Investigating cotton dyeing using exotic wood waste sawdust

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Abstract: Investigating cotton dyeing using exotic wood sawdust. The study investigated the possibility of using extracts from various species of exotic wood, obtained from waste sawdust, for dyeing cotton fabrics. Material from Obeche, Iroko, Merbau, Apple tree, American walnut, Tulip tree, Cumaru and Rosewood were used for the tests. Solutions of oxalic acid, tin chloride, aluminum sulfate and ferric chloride were used as mordants, along with no-mordant tests. The color was determined in the CIE L*a*b* coordinate system. Color fastness was tested using n-hexane for dry cleaning and acidic, alkaline and hydrogen peroxide bleach solutions. The results showed a strong dependence on the type of wood, the mortar used and the washing agent and covered a wide spectrum of color intensity and fastness. The best results were obtained for Rosewood, Merbau and Apple trees, while Tulip tree and American walnut turned out to be practically useless. Among the mordants used, the most intense colors were produced by metal salts, including ferric chloride, which strongly darkened fabrics. Dry cleaning proved to be the mildest of those tested, with the least effect on color fading.

Keywords: exotic wood, mordant, cotton, dyeing, color fastness

INTRODUCTION

In the wood industry, huge amounts of material are wasted every day in the manufacturing process. Semi-finished products such as wood chips, shavings and unsuitable components are often used to produce wood-based panels or gallantry. Despite the efforts of large companies, a sizable portion of the residue continues to go unused. In addition, a large part of wood waste is chips from the processing of exotic wood, which is very valuable in terms of aesthetic qualities, technological features and biological aspects - cutting down ecosystems in tropical countries. Once the material has been harvested, we should make every effort to use its potential in 100 percent. In the era of global trade, dozens of exotic woods from various parts of the world, including Asia, Australia and Oceania, are permanently available on the Polish market, and several hundred more can be imported by special order (Jankowska 2021).

The production of natural dyes for coloring upholstery materials from post-production chips allows maximum consumption of the material used. As an additive, one can move away from using artificial dyes that are often harmful to the environment and human or animal health (Hosen 2021, Brzozowska 2010). At the time of dye production, it is not important whether the material used has a desirable arrangement of fibers, uniform color, even shape or large size. Instead, what is important is that it is uncontaminated and that it can be processed to chips and wood dust.

Natural dyes were used as far back as antiquity extracted from fruits, flowers or tree bark (Chandrasekaran 2019), until they were displaced by the popularization of artificial dyes due to their lower price and greater range of colors available on the market (Ragheb 2017). Despite the several advantages of synthetic dyes, they produce harmful effluents and a carbon footprint harmful to the planet (Umbreen 2008).

To strengthen the color of the extract and its resistance, various natural and chemical mordants can be used (Vankar 2017) such as oxalic acid, alum, ferric or stannous salts. By using these mordants, several different shades and even completely different colors can be obtained from a single species. Some of them, like ferric salt are recognized not only as mordants, by the color modifiers as well.
MATERIALS AND METHODS

The list of species used in the study is showed in the Table 1. The moisture content of the chips was determined by oven-drying at 105 °C. The non-exotic Apple tree chips were tested for comparison.

Table 1. Results of dyeing cotton fabric with exotic wood extracts.

<table>
<thead>
<tr>
<th>Trade name</th>
<th>Scientific name</th>
<th>Region of origin</th>
<th>Moisture content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Obeche</td>
<td><em>Triplochiton scleroxylon</em></td>
<td>Africa</td>
<td>13.3%</td>
</tr>
<tr>
<td>Iroko</td>
<td><em>Chlorophora excelsa</em></td>
<td>Africa</td>
<td>17.8%</td>
</tr>
<tr>
<td>Merbau</td>
<td><em>Intsia sp.</em></td>
<td>Asia</td>
<td>10.3%</td>
</tr>
<tr>
<td>Apple tree</td>
<td><em>Malus domestica</em></td>
<td>Europe</td>
<td>9.3%</td>
</tr>
<tr>
<td>American walnut</td>
<td><em>Juglans nigra</em></td>
<td>North America</td>
<td>7.5%</td>
</tr>
<tr>
<td>Tulip tree</td>
<td><em>Liriodendron tulipifera</em></td>
<td>North America</td>
<td>10.1%</td>
</tr>
<tr>
<td>Cumaru</td>
<td><em>Dipterix odorata</em></td>
<td>South America</td>
<td>8.6%</td>
</tr>
<tr>
<td>Rosewood</td>
<td><em>Dalbergia sp.</em></td>
<td>South America</td>
<td>7.3%</td>
</tr>
</tbody>
</table>

The collected material was ground and 5 gram of each material was weighed for Soxhlet extraction according to Krutul (2002). Line-EtOH (Linegal, 96 % EtOH, 3 % Et$_2$O, 1 % t-Bu-O-Me) was used as a solvent for extraction in total volume of 170 cm$^3$. The extraction was conducted in a water bath for 10 hours with an overflow every 8-10 minutes.

The commercial bleached cotton fabric was used. The fabric was cut into 50×50 mm pieces and subjected to mordant treatment in a water bath (20 min, 95 °C, with occasional stirring). The mordants used were 2 % solutions of respectively: oxalic acid (anhydrous, (COOH)$_2$, Avantor), stannous chloride (SnCl$_2$·6H$_2$O, POCh), potassium aluminum sulfate (alum, KAl(SO$_4$)$_2$·12H$_2$O, POCh), ferric chloride (FeCl$_3$·6H$_2$O, POCh). Next, the fabric samples were pressed to remove excess of mordant solution, placed in glass vials, poured with appropriate wood extract and dyed in a water bath for next 20 min at 95 °C, with occasional stirring. A series of non-treated fabric samples were used for dyeing as well. After dyeing, the samples were rinsed with distilled water and air-dried at ambient temperature.

Coordinates in the CIELAB color space of raw and dyed fabrics was determined using ERICHSEN SPECTROMASTER 565-D. Total color change $\Delta E$ was calculated as the distance between points in the CIELAB color space (Molenda et al. 2021). All color determinations were taken as an average of measurement results at 5 points for each fabric sample.

Color fastness determination was limited to the selected set of samples with the most intense color. The samples were put into glass vials, poured with appropriate solution, and placed in a water bath for 30 min. The agents tested were as follow:

- Organic solvent for “dry cleaning” – n-hexane (C$_6$H$_{14}$, ChemPur),
- Acid – 0.04 M solution of sulfuric acid (H$_2$SO$_4$, prepared from 95% soln. ChemPur),
- Base – 1% solution of washing soda (Na$_2$CO$_3$, POCh),
- Bleach – 1% solution of hydrogen peroxide (H$_2$O$_2$, prepared from 30% soln. ChemPur).

In the case of hexane, the tests were conducted at 37 °C, in the case of water solutions at 98 °C. The samples were rinsed with distilled water and air-dried at ambient temperature. Then color coordinates of the samples were determined as described above, and $\Delta E$ was calculated.
RESULTS

Dyeing

The color of the fabric used was determined as $L^* = 85.54 \pm 0.19$, $a^* = -0.07 \pm 0.23$ and $b^* = 1.09 \pm 0.29$. The results showed good color uniformity of the fabric, thus color deviation due to dyeing can be precisely measured.

The results of dyeing are presented separately for each CIE $L^*a^*b^*$ coordinate in the following figures, while the Old World (Europe, Asia and Africa) species were presented separately from the New World (South and North America) ones.

The Old World Species

![Graph showing the results of dyeing for Old World species](image1)

Figure 1a. Colors of dyed fabrics – $L^*$ coordinate of CIELAB.

The New World Species

![Graph showing the results of dyeing for New World species](image2)

Figure 1b. Colors of dyed fabrics – $L^*$ coordinate of CIELAB

The results of dyeing are found to be strongly dependent on tree species. In the case of obeche and tulip tree, there are no visible changes in fabric lightness ($L^*$), except using ferric chloride as mordant (Figures 1a & 1b). Little darkening of fabrics was noticed for iroko, American walnut and cumaru, stronger for apple tree and merbau, and the strongest for rosewood. There were differences in mordant-color dependence as well, but there is no simple
relationship for all tree species, except of oxalic acid, which gave generally lighter color than raw fabric and ferric chloride, which gave much darker one.

In the case of a* coordinate (Figures 2a & 2b), stronger relative differences between tree species was observed. The lowest changes were obtained for tulip tree extract, independently on the mordant used, with a sole exception for the alum. Obeche extract was the second worst, giving only a small shift toward red color, observable for no mordant and ferric chloride, while for alum the change was noticeably stronger. Similar results were observed for Cumaru extract. In this case no mordant gave the only case of slight color change toward green, while oxalic acid and alum gave significantly stronger effect then the other mordants.
In the case of b* coordinate (Figures 3a & 3b), the most noticeable changes were generally observed, still strongly dependent on tree species and mordant used. Obeche extract caused the lowest changes in color, giving barely visible yellowish color, except of mordanting with alum, which gave fairly strong change. Among the American species, the lowest changes were observed for Tulip tree, with the exception again for the alum – in this case the highest b* coordinate change was observed. All American species caused rather small changes, especially in comparison to some of the Old World ones. Rosewood and Cumaru extracts gave more pronounced coloration then American walnut. Iroko extract gave clear change toward yellow color, which was in turn almost independent of the mordant used. Merbau and Apple tree extracts caused the highest changes in b* coordinate, giving very clear coloration. In both cases the strongest color was observed for the samples mordanted with tin and ferric chlorides.
Color fastness

The next step of investigation was to test color fastness. The samples of the most intense colors were selected and thus 11 dyed fabric series were tested, all samples of Merbau dyed, 4 of Rosewood dyed, as well as Iroko and Cumaru, treated with ferric chloride before dyeing. The results are presented as ΔE dependence on the test agent type, which is the best way for estimating color fading.

![Graph showing ΔE values for different dyed fabrics and treatments](image)

**Figure 4.** Colors of dyed fabrics – b* coordinate of CIELAB.

In the Figure 4 there are the results for Merbau and Rosewood at the weak dye-fiber connection, i.e. non-mordanted or treated with oxalic acid. No straight dependence was observed, as the lowest fastness was obtained for various agents. Organic solvent was the only agent, which gave small changes in all cases, though still over JND (just noticeable difference; Molenda et al. 2021). The strongest difference were observed where compared acid fastness – in this case fabrics mordanted with oxalic acid showed excellent color fastness, while non-treated – one of the worst. The influence of acid treatment on acid-fastness is thus significant. Alkali and oxidizer fastness are generally medium. Non-treated and Rosewood extract dyed fabric showed the worst peroxide fastness, while oxalic acid treated prior to dyeing showed significantly better. On the other hand, in the case alkali fastness inverse dependence was observed.

In the Figure 5 the results of color fastness using Lewis acid (except the ferric salt) were presented. Merbau dyed fabric showed generally lower fastness, while Rosewood dyed – one of the best. More pronounced dependences are observed in this case – the fabrics showed better fastness to dry cleaning and oxidizer, while much worse to acid and especially alkali treatment.
In the Figure 6 the results of color fastness using ferric chloride mordant were presented. Such a treatment gave intense dark coloration, thus more susceptible to fading. All samples tested showed the medium fastness, except of Merbau dyed ones, featuring very weak fastness unless dry cleaning. Rosewood dyed fabrics surprisingly showed the best fastness to alkali treatment, which can be connected to possible ferric salt or complexes formation with extractives.
CONCLUSIONS

The investigation presented proved the potential of exotic wood waste in textile manufacturing, though strongly dependent on the species. Thus Rosewood treated fabrics showed the best coloration, followed by Merbau and Apple tree. On the other hand, Tulip tree and American walnut gave quite unsatisfactory results. Strong dependence on used mordant type was observed as well. There is no universal recipe to obtain intense color, as the individual connecting of wood species and mordant type should be considered.

As a thumb rule, non-treated and oxalic treated fabrics were the least susceptible to dyeing, while the samples showed medium fastness. Alum and tin chloride as mordants gave intense and bright colors, but as a rule the fabrics showed poor fastness. On the other hand, ferric chloride gave intense dark color with good to medium fastness.

Color fastness strongly depends not only on wood species and mordant, but the agent as well. Dry cleaning showed generally to be the mildest, as gave the best fastness. Other, water-solution agents proved to be more aggressive, while some clear exceptions were observed, especially in the case of:

- Oxalic acid treated fabrics, which showed good fastness to acid,
- Tin chloride treated and Rosewood extract dyed fabric, which showed excellent fastness to peroxide,
- Ferric chloride treated and Rosewood extract dyed fabric, which showed excellent fastness to alkali.

There is still a long way to industrial application, but the results showed the suitability of some exotic wood species for eco-dyeing. Detailed selection of pre-treatment conditions in connection to fabric working and washing conditions is necessary though. UV exposure should be investigated further as well.

REFERENCES

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Streszczenie: Badanie możliwości wykorzystania odpadowej frakcji drewna egzotycznego do barwienia tkanin bawełnianych.

W pracy zbadano możliwość zastosowania ekstraktów z drewna egzotycznego różnych gatunków, pozyskanych z odpadowych trocin, do barwienia tkanin bawełnianych. Do badań użyto materiału z Obeche, Iroko, Merbau, jabłoni, orzecha amerykańskiego, tulipanowca, Cumaru i palisandru. Jako zaprawy użyto roztworów kwasu szczawiowego, chlorku cyny(II), siarczanu glinu i chlorku żelaza(III), równolegle przeprowadzono próby bez zaprawy. Barwa oznaczana była w systemie współrzędnych CIE L*a*b*. Trwałość barwy badano stosując n-heksan do prania chemicznego oraz roztwory kwaśny, alkaliczny i nadtlenku wodoru jako wybielacza. Wyniki wskazały silną zależność od gatunku drewna, zastosowanej zaprawy oraz czynnika piorącego i objęły szerokie spektrum intensywności i odporności barw. Najlepsze wyniki uzyskano dla drewna palisandru, Merbau i jabłoni, natomiast tulipanowiec i orzech amerykański okazały się praktycznie nieprzydatne. Spośród zapraw najintensywniejsze barwy dawały sole metali, w tym chlorek żelaza dawał silne ściemnienie tkanin. Pranie chemiczne okazało się najłagodniejsze ze zbadanych, najmniej wpływając na blaknięcie koloru.

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