B149 Combinatorial Optimization of the Extraction of Dyes From Textile Fibers

Amy R. Stefan, BS, Christopher R. Dockery, BS, Alexander A. Nieuwland, PhD, Samantha N. Roberson, James E. Hendrix, PhD, and Stephen L. Morgan, PhD, University of South Carolina, Department of Chemistry and Biochemistry, 631 Sumter Street, Columbia, SC 29208; and Mark L. Miller, PhD, FBI Laboratory, Counterterrorism and Forensic Science Unit, FBI Academy, Quantico, VA 22135*

The goal of this presentation is to report the development of automated procedures for the extraction of dyes from textile fibers and analysis of dye extracts by spectroscopic methods for possible adaptation to forensic casework.

This presentation will impact the forensic community and/or humanity by demonstrating how optimization of extraction conditions can provide the analyst with the maximum amount of dye, possibly providing additional discrimination between forensic fiber evidence.

This presentation will report the use of a Beckman-Coulter BioMek® 2000 laboratory automation workstation to systematically investigate a combinatorial approach to the development of micro-scale extraction techniques for a broad range of dye classes from a range of textile fiber types. The laboratory workstation was programmed to extract dyes from small fiber samples. The resulting extracts were used to quantitatively determine the extraction efficiencies of different extraction solvents. Details of the programming and extraction technology will be presented along with experimental results, conclusions, and potential impact on forensic science.

Use of an automated workstation to extract dyes from fibers presents several advantages. Both speed of analyses and sample throughput are drastically increased versus manual extraction. In addition, the automated workstation gives the ability to program combinatorial experiments for the determination of the solvent mixtures that will provide the most efficient extraction conditions possible for a given fiber-dye interaction. Extraction efficiencies were quantitatively determined via designed combinatorial experiments and generation of surface response models.

Knowledge of the chemistry of fibers and fiber dyes is important to developing extraction techniques. Depending on the chemistries of the dye-fiber interaction, dyes may be loosely associated with fibers (e.g., direct dyes), bound by salt linkages (e.g., acid dyes), covalently bound to the fiber (e.g., reactive dyes), or simply dispersed as finely divided pigments in the fiber (e.g., disperse dyes). Dyes may also be applied during melt spinning of thermoplastic fibers (e.g., pigment coloration of nylon, polyolefins and polyester) or adhered to the fiber surface with adhesives (e.g., pigment dyeing of bedding and apparel fabrics).

The following fiber/dye combinations have been successfully extracted using automated extraction techniques: acid dyes on nylon, basic dyes on acrylic, direct, vat, or reactive dyes on cotton, and disperse dyes on polyester. Acid dyes usually contain sulfonic or carboxylic acid groups that form salt linkages to the nylon. Basic dyes on acrylics, also called cationic dyes, contain basic groups that form salt linkages to the acrylic. Cotton is usually dyed using direct, vat, or reactive dyes. Direct dyes form hydrogen bonds with the cellulose, and usually contain sulfonic or carboxylic acid groups, rendering them soluble in water. Vat dyes, which are usually quinone structures, are reduced to their water-soluble leuco form for application to the fibers, and re-oxidized to form insoluble pigments to improve wash fastness. Reactive dyes contain functional groups that form covalent bonds to the cellulosic hydroxyl groups. Lastly, disperse dyes on polyester are finely divided organic pigments dispersed throughout the fiber.

Dyes were extracted from fibers using the automated workstation, and loaded into a 96-well plate system. In this 96-well plate system, each well contains a 500-1L glass insert. A Teflon liner was placed between the glass inserts and a plastic lid to minimize evaporation of the extraction solvent during high-temperature extractions. To further minimize evaporation of solvents during extraction, the assembled plate was clamped in an aluminum press to improve the seal between the Teflon liner and the glass inserts. Extracted dyes were analyzed by capillary electrophoresis (CE) with a PACE-MDQ CE system using diode array detection. Optimization of extraction conditions provides the analyst with the maximum amount of dye, possibly providing additional discrimination between forensic fiber evidence.

Textile Fibers, Dyes, Automated Extraction

 ** Presenting Author*