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A study on the thermal behaviours of Eri, Muga and Pat fibres

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Abstract: Thermal behaviours of Eri, Muga and Pat fibres, endemic to the North-Eastern region, under different conditions have been investigated by various experimental methods: Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), Thermogravimetry (TG) and Derivative Thermogravimetry (DTG) analyses. The experiments have been carried out over the temperature range 25°C to 400°C. The activation energy (E_A) of the dehydration and decomposition steps have been calculated. Significance of these observations has been discussed with reference to the suitability of such materials for application particularly in textile industry.

Keywords: Natural fibre, differential scanning calorimetry, differential thermal analysis, thermogravimetry, activation energy.

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1. Introduction

The natural silk fibres Eri, Muga and Pat, are hygroscopic and semicrystalline in nature. Due to their essential characteristics of external forms and of physico-chemical behaviours, they have great utilities in various textile industries. It is evident that reaction kinetics, enthalpy etc. of a material are based on its physico-chemical properties. Therefore, the study of the thermal behaviours of these natural silk fibres certainly have great importance in textile technology.

Many attempts have been made to study thermal behaviours of cellulose materials (Shafizadeh and Bradhury 1979, Antal *et al* 1980, Gregorski and Pavlath 1981). Studies on thermo-physical properties of various silk fibres have been made by some investigators (Kim and Chur 1982, Venger *et al* 1982). Further investigations on the reaction kinetics of cellulose, polymers and some fibres with Differential Scanning Calorimetry have been made by some workers (Gregorski and Pavlath 1982, Duswalt 1974, Connelly and O'Reilly 1982, Wagers and Bayer 1985). But no such studies on these types of natural silk fibres endemic to this North-Eastern region of India have been noticed.

Thermogravimetric (TG), Differential Thermal Analysis (DTA) and Derivative Thermogravimetry (DTG) have also been found to be the most suitable techniques for thermal decomposition studies by some workers (Dev *et al* 1989a, 1989b).

process. These peaks correspond to the DTG thermogram peaks (Figure 2) at the temperature ranges of about 45-132°C for the fibres.

Table I. DTA and DTG data (peak temperatures at the transition period) of silk fibres.

Sample	Step	DTA		DTG	
		Temp. range T (°C)	Peak temp. ΔT (°C)	Temp. range T (°C)	Peak temp. ΔT (°C)
Muga	1	60——125 (endo)	95.2	48——130 (endo)	82.8
	2	295—— 345 (exo)	312.6	270 350 (endo)	346.0
Eri	1	75 ——120 (endo)	94.0	45 — 132 (endo)	80.2
	2	300——340 (exo)	315.2	265——366 (endo)	320.0
Pat	1	68——138 (endo)	92.8	55——125 (endo)	70.5
	2	280——340 (exo)	312.0	240——340 (endo)	310.0

The TG curves (Figure 3) show beginning of the weight loss at 58°C for Muga, 46°C for Eri and 49°C for Pat. The process becomes rapid at about 120°C for all the fibres. The inflexion about 115°C for the fibres indicates the loss of all absorbed water. This dehydration stage was evident in the DSC thermograms (Figure 4) by endothermic peaks at 150-170°C (ΔT_{min} = 156.2°C) for Muga,

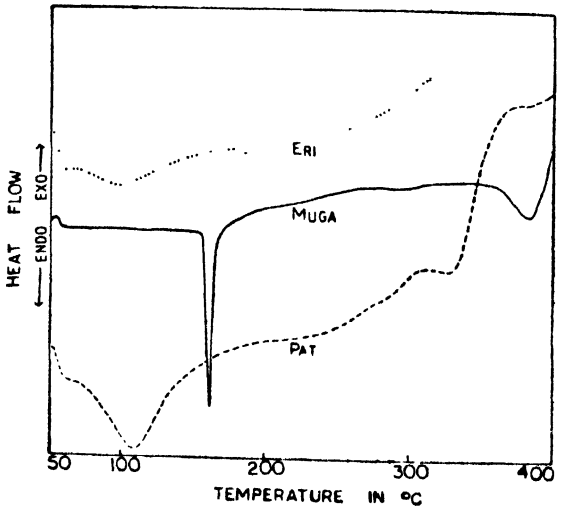

Figure 4. DSC Thermograms of silk fibres in air atmosphere. (a) Muga; (b) Eri; (c) Pat.

Table 2. Reaction kinetic data of DSC curves at the transition period.

Sample	Step	Temp. range T (°C)	Peak temp. ΔT (°C)	Wt. loss (×)	Activation energy E_A (Kjm^{-1})	Enthalpy ΔH	Order of reac.	Reaction
	1	150–170	156.2		128.01	450.13	1.72	Dehyd.
Muga				37.28				
	2	360–420	382.3		387.66	354.52	1.14	Decomp.
	1	70–130	98.3		117.80	546.55	1.63	Dehyd.
Eri				49.30				
	2	370–410	387.3		392.58	393.43	1.02	Decomp.
	1	73–160	108.5		101.08	901.43	1.87	Dehyd.
Pat				52.00				
	2	310–360	304.3		301.82	88.77	1.28	Decomp.

70-130°C (ΔT_{min}-98.3°) for Eri and 79-160°C (ΔT_{min}= 108.5°C) for Pat respectively at the heating rate of 10 deg min^{-1}.

With the use of TG data, the activation energy (E_A) of dehydration, calculated on the basis of the Freeman and Carroll equation (Freeman and Carroll 1958) was found to be 138 KJ mol^{-1} in the air atmosphere for Muga fibres. This value is found to be in good agreement with the value of 128.01 for the sample obtained from DSC thermograms (Figure 4) for the first transition peak temperature. From the first endothermic peaks of all thermograms, it is evident that heat is absorbed by the samples to decompose the water molecules mostly embedded in the

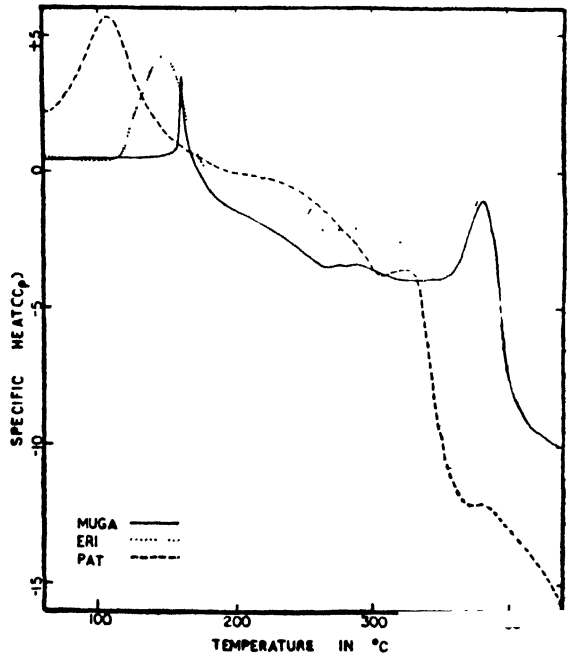

Figure 5. Plot of specific heat (C_p) values versus temperature of silk fibres. (a) Muga; (b) Eri; (c) Pat.

amorphous region of the fibres. During this period, structural set-up of the fibres remains unaltered as indicated by the return of the DSC thermograms to the base line. These results are also supported by X-Ray Diffraction (XRD) study made elsewhere (Baruah and Born 1988).

The rapid falling of the anhydrous was shown in TG curves (Figure 3) to take place beyond 260°C, 255°C and 240°C for Muga, Eri and Pat fibres respectively indicated the thermal decomposition processes. The exothermic peaks in DTA thermograms shown in the Table 1 indicated these decomposition processes. These peaks corresponded to the DTG thermogram peaks in the temperature ranges 270-350°C, 265-366°C and 240-340°C for Muga, Eri and Pat respectively. The degradation stage was accounted in the DSC thermograms (Figure 4) by the second

endothermic peaks at 360-420°C (ΔT_{min}=382°C), 370-410°C (ΔT_{min}=387°C) and 310-360°C (ΔT_{min}=304.3°C) with weight losses 37%, 49% and 42% at about 400°C for Muga, Eri and Pat fibres respectively. The activation energy (E_Δ) for

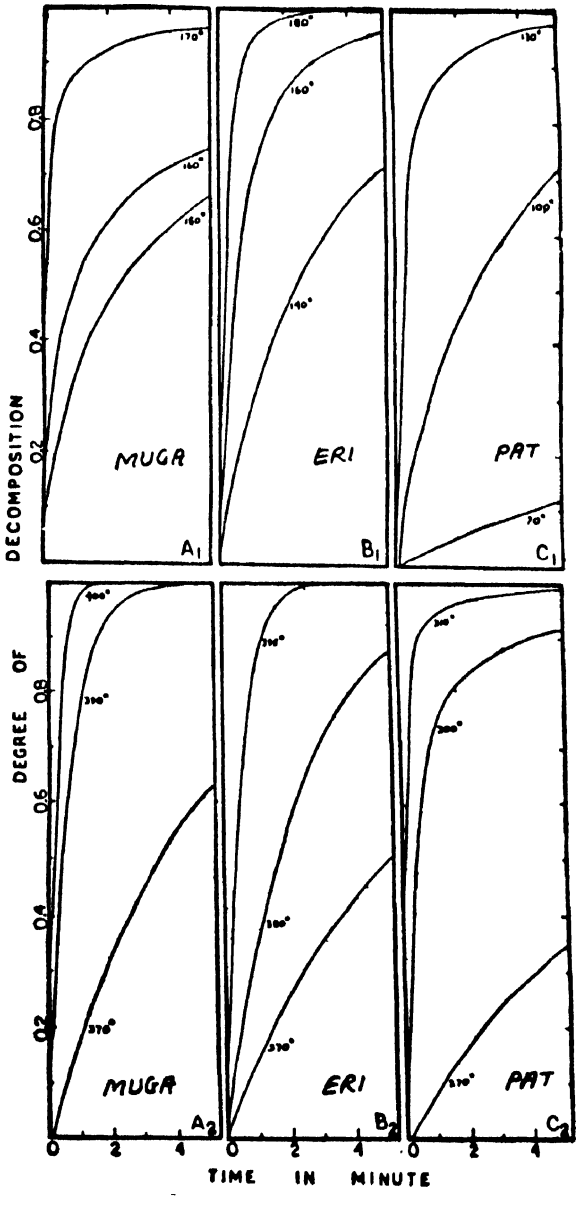

Figure 6. Plot of α (fraction of reaction completed) versus time at three temperatures around the transition points.
(a) Muga; (b) Eri; (c) Pat.
(A_1, A_2) (B_1, B_2) (C_1, C_2)

those second endothermic peak temperatures was found to be 387.66 KJ mol^{-1}, 392.56 KJ mol^{-1} and 301.82 KJ mol^{-1} for Muga, Eri and Pat fibres respectively.

Other reaction kinetic parameters of the samples are computed from the isothermal thermograms and the results are presented in the Table 2. They are different for different silk fibres at the transition states. Scanning at different rates showed that the higher the scanning rates, the higher the temperature of decomposition.

The plot of specific heat (C_p) as a function of temperature recorded at the heating rate 10°C min^{-1} for Muga, Eri and Pat fibres were recorded (Figure 5). Sudden change in specific heat at the transition temperature causes a sharp change in the position of the base line.

The plot of α (fraction of the reaction completed) versus time at three different temperatures for each transition were also recorded (Figure 6). These results showed that the cause of reaction was increased at regular rate (exponentially) with time for any transition temperature.

The first transition points represented by all the thermograms for the hygroscopic silk fibres indicate great significance for studying their suitability particularly in textile industry. Repeated thermograms for TG, DTA and DSC analyses have been taken for all the fibres annealed upto 160°C. Same transition peaks obtained under normal and annealed conditions reveal that the fibres under study possess adsorption and desorption characteristics. The adsorption and desorption of atmospheric moisture with the consequent evolution and absorption of heat add to the comfort of clothing of these fibres.

4. Conclusion

The first transition points of all the thermograms of the hygroscopic silk fibres represents the dehydration processes. These are governed by a mechanism involving dissociation of the water molecules mostly embedded in the amorphous region of the semi-crystalline fibres. The second transition points of all the thermograms represent thermal decomposition and degradation of crystalline set-up of the silk fibres. Further, the results show that the Muga and Eri fibres are thermally more strengthened than the Pat fibre.

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