Zero Waste Dyeing?- Application and Colorfastness of Spray versus Vat Indigo Dyeing

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Introduction & Literature Review. Indigo has been one of the most important dyes in the world (Encyclopædia Britannica, 2019). Natural indigo vat dye has maintained a small market share and one that is forecasted to grow in the next few years (Cision, 2021). Despite being a natural dye, indigo is not immune to sustainability concerns. Prominent among these concerns are the chemicals used and the amount of wastewater vat dyeing generates (Saikhao et. al., 2018). As a plant based dye, inconsistencies in hue and intensity can be observed in natural indigo (Ziarani et. al., 2018). Unique to indigo among natural dyes is the ability to be substantive toward cellulosic fibers without a mordant. Indigo dyeing involves an oxidation reduction chemical reaction (Saikhao et. al., 2018). In this reaction, the water insoluble blue indigo is reduced to a yellow leuco form with reducing agents under alkaline conditions, typically in a pH range between 11-14 (Komboonchoo et. al., 2009). The fabric introduced to the vat indigo is then re-oxidized by air drying, which converts the leuco dye back to the water-insoluble indigo (Saikhao et. al., 2018). This results in a blue shade trapped in the fibers (Saikhao et. al., 2018).

To reach the needed alkaline conditions in the dyeing vat, strong chemicals, such as sodium dithionite (hydrosulfite, $Na_2S_2O_4$), are used (Komboonchoo et. al., 2009). $Na_2S_2O_4$, is most commonly used in industry to reduce indigo due to its efficacy at quickly reducing indigo and ability to provide a stable dyebath for continuous dyeing (Saikhao et. al., 2018). However, concerns surrounding the chemical's use include it's instability in alkaline solutions at elevated temperatures and the toxic, corrosive nature of the sulfite and sulfate ion by-products of the reaction (Saikhao et. al., 2018; Blackburn et. al., 2009; Meksi et. al, 2012).

Recently, DyStar and RotaSpray developed a spray indigo dyeing procedure for mass production which allows denim producers to reduce their environmental impact (DyStar, 2018). The technology reportedly has high flexibility for dyeing small lots, reduced water usage and reduced effluent discharge (DyStar, 2018). However, the indigo solution used for this technology still requires hydrosulphite in the dyeing process (Warren, 2020). This technology does reduce the amount of sulfites in the wastewater (Warren, 2020). This research explored the possibility of spray application, reduced sustainably, in indigo dyeing.

Experiment Methodology. The fabric used was 100% cotton, Style #400M (Testfabrics, Inc.). Scouring had a 59:1 liquor-to-goods ratio of deionized water, with professional textile detergent and soda ash by weight of fabric. Temperature was raised to 55 C and maintained for 60 minutes. Fabric was then rinsed with cool deionized water. Indigo was prepared by heating 3000 mL of deionized water to 50 C. Maintaining temperature, 15g indigo, 36g NaOH and 30g fructose was added while slowly stirring (Saikhao et. al., 2018). Dye solution was adjusted to the volume of a 50:1 liquor ratio. Dye continued to be processed for 60 minutes, with stirring occurring every 5 minutes and pH being kept below 12. After the indigo reduction occurred, the dye solution was heated to 40 C. Once temperature was reached, the vat dyed sample was dipped and worked through the indigo solution for 15 minutes. Afterwards, the sample was oxidized for 60 minutes.

In the spray condition, the indigo was injected into the sprayer and sprayed front to back until the sample was saturated. The sample was oxidized for 60 minutes and rinsed with cool water.
Results. Both application methods were oxidized, with less depth of shade in the spray condition

Spray Application

Vat Application

Colorfastness to Crocking: Dry and wet crocking colorfastness evaluation was done in accordance to AATCC 8. Fabric specimens were cut to 50mm by 130mm, cut on an oblique 30 degree angle. 50mm by 50mm cotton crocking squares rubbed the surface for 10 turns at 1 turn per second. Water was dropped onto wet testing squares until moisture regain of 65 +/- 5% was achieved. Change in color as a result of crocking in 3 samples was evaluated with AATCC EP-2 Staining Scale for Color Change using .5 rating gradations. Wet and dry crocked samples were evaluated against non-crocked cotton plain weave cloth. At least two evaluators analyzed each sample in a light box using the daylight setting. Ratings used scored within .5 of one another on the change scale. The average change ratings for all dry crocking samples from the spray application group were 4.25. A range in the dry crocking samples for the vat dye application group was 4.5 to 4.75. Both results indicate little to no color change from dry crocking in the spray and vat application groups. In the wet crocking test, a range of 3.67 to 4.25 was observed for the spray application group. The range for application group was 3.00 to 4.25. The results from both application groups indicate moderate to slight color change. *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. Fabric specimens were 70mm by 120mm with an exposure area of 47mm by 47mm. Samples were mounted in the circular lamp and exposed to continuous light for 96 hours before being cooled and conditioned. All samples were then compared to unexposed samples in a light box using the daylight setting. Color change was rated with AATCC EP-1 Grey Scale for Color Change using .5 rating gradations. The average color change for the spray group was 3.75 to 4.5, while the vat group ranged from 4.17 to 4.67. These results indicate slight change from continuous exposure to light, with more change registering in the spray group.
Colorfastness to Perspiration: Colorfastness evaluation was done in accordance to AATCC 15. Fabric specimens were cut to 152mm by 152mm. Samples were wetted for 15 minutes, wrung and wetted again for 15 minutes. Samples were then weighed to ensure a weight 2.25 +/- .05 times original weight. Samples were stacked on plexiglass plates, evenly distributed in a horizontal perspiration tester and heated to 38 +/- 1 degree Celsius for 6 hours. All samples were then compared to unexposed samples in a light box using the daylight setting by at least two evaluators. The average color change from perspiration exposure for the spray group was 2.25 to 3.17, while the vat group ranged from 3.83 to 4.75. These results indicate substantial to slight change from perspiration, with more change in the spray group.
Conclusion. These results indicate that indigo is an effective colorant in both vat and spray applications. The spray samples displayed fairly good to very good performance in the crocking test, fairly good to very good in the light exposure test and fairly poor to moderately fair in the perspiration test. Future research may investigate variables unaccounted for here including angle and distance of spray application, pre-wetting spray fabric and additional spraying and oxidation.

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