Identification of Natural Dyes in Ancient Peruvian Mummy Cloths using LC/MS/MS

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Artifact Introduction
- Collected in the 1960s by researchers from and donated to the museum in the 1970s
- Undetermined if the researchers violated UNESCO protocols
- Made of wool and dyed with a variety of natural dyes that are made of plants or insects
- Have different colors and patterns that represent status of the Peruvian mummy
- Possibly thousands of years old

Chemical Introduction
- The spun yarn is boiled with the dyestuff and mineral salts are added to help the color stick to the wool
- Dyestuff in ancient Peru were often made of plant or animal materials
- Many natural wool colors such as black, brown, and tan are often just washed with a natural detergent made from the Jabonera plant

Dyes
- The common dyes used in Peru are listed below
  - Red-Cochineal Beetle
  - Yellow- Safflower

Dye Extraction Method
- An acid extraction was performed on the fabrics because the acid can hydrolyze dye molecules from their inorganic mordants
  1. Small (1 cm) pieces of fabric were placed into micro test tubes with a 250μl solution of HCl/MeOH H₂O (2:1 v/v)
  2. The samples were placed in 105°C water and heated for 10 minutes
  3. Then evaporated inside a centrifuge under vacuum at 40°C
  4. The residue of the samples were rehydrated with 200μl solution of MeOH H₂O (1:1 v/v)
  5. They were then extracted and placed into MS vials for analysis

Table Summary

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Compounds Found</th>
<th>Identified</th>
<th>Key Compounds Identified</th>
</tr>
</thead>
<tbody>
<tr>
<td>1970.9.0019 (Tan)</td>
<td>12</td>
<td>34 Found</td>
<td>N/A</td>
</tr>
<tr>
<td>1970.9.0019 (Red)</td>
<td>6</td>
<td>51 Found</td>
<td>Carminic Acid</td>
</tr>
<tr>
<td>1970.9.0024 (Tan)</td>
<td>13</td>
<td>23 Found</td>
<td>Salicylaldehyde</td>
</tr>
<tr>
<td>1970.9.0024 (Black)</td>
<td>7</td>
<td>52 Found</td>
<td>L-Histidine</td>
</tr>
<tr>
<td>1970.9.0025 (Brown)</td>
<td>8</td>
<td>18 Found</td>
<td>Vanillin</td>
</tr>
<tr>
<td>1970.9.0025 (Red)</td>
<td>11</td>
<td>25 Found</td>
<td>Salicylaldehyde</td>
</tr>
<tr>
<td>1970.9.0031 (Brown)</td>
<td>9</td>
<td>36 Found</td>
<td>Carminic Acid</td>
</tr>
<tr>
<td>1970.9.0032 (Brown)</td>
<td>10</td>
<td>17 Found</td>
<td>Vanillin</td>
</tr>
<tr>
<td>1970.9.0034 (Brown)</td>
<td>5</td>
<td>15 Found</td>
<td>Vanillin</td>
</tr>
<tr>
<td>1970.9.0034 (Tan)</td>
<td>14</td>
<td>24 Found</td>
<td>Chymostatin</td>
</tr>
</tbody>
</table>

Example Structures of Identified Natural Products

Conclusions
- In the brown samples, they have been dyed with a type of plant
- The red samples have been dyed with Cochineal Beetle
- The black sample was most likely made of natural black wool

Future
- Make a larger library of dyes so they can be identified by the auto MS/MS

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Citations
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