E. M. BELAVTSEVA, K. Z. GUMARGALIEVA, A. I. KITAIGORODSKII

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Abstract

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E. M. BELAVTSEVA, K. Z. GUMARGALIEVA, A. I. KITAIGORODSKII

ELECTRON-MICROSCOPIC STUDY OF THE STRUCTURE OF CAPRON AND LAVSAN FIBERS TREATED WITH PHOSPHOTUNGSTIC ACID

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The main structural problem in the field of polymeric substances is the question of the shape, size, and combination of regions with different degrees of order. Methods of X-ray structural analysis prove not entirely adequate for solving this problem. We believe that the principal advances should be expected from high-resolution electron microscopy. Here, however, there are also difficulties. They are due chiefly to the limited possibilities of the various methods of shading and contrasting, as well as to the appearance of artifacts.

In this article we wish to point out a number of advantages of the method of contrasting with phosphotungstic acid, which we used in order to establish a number of structural features of capron and lavsan fibers.

Contrasting of objects with phosphotungstic acid (PTA) has become widespread in biology. In recent years the so-called method of negative contrasting, or negative staining, has appeared, in which the specimen is treated with neutralized PTA ([1]). Treatment of polymer fibers with phosphotungstic acid in a neutral medium to obtain negative contrast had not previously been carried out. There are, however, studies on staining perlon fibers ([2]) and nylon ([3]) with PTA in an acid medium, with preliminary treatment of the specimens with a solution of hydrochloric acid.

Our objects were contrasted with phosphotungstic acid in a neutral medium without preliminary treatment of the fibers, according to the procedure proposed by Brenner and Horne for biological objects ([1]), with some modifications. Fibers of capron and lavsan, dispersed mechanically for 3–5 min in distilled water, were studied. After this they were deposited on a grid covered with a formvar supporting film and dried in air.

Contrasting of the object with PTA was achieved in the following manner. A drop of a 2% aqueous solution of PTA, neutralized to pH 7.0–7.2 with a 1N KOH solution, was applied to the film with the object. After 3–5 min the drop was removed with filter paper, leaving a small amount of solution on the film. After drying, the objects were suitable for observation in the electron microscope. Another portion of the dispersed fibers, not treated with PTA, was contrasted by vacuum coating of the objects with a gold-palladium alloy. For comparison, electron micrographs were obtained of uncontrasted capron and lavsan fibers. The objects were studied in a JEM-5Y electron microscope.

Electron-microscopic images show that after dispersion capron and lavsan are broken up into individual fibers several microns long. Figure 1a gives an electron micrograph of an uncontrasted capron fiber at a magnification of 30,000×. From this figure it is evident that the internal structure of the synthetic fiber is not revealed because of insufficient contrast. When capron and lavsan fibers are shadowed with metal, in a number of cases a fibrillar structure of the objects is observed, the size of the individual fibrils being different. In Fig. 2a is pres—

Fig. 1. Dispersed capron fibers. *a*—object not contrasted; 30,000×; *b*—object treated with PTA at pH 7.2, 110,000×; *v*—same, 140,000×

Fig. 2. Dispersed lavsan fibers. *a*—object stained with a gold-palladium alloy, 35,000×; *b*—object treated with PTA at pH 7.2; 150,000×

A microphotograph of a Lavsan fiber shadowed with a gold-palladium alloy at a magnification of 35,000× was presented. However, in this case as well it is not possible to reveal the internal structure of the fiber, but only to obtain an idea of the surface of the object.

Treatment of specimens with phosphotungstic acid in a neutral medium makes it possible to reveal the internal structure of the fiber over a depth of several layers. Figures 1b and 2b show electron micrographs of dispersed Kapron and Lavsan fibers, contrasted with PTA, at magnifications of 110,000× and 150,000×, respectively. In these images one can see fibers consisting of individual fibrils; the dark regions correspond to phosphotungstic acid, the light ones to fibrils. The fibril width for Kapron averages 50-70 Å, and for Lavsan 80-100 Å. For Lavsan, a periodicity of 100-110 Å is clearly revealed in the alternation of dark and light regions across the fiber. As is known, small-angle X-ray diffraction shows a periodicity of 100-150 Å for Lavsan. Thus, the results obtained by us for Lavsan with the aid of electron microscopy agree with the X-ray data. For Kapron, electron-microscopic images show a large scatter in the periodicity of alternation of dark and light regions across the fiber (100-200 Å).

In the micrographs presented in Figs. 1b and 2b, it is seen that individual fibrils do not continue along the entire length of the fiber. Their length for Kapron and Lavsan is 500-3000 Å. The PTA regions located between the fibrils have approximately the same length.

In individual cases, for Kapron a periodicity along the fibril was observed in the form of globules with an average size of 120-150 Å. The globular structure of individual fibrils was observed mainly in regions with a high PTA concentration (Fig. 1b). It may be assumed that in this case phosphotungstic acid penetrates into disordered regions and reveals the periodicity along the fibril. Probably much more often PTA merely rims the fibril, owing to which the form of an individual fibril with varying width along its length is observed.

Kapron and Lavsan fibers after treatment with PTA differ in the arrangement of the individual fibrils. The Lavsan fiber consists of individual fibrils arranged predominantly parallel to one another. In Kapron, on the contrary, regions consisting of intersecting fibrils are often encountered. The fibrils of Kapron fiber, as a rule, are separated by broader interlayers of PTA. However, it remains unclear whether such a difference existed in the original fibers or appeared as a result of the different staining action of PTA on Kapron and Lavsan.

Thus, treatment of Kapron and Lavsan specimens with phosphotungstic acid, which strongly scatters electrons, made it possible to reveal the internal structure of the fiber owing to a sharp increase in contrast. With such treatment the object in the image appears light against a dark background, i.e., an anomalous "staining" effect takes place (1). Consequently, for synthetic fibers, by means of neutralized phosphotungstic acid it is possible to obtain negative contrast. The negative-contrast method gives more detailed information on the morphological structure of Kapron and Lavsan fibers than does shadowing of objects with heavy metals. In addition, contrasting of polymers by means of neutralized phosphotungstic acid is convenient because of the simplicity and speed of specimen preparation.

Institute of Organoelement Compounds
Academy of Sciences of the USSR

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CITED LITERATURE

1. E. Brenner, R. Horne, *Biochim. et biophys. acta*, **34**, 103 (1959).
2. K. Hess, E. Cütter, H. Mahl, *Koll. Zs.*, **168**, 37 (1960).
3. B. J. Spit, Fifth Intern. Congr. for Electron Microscopie, **1**, BB-7, Philadelphia U. S. A., 1962.

Note: Figure translations are in progress. See original paper for figures.