

A New Purple?: Color Exploration and Extraction from the Catkins of Cottonwood Trees

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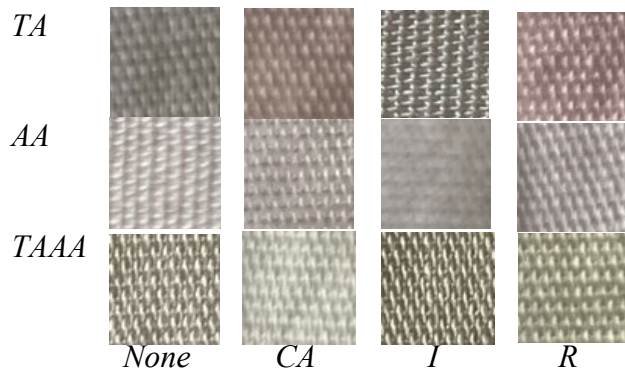
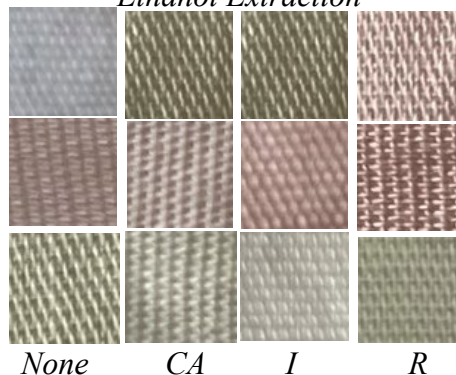
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Literature Review. Though the textile industry is an economic powerhouse, it remains one of the largest polluters (Desore & Narula, 2018). In the manufacturing process, vast amounts of water, fuel and various chemicals are utilized (Desore & Narula, 2018). Currently, an estimated 35% of the chemicals released into the environment are directly tied to textile processing and dyeing (Thiry, 2011). Waste water often contains compounds that are not biodegradable and potentially carcinogenic (Manzoor & Sharma, 2020). Consumer exposure could cause health complications, including respiratory issues and skin irritation (Manzoor & Sharma, 2020).

These issues are likely contributing to a renewed interest in the use of natural dyes for cellulosic fibers (Haar et. al., 2013). However, natural dyes have a number of limiting factors. The variability of plant based dyes means a high degree of skill in application to achieve the desired color (Ziarani et. al., 2018; Saikhao et. al., 2018). Second, most natural dyes need a mordant for colorfastness. The color and colorfastness become related to both the dyestuff and the mordant, and concentrations (Arroyo-Figueroa, 2011). Finally, as the natural dyeing process requires two steps, there will likely be more water used, but lower toxicity, in the effluent.

Many plant based dyes were used in dyeing fabric throughout history, though much of this knowledge has been lost with the adoption of synthetic dyes. Cottonwood trees were used to produce brown and yellow dyes (USDA, 2021). Cottonwood trees are members of the genus *Populus* (OSU, 2021). Recent interest in natural dyes includes *populus deltoides* in dyeing fabric (Geelani et. al., 2017; Harmon, 2021). When dyeing cotton with Indian Bat Tree leaves, good washing, light exposure and perspiration fastness was observed (Kumbhar et. al., 2019). Additionally, using ethanol and water to extract natural dye from plant matter has previously been successfully explored, showing little increase past 75% ethanol concentration for extracting polyphenols (Shafiq et al., 2021). This research used the cottonwood's purple catkins with cotton fabric, in two extraction methods, three mordant and four dye fixative conditions.

Experiment Methodology. 100% cotton, Style #400M (Testfabrics, Inc.) material was scoured with hot water, rinsed and air dried. Catkins were collected the first days of their blooming cycle, before covering with the extraction solutions. Color was extracted using water and a 30% water, 70% ethanol mix, with adjusted processing temperatures and times for different boiling points. Mordants used were tannic acid (TA), alum acetate (AA) or both (TAA). Fabric was wetted out, mordants were 8% weight of fabric, added to distilled water, 40:1 liquor to dry goods ratio. Mordants dissolved, while heat increased to 30 C. Heat increased to 80 C in an hour, lowered to 60 C for 2 hours, stirring every 10 minutes. Fabric cooled for 20 minutes, was rinsed with warm water & air dried. Fabric samples were dyed with dye extracts and distilled water in a liquor ratio of 1:40, temperature going from 40 C to 80 C over 90 minutes, stirring every 10. Dye fixative conditions were none, citric acid (CA), retayne (R) and iDye.(I) Three fixatives had 8% weight of dry fabric added to 60 C dye, stirred every 10 minutes for 30, followed by a rinse and air dry.

Results. Colors Achieved- Water Extraction**Ethanol Extraction**

Two evaluators rated the conditioned samples in a light box set to daylight setting using either the AATCC EP-2 Staining Scale for Color Change or AATCC EP-1 Grey Scale for Color Change, using .5 rating gradations. *Colorfastness to Crocking*: Dry and wet crocking evaluation was done in accordance to AATCC 8. In dry and wet crocking for samples dyed from the water and water/ethanol extracted dye, all ratings ranged from 4.5 to 5 across all mordant and fixative conditions indicating very good to excellent resistance to staining from crocking. *Colorfastness to Artificial Light*: AATCC 16.2 was used as an evaluation guide with a custom built light exposure device modeled after Australian test method 2001.4.21: Determination of colorfastness to light using an artificial light source. Samples were mounted in the circular lamp and exposed to continuous light for 72 hours. In both extractions, samples with no fixatives and the citric acid treatment performed modestly ($M = 1.25$ to 3.00), indicating poor to moderate colorfastness, while R and I averages ranged from 1.50 to 4.00 . *Colorfastness to Perspiration*. Colorfastness evaluation was done in accordance to AATCC 15-2013. Artificial perspiration pH for these samples was 6.0 . Samples were wetted for 15 minutes, wrung and wetted again. Samples were then weighed to ensure a weight $2.25 \pm .05$ times original weight. Samples were evenly distributed on plexiglass plates in a horizontal perspiration tester, and heated to 38 ± 1 degree Celsius for 6 hours. Ratings for the water extraction samples averaged from 3.75 to 5 , indicating fairly good to excellent colorfastness to perspiration. Ratings for the ethanol extraction group averaged 2.75 to 4.75 , indicating moderate to very good. Dye fixatives in the water extraction samples from AA mordant produced a color hue change, though value remained consistent. *Colorfastness to Laundering*. To evaluate laundering, AATCC 61-2013 Test No. 1A was used. Fabric specimens were added to preheated ($40 \text{ degrees} \pm 3 \text{ C}$) canisters with 10 steel balls, .37% AATCC standard reference detergent without brightener and 200 ml distilled water. The laundrometer was run for 45 minutes which was followed by 3 individual rinses with water heated to 40 ± 3 degrees C. Samples were air dried. The addition of the dye fixatives improved colorfastness to laundering. Specifically, water and water/ethanol extracted samples rated 1.75 to 3.50 with no fixative, while CA, R and I samples rated 2.00 to 4.50 on average.

Conclusion. In this experiment, the tannic acid only mordant was most successful in producing a purple spectrum hue, in both extraction conditions. Both extraction conditions led to poor light colorfastness without dye fixatives, while the addition of fixatives improved colorfastness to laundering. Future research may want to consider comparing time of catkin harvest, alkaline dye extraction methods, and the addition of dye bath chemical modifiers during dyeing.

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