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XRD Studies of Some Cellulose Fibers at Different Temperatures

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ABSTRACT
The X-ray diffractogram of raw ginger (Zingiber officinale), turmeric (Curcuma longa) and eri (Samia cynthia ricini) fibers are taken at different temperatures (313, 353, 393, 433 and 473 K) in an X-ray powder diffractometer (Phillips X'Pert Pro) with high temperature attachment (Anton Paar, HTK-16). It is found that the percentage of degree of crystalinity first increases from its value at 313K to the value at 353K, and then decreases at higher temperature for all the experimented raw fibers. The degree of crystallinity finds to be maximum at temperature 353K. The degree of crystallinity of chemically treated ginger fibers also finds to decrease with the rise of temperature with maximum at 353K. The degree of crystallinity of ginger fiber finds to increase due to degumming at a certain temperature.

Keywords: *Zingiber officinale, Curcuma longa, Samia cynthia ricini, Degree of crystallinity, x-ray diffractogram.*

I. INTRODUCTION

The cellulose fibers such as ginger, turmeric and eri silk fibers are semicrystalline in nature. The thermophysical properties of these fibers have great importance in textile technology. The thermal properties of various cellulose materials have been studied by various workers by Differential Scanning Calorimetry (DSC) method[1-6]. The action of absorbed water and influence of gummy matter on crystalline structure of some cellulose fibers has been studied[7-9]. The crystallographic behaviours of varioius fibers have been observed at room temperature as well as at different temperatures at which they are quenched and annealed[10-11]. In our present investigation, the raw fibers of ginger, turmeric, eri silk and degummed ginger fibers are subjected to X-radiation at different temperatures (313, 353, 393, 433 and 473K) to study their crystalline behaviour with rise of temperatures.

II. SAMPLE PREPARATION

Ginger (zingiber officinale), turmeric (curcuma longa) plants and eri (samia cynthia ricini) cocoons have been collected from various parts of the North-Eastern region of India. The fibers have been extracted from the middle portion of the stem of these plants and the eri cocoons. One part of the raw ginger fibers were immerged in a mixture of Benzene and Alcohol (ratio 1:1) for six hours and then washed with distilled water and dried at room temperature. This part is then divided into two parts, one of which is boiled with NaOH solutions at 6% concentration and the other at 2% concentration for 30 minutes. These fibers were then again washed with distilled water and dried normally. These raw and treated fibers are then cut into very fine powder. The samples so prepared were then taken into XRD analysis.

III. EXPERIMENTAL ARRANGEMENT

X-ray powder method is a suitable method to study the cellulose crystallinity, due to separate able diffraction from peaks caused by cellulose crystals and diffraction from amorphous cellulose, hemicellulose, lignin, pectin and minerals. For XRD, the crystalline diameter can also be estimated with X-ray diffraction based on the width of peaks representing the directions perpendicular to the fiber axis. The crystalline length can also be estimated on the width of peaks representing directions parallel to the fiber axis.

IV. MEASUREMENT

The finely cut fiber samples were subjected to X-radiation (Cu Kα, wavelength= 1.54 Å). The applied voltage and current to the X-ray tube were 40 KV and 20mA respectively. Programmable divergence slit with irradiated length of 8 mm were used during measurement with programmable receiving slit being used at the other end with slit height of 0.1 mm; the incident beam mask was fixed at 10 mm. the diffracted beam mask was not used. Diffracted beam soller slit was fixed at 0.04 rad. Pulse height distribution for the detector at upper and lower levels were fixed at 80% and 35% respectively. The scanning range was taken from 10^0 to 30^0.

The high temperature measurements were done in a X-ray powder diffractometer of Philips make (X'Pert Pro). The samples were scanned at constant temperatures 313, 353, 393, 433 and 473 K respectively with step size 0.04^0 and time per step 0.5 s. The samples were heated at the rate of 20^0 per minute.

V. RESULTS AND DISCUSSION

The X-ray diffractogram spectra of raw ginger, turmeric and eri fibers at different temperatures have been shown in fig. 1. The identical XRD spectra of the samples indicate that all these fibers possess similar crystallographic characteristics. It is observed that the peak positions of these fibers are nearly same due to heat treatment, whether the XRD peaks shifted to smaller angle when the measurements was carried out at 473 K. This shift could be attributed to the slight increase of the intersheet spacings of the crystals.

The interplaner spacings (d_{hkl}), peak intensity (I_{max} , I_{min}) and degree of crystallinity (DC) of raw ginger, turmeric and eri fibers from XRD spectra are shown in table 1. The method used to calculate the degree of crystallinity is same as used by Bora et al[11].

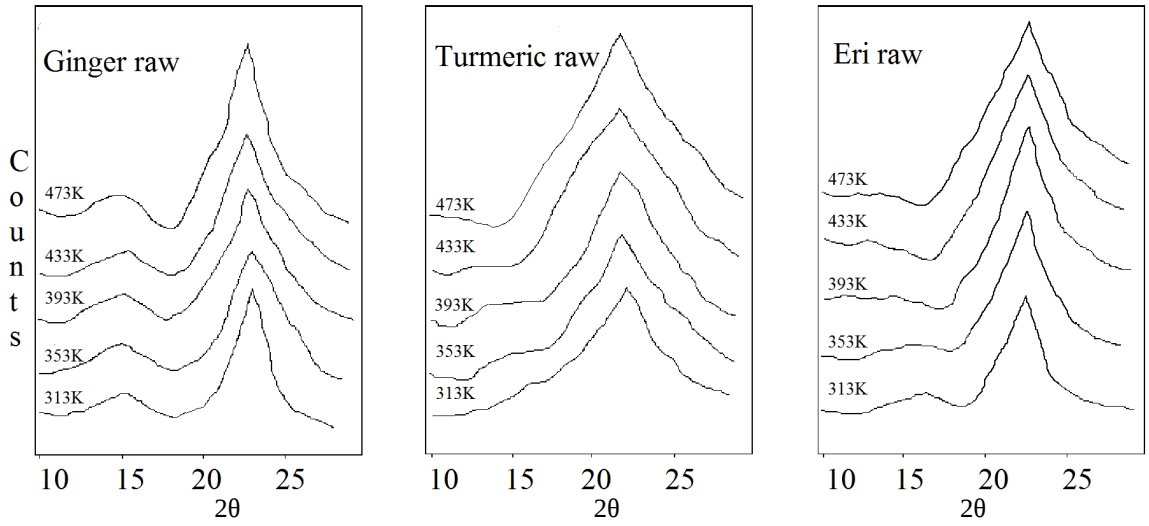

Fig 1- XRD of raw ginger, turmeric and eri fibers at different temperatures

Table -1: parameters obtained from XRD spectrum of raw ginger, turmeric and eri fibers

Samples	Temperature (K)	2θ (degree)	d_{hkl} (Å)	I_{max} (counts)	I_{min} (counts)	DC (%)
Ginger	313	22.8	3.9226	98	34	65.3
	353	22.5	3.9246	106	35	66.9
	393	22.4	3.9437	103	40	61.5
	433	22.4	3.9459	100	39	61.0
	473	22.3	3.9459	104	41	60.6
Turmeric	313	23.3	3.8752	86	27	68.6
	353	23.1	3.8862	94	28	70.2
	393	23.0	3.8871	94	31	67.0
	433	22.4	3.9322	90	32	64.4
	473	22.4	3.9322	87	32	63.2
Eri	313	23.4	3.6978	120	35	70.8
	353	23.2	3.7971	128	36	72.0
	393	22.7	3.7975	124	40	67.7
	433	22.5	3.7975	114	39	65.8
	473	22.4	3.7978	110	39	64.5

From the table it is seen that the degree of crystallinity decreases from 65.3% at temperature 313K to 60.6% at higher temperature for ginger raw fiber, however, it is found to increase at 353 K. For turmeric fiber the degree of crystallinity decreases from 68.6 at 313 K to 63.2 at higher temperature, with exception at 353 K. The degree of crystallinity of eri fiber decreases from 70.8 at 313 K to 64.5 at higher temperature. For all these fibers, it is found that the degree of crystallinity increases first from 313 K to 353 K, then decreases with the rise of temperature. The maximum value of DC% has been found at temperature 353 K. these might be due to presence water molecules in the amorphous region of hygroscopic fibers, which restrict the alignment of the chain molecules and gives rise to strain in the crystalline region. On removal of the water molecules the restrictive force being withdrawn, the molecular chain are free to rearrange. The decrease of degree of crystallinity for the fibers heated from 353 K to 473 K attributes gradual degradation of crystallinity of the fiber samples. These

findings coincide with the observation of workers for some annealed and quenched cellulose fibers[12].The interplaner spacings of all these fibers are almost same.

Fig.2 shows the comparison of XRD spectra of different raw and degummed ginger fibers at different temperatures.

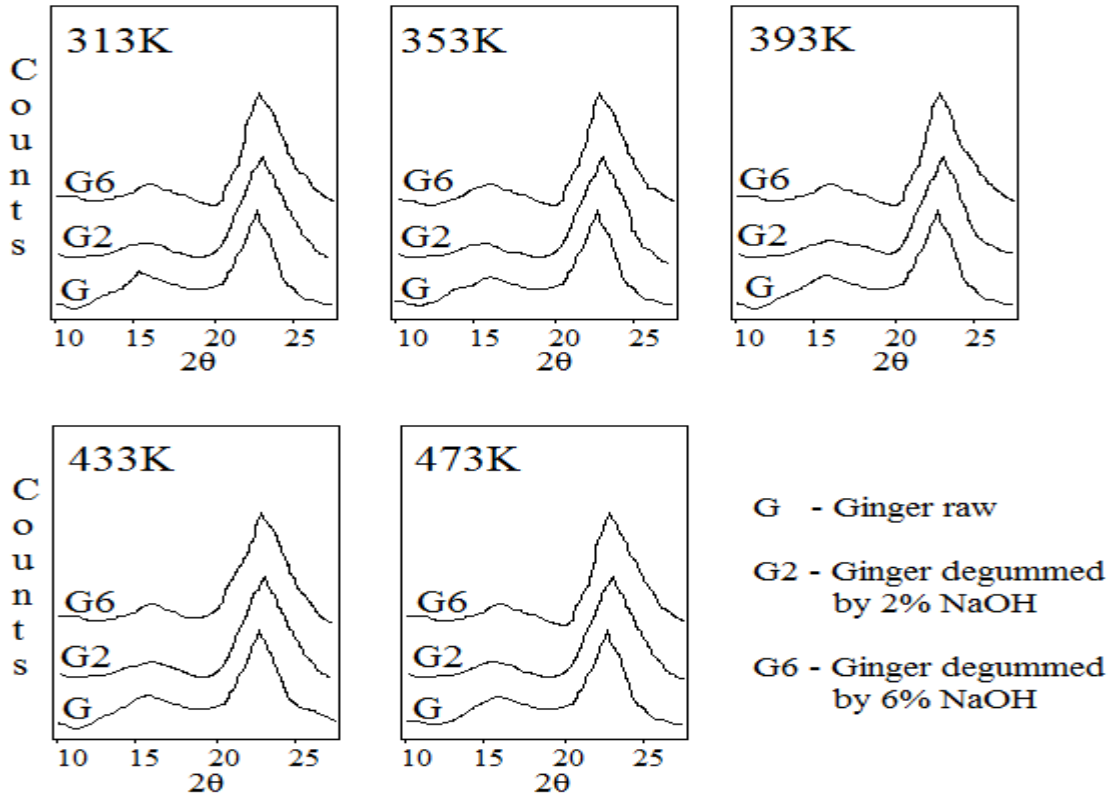

Fig. 2- Comparision XRD of raw and degummed ginger fibers at same temperature.

The interplaner spacings (d_{hkl}), peak inmtensity (I_{max} & I_{min}) and the degree of crystallinity (DC) of raw and degummed ginger fiber from XRD spectra are shown in table 2.

From the table 1 and table 2, it is seen that the degree of crystallinity of ginger fiber, at all the temperature, increases due to degumming. This may be due to the fact that, after degumming, the molecular chains of the fibers, which were restricted by gum, become free to rearrange and have more crystalline form.

It is seen that the degree of crystallinity increases at 353K, for all the fibers, due to removal of water molecules. The presence of water molecules in the amorphous region of the fibers may restrict the alignment of the chain molecules and thus give rise to strains in the crystalline region.

Table- 2: Parameters obtained from the XRD of degummed ginger fibers

Sample	Temperature (K)	2θ (degree)	d_{hkl} (Å)	I_{max}	I_{min}	DC%
Ginger	313	23.3	3.7911	87	22	74.7
Degummed by 2% NaOH	353	23.3	3.7911	90	21	76.7
	393	23.4	3.7841	92	22	76.1
	433	23.3	3.7936	86	21	75.6
	473	23.2	3.8104	84	22	73.8
Ginger degummed by 6% NaOH	313	23.1	3.8312	112	25	77.6
	353	23.2	3.8034	120	19	84.2
	393	23.2	3.8034	118	20	83.1
	433	23.1	3.8312	116	20	82.7
	473	23.2	3.8032	117	22	81.2

The change in degree of crystallinity of raw and degummed ginger fibers with the change of temperature is shown in fig. 3. It is seen that the degree of crystallinity percentage for raw ginger fiber decreases rapidly from temperature 350K than the two degummed ginger fibers. This may be due to the lowering of thermal degradation due to degumming. Thus, from the figure it can be stated that the thermal stability increases due to degumming.

In fig.3, the change of degree of crystallinity of raw and degummed ginger fiber with change of temperature is shown.

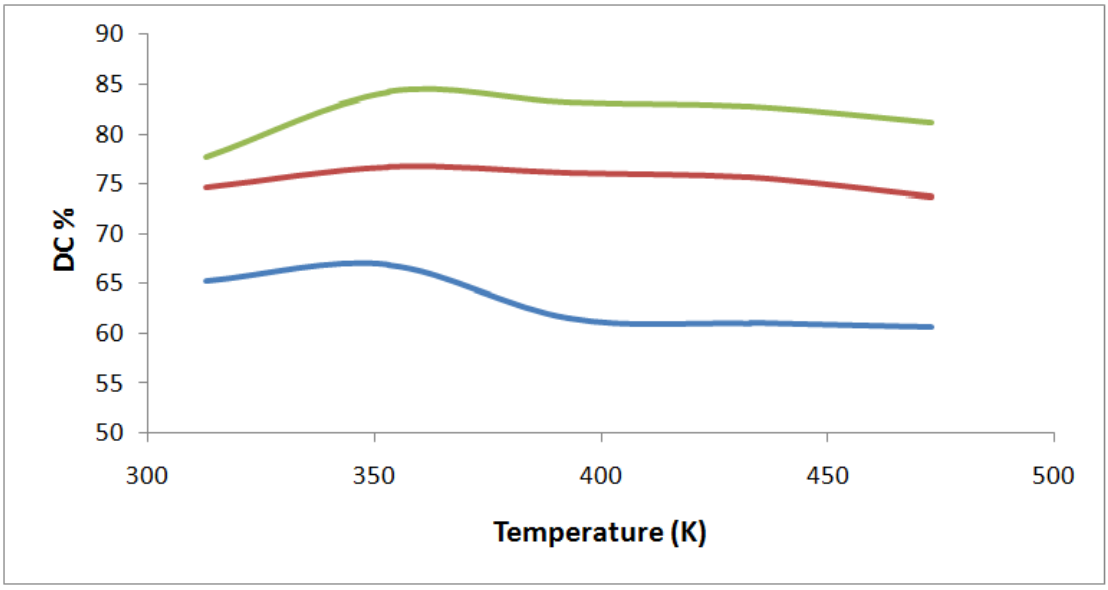

Fig.3- Change DC% of raw and degummed ginger fibers with temperature.

It is seen that the degree of crystallinity percentage decreases rapidly for raw ginger fiber from 350K than the two degummed ginger fibers. This may be due to lowering of thermal degradation due to degumming. Thus the fig. says that the thermal stability increases due to degumming.

VI. CONCLUSION

The observation states that the degree of crystallinity of different raw and degummed fibers is different. The degree of crystallinity of turmeric fiber is higher than ginger fiber, but less than eri fiber. At temperature 353K, the degree of crystallinity is highest for all the fibers. This is due to the evaporation of water molecules from the hygroscopic fibers. The degree of crystallinity decreases from 353K to higher temperature for all the fiber samples due to the thermal degradation of the fiber molecules. Due to degumming the thermal stability of the fibers increases.

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