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Identification of Dye Compounds in Chinese Artifacts by GC MS

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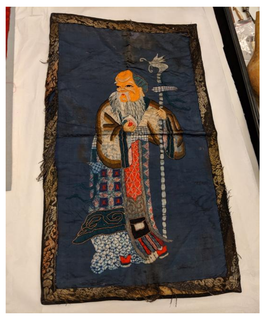
BACKGROUND

- UNI Museum homes wide variety of textile artworks.
- Many of them have historical significance. Two of them are UNIM 2005.2.24 (Tapestry) and UNIM1986.14.1985.1.124 (Hanging).



- Rectangular Textile
- Dimension: 36cm × 69cm
- Dark blue silk embroidery of an old bearded man believed to be Chinese god, Shou-Hsing
- Contrasting floral designed 1'' border
- "Made in China" stamp on the plain cotton fabric back

Figure 1: UNIM 2005.2.24 (Tapestry)

- Silk fabric
- Dimension 158cm × 74cm
- Dimension: 36cm × 69cm
- Depiction of an elderly man like the one in Chinese folklore about prosperity
- Assumed to be of Chinese origin

Figure 2: UNIM 1986.14.1985.1.124 (Hanging)

OBJECTIVES

- Identify the dye compounds in textiles.
- Determine if the dyes used in textiles are natural or synthetic.
- Date the textiles based on dye compounds.

SAMPLE PREPARATION

- Two different methods: HCl method and EDTA method were used for extraction of sample for GC-MS analysis.
- For HCl method, 0.2 mg sample was placed in 400µL of 37% HCl+ methanol + water in the ratio of 2:1:1 in microcentrifuge tube. The solution was heated at 100 °C for 10 minutes.
- For EDTA method, 0.2-mg sample was placed in microcentrifuge tube with 400µL of 0.001 M aqueous H_2EDTA + acetonitrile + methanol in the ratio of 2:10:88. The solution was then heated at 60 °C for 30 minutes.

Figure 3: Vacuum Desiccator

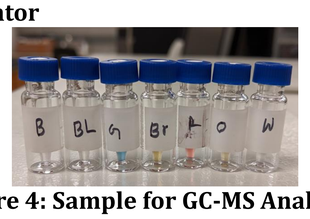


Figure 4: Sample for GC-MS Analysis

- The solution was dried under vacuum desiccator for both methods.
- The residues were dissolved in 50µL of methanol +water in the 1:1 ratio with the aid of a vortex mixer.
- The tubes are then centrifuged, and the solution is removed for the analysis.[3]

INSTRUMENTATION

- GC-MS is the combination of two different analytical techniques.: Gas Chromatography(GC) and Mass Spectroscopy(MS)
- GC separates the components in gas and MS identifies the compound.
- The samples are vaporized in injector port and carried by carrier gas through the column.
- The sample moves through column placed inside the temperature-controlled oven with stationary phase coated on the inner wall .
- The sample gets separated based on their volatility and size.
- They pass through the transfer line exiting GC to the MS.
- The samples get ionized and are detected based on their charge to mass ratio.
- The data is then analyzed using the library of samples to identify the compound.

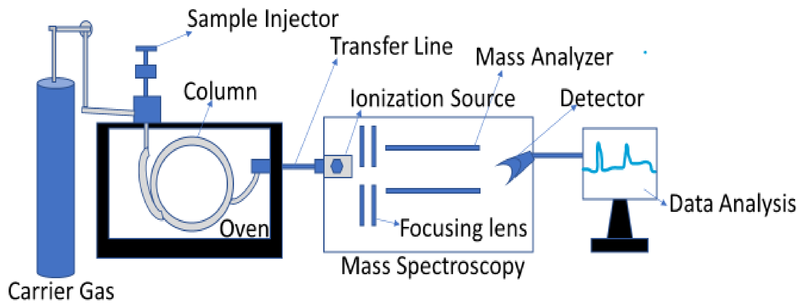


Figure 5: Schematic of GC-MS

RESULTS

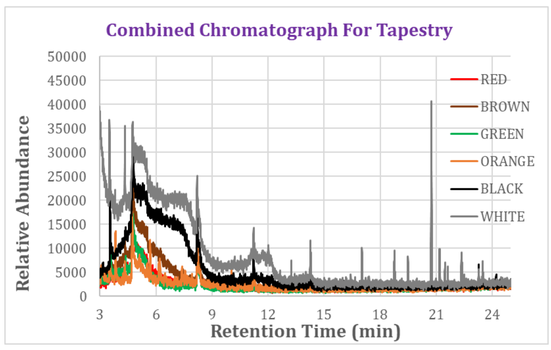


Figure 6: Chromatograph of Tapestry Samples

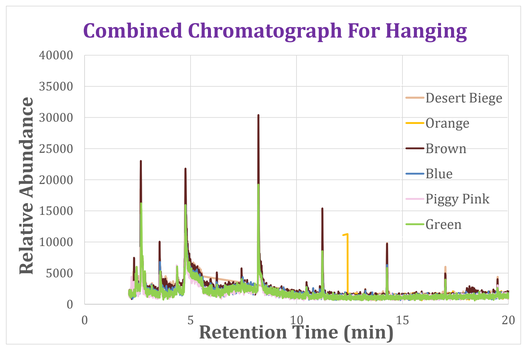


Figure 9: Chromatograph of Hanging Samples

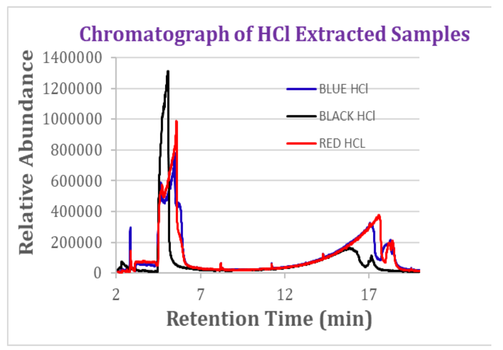


Figure 12: Chromatographs of Samples Extracted by HCl

- All the HCl extracted samples did not result in useful identifying data (Figure 12).

- Figure 6 and Figure 9 shows the chromatograms of all the samples extracted by EDTA method for Tapestry and Hanging respectively.
- Common Peaks present in all samples are assigned as background.

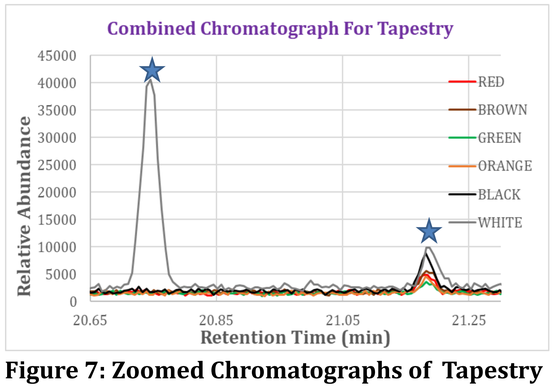


Figure 7: Zoomed Chromatographs of Tapestry

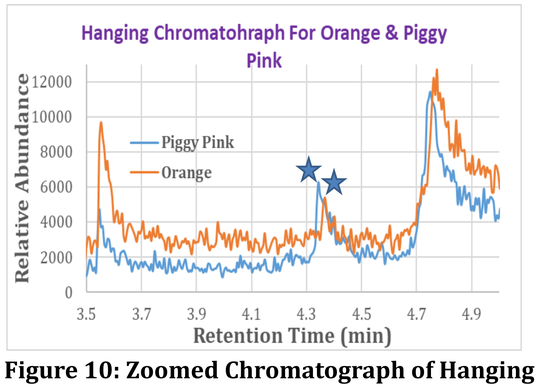


Figure 10: Zoomed Chromatograph of Hanging

- Unique dye compounds are labeled with stars in figures 7, 8, 10 & 11 as case examples.
- Dye components like triazine, anthraquinone, imidazole, quinoline, triphenylamine, indole, triphenyl methane, and anthracene were found in the samples from both textiles. These are known components of industrial dying techniques.

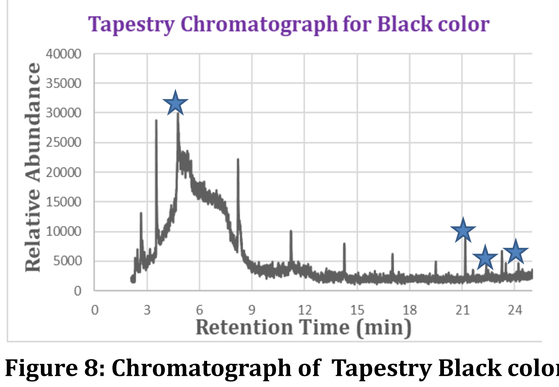


Figure 8: Chromatograph of Tapestry Black color

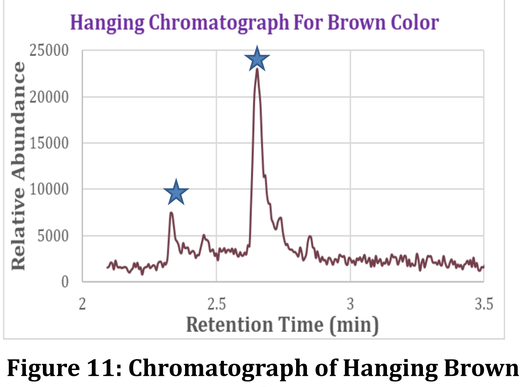


Figure 11: Chromatograph of Hanging Brown Color

CONCLUSIONS

- Dye compounds are identified in the sample of both textile artifacts: Tapestry and Hanging analyzed.
- All the dyes identified in both textiles are synthetic dyes.
- As the development of synthetic dye first began in 1856 and were imported in China in 1871, it can be concluded that the artifacts were made in late 19^{th} century .[2]

ACKNOWLEDGEMENT

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REFERNCES

1. Granger, R. M.; Yochum H. M.; Granger, J. N.; Sienerth, K. D.; Instrumental Analysis*:* Oxford University Press: Oxford. 2017.
2. Jing, H. The Historical and Chemical Investigation of Dyes in High Status Chinese Costume and Textiles of the Ming and Qing Dynasties (1368-1911). PhD thesis, University of Glasgow. 2016; 43-44.
3. Zhang, X.; Laursen, R.A. Development of Mild Extraction Method for the Analysis of Natural Dyes in Textiles of Historical Interest Using LC-Diode Array Detector -MS. *Anal.Chem.* 2005, 77, 2022-2025.