Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602102285

STAINING AND OTHER MICROSCOPIC TECHNIQUES FOR TEXTILES

E. K. Boylston

Southern Regional Research Center, P. O. Box 19687, ARS, USDA, New Orleans, LA 70179

Microscopical procedures for fibers are important to the textile industry in evaluating natural fiber maturity, mixed fiber blends, dyes and chemical finishes on fabrics. Staining (color for light microscopy and electron dense staining for TEM) of textile fibers as well as solubility and swelling tests along with special embedding methods for image analysis and Fourier Transform Infrared microscopy/spectroscopy are all essential in determining fiber properties and finishes. The Goldthwait test (ASTM D-1461) for determining maturity of cotton fibers is a differential dye test which reflects differences in effective pore size by using a combination of two dyes, Direct Green 26 (C.I. 34045) and Direct Red 81 (C.I. 28160). Immature, thin-walled fibers dye green, while mature, thick-walled fibers dye red. There are a number of "universal stains" composed of dye mixtures which have been developed for the identification of textile fibers. Testfabrics Inc. has several stain identification products that will stain different fibers (acetate or viscose rayon, acrilan, arnel , cotton, creslan, dacrons, nylons, orlon, silk, wood, wool, etc.) distinctive colors which can be identified from the supplied standard fiber strip. All natural fibers have a distinctive cross-sectional shape which also helps in identification by light microscopy. In many cases evidence obtained microscopically may be useful in the study of fiber damage and its causes. Types of suspected damage are abrasion, compression, tensile break and heat damage, as well as microbial damage due to enzymatic attack of microorganisms, or chemical damage. Electron microscopy of nearly all organic material requires some type of method to improve contrast because of low atomic masses present. This is especially true in the study of the substructure of cellulosic fibers such as cotton. The fiber is made up of concentric cellulose layers composed of an aggregate of fibrils, further divided into even smaller elementary fibrils. Exact measurement of fibrillar size is extremely difficult because of the inherent lack of contrast within the cellulose polymer due to low atomic weight. Electron staining encompasses two general approaches: a) negative staining with a substance such as phosphotungstic acid, where the electron-dense substance is deposited around the material, creating a negative contrast (Figure 1.), and b) positive staining, where the heavy metal is chemically bound to the material being stained. With protein fibers, positive staining is not a problem, but in cellulosic fibers, normally the cellulose polymer must be modified by attaching acidic, basic or unsaturated groups (reactive to heavy metal compounds) to the cellulose chain (Figure 2).

The expansion method is a microscopical procedure whereby cotton fibers are embedded wet in a low molecular weight methacrylate polymer[1]. Water in the fibers is replaced by the liquid plastic which is then further polymerized to a solid, which forces the layers of cellulose within the fiber apart. This procedure is useful in determining a) inter-fibrillar cross-linking (cross-linking between cellulose layers) - no swelling, b) fiber mercerization (swelling pattern of the fiber), and c) finishing agent deposition within the fiber, and particularly those finishes which do not contain elements easily detected by EDX.

Cuene, a cellulose solvent, is used to determine cross-linking of cellulose in cotton fabric treated for durable press, flame retardant finishing, etc.[2]. Cross-sections are mounted on TEM grids, covered with a drop of 0.1M cuene solution for 30 min., rinsed and examined in the TEM. Cotton fibers that are crosslinked will not dissolve in cuene solutions. Intra-fibrillar cross-linking (cross-linking within a

cellulose layer) is apparent when fiber cross-sections have expanded but do not dissolve in cuene.

Three special embedding methods have been developed for cotton and cotton fiber blends[3]. 1) A rapid embedding method employing UV polymerization reactions has been devised for embedding fibers in acrylic and methacrylate media. After 10 min., the resultant thin, flat embeddings are suitable for both light and electron microscopy. 2) A process for the evaluation of yarns by light microscopy/image analysis has been developed. Approximately 2000 fibers before spinning, 50 yarn segments after spinning, or yarns removed from fabric after processing, can be encased in a tube, embedded in methacrylate plastic, quickly UV polymerized, and sectioned. All yarns are discretely separated and individual fibers are distinct so that image analysis and statistical evaluations are possible. Fiber maturities, yarn evenness, dye penetration, and position of different types of fibers in blend yarns are all visible and readily quantified. 3) A procedure for embedding cellulosic textiles has been developed for FT-IR microscopy whereby fibers are embedded in polystyrene. This polymer does not absorb in the same regions of the infrared spectrum as cellulose or traditional acrylate and epoxy resins that contain chemical groups in common with cellulose. Additionally, use of cross-sections mounted on a KBr disk has the advantage of better resolution than grinding and pressing fibers in a KBr disk.

References

1. E. K. Boylston and L. L. Muller, Journal of Applied Polymer Science, 19(1975)1079.
2. E. K. Boylston, et al., Textile Research Journal, 45(1975)790.
3. E. K. Boylston, et al., Biotechnic and Histochemistry, (1991)122.

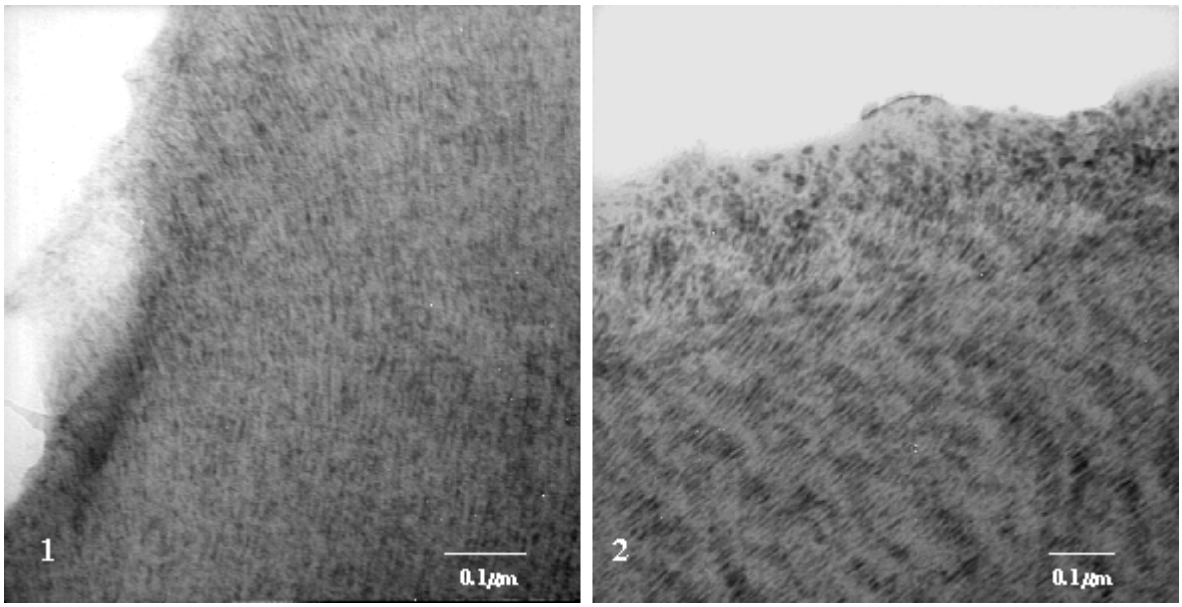

Fig. 1. Cotton fiber longitudinal section negatively stained with 2% phosphotungstic acid.
Fig. 2. Cotton fiber longitudinal section positively stained with 2% OsO_4 after treatment with sorbyl chloride.

https://doi.org/10.1017/S1431927602102285 Published online by Cambridge University Press