Fine Structure Investigation of *Sansevieria Roxburghi* Fiber by Small-Angle X-Ray Methods

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Synopsis

Small-angle x-ray methods were used to evaluate macromolecular parameters such as specific inner surface of the dispersed phase; transversal lengths such as length of inhomogeneity and length of coherence; and the air fraction of the scattering particles in *Sansevieria roxburghi;* these parameters were found to be 12.76×10^{-6} Å^{-1}, 549.9 Å, 15.21 Å, and 0.17%, respectively. A small-angle Kratky camera was used for the experimental measurements, and the theories of Kratky and Kratky and Porod were utilized to evaluate these parameters. The sample under investigation is treated as a densely packed colloidal system belonging to general micelle systems.

INTRODUCTION

The study of small-angle x-ray diffraction was introduced by several workers[1-4] when it became desirable to detect large lattice spacings of the order of hundreds and thousands of interatomic distances. Guinier[5,6] has put forward a theory which is applicable to colloidal particles in dilute systems where interparticle interference is neglected. Hosemann[7] applied the same theory to densely packed colloidal systems where he also eliminated interparticle interference.

However, Kratky[8,9] has pointed out the importance of interparticle interference in small-angle scattering by densely packed systems. Heyn[10] has supported the above view point and treated micellar systems in terms of interparticle interference. Porod[11] gave a rigorous theoretical analysis of small-angle scattering for particles of various shapes and sizes. Sellen[12] had applied light scattering methods to determine the macromolecular size of Ludox (an aqueous suspension of silica). Estimation of the microcell sizes would lead to the understanding of the buildup of the macromolecules in the fiber from a large number of microcells. Wide-angle studies of microcell dimensions of mung bean roots have been reported by Preston.[13] The Kratky[14] camera, having a resolution corresponding to a Bragg value of 20,000 Å, was used for the experimental measurements. Since the fiber sample belongs to a densely packed colloidal system, a pore analysis of the substance only is possible, and hence macromolecular parameters such as specific inner surface, transversal lengths, and percentage of air present in the substance were determined.

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EXPERIMENTAL

Sample

Numerous species of the genus *Sansevieria* occur as wild plants in various parts of tropical Africa and Asia.[15] In monocots such as *Sansevieria* the fibers are arranged in several characteristic patterns.[16] These plants are also cultivated. The plants yielding these strong fibers, if properly developed, may form sources of earning foreign exchange for tropical countries, and hence the importance of these investigations.

The leaf of *Sansevieria roxburghi* was taken and physically decorticated to extract the fiber. It was then washed in water and dried in air. This fiber sample was dewaxed for 12 hr according to the procedure laid down by Roy[17] in a 1:2 mixture of alcohol and benzene under gradual rise and fall of temperature from 30° to 55°C. It was then washed in distilled water and dried in air. This treatment reduced the sample to a hohlraum system, that is, substances lying in layers with free spaces in between.

Method and Apparatus

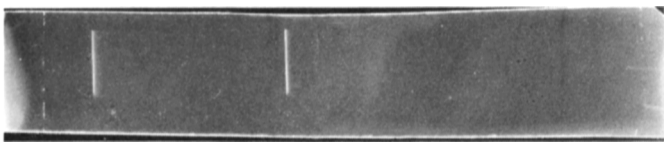
The sample so prepared was mounted in a mark-capillary tube of diameter 0.15 cm and placed in the Kratky camera so that the length of the x-ray beam was parallel to the fiber axis. Therefore the diffraction pattern should correspond to the equator representation of the fiber diagram of Polanyi.[18]

A Machlett A-2 x-ray diffraction tube with Cu anticathode coupled with a highly sensitive stabilizer was run at 30 kV, 20 mA. It gave an intense beam of x-rays which was focussed to irradiate the sample by the bent crystal monochromator after Johansson and Guinier.[19] The photographic technique was adopted for recording the intensities. As it was not possible to obtain the whole range of scattering in a single exposure it was photographed in stages for 3 hr and 6 hr, respectively. The two scattering patches can be seen from Fig. 1. These scattering patches were micophotometered, and then the final scattering curve (Fig. 2) was built up giving due consideration to the time of exposure. The angular range studied is from 0.478×10^{-3} to 9.17×10^{-3} radian. Here we have represented the scattering angle 2θ by a quantity X, defined by

$$X = 2\theta ap$$

where a is the film sample distance = 23 cm and p is the transformation factor of the microphotometer = 100.

The sample is a natural fiber having a good degree of orientation as could be easily inferred from the wide-angle x-ray pattern (Fig. 3). The fiber diagram



(a) (b)

Fig. 1. Small-angle scattering patches of *Sansevieria roxburghi* fiber through Kratky camera for (a) 3 hr and (b) 6 hr of exposure.

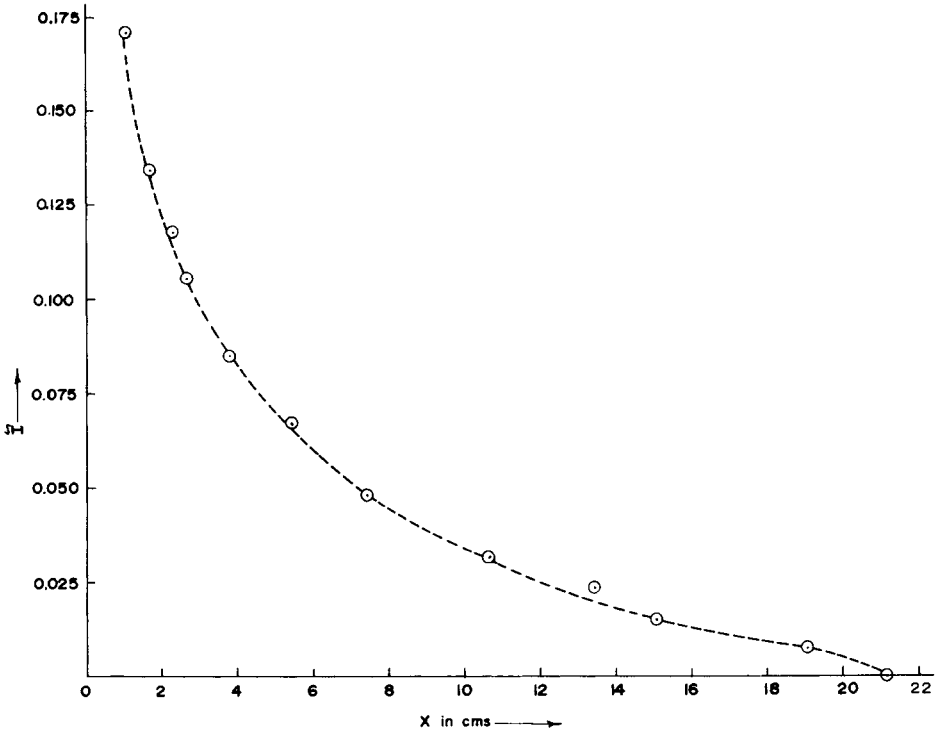


Fig. 2. The scattering curve.

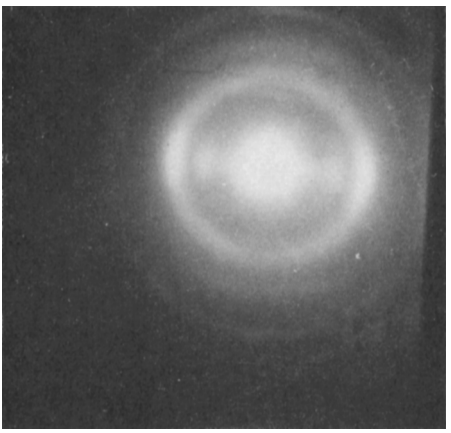


Fig. 3. Wide-angle scattering photograph of fiber showing a high degree of orientation.

is similar to those having a good degree of orientation.[20] Therefore, no slit correction to the scattering curve is required, and one can proceed to estimate the macromolecular parameters from the smeared out scattering intensities. According to the theories of Porod[11] the tail end of the intensity curve of a general two-phase system is proportional to x^{-3} (Fig. 4). This can also be seen from the

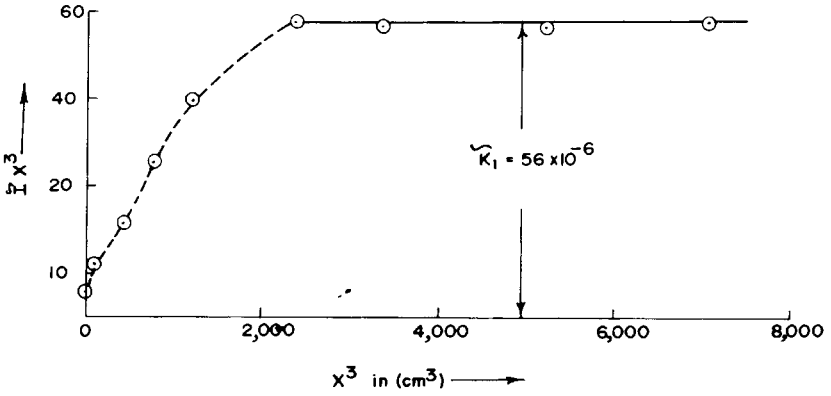


Fig. 4. The run constant $\bar{k}_1$.

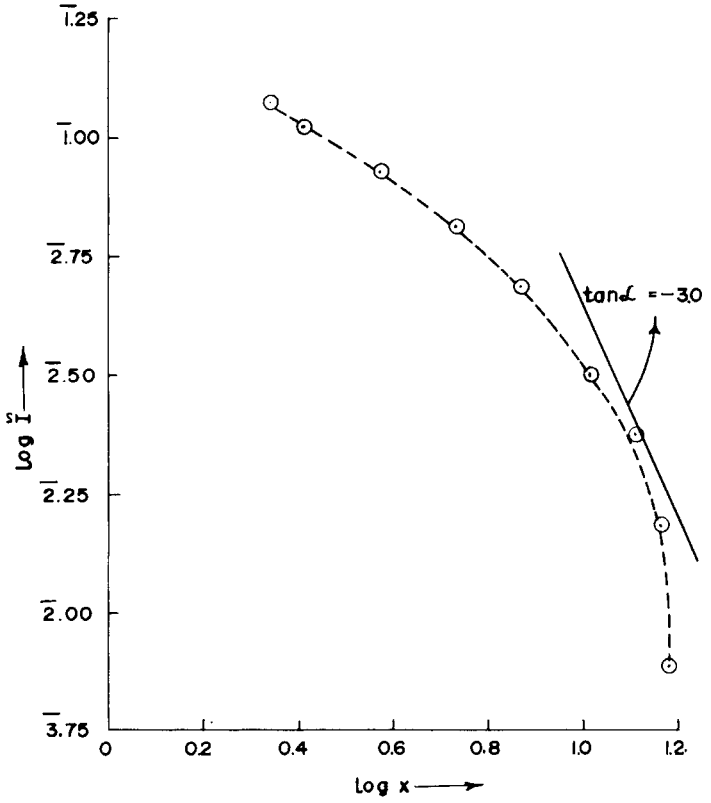


Fig. 5. Double logarithmic plot.

double logarithmic plot (Fig. 5). Therefore, the sample can be treated as one belonging to two phases.

The primary beam was exposed for 15 sec employing the film-shifting device.[21] This was microphotometered, and then the intensity-versus-X curve was drawn and its area planimetered. After this P_0, the primary beam intensity, was cal-

culated using the relation

$$P_0 = \frac{\text{area under the curve of primary beam}}{\text{time of the exposure of the primary beam}} \times \frac{\text{maximum time of exposure of the sample}}{\text{microphotometer transformation factor}}$$

The value of P_0 thus calculated is 0.936.

THEORY AND EVALUATION OF PARAMETERS

Air Fraction

For a densely packed system having two phases, the invariant $\tilde{Q}$ of the scattering curve introduced by Debye[22] and also by Porod[11] is given by

$$\tilde{Q}_{\text{exp}} = \int_0^\infty \tilde{I}(X) X \, dx$$

for smeared out intensity. This is evaluated by planimetering the area under the curve (Fig. 6), which yielded the value of the experimental invariant to be 54.1×10^{-4} cm^2.

The effective sample thickness D is given by Kratky[23] as

$$D = \frac{\delta_a}{\delta_c} \phi$$

where δ_a is the apparent density of the sample = 1.35 g/cc, δ_c is the compact density of cellulose = 1.6 g/cc; and ϕ is the diameter of the mark-capillary tube = 0.15 cm. Substituting these values we get D = 0.120 cm.

The electron density of cellulose is given after Kratky[24] as

$$\rho = \delta_c \frac{\Sigma 0}{\Sigma A} = 0.8480,$$

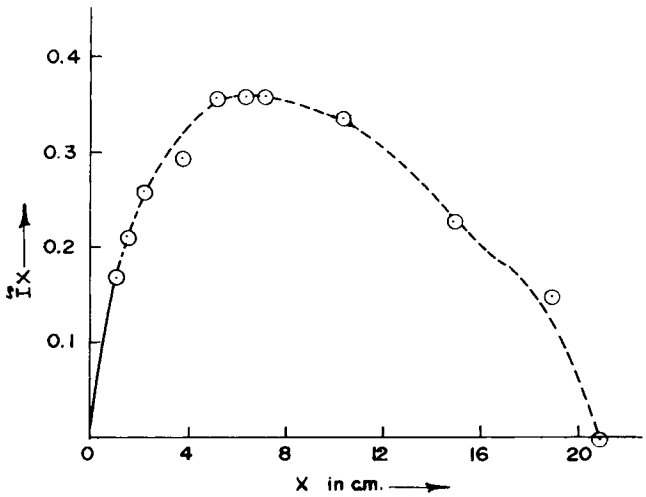


Fig. 6. The invariant $\tilde{Q} = \int \tilde{I}(x) x \, dx$.

where $\Sigma 0$ is the sum of atomic numbers and ΣA is the sum of the atomic weights.

The theoretical invariant $\tilde{Q}_{th}$ is given by

$$\tilde{Q}_{th} = \tfrac{1}{2}\pi(e^2/mc^2)^2\lambda^3 N^2 P_0 Dap\rho^2 w_1 w_2$$

where e is the electronic charge, m is the electronic mass, c is the velocity of light, λ is the x-ray wavelength = 1.5418 Å, N is Avagadro's number, w_1 is the volume fraction of void, and w_2 is the volume fraction of matter in a scattering inhomogeneity.

Equating $\tilde{Q}_{th}$ with $\tilde{Q}_{\text{exp}}$ and using the respective values we get $w_1 w_2 = 17.38 \times 10^{-4}$. Since $w_2 = 1$, the volume fraction of air or void contained in the sample is 0.17%.

Specific Inner Surface

The specific inner surface, that is, the phase boundary area per unit volume of the dispersed phase, is represented by Kratky[24] as

$$\frac{O}{V} = \frac{8\pi}{\lambda_a} w_1 w_2 \frac{\tilde{K}_1}{\tilde{Q}_{\text{exp}}} \qquad \tilde{K}_1 = \text{run constant (Fig. 4)}$$

Substituting the respective values yields the specific inner surface $O/V = 12.76 \times 10^{-6}$ Å^{-1}

Transversal Lengths

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

$$\bar{l}_1 = 4w_1/(O/V) = 544.9 \text{ Å}$$

$$\bar{l}_2 = 4w_2/(O/V) = 3.135 \times 10^5 \text{ Å}$$

Range of Inhomogeneity

The range of inhomogeneity l_r is given by

$$l_r = (4V/O)w_1 w_2 = 4(V/O)w_1 \because w_2 \simeq 1$$

or

$$l_r = \bar{l}_1 = 544.9 \text{ Å}$$

Length of Coherence

The coherence length l_c introduced by Guinier[25] is given by

$$l_c = (\lambda_a/\pi) \int_0^\infty \tilde{I}(X)\, dx \Big/ \int_0^\infty \tilde{I}(X)x\, dx$$

$$= (\lambda_a/\pi)(\tilde{E}/\tilde{Q}_{\text{exp}})$$

$$= 0.490a(\tilde{E}/\tilde{Q}_{\text{exp}})$$

where $\tilde{E}$ is the integrated scattered energy obtained by planimetering the area under the curve in Figure 2 and found to be equal to 0.73×10^{-2} cm^2. Substituting the respective values we obtain l_c = 15.21 Å as the coherence length.

Conclusions

The parameters determined are the air fraction, the specific inner surface, transversal lengths l_1 and l_2, and the coherence length l_c, which were found to be 0.17%, 12.76×10^{-6} Å^{-1}, 544.9 Å, 3.135×10^5 Å, and 15.21 Å, respectively. The scattering particles in cellulosic *Sansevieria roxburghi* may be lamillar in type.[26] These parameters may throw some light on the textile properties of the fiber.

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