ARCADIA UNIVERSITY
— FORENSIC SCIENCE PROGRAM —

Application and Analysis of Chemical Testing on Regenerated Cellulose Fibers

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Introduction

Fibers can be key pieces of associative evidence. One method that can be used in fiber analysis is chemical testing, a quick, easy, and relatively cheap presumptive method to qualitatively identify fiber type. Chemical tests such as zinc chloro-iodide, based on color change, and cupriethylenediamine hydroxide, based on solubility, are known tests for natural fiber cellulose. Regenerated cellulose fibers are a type of semisynthetic fiber made from the chemical processing of cellulose in order to make them more useful. Viscose rayon, cuprammonium rayon, modal, and acetate are the main classifications of regenerated cellulose fiber and they can be used to make items such as clothing, yarn, and thread.

The purpose of this research was to apply the chemical tests to various modern regenerated cellulose samples to test if actual results were consistent with expected results. While there is some information about the possibility to identify these fibers using zinc chloro-iodide and cupriethylenediamine hydroxide, actually putting these tests to use with modern samples shows any consistencies and discrepancies with previously published data. This helps determine the value of the zinc chloro-iodide and cupriethylenediamine hydroxide chemical tests in forensic science.

The fibers chosen were sourced from threads, garments, fabrics, and yarns of different dyes, textures, and manufacturers. These differences could all potentially affect how the fibers react. Samples were compared between classes and within classes. For the observation of false positives, synthetic fibers, which contain no cellulose, were also tested.

Materials and Methods

- **-Positive controls:** 100% white cotton and 100% off-white cotton samples (cotton contains ~85-90% cellulose)
- **-Negative control:** 100% off-white polyester (contains no cellulose)
- **-Semisynthetic regenerated cellulose fibers:** viscose (n=6), modal (n=11), rayon (n=8), acetate (n=7)
- **-Synthetic fibers:** polyester (n=8), acrylic (n=3), nylon (n=6)
- -Fibers were obtained from samples by using tweezers and scissors
- -12 regenerated cellulose blind samples were tested for accuracy of the tests
- -This method was repeated for all samples in three separate trials for an evaluation of reproducibility

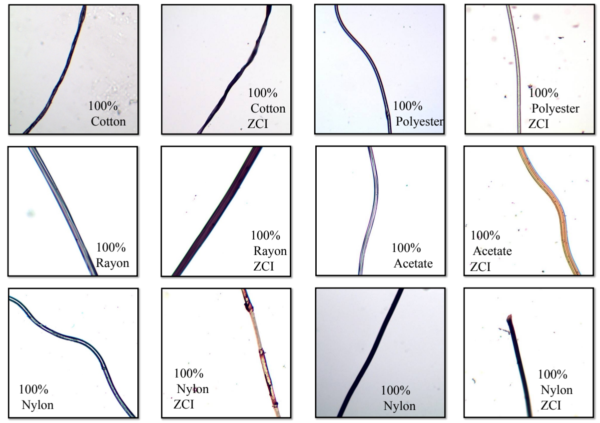
Zinc chloro-iodide:
Fibers (n=10) of each sample were put on a microscope slide without chemical reagent. More fibers (n=10) from the same sample were put on another microscope slide and reagent was added to these fibers. A few drops of zinc chloro-iodide were added to the fibers by using a glass pipette and bulb in the fume hood. The drops were enough to ensure all of the sample was covered with reagent. A glass coverslip was put on top of the sample while pushing down on it slightly so the reagent was evenly spread on all fibers. The slides were viewed immediately with a compound light microscope (Leica DM750P) with a total magnification of 100x and 200x. Color changes were observed, as well as the original color of the fiber, and recorded.

Cupriethylenediamine hydroxide:
Fibers (n=10) of the sample were put on a microscope slide without chemical reagent. More fibers (n=10) from the same sample were put on another microscope slide and reagent was added to these fibers. A few drops on 0.5M cupriethylenediamine hydroxide were added to the samples by using a glass pipette and bulb in the fume hood. The drops were enough to ensure all of the sample was covered with reagent. A glass coverslip was put on top of the sample while pushing down on it slightly so the reagent was evenly spread on all fibers. The slides were viewed immediately with a compound light microscope (Leica DM750P) with a total magnification of 100x and 200x. Solubility changes were observed, as well as the original structure of the fiber, and recorded. For fibers that appeared insoluble, they were observed for up to 5 minutes.

-Both procedures were repeated for all samples in three separate trials for an evaluation of reproducibility

-Digital photographs were taken with Motic BA310Pol, MotiCam 3.0MP, and Motic Image Plus 2.0 Software at 50x total magnification

Results



Expected Results for Zinc chloro-iodide: Blue-violet to wine-red on purer cellulose, blue-green for viscose rayon, brown for cuprammonium rayon, violet for cotton, yellow for acetate. Most other fibers will turn yellow-brown shades.

Actual Results for Zinc chloro-iodide:
- **-Positive Controls:** Maroon-purple, maroon-brown, dark purple, consistent with expected results
- **-Negative Control:** Slight yellow to yellow-brown tint, consistent with expected results

Semisynthetic Regenerated Cellulose:
- **-Viscose** Maroon, brown, purple, consistent with positive controls, inconsistent with expected results for viscose rayon, but consistent with other expected results
- **-Modal:** Maroon, brown, purple, consistent with positive controls and expected results
- **-Rayon**: Maroon, brown, purple. Some variation with a lot of grey also being observed that wasn't as prominent in the other fiber types, consistent with positive controls as the main color, consistent with viscose rayon with grey color closely coinciding with a blue-green expected result, but unknown if viscose or cuprammonium process was used in manufacturing
- **-Acetate:** Orange-yellow to orange, inconsistent with positive controls and consistent with expected results. Also a distinct color difference from the other regenerated cellulose samples
- **-Pink Samples** of viscose, modal, and rayon also exhibited a pink color in some fibers. The pink sample of acetate did not exhibit this reaction

Synthetic:
- -Most had yellow tint or faded color, consistent with negative control and expected results

False Positives:
- **-Nylon:** Some samples behaved as the negative control in the body of the fiber, but the end turned yellow/orange, while some other nylons had alternating bands of negative control color and yellow/orange, inconsistent with negative control, false positive for acetate
- **-Orange-red Polyester:** Orange, inconsistent with negative control, false positive for acetate
- **-Victorian-rose Acrylic:** Orange, inconsistent with negative control, false positive for acetate
- **-Dark Country Blue Acrylic:** Grey, grey-brown, inconsistent with negative control, false positive for rayon

Expected Results for Cupriethylenediamine hydroxide: Solubility. Dissolves cellulose material.
Actual Results for Cupriethylenediamine hydroxide :
- **-Positive Controls:** Immediately soluble, consistent with expected results
- **-Negative Control:** Insoluble after 5 minutes, consistent with expected results

Semisynthetic Regenerated Cellulose:
- **-Viscose and Modal:** Immediately soluble, consistent with positive controls and expected results
- **-Rayon:** Slow solubility for 30 seconds, then quickly dissolved, slight variation from positive controls, consistent with expected results
- **-Acetate:** Slow solubility, inside of fiber becomes slightly soluble, while structure of fiber left intact after 5 minutes, difficult to distinguish for some samples, variation from positive controls, slightly inconsistent with expected results

Viscose Thread vs Modal Garment: Similar color change and solubility, different sources did not affect results

Results for Blinds:
The unknowns which were acetate were correctly presumed. The unknowns which were modal and rayon were correctly presumed as not acetate.

Conclusion

This research shows how fiber type and dyes influence results of these chemical tests. There was variation across the regenerated cellulose classes. This can be seen with the acetate and rayon samples with both the zinc chloro-iodide and cupriethylenediamine hydroxide tests. There was also variation within the classes since not every fiber exhibited the same exact color change when zinc chloro-iodide was applied, however, they were very similar. Fibers need to be light in color to see the color change because the color change is not evident on fibers that are too dark. Fibers also need to be dyed different colors than the positive color changes to avoid false positives. Even though the negative fibers were all pure synthetics, they did not test the same as the pure synthetic negative control. While cupriethylenediamine hydroxide dissolves cellulose and is a known solvent, the acetate samples were difficult to distinguish and had very slow solubility which could lead to a false negative.

These tests may assist in helping determine if a fiber is a regenerated cellulose fiber, however, it may not be able to distinguish the specific class. This can been seen when testing the blinds since only acetate could be eliminated as a class. Thus, chemical processes, dyes, and cellulose percent content are all likely contributors to the variation in color and solubility changes.

Chemical tests are a destructive technique, but can be used when multiple fibers are present in a forensic setting. In conclusion, zinc chloro-iodide and cupriethylenediamine hydroxide are beneficial presumptive tests used to assist in qualitatively identifying fiber classes, but other tests are still needed to confirm identities.

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