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## Comparison of Aluminum Mordants on the Colorfastness of Natural Dyes on Cotton

*Sherry Haar, Erica Schrader, and Barbara M. Gatewood*

Keywords: natural dyes, mordants, colorfastness, aluminum acetate, aluminum potassium sulfate

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*Two mordanting agents, aluminum potassium sulfate and aluminum acetate, in three concentrations (5%, 10%, and 20% owf) were evaluated for colorfastness to laundering and light of natural dye extracts (madder, weld, and coreopsis) on cotton print cloth. The type of aluminum mordant had a greater influence on colorfastness to laundering, whereas dye type had a greater influence on fastness to light. Aluminum acetate at 5% owf concentration gave slightly higher Gray Scale ratings for colorfastness to laundering of coreopsis and weld. All treatments had negligible to no staining on cotton. Weld had slightly better colorfastness to light ratings than the other dye types with 20% aluminum potassium sulfate rating highest. Even though the aluminum acetate mordant improved the colorfastness to laundering on weld and coreopsis at the 5% and 10% owf concentrations, it did not improve fastness to light and resulted in slightly lower fastness to light grades on coreopsis.*

The renewed interest in natural dyes, especially for cellulosic fibers, has been attributed to the potential health and environmental risks of synthetic dyes, emphasis on 'eco-friendly' textiles, and developing markets for alternative cellulosic fibers. Because most natural dyes are water-soluble compounds, except for indigo and other vat dyes, they usually are applied in conjunction with mordanting agents to increase affinity, substantivity and fastness properties. Aluminum containing mordant agents, aluminum potassium sulfate (APS) and aluminum acetate (AA), are the most widely used for dyeing cotton with natural dyes. While APS has been used as a mordant for both protein and cellulosic textiles, AA has been recommended as a better mordant for cotton textiles (Brown, de Souza, & Ellis, 2010; Dean, 2009; Liles, 1990; Wipplinger, 2005). However, no reports were found that compared the aluminum mordant agents for colorfastness properties. The purpose of this study was to determine if an AA mordanting agent could enhance the colorfastness to both laundering and light of natural dyes on cotton compared to APS.

## **Mordanting Agents**

Mordanting agents have been used since antiquity in dyeing both cellulose and protein fibers. Historically, many different types of inorganic (e.g., minerals, salts, crusts, mud, etc.) and organic substances (e.g., urine, excrement, blood, animal and vegetable fats, plant juices, wood ash, etc.) have been used as mordanting or fixing agents, resulting in greater depth of shade and colorfastness (Cardon, 2007; Casselman, 1993). Chemically, mordants are defined as polyvalent metallic compounds that can form coordinate and covalent complexes with certain dyes and fibers (Baker, 1958). Thus, the metal atom typically forms both a covalent bond with hydroxyl (or carboxyl) oxygen on the dye (or fiber) and a coordinate bond with an adjacent lone pair of electrons on a double bonded oxygen. Mordants can chelate several dye molecules together to create a larger complex and provide a link between the dye and fiber. These complexes help the fiber retain color, thus increasing depth of shade and fastness to wet and mechanical treatments (Gordon & Gregory, 1983). However, mordants also can influence lightfastness.

Most mordanting agents are metallic salts of chromium, tin, iron, copper, and aluminum. Copper and chromium containing compounds (e.g., copper sulfate and potassium dichromate) were widely used as mordants, but their usage has declined because of toxicity concerns. Likewise, iron and tin mordants (e.g., iron sulfate and tin chloride) can affect the color and tactile qualities of the dyed textile. Aluminum mordanting agents are commonly used and considered among the safest in the application of natural dyes to cellulosic textiles.

The three methods for applying mordants are by pre-mordanting, the one bath (on-mordant or simultaneous) process, and post-mordanting after dyeing. The pre-mordanting method often yields the best results and is the most widely used (Böhmer, 2002; Cardon, 2007; Dean, 2009). Advantages of pre-mordanting are that multiple batches of fabric can be prepared

in advance and the dye bath is not chemically altered as with the one bath process (Cardon, 2007); however, the mordanting method also has been shown to have minimal influence on colorfastness to laundering (Sarkar & Seal, 2003). Hence, pre-mordanting was the application method used in this study.

### **Aluminum Mordants**

Aluminum is the most abundant of the earth's metals and is a constituent of most silicon-containing soils and rocks, such as clay, feldspar, and bauxite. Various aluminum compounds (e.g., APS, AA, aluminum sulfate, and aluminum hydroxide) have been used as mordants in textile dyeing throughout history (Liles, 1990; Wipplinger, 2004). Aluminum ions have a strong affinity for both cellulose and protein fibers and readily serve as a bridge between multiple dye molecules and/or between the fiber and dye.

**Aluminum potassium sulfate.** Since antiquity, APS ( $\text{AlK}(\text{SO}_4)_2$ ), also referred to as alum, potassium alum, potash alum, or potassium aluminum sulfate, has been used in textile dyeing. APS is a mineral salt historically obtained from natural deposits of aluminous rocks (e.g., slate and shale) (Cardon, 2007). Today, it is made by the refinement of bauxite, an unprocessed form of aluminum ore (Burgess, 2011).

Procedures for pre-mordanting cotton textiles vary greatly in the amount and type of assist. Reported concentrations of APS have ranged from 2% to 100% owf (on-weight-of-fabric); however, 20% owf concentration was often reported (Crook, 2007; Dean, 1999; Samanta, Singhee, & Sethia, 2003). Sodium carbonate (soda ash or washing soda) and tannic acid have been recommended as assists for pre-mordanting cotton (Cardon, 2007; Dean, 2009; Liles, 1990) but are not always used (Angelini, Bertoli, Rolandelli, & Pistelli, 2003; Samanta, et al., 2003). A common procedure for mordanting cotton is a pre-treatment in a tannic acid bath, followed by

one or two APS baths (Brown et al., 2010; Cardon, 2007; Dean, 2009). In addition, Liles (1990) recommended applying a mordant fixative of calcium carbonate prior to dyeing.

**Aluminum acetate.** Three AAs (aluminum salts of acetic acid) are reported in the literature: neutral aluminum triacetate,  $\text{Al}(\text{OOCCH}_3)_3$ , basic aluminum diacetate,  $\text{HOAl}(\text{OOCCH}_3)_2$ , and basic aluminum monoacetate,  $(\text{HO})_2\text{AlOOCCH}_3$ . The diacetate form is used in mordant dyeing, dye manufacturing, in water- and fire-proofing fabrics, and in antiperspirant and antibacterial formulations (Budavari, O'Neil, Smith, Heckelman, & Kinneary, 1996). AA has been used as a mordanting agent in textile dyeing and printing of cotton since the 18th century. (Liles, 1990). Typically, a thickened AA mordant-dye solution, prepared from aluminum hydrate and acetic acid or aluminum sulfate and lead acetate, was printed on calico fabric, heat set, steamed-cured, then after-scoured to set the mordant (Hummel, 1896). Liles (1990) and Garcia (as cited in Rich, 2009) modified historical recipes to create AA, from aluminum sulfate and calcium acetate, for dyeing and printing. Dean (2009) and Wipplinger (2005) utilized prepared AA at 5% owf concentration for pre-mordanting cotton textiles.

### **Influence of Mordants on Dyeing and Colorfastness**

Numerous researchers investigating the application of natural dyes and mordants have examined dye exhaustion, color quality, and colorfastness properties with varied results, depending on the complex interaction among fiber type, mordant agent, dye type, and application parameters (e.g., substrate preparation, component concentration, time, temperature, etc.). The mordant type can influence dye-fiber association and thus colorfastness to wet and mechanical treatments (e.g., crocking) as well as lightfastness. Crews (1982) reported that mordant type had a greater influence on fastness to light than dye type for yellow natural dyes on wool. In addition, the rate and extent of color change were greater with aluminum and tin mordants than for

chrome, copper, and iron mordants. On cotton, fastness to light of natural mordant dyes, regardless of mordant type, were poor-to-moderate (Angelini et al., 2003; Samanta et al., 2003; Sarkar, Mazumdar, Datta, & Sinha, 2005). When researchers reported color change ratings in laundering of mordant natural dyes on cotton, ratings varied depending on dye (Sathianarayanan & Bhat, 2009), mordant (Sarkar et al., 2005; Vastrad, Naik, & Mamatha, 1999) or post-treatment (Samanta et al., 2003), with ratings of 4-5 (slight-to-no change) to 2 (considerable change) on the Gray Scale for Color Change. Staining ratings also ranged from 4-5 to 2.

There have been fewer comparisons of the influence of various aluminum mordants on the uptake and fastness properties of natural dyes on cotton fabrics. Brown et al. (2010) evaluated aluminum mordants with and without carbonate fixatives and tannic acid presoaks and reported that the 5% owf AA mordant had comparable or better visual results than the APS. Colorfastness properties were not evaluated.

### **Natural Dyes**

This study focused on three natural dyes that can be grown readily in the Midwest. Madder (*Rubia tinctorum*) and weld (*Reseda luteola*) have been used throughout the history of dyeing, and even though coreopsis (*Coreopsis tinctoria*) has been used in the Americas (Richards & Tyrl, 2005), it is not as well known. Madder is a red or coral anthraquinone dye extracted from the roots of the madder plant (Cardon, 2007). Weld is a yellow flavonoid dye extracted from the plant leaves (Angelini et al., 2003). It is noted as a colorfast yellow natural dye, even though flavonoids typically have poor colorfastness (Cristea & Vilarem, 2006). Coreopsis flowers produce a reddish orange flavonoid dye, called anthochlors (Cardon, 2007).

### **Research Questions**

With the aim of comparing aluminum mordants on naturally dyed cotton for colorfastness properties, this study addressed the following research questions:

1. Does an AA pre-mordant provide improved colorfastness to laundering, staining, and light compared to APS?
2. What is the effect of increasing mordant concentration on the colorfastness to laundering, staining, and light?
3. How does mordant type and concentration influence dye hue prior to and after laundering tests?
4. What recommendations can be made for mordant use on cotton dyed with madder, weld, and coreopsis?

## **Experimental Procedure**

### **Sample Preparation**

The fabric selected for this study was 100% bleached cotton print cloth, Style #400 (Testfabrics, Inc.) weighing 102 g/m<sup>2</sup>. It was pre-scoured in an aqueous RO (reverse osmosis) bath with 5% owf Professional Textile Detergent (Dharma Trading Company) and 2% owf sodium carbonate (Hillcreek Fiber Studio) at a 40:1 liquor-to-goods ratio in a 15 L, covered stainless steel container heated on an electric hot plate. The scouring commenced at 25 °C and was raised to 80 °C over 30 min and held for another 30 min with stirring at 5 min intervals. After cooling, the fabric was rinsed with warm, running tap water and air-dried.

The scoured fabric was cut into 54 samples weighing  $7 \pm 0.02$  g for pre-mordanting and dyeing. Fabric samples were pre-mordanted with one of two mordants (APS and AA) at three concentrations (5%, 10%, and 20% owf) before being dyed with one of three natural dyes (madder, weld, and coreopsis). Three replications of each mordant x concentration x dye

combination were prepared. Thirty minutes prior to mordanting, samples were wetted out (i.e., soaked) in RO water. Individual samples were mordanted in Atlas Launder-Ometer stainless steel canisters with 560 ml RO water, which was equivalent to an 80:1 liquor-to-dry goods ratio. The Atlas Launder-Ometer rotates the samples in closed, stainless steel canisters in a thermostatically controlled water bath. The apparatus ran for 90 min with the temperature increasing from 25 °C to 80 °C. After cooling for 20 min, the mordanted samples were rinsed under a RO water faucet by hand for 30 sec to remove unabsorbed surface mordant. Samples were air-dried.

The natural dye extracts used were obtained from Couleurs de Plantes. Stock dye solutions were prepared from the extracts in stainless steel containers with extract amounts of 3% owf for madder and weld, while coreopsis had 8% owf for APS samples and 12% owf for AA samples. Pretests showed that these concentrations were needed to achieve a medium dye shade across dye types, which was equivalent to a 1/6 standard depth of shade based on the American Association of Textile Chemists and Colorists (AATCC, 2009) standard depth scale. An 80:1 liquor-to-dry goods ratio was used because pretests also showed that this level produced the desired evenness and shade reproducibility. The temperature of the stock dye solutions was raised from 25 °C to 80 °C over 20 min and maintained for 1 hr. The purpose of this step was to insure that the dyestuffs were completely dissolved prior to dispensing aliquots for dyeing.

Prior to dyeing, the samples were wetted out for 30 minutes in RO water. The individual dyebaths were prepared by pouring 560 ml of dye solution into individual Launder-Ometer stainless steel canisters, followed by the wetted out, pre-mordanted,  $7 \pm 0.02$  g sample. During dyeing the temperature in the apparatus was raised from 25 °C to 80 °C over 90 min, then cooled

to 25 °C over 20 min. The dyed samples were rinsed under a RO water faucet by hand for 30 sec and laid flat on towels to air dry.

### **Tests for Colorfastness to Laundering and Light**

After pre-mordanting and dyeing, individual specimens were cut from the 54 fabric samples for testing colorfastness to laundering and light. The specimens for the laundering and light exposure measured 50 x 150 mm and 80 x 120 mm, respectively. Three replications of each mordant (2) x concentration (3) x dye (3) combination were prepared. A comparison specimen was also cut from each sample to serve as the untested fabric for evaluation between unwashed and washed specimens. For light testing the untested fabric was the masked portion of the specimen. The specimens for the laundering tests were serged with polyester thread, then a 50 x 50 mm piece of multifiber test fabric No. 1 (Testfabrics, Inc.) was stitched to the surface to test for staining on the cotton strip.

Colorfastness to laundering testing of the specimens was conducted in accordance with AATCC Test Method 61-2007, Colorfastness to Laundering, Test No. 2A which simulates five home launderings at a warm temperature of  $49 \pm 3$  °C (AATCC, 2009). The Launder-Ometer was pre-heated to 49 °C and held at this temperature throughout testing. The canisters were filled with 150 ml of RO water, 50 stainless steel balls (6 mm), and 0.15% total volume or 0.23 g of 1993 AATCC Standard Reference Detergent. The canisters were pre-heated in the Launder-Ometer for 2 min, then the machine was stopped, and one dyed test specimen was added to each canister. Once the machine was fully loaded, it was run for 45 min. After laundering, the specimens were removed and rinsed three times for 1 min each in individual beakers with 150 ml RO water at 25 °C. The specimens were dried in a laundry dryer at the temperature for regular fabrics for 20 min, then laid flat to complete drying.

Light exposure of the dyed specimens and evaluation of color change was conducted by Professional Testing Labs, Dalton GA, following AATCC Test Method 16-2004, Colorfastness to Light, Option 3 (AATCC, 2009). The specimens were exposed to continuous light for 20 AATCC fading units (AFU) in an Atlas Xenon Weather-Ometer maintained at  $63 \pm 1$  °C and  $30 \pm 5\%$  RH, simulating exposure behind glass.

### **Color Evaluation Procedures**

Color change in the laundered and light-exposed specimens were visually evaluated following AATCC Evaluation Procedure 1, Gray Scale for Color Change, whereas staining or color transference in laundering was evaluated following AATCC Evaluation Procedure 2-2007, Gray Scale for Staining (AATCC, 2009). The test and untested specimens were compared and given a grade representative of a Gray Scale step with the same color or contrast difference. The Gray Scale grades for color change or difference are 5 negligible or no color change was perceived, with lower numbers representing increasing amounts of color change. The Gray Scale grades for staining also use a comparable 5-step scale. Even though multifiber test fabric was used, only the cotton strip was evaluated for staining. Evaluations for laundering were conducted by three evaluators who had normal color vision with their responses averaged. Exposure to light evaluations were conducted by three evaluators from Professional Testing Labs with the mode reported.

In addition, hue differences between the AA and APS specimens were evaluated prior to and after laundering at each concentration. Differences in hue are noted as bluer, greener, redder, or yellower according to AATCC Evaluation Procedure 1-2007 (AATCC, 2009). This information was gathered to supplement the color change data, which evaluated just the amount of color difference between the pre-mordanted unlaundered and laundered specimen within

mordant type. Examining the influence of mordant on hue is important to dyers, who often strive to achieve a specific color. Evaluations for hue differences were conducted by two evaluators who had normal color vision with the responses negotiated.

### **Statistical Analysis**

A General Linear Model (GLM) of analysis of variance (ANOVA) was applied to the colorfastness to laundering data to examine the influence of mordant type and concentration on Gray Scale grades for color change in laundering. Pairwise comparisons of least squares means were conducted to compare effect of concentration within dye and mordant types. Colorfastness to light data was not statistically analyzed as the Gray Scale grade reported from Professional Testing Labs was the mode grade of three evaluators instead of the mean.

## **Results**

### **Colorfastness to Laundering**

For madder dyed cotton the GLM ANOVA with mordant (APS and AA) and concentration (5%, 10%, and 20% owf) as independent variables and mean Gray Scale color change rating as the dependent variable yielded a significant interaction between mordant and concentration (Table 1). Overall the color change for madder dye specimens in the laundering test were considerable with grades for APS (1.69, 2.00, and 2.11) and AA (2.02, 1.86, and 1.67) applied at 5%, 10%, and 20% owf, respectively (Table 2). The mean difference between mordants was .08 with the rank order of colorfastness means reversed at the higher concentrations. As shown in the pairwise comparisons, there was a significant difference between 5% and 20% owf APS with the 20% concentration rating higher (2.11 to 1.69). There were no significant differences between the AA concentrations of madder dyed cotton. The colorfastness to laundering findings for madder were lower than that reported by Samanta et al.

(2003) where madder on cotton pre-mordanted with 20% owf aluminum sulfate were 3-4 or noticeable-to-slight color change.

Table 1

*GLM ANOVA Results on Mean Gray Scale Color Change Ratings for Colorfastness to Laundering of Natural Dyes on Cotton Fabric with Aluminum Mordants at Three Concentrations*

Dye	Source	df	Type III SS	MS	F	Pr > f
Madder	Mordant	1	0.031	0.313	0.71	0.4155
	Concentration	2	0.014	0.007	0.16	0.8531
	Mordant x Concentration	2	0.452	0.226	5.14	0.0244*
Weld	Mordant	1	2.347	2.347	218.12	<.0001**
	Concentration	2	0.490	0.245	22.76	<.0001**
	Mordant x Concentration	2	0.456	0.228	21.16	0.0001**
Coreopsis	Mordant	1	7.762	7.762	1675.21	<.0001*
	Concentration	2	0.034	0.017	3.68	0.0565
	Mordant x Concentration	2	0.013	0.007	1.45	0.2730

Note. GLM = general linear model; ANOVA = analysis of variance; SS = Sum of Squares; MS = Mean Square. \* $p < .05$ . \*\* $p < .01$ .

Table 2

*Gray Scale Means and Standard Errors of Mordant and Concentration for Colorfastness to Laundering of Natural Dyes*

Dye	Mordant	Color Change Means*			Mordant Overall Mean	Standard Error
		5	10	20		
Madder	aluminum potassium sulfate	1.69 <sup>a</sup>	2.00 <sup>a,b</sup>	2.11 <sup>b</sup>	1.93	0.12
	aluminum acetate	2.02 <sup>a</sup>	1.86 <sup>a</sup>	1.67 <sup>a</sup>	1.85	
Weld	aluminum potassium sulfate	1.25 <sup>a</sup>	1.00 <sup>b</sup>	1.17 <sup>a,b</sup>	1.14	0.06
	aluminum acetate	2.17 <sup>a</sup>	1.97 <sup>b</sup>	1.44 <sup>c</sup>	1.86	
Coreopsis	aluminum potassium sulfate	1.81 <sup>b</sup>	1.75 <sup>b</sup>	1.67 <sup>b</sup>	1.74	0.04
	aluminum acetate	3.11 <sup>a</sup>	3.00 <sup>a</sup>	3.05 <sup>a</sup>	3.06	

*Note.* owf = on-weight-of-fabric. \*Gray Scale for Color Change: 5=no change, 4=slight, 3=noticeable, 2=considerable, and 1=much. Means with the same superscript letter, within a row, are not significantly different at  $\alpha=0.05$ .

Weld dyed cotton had significant differences by mordant, concentration, and the interaction between mordant and concentration (Table 1). AA had significantly higher gray scale scores for colorfastness to laundry compared to APS. The overall mean average for AA was higher than APS by .72 (Table 2). The three concentrations of AA were significantly different from one another with the 5% owf concentration grade (2.17) slightly higher than 10% and 20% owf (1.97 and 1.44). The overall colorfastness to laundering for weld of much-to-considerable color change, was much lower than the 4.8 or slight-to-no color change rating reported by Angelini et al. (2003) for cotton yarns pre-mordanted with 20% owf potassium alum. However, those excellent ratings seem atypical for a natural dye and require further investigation.

The coreopsis dyed cotton specimens were significantly different between aluminum mordants, with the AA pre-mordant rating higher (Table 1). The overall mean difference between mordants was 1.32 (Table 2). The Gray Scale grades for the AA pre-mordant were 3.11, 3.00, and 3.05 (5%, 10%, and 20% owf respectively) or noticeable with no significant difference across concentrations. As no prior published research was found on the colorfastness to laundering of coreopsis, a comparative assessment cannot be made; however, this study does present preliminary findings.

### **Staining Evaluation**

Among the three dye types, only the cotton dyed with madder exhibited slight staining on cotton with staining ratings  $\geq 4.7$  (Table 3). No appreciable differences were observed for the mordant types and concentrations. The results for staining on cotton by madder were similar to Samanta et al. (2003) who reported a grade of 4.5.

Table 3

*Gray Scale Means of Mordant and Concentration for Staining Evaluation of Natural Dyes on Cotton*

		Staining on Cotton*		
		Mordant Concentration, % owf		
Dye	Mordant	5	10	20
Madder	APS	4.8	4.8	4.8
	AA	4.7	4.8	4.8
Weld	APS	5	5	5
	AA	5	5	5
Coreopsis	APS	5	5	5
	AA	5	5	5

*Note.* owf = on-weight-of-fabric; APS = aluminum potassium sulfate; AA = aluminum acetate.  
\*Gray Scale for Staining: 5=equal or no perceived staining, 4=slight, 3=noticeable, 2=considerable, and 1=much.

### **Colorfastness to Light**

The cotton dyed with madder specimens all had a Gray Scale color change grade of 1.5 or much-to-considerable color change for both aluminum mordants and all three concentrations. Although these ratings seem low, fair-to-poor colorfastness to light of natural dyes on cotton mordanted with APS have been noted in the literature (Crews, 1982). However, our ratings for madder were lower than the rating of 3 reported by Samanta et al. (2003) for madder on cotton pre-mordanted with 20% owf aluminum sulfate.

Table 4

*Gray Scale Modes of Mordant and Concentration for Colorfastness to Light of Natural Dyes*

		Color Change*			Mordant Overall Mean
		Mordant Concentration, % owf			
Dye	Mordant	5	10	20	
Madder	APS	1.50	1.50	1.50	1.50
	AA	1.50	1.50	1.50	1.50
Weld	APS	2.00	2.00	2.50	2.16
	AA	2.00	2.00	2.00	2.00
Coreopsis	APS	1.50	1.50	2.00	1.67
	AA	1.00	1.00	1.50	1.17

*Note.* owf = on-weight-of-fabric; APS = aluminum potassium sulfate; AA = aluminum acetate. \*Gray Scale for Color Change: 5=equal or no perceived color change, 4=slight, 3=noticeable, 2=considerable, and 1=much.

The colorfastness to light ratings for weld were 2.0 or considerable change for AA pre-mordanted specimens at all three concentrations. APS applied at 5% and 10% owf were also 2.0, while at 20% owf a slightly higher grade of 2.5 or considerable-to-noticeable was obtained. Our colorfastness to light ratings for weld were similar to those poor reported by Angelini et al. (2003).

Coreopsis had the poorest colorfastness to light with grades of 1.0, 1.0, and 1.5 (much-to-considerable change) on cotton pre-mordanted with AA at 5%, 10%, and 20% concentrations. The corresponding grades for APS were 1.5, 1.5, and 2.0. As coreopsis contains flavonoids, which are not good dye receptors, the overall lower ratings for colorfastness to light were expected.

### **Differences in Hue Direction between Mordant Type**

As explained in the procedure, similar color depth of specimens across dye type, mordant type, and concentrations was established during pre-testing by adjusting the dye concentrations until specimens were visually similar in terms of lightness and darkness. However, the APS and AA pre-mordanted specimens did have slight differences in color direction of hue between mordant type for each dye prior to laundering with some differences remaining after laundering.

The madder dyed unlaundered specimens at all concentrations pre-mordanted with AA were slightly bluer (4 Gray Scale grade) compared to the APS specimens which had a slightly yellower cast across all three concentrations (see Table 5). After exposure to laundering, madder specimens pre-mordanted at 5% and 10% owf for both mordants retained the hue direction;

however, the difference was lessened to a 4.5 Gray Scale grade while the 20% concentration for both mordants no longer had visible hue direction differences.

Table 5

*Hue Differences of Naturally Dyed Cotton Between Aluminum Mordants Before and After Laundering*

			Color Change and Hue Descriptor		
			Mordant Concentration, % owf		
Dye	Laundering Test	Mordant	5	10	20
Madder	Pre	APS	4.0 Yellower	4.0 Yellower	4.0 Yellower
		AA	4.0 Bluer	4.0 Bluer	4.0 Bluer
	Post	APS	4.5 Yellower	4.5 Yellower	5.0
		AA	4.5 Bluer	4.5 Bluer	5.0
Weld	Pre	APS	5.0	5.0	5.0
		AA	4.5 Redder	4.5 Redder	4.5 Redder
	Post	APS	5.0	5.0	5.0
		AA	4.0 Redder	4.0 Redder	4.5 Redder
Coreopsis	Pre	APS	4.5 Yellower	4.5 Yellower	4.0 Yellower
		AA	4.5 Redder	4.5 Redder	4.0 Redder
	Post	APS	4.75 Yellower	4.75 Yellower	4.75 Yellower
		AA	4.75 Redder	4.75 Redder	4.75 Redder

*Note.* Gray Scale grade: 5=equal, 4=slight, 3=noticeable, 2=considerable, and 1=much.

The weld dyed unlaundered specimens pre-mordanted with AA were slightly redder compared to the APS unlaundered specimens that appeared clear yellow with no visible hue direction. After the laundering test, a slight red cast for the AA specimens was still visible with ratings of 4.0 redder for the 5% and 10% owf concentrations and 4.5 redder for the 20% owf concentration. There remained no hue difference in the weld specimens pre-mordanted with APS after laundering.

The coreopsis dyed unlaundered specimens pre-mordanted with AA had a slightly redder hue compared to the APS pre-mordant, the specimens of which had a hue difference towards

yellow. The slight difference towards red and yellow were barely visible after the colorfastness to laundering testing.

### **Discussion**

The purpose of this study was to determine if an AA mordanting agent could enhance the colorfastness to both laundering and light of natural dyes on cotton compared to APS. The dyestuffs evaluated (madder, weld, and coreopsis) have regional importance and have been widely used by dyers for centuries for dyeing both cellulose and protein fibers. The aluminum mordants were applied by a pre-mordanting procedure at three concentrations (5%, 10%, and 20% owf). All of the treatment combinations (mordant type (2) x mordant concentrations (3) x dye (3) x replication (3)) were exposed to accelerated laundering and lightfastness (20 AFU's of Xenon light) tests. The results of these tests were used to answer the research questions proposed by this study.

1. Does an AA mordant provide improved colorfastness to laundering, staining, and light compared to APS? Results from the colorfastness to laundering tests showed that the weld and coreopsis dyes had better colorfastness or less color loss with the AA pre-mordant, whereas fewer differences were observed for madder. However, even the highest color change mean of 3.11 for coreopsis indicated noticeable color loss. Mordant type did not influence colorfastness ratings for staining, and all of the dyed specimens with either AA or APS had negligible to no staining on cotton. The appreciable color loss and lack of staining during laundering are indications that cotton fibers have relatively low affinity for these natural dyes.

Overall the color change after 20 AFU's of Xenon light exposure was much-too-considerable for all treatments with mode Gray Scale for color change ratings of 1.0 to 2.5. Comparable colorfastness to light ratings were obtained for the two mordant types, except for the

coreopsis specimens pre-mordanted with AA that resulted in a half-step lower grade compared to the APS.

To place these ratings in an applicable context, the American Society for Testing and Materials (ASTM) requires a laundering grade of 4, staining grade of 3, and light grade of 4 of fabrics for apparel and home furnishing end use. Such standards include the Standard Performance Specification for Woven Blouse, Dress, Dress Shirt & Sport Shirt Fabrics (ASTM D 7020, 2005) and, for items such as quilts, the Standard Specification for Woven and Warp Knitted Comforter Fabrics (ASTM D 4769, 2005). Thus, only the evaluation of staining rated high enough to meet these apparel and home furnishing fabric end use standards.

2. What is the effect of increasing mordant concentration on the colorfastness to laundering, staining, and light? Results from the colorfastness to laundering tests showed that the weld and coreopsis dyes had slightly higher colorfastness ratings at 5% owf concentration compared to the 10% and 20% owf concentrations with both the AA and APS mordants. For madder dyed cotton specimens APS performed better at 20% owf concentration and AA at 5% owf concentration. Concentration of mordant did not influence colorfastness to staining. The findings from the laundering test of AA rating slightly better at the 5% owf concentration supports the recommended concentration for AA at 5% in the literature.

The colorfastness to light grades for weld and coreopsis were highest at 20% APS with grades of 2.5 and 2.0, respectively. There was no influence of concentration for weld dyed cotton pre-mordanted with AA. For coreopsis, the AA pre-mordant rated slightly higher at 20% owf concentration. For madder there were no differences in colorfastness to light ratings between mordant or concentration.

3. How does mordant type and concentration influence dye hue prior to and after laundering tests? Mordant type had a slight influence on hue with APS pre-mordanted specimens appearing slightly yellower, except for weld which had no hue difference. The AA pre-mordanted specimens had a slightly redder (weld, coreopsis) or bluer (madder) hue difference. The hue differences were typically lessened following the laundering test, except for weld pre-mordanted with AA, which showed slightly more red in the post test at 5% and 10% owf concentrations. Concentration had limited influence on hue differences. The 20% owf concentration showed slightly reduced hue differences for laundered madder and weld.

Even though the differences in hue direction were slight, such information may influence a dyer who is aiming to obtain a particular color. The polychromatic effects of natural dyes with different mordanting agents (e.g., alum, tin, copper, iron) and modifiers (e.g., acid, alkali, iron, copper) is documented in the literature (Dean, 2009; Richards & Tyrl, 2005); however, this study is seemingly the first to note hue differences between two aluminum mordants.

4. What recommendations can be made for mordant use on cotton dyed with madder, weld, and coreopsis? As madder had no significant difference between mordant type for colorfastness to laundering and negligible-to-no difference on staining and colorfastness to light, it could be recommended to use 10% owf concentration if applying APS and 5% owf concentration when mordanting with AA. For weld and coreopsis dyed cotton, the recommendation differs by whether colorfastness to laundering or light is more important. Colorfastness to laundering ratings were significantly higher for the AA pre-mordant and at 5% owf concentration. However, colorfastness to light ratings for weld and coreopsis were highest with APS at 20% owf concentration.

In summary, the type of aluminum mordant had a greater influence on colorfastness to laundering, whereas dye type had a greater influence on colorfastness to light of the cotton print cloth dyed with madder, weld, and coreopsis. Mordant type had a greater influence on the colorfastness to laundering ratings of coreopsis and weld with the AA giving slightly higher Gray Scale ratings. Even though the AA mordant improved the colorfastness to laundering on weld and coreopsis, it did not improve colorfastness to light and resulted in slightly lower grades on coreopsis.

### **Limitations and Further Study**

A limitation of the study was that the dye specimen replications were not actually true replications as one dye bath was divided between the accelerated laundering canisters for dyeing, instead of individual dye concentration in each canister. Hence, less variation was observed within than between the treatment combinations. Replications for dyeing are recommended in future studies. Because the colorfastness to laundering and light grades were below acceptable ASTM performance standards for applicable apparel and home furnishing end use, more work is needed on pre- and post- treatments for improving the colorfastness properties of cotton naturally dyed with aluminum mordants.

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