate and these have motivated us to investigate some thermophysical properties of these fibres.

The degumming trials and heat treatment carried out on these fibres by the method describe in different publish research papers for

II. SAMPLE PREPARATION FOR THIS STUDY

For the ladies finger fibre, the stems are retted in water in order to free the bark and to loosen the fibre from the gummy and other adhering materials. After the retting is complete, the fibre are stripped from the stems. Then the fibre is washed in clean water and dried.

The Dagger fibre is prepared from the leaf. To obtain the fibres, the green leaves are cut close to the trunk of the plant, the spines are cut off from the sides, and the pulp is scraped away, leaving the fibres. Then it was washed and dried.

The betel nut fibres that surround the seeds of betelnut are collected from the seeds. Then it retted and after the retting is completed the fibre is washed in clean water and dried in the sun.

III. CHEMICAL DEGUMMING AND HEAT TREATMENT

Chemical treatments are recommended for the removal of gummy materials from natural fibres [10]. Therefore, a series of degumming trials on some natural fibres such as Dagger, lady's finger, Betel Nut, etc have been carried out using the commonly available degumming chemicals, so as to assess the extent to which these fibres could be made finer.

different plant fibres [2], [5], [6], [9]. The heat treatment consists of annealing and quenching. After degumming trials and heat treatment, the

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corresponding fibres were used for different physical, crystallographic and industrial properties.

IV.OBSERVATION AND RESULTS

In the data taken for this study, X-ray diffraction of the fibres were carried out in the APD 1700 (Automatic powder diffractometer) of Philips make, coupled with PM8230-A one line recorder unit. This was used for both annealed and quenched samples.

For taking the reading the automatic powder diffractometer is checked with standard Si samples before inserting the samples inside the Goniometer. Then the APD was set to scan the samples from 5 θ to 30 θ .

The value of 2θ corresponding to the different peaks were measured from the diffractogram records, plotted by the One Line Recorder.

The raw fibres of Lady's Finger and Dagger are pale white while that of Betel Nut are light yellow. After degumming with sodium hydroxide, the fibres of Lady's Finger and Betel Nut changed their colour to pale brown and brown respectively. On the other hand, the dagger fibres become bright after degumming. Due to degumming with sodium hydroxide the ramie fibres become snow white.

The change of colour of the fibres may be due to the change of degree of crystallinity of the fibres due to degumming and heat treatment. The change of colour with the change of degree of crystallinity is observed by some worker for the case of some soil samples.

The X-ray diffractogram of the natural plant fibre such as Dagger, Lady's Finger and betel nut are shown in figure 1.1.1 to 1.1.3. From the figures it is observed that the peak positions of all the plant fibres are almost the same. This indicates that the structural conformations of all the plant fibres are the same.

In figures 1.1.1(a), the X-ray diffractograms of raw and degummed Dagger are displayed. The X-ray diffractograms of annealed and quenched Dagger samples at different thermal conditions are displayed from figure 1.1.1(b) to 1.1.1(d).

In figures 1.1.2(a), the X-ray diffractograms of raw and degummed Lady's Finger are displayed. The X-ray diffractograms of annealed and quenched Lady's Finger samples are displayed from figure 1.1.2(b) to 1.1.2(d).

In figures 1.1.3(a), the X-ray diffractograms of raw and degummed Betel Nut are displayed. The X-ray diffractograms of annealed and quenched Betel Nut samples are displayed from figure 1.1.3(b) to 1.1.3(d).

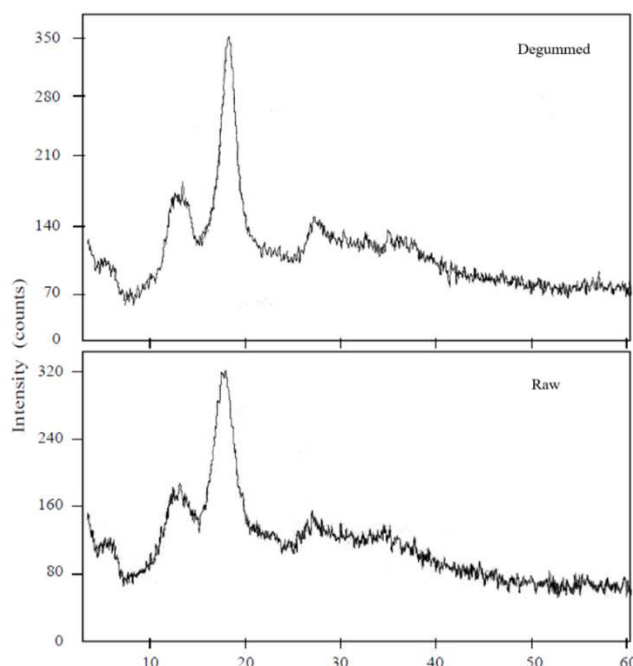


Figure 1.1.1(a): X-ray thermograms of dagger fibres

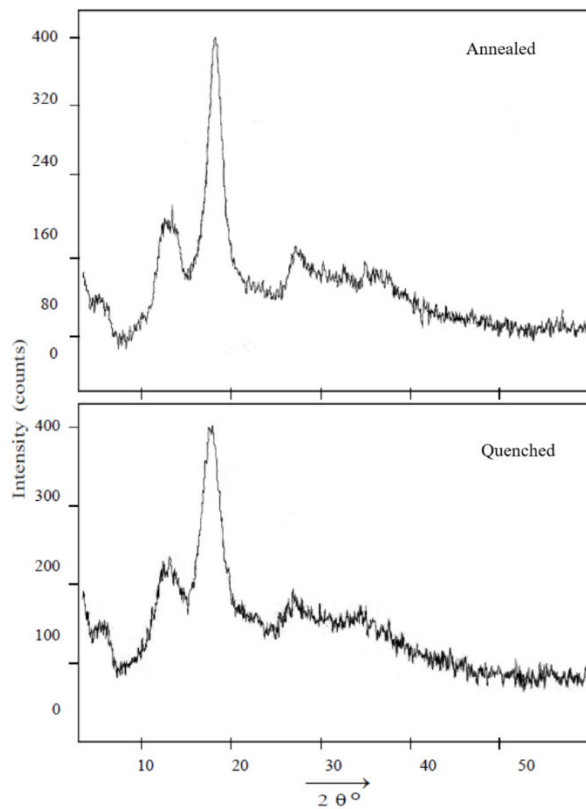


Figure 1.1.1(b): X-ray thermograms of dagger fibres, annealed and quenched from 373 K

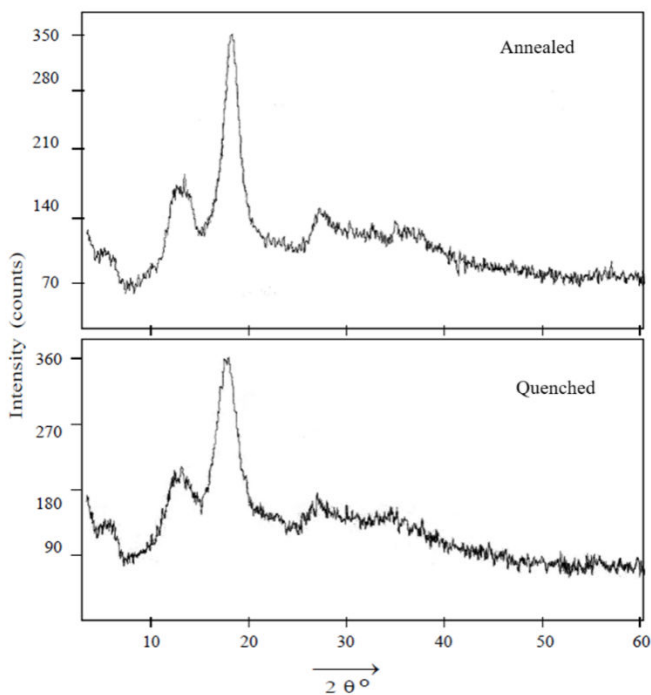


Figure 1.1.1(c): X-ray thermograms of dagger fibres, annealed and quenched from 473 K

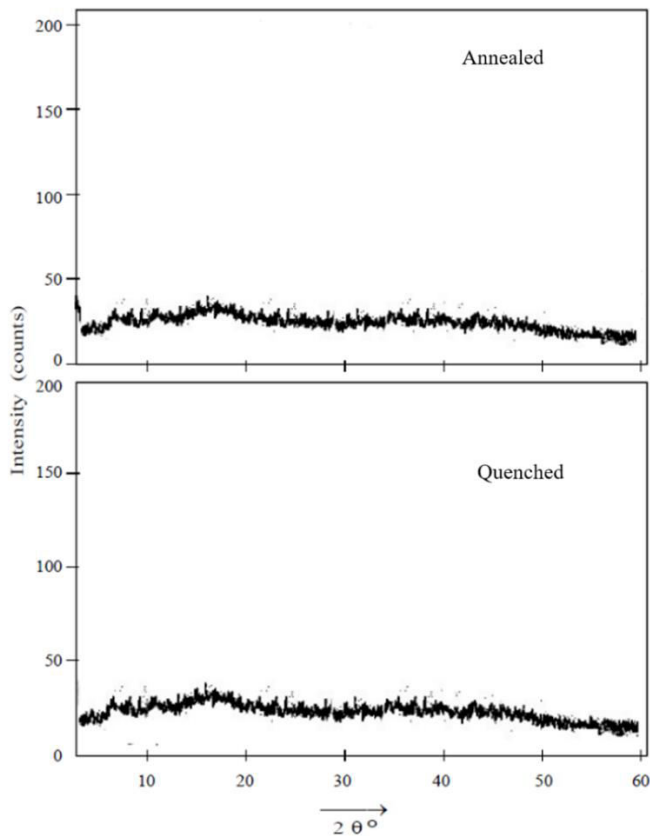


Figure 1.1.1(d): X-ray thermograms of dagger fibres, annealed and quenched from 573K

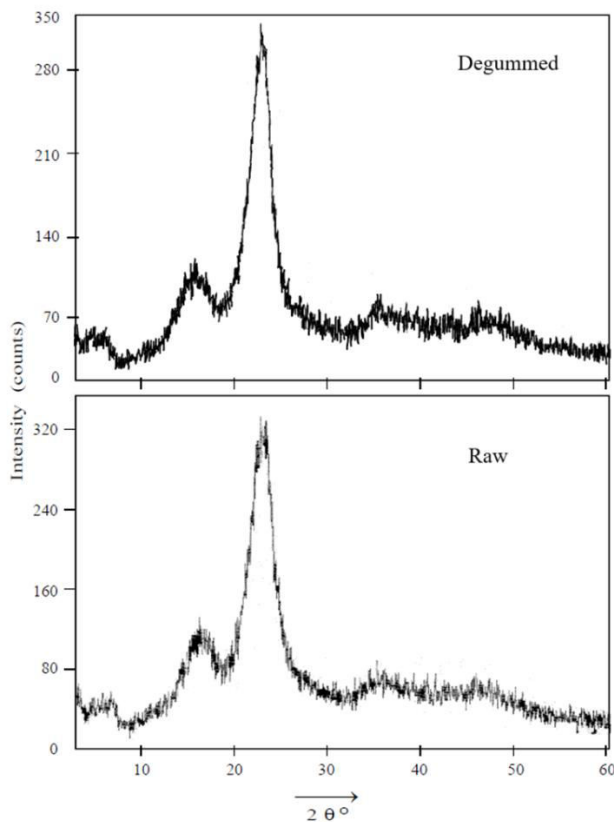
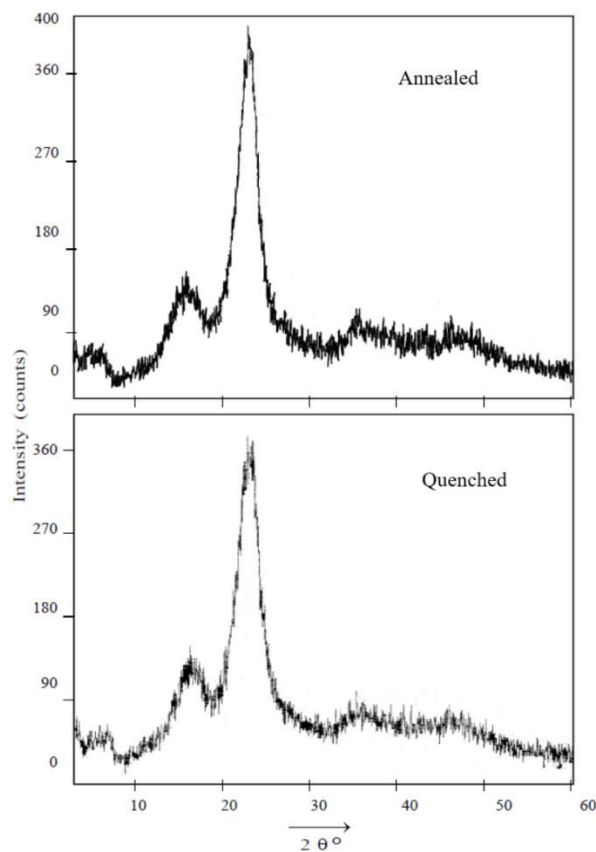


Figure 1.1.2(a): X-ray thermograms of lady's finger fibres



211

Figure 1.1.2(b): X-ray thermograms of lady's finger fibres, annealed and quenched from 373 K

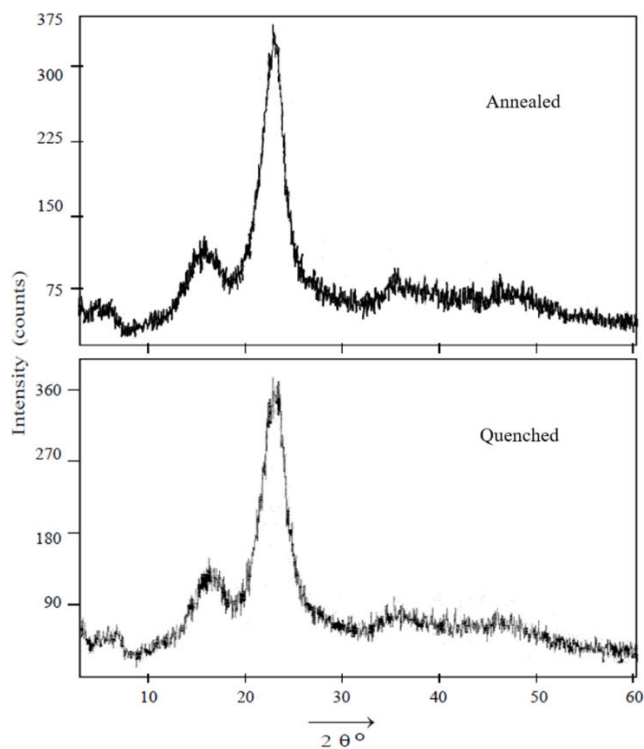


Figure 1.1.2(c): X-ray thermograms of lady's finger fibres, annealed and quenched from 473 K

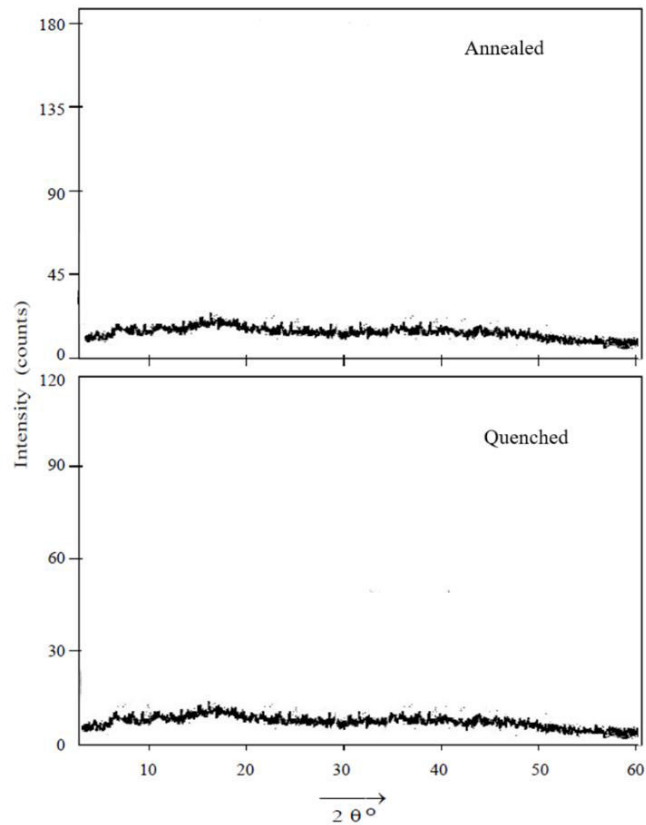


Figure 1.1.2(d): X-ray thermograms of lady's finger fibres, annealed and quenched from 573 K

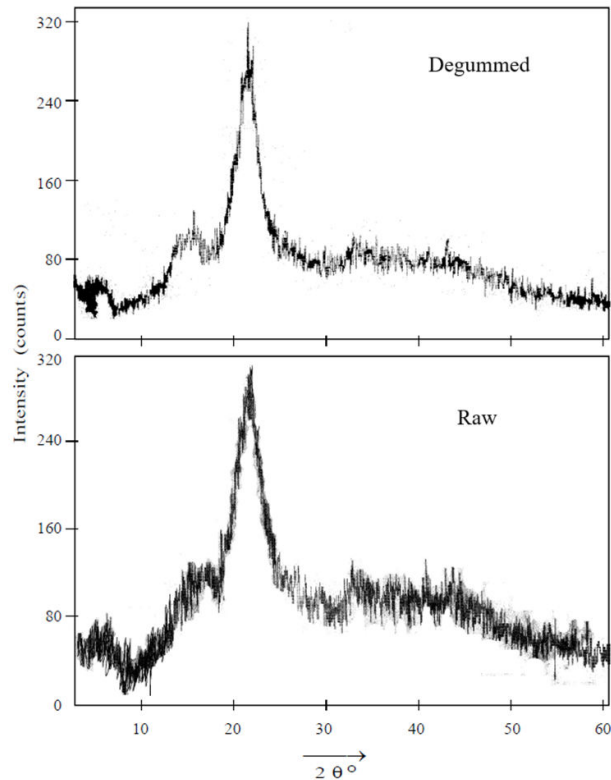


Figure 1.1.3(a): X-ray thermograms of betel nut fibres.

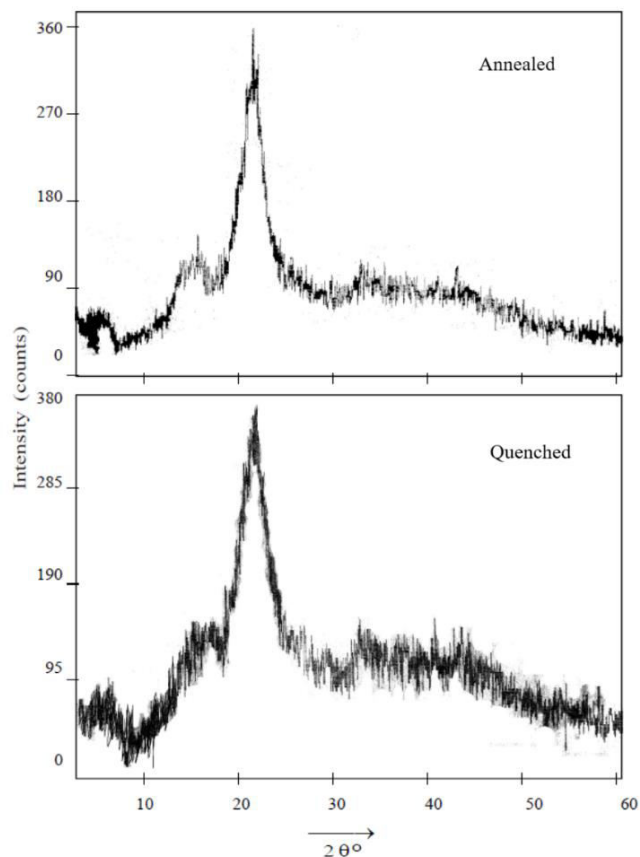


Figure 1.1.3(b): X-ray thermograms of betel nut fibres, annealed and quenched from 373 K

213

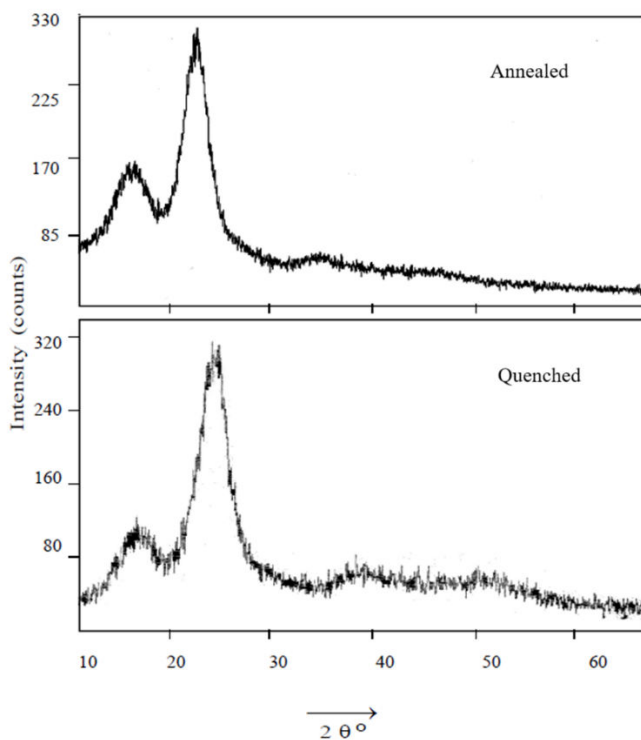


Figure 1.1.3(c): X-ray thermograms of betel nut fibres, annealed and quenched from 473 K

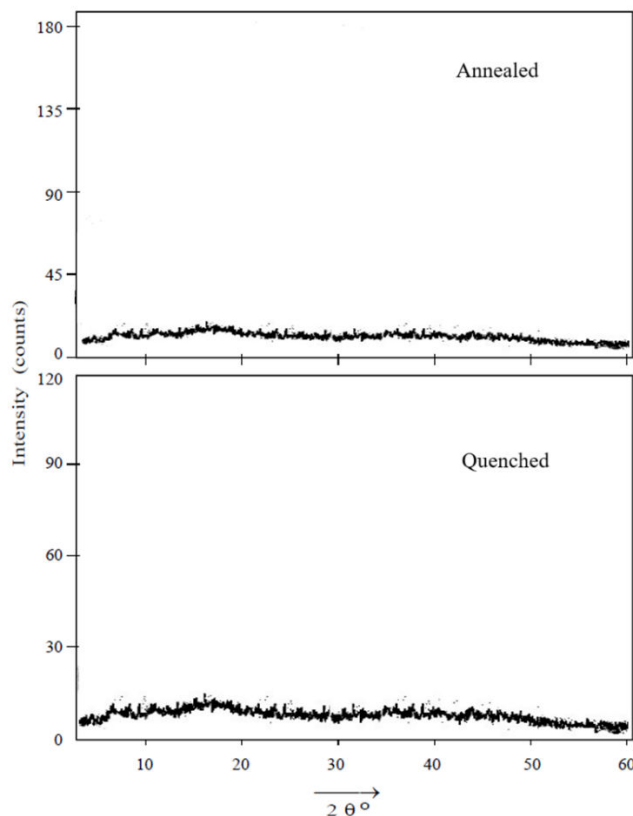


Figure 1.1.3(d): X-ray thermograms of betel nut fibres, annealed and quenched from 573K

Table 1.1.1: Degree of Crystallinity (DC%) of Raw, Degummed, annealed and quenched Dagger fibres:

Specimen	Crystallinity (%)
Raw	71.11
Degummed	73.33
Annealed from 373 K	74.87
Quenched from 373 K	74.54
Annealed from 473 K	72.17
Quenched from 473 K	71.76
Annealed from 573 K	-
Quenched from 573 K	-

Table 1.1.2: Degree of Crystallinity (DC%) of Raw, Degummed, annealed and quenched Lady's Finger fibres:

Specimen	Crystallinity (%)
Raw	74.38
Degummed	75.65
Annealed from 373 K	77.37
Quenched from 373 K	77.37
Annealed from 473 K	75.71
Quenched from 473 K	75.42
Annealed from 573 K	-
Quenched from 573 K	-

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Table 1.1.3: Degree of Crystallinity (DC%) of Raw, Degummed, annealed and quenched Betel Nut fibres:

Specimen	Crystallinity (%)
Raw	73.33
Degummed	74.38
Annealed from 373 K	76.71
Quenched from 373 K	76.71
Annealed from 473 K	74.15
Quenched from 473 K	74.46
Annealed from 573 K	-
Quenched from 573 K	-

The X-ray diffractograms shows the increase of intensity of all the observed fibres after degumming.

The degree of crystallinity percent (DC%) of raw and degummed Dagger, Lady's Finger and betel nut fibres are measured and calculated from X-ray diffractograms and these calculated values of degree of crystallinity percent (DC%) listed in table 1.1.1. to 1.1.3. These values were calculated on the basis of the model used by Teh and Rudin. From the table it is observed that the crystallinity percent (DC%) increases for all the plant fibres after degumming. These may be due to the increase of cellulose II in the fibre due to degumming. This means, when the fibres are degummed, the gum portion of the fibre is removed and the macromolecules are rearranged in more ordered form.

Again, from the table it is observed that the degree of crystallinity percent (DC%) is highest for Lady's Finger fibres and least for Dagger fibres.

The identical diffractograms of the samples indicate that all these three plant fibres possess similar crystallographic characteristics. The macromolecules are linked up end to end in chains and the binding forces between these packed molecules cause unidirectional orientation of the crystallites in the fibre. The lattice parameters and space groups of the samples are computed from front reflection and oscillation X-ray photographs. From these crystallographic data, it is inferred that these plant fibres possess orthorhombic unit cell.

The data from diffractograms listed in table 1.1.1, 1.1.2 and 1.1.3 show that it is almost immaterial whether the fibrous samples are annealed or quenched (from 373K or 473 K). However, the crystallinity increases slightly when annealed from 373 K. This might be due to presence of water molecules in the amorphous region of hygroscopic fibres, which restrict the alignment of the chain molecules and thus give rise to strains in the crystalline region. On removal of water molecules, the restrictive force being withdrawn, the molecular chains are free to arrange. The decrease in intensity of the basal reflections heated to 473 K attributes to the gradual degradation of crystallinity of the fibre samples. Disappearance of basal reflections at about 573 K indicates the transformation of fibres from crystalline to amorphous state.

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