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Fine Structural Characteristics and Physical Properties of Raw Silk

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SUMMARY

The theories of KRATKY, DEBYE and BUECHE and POROD have been applied to evaluate macromolecular parameters which speak of the fine structural characteristics of raw silk – a natural polymer in the solid state. The small-angle KRATKY camera has been utilised for the measurements of the scattering intensities. The macromolecular parameters evaluated are the percentage of void (w_1), the specific inner surface (O/V), length of coherence (l_c), range of inhomogeneity (l_r), transversal length -s – $\bar{l}_1$ and $\bar{l}_2$ which were found to be equal to 0.13%, 25.15 x 10^{-4} Å^{-1}, 21.84 Å, 2.11 Å, 1.59 x 10^3 Å and 2.11 Å respectively The physical properties as evaluated by Scott's IP2 Tester have been reported.

INTRODUCTION

In all scattering phenomena the scattering angles are inversely related to the dimensions of the scattering particles. As macro -molecules are giant, compared with normally used CuK_α wave length ($\lambda = 1.54$ Å), we find the scattering of the particles as a whole at correspondingly small angles, in fact, X-ray small-angle scattering occurs. This is a process to explore the size, shape and orientation of particles of colloidal dimensions as well as in the two phase systems, the volume-share of the two phases.

In case of fibers, both crystalline and amorphous zones, are regarded as particles because of their different densities. The volume fraction of the crystalline, amorphous zones as well as the volume of the holes are of equal importance to the textile technicians as the statement of quality of fiber structure. Therefore our final goal is to establish a connection between the fine structural characteristics and textile properties. This will be only possible through diversified and intensive research of X-ray small-angle scattering on a great number of fibers (KRATKY, 1972). In this piece of work we have reported the fine structural characteristics and physical prop -erties of a protein fiber – raw silk, whereas a large number of other protein fibers are under our investigation.

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EXPERIMENTAL

Raw silk - a natural protein fiber, was obtained in its pure form from Central Sericulture Research and Training Institute, Government of India, Mysore. The sample was treated with a 3:1 mixture of alcohol and ether for 24 hours, in cyclohexene for 8 hours and finally in benzene for 8 hours to remove completely fats present in the sample (RATHO and PATEL, 1975). The sample was then packed in a Mark Capillary tube of 0.09 cm diameter and was mounted in the small-angle KRATKY camera which was coupled with proportional counter in combination with a pulse height discriminator set to count the CuK_α - radiation only. For automatic operation a programmable electronic step scanning device was used. The sample was so mounted that the length direction of the slit was parallel to the fiber axis. The scattering intensities were measured at different scattering angles in the range from 2.25×10^{-3} to 3.42×10^{-2} radians. The intensities were corrected for the statistical error and finally the scattering curve $\tilde{I}(X)\sim X$ (Fig.1), was constructed where $X = 2a\theta$. Here 'a' (20 cms.) is the distance between the sample and the plane of registration of intensities and 'θ' is the half scattering angle. A second curve (Fig.2) $\tilde{I}(X)X^3 \sim X^3$ was plotted to determine the run constant K_1 and the background correction constant K_2 which were found to be equal to 1.10×10^2 and 1923.077 respectively (KRATKY, 1966). A third curve (Fig.3) between $\tilde{I}(X)X^4 \sim X$ was plotted to find out the experimental value of the invariant (RATHO et al, 1974).

The physical properties like breaking strength, percentage of elongation at break, linear density, tenacity, standard deviation (δ) and percentage coefficient of variation (% C.V.) at breaking load and standard deviation (δ) and percentage coefficient of variation (% C.V.) at percentage of elongation at break of defated raw silk were evaluated by using Scott's IP2 inclined plane tester, in the load range of 250 gms to 500 gms at a constant 65% rh. This type of constant rate of loading instrument has been utilised for the experimental measurements of the physical properties of defated raw silk because of accurate scientific work and satisfactory interpretation of results (SUNDARAM, 1979).

RESULTS AND DISCUSSIONS

Silk being a natural fiber, one can proceed with the estimation of parameters from the smeared out scattering curve (RATHO and SAHU, 1971) and can make a pore analysis of the sample. According to theory of POROD (MITTELBECH and POROD, 1965), the tail end of the intensity curve of a general two phase system is proportional to X^{-3} (Fig.2) and this is based on the fact that there is homogeneous electron density distribution in each phase.

Po, the primary beam intensity; ρ, the electron density; D, the effective sample thickness; δ_c, the compact density; δ_a the apparent density; Q_{expt}, the experimental value of the invarient; E, the integrated scattered energy; were found to

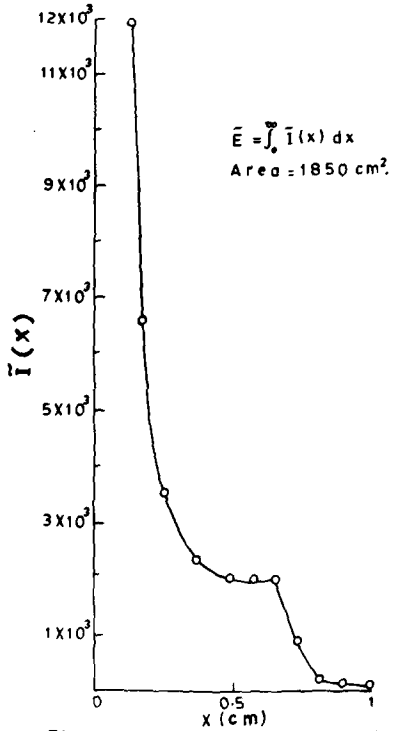

Fig-1. The scattering curve giving the value of $\tilde{E}$.

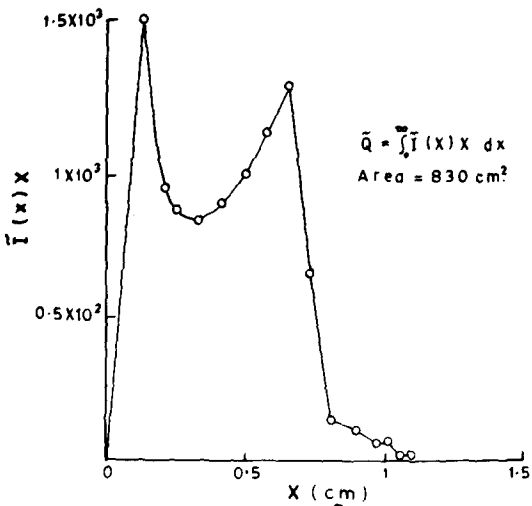

Fig-3. The invariant ($\tilde{Q}$) curve.

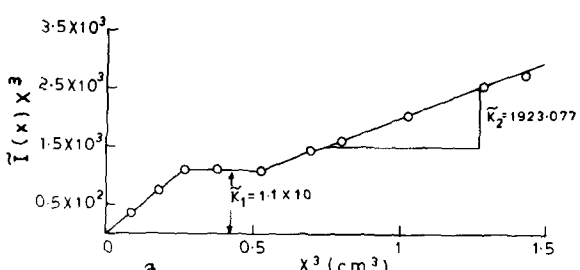

Fig-2. $\tilde{I}(x)X^3$ Vrs X^3 Plot giving the RUN constant $\tilde{K}_1$ and the correction constant $\tilde{K}_2$ for background scattering.

be equal to 4031.59×10^4, 0.72, 0.08 cm, 1.35, 1.25, 830 cm^2, 1850 cm^2, respectively. Utilising these values in the equations described in our previous paper (PATEL et al, 1980) the follow -ing macromolecular parameters of defated raw silk were evaluated and tabulated in TABLE -I with the corresponding values of Sisal, Wool, Jute and Xylen.

(a) Specific Inner Surface (O/V): It is the phase boundary area of unit volume of the dispersed phase.

(b) Percentage of Void (w_1): It is the volume fraction of air or void contained in the sample.

(c) Length of Coherence (l_c): It is the mean occupied length in a line of length drawn from a point in matter.

(d) Transversal Lengths ($\bar{l}_1$ and $\bar{l}_2$): For calculation of transversal parameters we propose a model of a system consisting of slightly irregularly distributed like or unlike particles with random orientations. These are separated by void or free spaces. Therefore if we shoot arrows through the systems in all directions and measure the average intersectional lengths of the arrows with the two phases, we get the transversal lengths $\bar{l}_1$ and $\bar{l}_2$.

(e) Range of Inhomogeneity (l_r): $\bar{l}_1$ equal to l_r, the range of inhomogeneity which corresponds to the reduced mass in mechanics.

TABLE-I

Sample	O/V $\times 10^{-4}$ in Å^{-1}	% of void	l_c in Å	l_r in Å	$\bar{l}_1$ in Å	$\bar{l}_2 \times 10^3$ in Å
raw silk	25.15	0.13	21.84	2.11	2.11	1.59
Sisal (RATHO et al,1974)	4.06×10^{-2}	0.01	342.2	107.31	107.31	985.0
Wool(Marino) (RATHO and PANDA, 1967)	2.082	0.70	423.6	137.5	137.5	-
Jute (SAHU and MISHRA,1973)	0.06	0.02	524.5	137.7	137.7	644.2
Xylen (KAHOVEC et al,1953)	-	0.11	695.0	305.0	305.0	-

The physical parameters of defated raw silk were also evaluated using the Scott's IP2 (CRL Type)instrument as per the methods suggested by GROVER and HAMBY (1979) and the results were tabulated in TABLE-II.

TABLE-II

Sample	Breaking strength in gms.	% of elongation at break	linear density in denier	tenacity gm/denier	At breaking load δ	At breaking load % C.V.	At % elongation at break δ	At % elongation at break % C.V
raw silk	131.25	20.42	30.72	4.27	11.81	9.00	1.76	8.62

CONCLUSION

The sample raw silk is considered as the densely packed two phase system belonging to general micelle system. Hence the theories of KRATKY (1966), DEBYE and BUECHE (1949) and MITTELBECH and POROD (1965) have been utilised to make a pore analysis of raw silk, and the corresponding macromolecular parameters have been evaluated. The scattering curve (Fig.1) exhibits a sharp fall of the scattering intensity because of the short range order of electron-density distribution in the crystalline and amorphous phases of the raw silk. This can also be utilised to calculate the extent of crystalline and amorphous zone in raw silk if a wider angular range is studied (PRASAD and GOWDA, 1980). The fine structural characteristics and the physical properties of raw silk may throw light on structure function correlation.

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