Polymer Bulletin 2, 189–194 (1980)

Polymer Bulletin

Small-Angle X-Ray Scattering Studies of a Natural Fiber: Agave Cantala

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SUMMARY

Agave cantala, a natural fiber is considered as a densely packed colloidal system belonging to the general micelle system and the well known theories of KRATKY and POROD have been utilized to evaluate some macromolecular parameters of the scattering inhomogeneities in it. The small-angle KRATKY Camera of the latest design has been used for experimental measurements. The parameters evaluated are the specific inner surface, transversal lengths like the length of inhomogeneity and the length of coherence and the air fraction of the scattering particles in Agave cantala fiber; these were found to be

1.8137 x 10^{-6} Å, 375.066 Å, 502.7223 Å, and 0.017%, respectively.

INTRODUCTION

The small-angle X-ray scattering (SAXS) was introduced when it became desirable to detect large lattice spacings of the order of hundreds and thousands of interatomic distances found in some particular minerals and in certain complex molecules such as high polymers or proteins. To satisfy BRAGG's relation ($n\lambda = 2d \cdot \sin\theta$) for high values of lattice spacings, the angle of diffraction must be extended to include extremely small angles even up to small fractions of a degree and fortunately the KRATKY Camera (KRATKY and SKALA, 1958) of the latest design can measure the angle corresponding to a BRAGG value of 20.000 Å.

The theory of small-angle scattering was developed by GUINIER (GUINIER, 1937) and subsequently by HOSEMANN (HOSEMANN, 1950). KRATKY (KRATKY, 1938, 1963) took account of interparticle interference. POROD (MITTELBECH and POROD, 1965) has given a rigorous theoretical analysis of small-angle X-ray scattering for densely packed colloidal systems.

0170-0839/80/0002/0189/$01.20

EXPERIMENTAL

The sample Agave cantala fiber was obtained in its pure form from Sisal Research Station, Indian Council of Agricultural Research, Bamara, Orissa, India. Agaves are now the World's most important leaf fiber and most of the "soft" currency countries have a possibility of earning "hard" currency through exports of this material. Agaves are increasingly industrially utilized for a very wide and varied range of products.

The sample was dewaxed to show a "Hohlraum" character, i.e. the material is packed in layers with free spaces in between. This 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.

CuK_α radiation from a MACHLETT-A_2 diffraction tube, running at 3o KV and 2o mA was monochromatised by a curved crystal monochromator and allowed to pass through a rectangular slit of width 1ooμ fitted to the KRATKY Camera which is designed with special precautions to eliminate parasitic scattering. The beam is intercepted by the sample which is placed such that the fiber axis is parallel to the length of the primary beam. The time of exposure was varied from 3hr to 6hr. The scattering curve $\tilde{I}$ vs.X (Fig.1) was constructed and the graph $\tilde{I}(X)X^3$vs.X^3plotted (Fig.2) to determine the run constant $\tilde{K}_1$ and the correction constant $\tilde{K}_2$ which were found to be $3.00x1o^{-6}$ and o.oo75752, respectively.

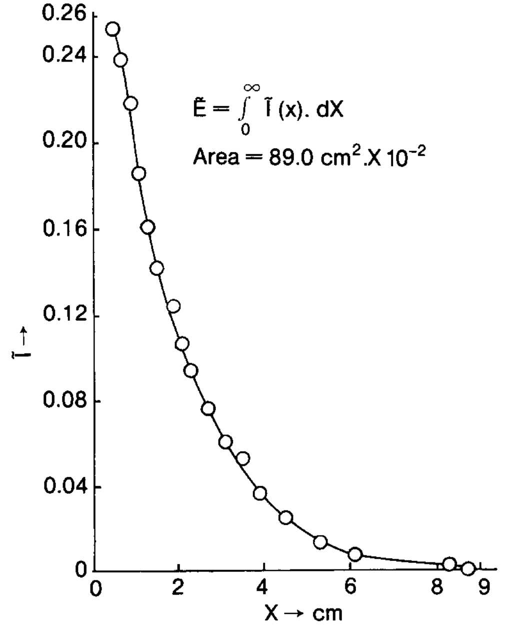

Fig.1: The Scattering Curve Giving the Value of $\tilde{E}$

Here we have represented Θ, half the scattering angle, by a quantity X where X=2apΘ; 'a' is the film sample distance =23cm and 'p' the transformation factor of the microphotometer =1oo. According to the 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). This can also be seen from the limiting slope of -3 of the double logarithmic plot in Fig.3.

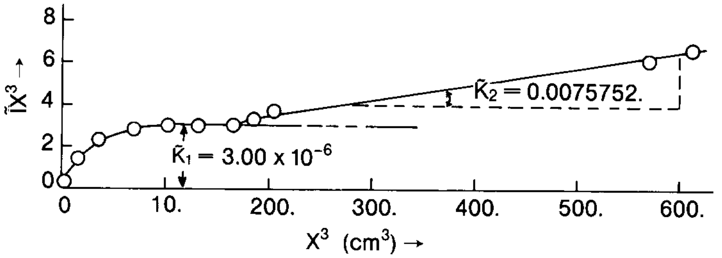

Fig. 2: $\tilde{I}X^3$ vs.X^3 Plot Giving the Run Constant $\tilde{K}_1$ and the Correction Constant $\tilde{K}_2$ for Back and Ground Scattering.

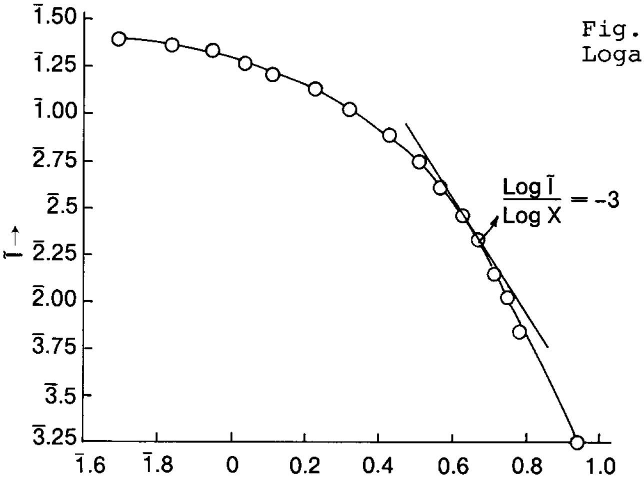

Fig.3: Double Logarithmic Plot

RESULTS AND DISCUSSION

The invariant $\tilde{Q}$ of the scattering curve introduced by POROD (POROD,1951)is given by

$$\tilde{Q}exp = \int_0^\infty \tilde{I}(X)X\,dX \quad (1)$$

for smeared-out intensity (Fig.4), while $\tilde{Q}_{theor}$ is given by

$$\tilde{Q}_{theor} = (1/2\pi)\ (e^2/mc^2)^2\ N^2\lambda^3 DP_o a\rho^2 w_1 w_2 \quad (2)$$

after KRATKY (KRATKY,1963) and POROD (POROD,1953) where w_1 and w_2 are the volume fraction of the void or air and matter, respectively , e is the electronic charge, m the mass of electron, c the velocity of light, λ the wavelength of X-rays=1.54Å , N AVAGADRO's number, D the effective sample thickness=o.1587 cm. P_0 is the intensity of the primary beam=26.84, a the film sample distance, ρ the electronic density of the scattering particle. The asymptotic behavior of large values of X in Fig.3 and the intensity which decreases proportional to X^{-3} in Fig.2 are based on the fact that there is a homogeneous electron density distribution in each phase. The $\tilde{I}(X)X^3$vs.X^3 plot yields the background scattering constant $\tilde{K}_2$ as the slope of the curve as suggested by KRATKY (KRATKY,1963).

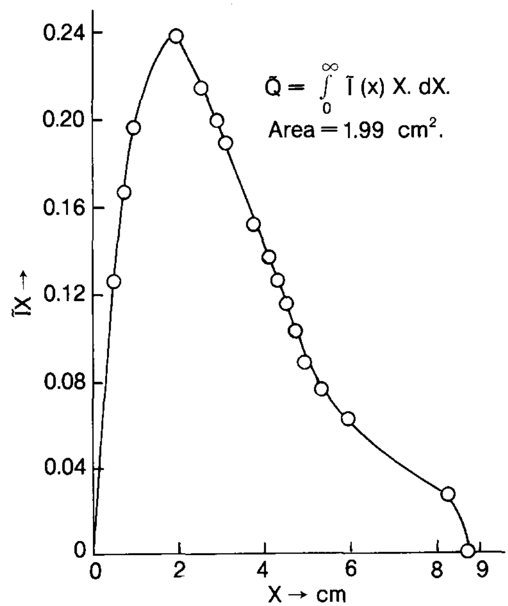

Fig. 4: The Invarient ($\tilde{Q}$) Curve

Air Fraction

The effective sample thickness D is given by

$$D = (\delta_a/\delta_c)\,\phi \qquad (3)$$

Here the compact density δ_c is taken to be the density of cellulose (1.6o gm/cm^3) (RATHO, PATEL and SINGHAL, 1974) and the apparent density δ_a is 1.4851 gm/cm^3. ϕ is the inner diameter of the capillary tube = o.171 cm; the value of D thus calculated is o.15871 cm.

The electron density of the substance is given by

$$\rho = \text{\textcent}\Sigma O/\Sigma A = 0.848 \qquad (4)$$

Where $\Sigma O/\Sigma A$ = ΣAtomic numbers/ΣAtomic weights = 0.53 for cullulose.

Now equating $\tilde{Q}_{th}$ with $\tilde{Q}_{exp}$ and taking the respective values one gets $w_1\, w_2 = 1.7006 \times 10^{-4}$. Since $w_2 \approx 1$, the volume fraction of air or void contained in the sample becomes 0.017%.

Specific Inner Surface

The phase boundary area per unit volume of the dispersed phase is given by

$$O/V = (8\pi/\lambda a) w_1 w_2 (\tilde{K}_1/\tilde{Q}_{exp}) \qquad (5)$$

Substitution of the respective values yields $O/V = 1.8137 \times 10^{-6}\ \text{Å}^{-1}$.

Transversal Lengths

If we shoot arrows through the system in all directions and measure the average intersectional lengths of the arrows with the two phases and call them transversal lengths $\bar{l}_1$ and $\bar{l}_2$, we get

$$\bar{l}_1 = 4w_1 / (O/V) = 375.066\text{Å} \qquad (6)$$

and

$$\bar{l}_2 = 4w_2 / (O/V) = 22.054 \times 10^5\text{Å} \qquad (7)$$

of this substance.

Range of Inhomogeneity

The range of inhomogeneity l_r is given by

$$l_r = (4V/O) w_1 w_2 = 4(V/O) w_1 \text{ since } w_2 \approx 1 \qquad (8)$$

or $l_r = \bar{l}_1$; thus, the range of inhomogeneity corresponds to the reduced mass in mechanics.

Coherence Length

The coherence length is given by

$$l_c = (\lambda a/\pi) \int_0^\infty \tilde{I}(X)\, dX / \int_0^\infty \tilde{I}(X)\, X dX \qquad (9)$$

or

$$l_c = (\lambda a/\pi) \tilde{E}/Q_{exp} \qquad (10)$$

where $\tilde{E} = \int_0^{\infty} \tilde{I}(X)dX$ is the integrated scattered energy (Fig.1) which is equal to 0.89×10^{-2}. Substituting the respective value we get l_c=5o2.7223Å.

CONCLUSION

The sample Agave cantala is considered as the densely packed two-phase system belonging to the general micelle system. Hence, the theories of KRATKY and POROD have been utilized to make a pore analysis and the corresponding macromolecular parameters have also been evaluated. The parameters determined are percentages of the air or void specific inner surface, transversal lengths, and of the coherence length which were found to be equal to o.o17%, 1.8137×10^{-6} Å^{-1}, 375.oo6Å, 22.0542×10^{5}Å, and 5o2.7223Å, respectively.

ACKNOWLEDGEMENT

The authors gratefully acknowledge the financial support from U.G.C., New Delhi, to carry out this work.

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Received December 13, 1979/ January 7, 198o