COMPARISON OF ALUMINUM MORDANTED AND NONMORDANTED WOOL YARNS NATURALLY DYED WITH KANSAS BLACK WALNUT, OSAGE ORANGE, AND EASTERN REDCEDAR SAWDUST

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Abstract

This study compared the colorfastness of potassium aluminum sulfate (PAS) mordanted and nonmordanted 30/2 wool yarn, dyed with black walnut (*Juglans Nigra*), Osage orange (*Maclura pomifera*), and eastern redcedar (*Juniperus virginiana*) sawdust. Information from this study is intended to inform natural dye artisans and to increase the profitability of sawdust for farmers, ranchers, and mill owners who would otherwise find little use for this byproduct of timber manufacturing.

Pre-testing ensured dyeings of visually comparable color depth and dye concentrations were pre-tested to find a standard depth of shade between the same dye on PAS mordanted and nonmordanted wool yarns. Tests for colorfastness to light, laundering and staining were performed in accordance to AATCC test methods. Resulting colors for exposed and unexposed specimens were rated using CIE L^* a^* b^* values and AATCC gray scale for color change. GLM Anovas and two-sample t-tests were used to statistically analyze CIE L^* a^* b^* values. As expected, findings indicated that dye absorption was improved with the use of a PAS mordant, especially for black walnut and eastern redcedar. For yarns premordanted with PAS the dyewoods became yellower. A PAS mordant slightly improved colorfastness to light for black walnut and eastern redcedar, but did not influence Osage orange which had an unexpected color change from bright yellow to warm brown after exposure to light. Colorfastness to laundering was slightly improved with PAS for Osage orange, while black walnut and eastern red cedar had slightly less color change without the mordant. This research was supported by the Agricultural Research Experiment Station and Kansas State University.

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Chapter 1 - **Introduction**

Tree leaf, fruit, bark, and heartwood have long been a source of natural dye colorant. American Kiowa and Pima Indian tribes utilized the Osage orange tree as a source of bright yellow dye color (Cardon, 2007). The quebracho tree was used to dye and tan hides during Great Britain's Industrial Revolution (Haslam, 1989). A struggling 1941 American economy rediscovered the importance of natural dyes derived from trees, as many manufacturing facilities that produced coal tar for synthetic dyes were diverted for use in the growing defense industry (Tisdale, 1941). An article from that time period by the *American Dyewood Company* argued that extracts from logwood, fustic, Osage orange, and brazilwood would be a necessary product for the domestic consumer as the impending disaster of World War II loomed closer (Tisdale, 1941). Post war, synthetic dyes were once again the industry standard due to strong colorfastness properties, consistency between baths, low cost, and wide range of bright colors. However, many synthetic dye effluents pollute our environment with hazardous materials such as color residues, sulfur oxides, formaldehyde, and heavy metals (EPA, 1996). Consequently, these hazards have invigorated a renewed interest in natural dying.

The author's interest in dyewoods stemmed from an early exposure to native Kansas species of wood products, harvested for use in her father's woodshop. To develop a better understanding of mill waste (sawdust) from Kansas black walnut, Osage orange, and eastern redcedar, this project aimed to compare the differences of these dyes on mordanted and nonmordanted wool yarn. A mordant is a chemical treatment added to the fiber that helps adhere natural dye to the fiber; yet most trees contain tannic acid, a naturally occurring mordant that contributes to colorfastness without the addition of a chemical mordant treatment. Naturally occurring tannin in dyewood could possibly make for dyes that are durable without the aid of a mordant, which in turn could increase their importance to natural dyers and add value to a Kansas grown product. Furthermore, while it has been stated that black walnut is a substantive dye (i.e., does not require the aid of a mordant) due to high levels of tannic acid, the same has not been scientifically explored for other dyewoods, such as Osage orange and eastern redcedar (Cardon, 2007). This is important, as poor colorfastness has been a concern when considering use of natural dyes for textiles. This study compared colorfastness properties between mordanted and nonmordanted wool yarns, dyed with Kansas black walnut, Osage orange, and eastern redcedar to create a better understanding of sawdust waste as a colorant.

Project Aim

The purpose of this research was to examine the colorfastness of dyes derived from Kansas black walnut (*Juglans Nigra*), Osage orange (*Maclura pomifera*), and eastern redcedar (*Juniperus virginiana*) mill waste on wool yarn with and without a mordant treatment of potassium aluminum sulfate (PAS). Data from this study was used towards the scientific exploration of natural dyes by evaluating the colorfastness to light, laundering, and staining of timber sawmill waste. Furthermore, the researcher incorporated the knowledge gathered from this study to dye yarns for use in handwoven textiles. The handwoven textiles were displayed in the form of art garments during a culminating exhibit.

Research Questions and Hypotheses

This research compared the colorfastness of potassium aluminum sulfate (PAS) mordanted and nonmordanted 30/2 wool yarn, dyed with black walnut (*Juglans Nigra*), Osage orange (*Maclura pomifera*), and eastern redcedar (*Juniperus virginiana*) sawdust. A prior study by the researcher showed that a PAS mordant on wool fabric dyed with black walnut leafs, hulls, and bark improved lightfastness of these dyes but did not improve washfastness or staining qualities (Doty & Haar, 2013). Based on this knowledge the following research questions and hypotheses were formed.

Question A: Does a PAS mordant impact the dye concentration needed to achieve visually comparable dye depth of shade for walnut, Osage orange, and eastern redcedar?

- *H_1: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will require less dye concentration to achieve visually comparable depth of shade compared to wool yarn without a premordant.*

Question B: How does a PAS mordant affect overall color parameters when dyed with black walnut, Osage orange, and eastern redcedar and subjected to tests of colorfastness to light and laundering?

- *H_2: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly warmer (yellower or redder) when compared to wool yarn without a premordant before and after colorfastness to light testing.*
- *H_3: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly warmer (yellower or redder) when compared to wool yarn without a premordant before and after colorfastness to laundering testing.*

Question C: Does a PAS mordant improve colorfastness properties of black walnut, Osage orange, and eastern redcedar on wool yarn when subjected to tests of colorfastness to light, laundering, and staining?

- *H_4: On tests for colorfastness to light, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will have less color change and be significantly different compared to wool yarn without a premordant.*
- *H_5: On tests for colorfastness to laundering, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will have less color change and be significantly different compared to wool yarn without a premordant.*
- *H_6: On tests for colorfastness to staining, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will have similar color change ratings compared to wool yarn without a premordant.*

Question D: Will gray scale ratings for wool yarns dyed with black walnut, Osage orange, and eastern redcedar meet minimum American Society of Testing and Materials (ASTM) standards, requiring a light grade of 4, laundering grade of 4, and a staining grade of 3?

- *H_7: Minimum ASTM standards for colorfastness to laundering, staining, and light for apparel and home furnishing use will be met with wool yarns both premordanted with PAS and unmordanted and dyed with black walnut, Osage orange, and eastern redcedar dyewoods.*

Justification

The justification for this study was to increase knowledge of the application and colorfastness properties of local Kansas dyewoods for use as a sustainable alternative for textile coloration by natural dye artisans who use yarn as part of their craft (e.g., hand-weaving, knitting, crochet).

All dyewoods used in this study were grown and processed in Kansas and the resulting information may aid manufacturers looking to add value to a byproduct of timber processing. Furthermore, two of the three trees selected, eastern redcedar and Osage orange, are considered an invasive species. Research on eastern redcedar found the evergreen to be an invasive species with an increase in stock volume of 23,000% on Kansas lands from 1965 to 2005, effectively decreasing the value of undermanaged pastureland (USDA Forest Service, 2008). Also known for its potential as an invasive tree is the Osage orange, found in hedgerows around the state. Once planted for its excellent properties as a living fence line, the *bois d'arc* or Osage orange tree can be a nuisance to cattlemen, as they tend to invade pastureland (Rutter, 2013). Conversely, the growth and maintenance of black walnut trees are encouraged in Kansas as a cash crop, due to both a desirability as a hard wood and because of it edible fruit (USDA Forest Service, 2008). Whether any of the dyewoods are harvested due to the maintenance of Kansas's pastureland or because of its economic significance, sawmill waste from their timber could be a sustainable source of color for textiles while adding value to a Kansas byproduct.

Definitions

The following are terms and definitions as used in this study.

Mordant- Substances that bond the dye chromospheres to the fiber and can change the color outcomes and fastness properties of the natural dyes (Cardon, 2007; Burgess, 2011).

Treatment- Referring to specimens with a mordant treatment of potassium aluminum sulfate.

Exposure- To subject a test specimen to either colorfastness to light or laundering testing, AATCC 16-2004 and 61-2997 respectively.

Standard- Unexposed or untreated reference for comparison of color change.

Substantive Dyes- Natural dyes that do not require the addition of a mordant, due to their naturally occurring compounds (Cardon, 2007; Burgess, 2011).

Adjective Dyes- Natural dyes that require the addition of a mordant to aid in dye adherence (Cardon, 2007).

Tannic Acid- Also known as tannin, is a naturally occurring mordant found in substantial quantities in the bark, branches and fruits of trees and in some plants such as sage and sumac (Casselman, 1980; Burgess, 2011)

Potassium aluminum sulfate (PAS)- A metallic aluminum mordant created from the refinement of bauzite, resulting in a white granulated substance (Burgess, 2011; Wipplinger, 2005)

Chapter 2 - **Review of Literature**

Mordants

Natural dyes from trees can be considered either adjective or substantive. Adjective dyes require a mordant to bind the color to the fiber. Many colorants sourced from natural dyes have a weak chemical affinity to textiles, mordants remedy this naturally weak bond by helping the dye adhere to the textile and act as a bridge between the fiber and the dye, effectively changing the color outcomes and fastness properties of the natural dyes (Cardon, 2007; Flint, 2008). Mordants are required to fix adjective dyes to a textile but can also aid the colorfastness of substantive dyes. Mordants can also change a natural dye's light absorption, effectively changing the color of the dyed fiber and giving the dye artisan a wider array of color to choose from (Richards & Tyrl, 2005). This chemical bridge between the dye chromospheres and the textile can be either a mineral mordant (salts, efflorescences, mud, metal oxides) or an organic mordant (urine, blood, fats, plant juices) (Cardon, 2007). A majority of today's natural dyers use a nontoxic metallic salt, such as aluminum or iron to help adhere their wanted colors and improve colorfastness (Cardon, 2007; Richards & Tyrl, 2005). Mordants can be added before (pre-mordanting), after (post-mordanting) or during (simultaneous mordanting) the dye process but the most common practice is to pre-mordant the textile before dyeing (Flint, 2005). Conversely, substantive dyes do not require the addition of a mordant, due to naturally occurring compounds. One such compound is tannic acid that is found in high concentrations in plants such as black walnuts, acorns and sumac. Even though substantive dyes do not require a mordant, it can be used to modify the dye color and has also been found to increase colorfastness when subjected to light testing (Doty & Haar, 2012).

Tannin

Tree tannins have a long history as part of the leather tanning industry. During Great Britain's Industrial Revolution trees such as wattle, quebracho, and the acorn cups of the bearded oaks were imported to supplement a growing demand for hide tanning (Haslam, 1989). Today tree tannins for the leather industry have been replaced with synthetic tannins (Haslam, 1989; Hon & Shiraishi, 2000). Natural tannins can still be found in unripe fruit, beer, wine, coffee and tea; giving food and beverages their dry astringent flavor (Haslam, 1989; Hon & Shiraishi, 2000). At the molecular level, tannins are considered plant polyphenols and are divided into two categories, hydrolysable and condensed (Cardon, 2007; Haslam, 1989). Both types of tannin react with ferrous sulfate to produce gray or black colors and are responsible for the chemical reaction responsible for iron-gall inks (Cardon, 2007). Specific to condensed tannins is the color reaction causing a red-brown. The color is produced when condensed tannins are oxidized and is where cutch extracts and tea receive their signature hues (Cardon, 2007).

A naturally occurring mordant, tannin is found in substantial quantities in various parts (i.e. leaf, bark, heartwood, fruit) of trees and protects plants from various infectious microorganisms and prevents rotting (Casselman, 1980; Burgess, 2011; Hon & Shiraishi, 2000). Tannin amounts vary greatly between tree species and then amounts can vary within a single tree depending on the time of year (Pizzi, Conradie, & Jansen, 1986). As a general rule, hardwoods contain higher tannin amounts than softwoods (Pizzi, et al., 1986). Information regarding the amounts of tannin in black walnut, Osage orange, and eastern redcedar was hard to obtain. However, recent research looking at the discoloration of hardwoods suggests that black walnut heartwood contains 2% tannin (Hon & Shiraishi, 2000). While data from hide tanning research performed in the 1910's suggests that Osage orange native to Texas had upward of 10-11% tannin (Kressman, 1916). Research performed in 1901 by the pharmaceutical industry suggests tannin amounts in eastern redcedar at 2-8% depending on the time of year when samples were collected (Peacock, 1901). Since eastern redcedar is considered a softwood it is surprising that it would contain more tannin than black walnut, a hardwood. Either eastern redcedar is an exception to the general rule of softwoods containing less tannin or more modern methods might yield truer detection of tannin percentages.

Tannin can also be purchased in a refined form as a powder from dye suppliers, mainly marketed for use as a mordant for cellulosic fibers (Burgess, 2011; Cardon, 2007). It is important to note that tannins have a natural affinity to cellulosic fibers (Cardon, 2007). Yet the researcher of this study wanted to investigate the advantage or disadvantage of tannin on wool, due to wools improved affinity to take natural dye colors. Furthermore, a previous study performed by the researcher showed wool fabric dyed with walnut leafs, hulls, and bark had better colorfastness to light ratings than cotton fabric (Doty & Haar, 2013).

Extracted tannin does not produce clear colors with all natural dyestuffs, yet some dyers use tannin extracts in combination with aluminum mordants to enhance colorfastness properties (Casselman, 1980). While tannins will give a brown overcast to the dye color, some research suggests that tannin dyes and extracts might darken dyed textiles as they age, instead of the color fading due to exposure to washing and light (Dean, 2010; Cardon, 2007; Richards & Tyrl, 2005). Furthermore, previous research found that wool fabric pretreated with an PAS mordant and dyed with a tannin dye (black walnut bark) had improved colorfastness to light and met ASTM test standards for use in apparel and home furnishings with a gray scale rating of 4 for lightfastness and washfastness (Doty & Haar, 2013; ASTM, 2002).

Potassium Aluminum Sulfate

Aluminum, the most common metal found on the earth's crust, accumulates naturally in deserts and volcanic areas (Cardon, 2007). Its derivatives, potassium aluminum sulfate (also called alum, potassium alum, or aluminum potassium sulfate), aluminum sulfate, aluminum ammonium sulfate, and aluminum acetate are some of the most commonly sourced mordants for natural dye artisans and researchers, due to their benign nature and ability to produce bright clear dye colors (Cardon, 2007; Burgess, 2011; Dean, 2010). During the Middle Ages alum was mined out of shale formations and was exploited for use in the growing textile industry in Europe (Cardon, 2007). Chemists' of the 1800s found a method for re-extracting alum from left over mine slag utilizing sulphurous fumes (Cardon, 2007). Today's alum is no longer gathered from the land and is instead created from the refinement of bauzite, resulting in a white granulated substance (Burgess, 2011; Wipplinger, 2005). Potassium aluminum sulfate (PAS), can be found in baking soda, used for pickling, utilized in water treatment systems, and is used in paper sizing (Burgess, 2011). PAS is currently one of the most commonly used mordants for animal fibers, due to its relative non-toxicity (Dean, 2010).

Research has shown that the use of PAS as a textile mordant changes the color of natural dyes towards warmer and more yellow on cotton and wool textiles dyed with a variety of dyestuffs (Doty & Haar, 2013; Haar, Schrader, & Gatewood, 2013). Doty and Haar (2013) found that wool textiles mordanted with PAS and dyed with tannin rich black walnut hulls, bark and leafs had a color change towards yellow compared to specimens that had been left unmordanted. The same study showed that the lightfastness of wool dyed with black walnut bark improved with the aid of an aluminum mordant, while washfastness remained similar between PAS premordanted and nonmordanted wool samples (Doty & Haar, 2013). Conversely, Shams-Nateri, Hajipour, Dehnavi, and Ekrami (2014) found washfastness improved with the use of a PAS mordant while lightfastness gave mixed results between the mordanted and nonmordanted nylon fabrics dyed with tannin rich pomegranate peels.

Cream of Tartar

Mordant formulas for wool often include the option of using cream of tartar or tartaric acid alongside the mordant to keep protein fibers from becoming stiff or harsh (Casselman, 1980). Tartar is created during the fermentation of grapes for wine and is used in baking to maintain an even pH level (Wipplinger, 2005). While this additive softens wool fibers it can also affect the color outcome, causing dye to brighten or change in hue (Wipplinger, 2005). To enable the researchers to report reliable color results between mordanted and nonmordanted samples, the use of tartaric acid was not included in this study.

Dendrology

Dendrology is the study and science of wooded plants (trees), which consist of a root system, trunk, and a crown of branches producing leafs (Kelsey, 2010). This section will discuss in brief the basic anatomy of timber and its terminology.

Wood is composed of varying amounts cellulose, lignin and hemicellulose cells (Kelsey, 2010). A cross section of a tree trunk has pronounced layers wrapped around each other, consisting of the bark, phloem, cambium, heartwood, sapwood, pith, and growth rings. See Figure 2.1. Bark is the dry protective outermost ring that insulates the tree and keeps the phloem and sapwood from drying out; the next layer is the phloem which carries dissolved sugars and growth hormones from the leafs to growing parts of the tree (Kelsey, 2010; Forest Products Laboratory, 2007). Under the phloem is the microscopic cambium layer whose job is to form new wood and bark cells, followed by the sapwood that carries sap from the roots to the leafs (Miller, 2007, Chapter 2). The inner most layers are the heartwood and pith. Heartwood is sapwood that had become clogged with extractives and helps to support the trees structure. Heartwood is frequently darker, denser and more decay resistant than sapwood due to the extractives (Kelsey, 2010; Forest Products Laboratory, 2007). At the very center of a tree is the pith, a small core where initial wood growth takes place (Forest Products Laboratory, 2007). Running through the heartwood and sapwood are growth rings, which form during the growing season. These rings indicate the age of the plant with one ring forming for each year of growth (Forest Products Laboratory, 2007).

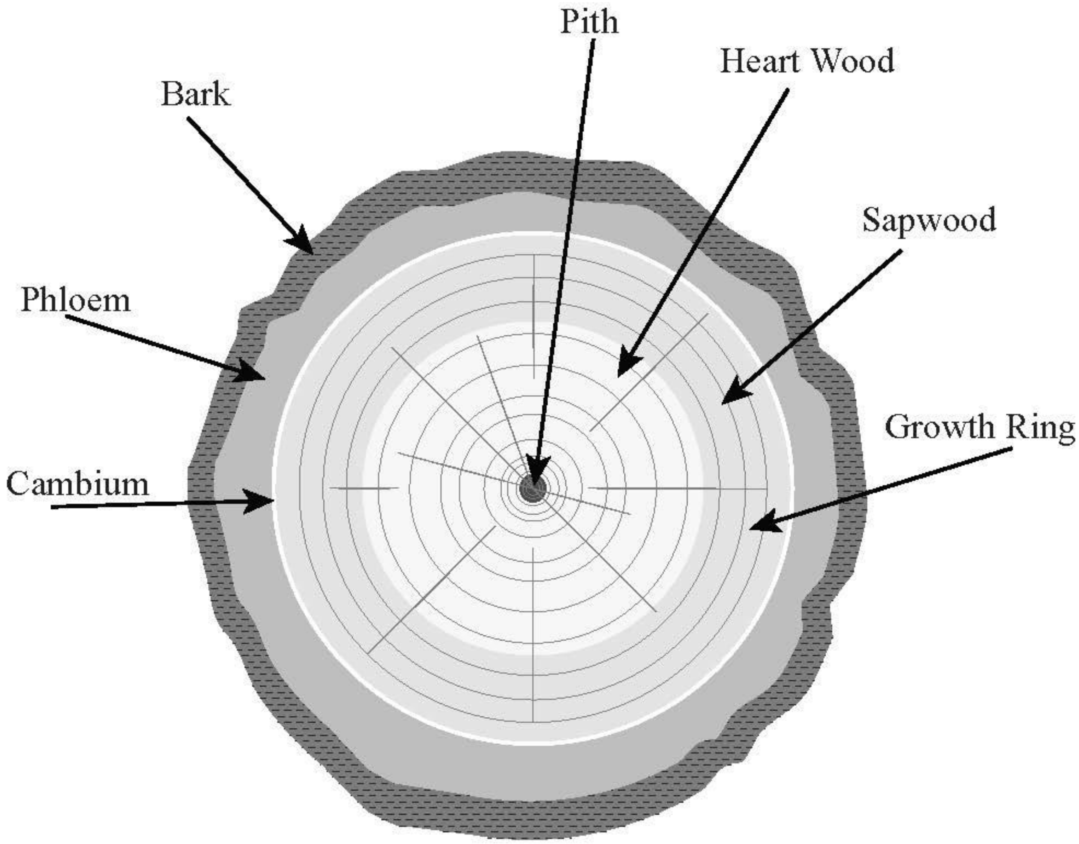

Figure 2.1. Cross-section diagram of a tree trunk[1]

[1] Image created by author and adapted from https://www.woodstairs.com/tradepage/how-to-evaluate-wood/tree-diagram/

Trees are categorized as either hardwoods or softwoods (Forest Products Laboratory, 2007). These terms tend to be a misnomer, not necessarily referring to the stability or density of the wood but instead describe the cellular structure of the lumber. Hardwoods have cells that are open at each end, forming a continuous tube that transports nutrients within the tree; this internal structure in turn produces a porous type wood (Forest Products Laboratory, 2007). In contrast, softwoods (usually conifers) are nonporous with tracheid type cells and typically produce cone type fruit, with needles in place of leafs (Forest Products Laboratory, 2007; Kelsey, 2010).

Dyewoods as a Natural Colorant

Natural dyers have long used numerous parts of trees (including their leafs, fruit, bark, and sawdust) and have found these dyes to be colorfast with the aid of a mordant (Cardon, 2007). Noted dyes from the differing parts of trees include eucalyptus leafs, black walnut fruit, Osage orange mill waste, logwood mill waste, pomegranate fruit, oak fruit, and the fruit of the ebony tree. These dyes give a range of color from reds (eucalyptus), purples (logwood), yellows (Osage orange) to dark gray and blacks (ebony and black walnut) (Flint, 2008). This use of varying parts of the tree as a dyestuff calls into question its sustainability as an ongoing and viable product. The fruit of trees could be considered the most sustainable, with new produce being grown each year. The fruit as dyestuff is also excellent in terms of being value added to the product, since the parts of the fruit that give the best color are usually inedible (e.g., black walnut hulls and pomegranate rinds). Less sustainable would be the bark, roots and heartwood of a tree. While it’s possible to collect roots and bark from trees without harming the plant, it does take skill and knowledge of a tree’s structural and nutritional needs. A healthier practice would be to collect these items from trees already being harvested for manufacturing or for timber control.

Black Walnut

Two different species of walnut are commonly used amongst dyers; *Juglans regina* L. (walnut) found in temperate regions of Europe and *Juglans nigra* L. (black walnut) commonly found in North America, both species of dyewoods (*regina* and *nigra*) are substantive due to naturally high levels of tannic acid (Cardon, 2007). Research on *Juglans regina L.* dye extracted from the green walnut hulls was found antimicrobial and the bark exceptionally colorfast to laundry and light when premordanted with alum, with a AATCC gray scale rating of 5 or no change on cotton yarn (Mirjalili & Karimi, 2013; Sharma & Grover, 2011). Black walnut and European walnut both belong to the Juglandaceae family and contain valuable compounds specific only to walnut trees, such as the quinone compound juglone and the hydrolysoble tannin compound juglandaceae (Cardon, 2007; Baker, 1958).

Research conducted on *Juglans nigra*, found lightfastness was enhanced with the aid of an aluminum mordant on wool textiles (Cardon, 2007; Doty & Haar, 2012). Leafs from black walnut (*Juglans nigra*) collected in the early summer give a golden tan, while the husks, bark, and mill waste gives a medium to dark brown color (Hardman & Pinhey, 2009; Richard & Tyrl, 2005). The brown dye is rendered darker and grayer with the addition of an iron modifier. Black walnut trees (*Juglans nigra*) belong to the hard wood family of tree and are mainly found in the eastern half of the United States, growing up to 40 m tall. This species produces a rich dark brown wood, heart shaped serrated leafs, and globose fruit with an outer husk concealing an edible nut (Cardon, 2007; Richards & Tyrl, 2005). See Figure 2.2.

Black Walnut in Kansas

Known as one of the most commercially valuable tree species in the Kansas, black walnut wood is used to produce furniture, gunstocks, and veneer (Atchison, 2005; Little, 1980;

Richards & Tyrl, 2005). The fruit of the black walnut tree is also considered valuable, as the nutmeat is edible with a strong distinct flavor (Atchison, 2005). Walnut is currently one of the top five most heavily harvested tree species in Kansas (at 14 %) and is encouraged as a cash crop by the Kansas Forestry Service (USDA Forest Service, 2008). Kansas growers should be aware of a disease spreading from Colorado amongst black walnut trees known as thousand cankers disease (Treiman, Atchison, McDonnell, Barden, & Moser, 2010). This disease is propagated by the walnut twig beetle, which in large populations spread a fungus through the tree's tissue (Treiman et al., 2010). Early signs of the disease is the yellowing of the upper crown, continued on with the death of the larger branches and foliage and the eventual death of the walnut tree (Treiman et al., 2010).

Figure 2.2. Ripe black walnut fruit (a). Black walnut leafs and fruit (b). Black walnut bark (c).[2]

[2] Photographs by author

Osage Orange

Dyes from Osage orange (*Maclura pomifera*) mill waste can range from bright yellow to olive yellow to khaki, depending on the mordant and post treatment. Osage orange dyes are derived from flavonoid compounds, which have been noted for poor colorfastness (Richards & Tyrl, 2005; Cardon, 2007; Cristea & Vilarem, 2006). This particular dyewood is well known to natural dyers and its dyestuff is sold as wood shavings from mill waste or as powdered or liquid extracts (Cardon, 2007). Furthermore, the dye is colorfast to light and laundering with the aid of a mordant (Cardon, 2007).

Osage orange is a dioecious medium sized tree with several trunks, branchlets producing stout, straight thorns and an irregular canopy (Little, 1980; Cardon, 2007; Smith & Perino, 1981). See Figure 2.3. Osage orange is considered a hardwood, with excellent rot and decease resistant qualities, having a life expectancy of 150 plus years. The female of the species yearly produces yellow-green fruit, large, globose in structure, and commonly referred to as a "hedge-ball" (Smith & Perino, 1981). When freshly cut, the heartwood is bright golden yellow, turning to darker yellow-brown when exposed to air. Osage orange is historically significant in the United States. Originating in Texas and Oklahoma, Osage orange trees were cultivated in the southern and midwestern states during the 1800s, due to its drought resistance and reliability as a natural hedge (Richards & Tyrl, 2005). Pioneers brought the Osage orange tree to the American Midwest and Kansas to create long hedgerows for field protection while also finding it a good dye source for yellow (Little, 1980; Richards & Tyrl, 2005; Cardon, 2007). Comanche Indians used Osage orange as an eye medication and the Kiowa and Pima tribes utilized the tree as a source of dye color (Cardon, 2007). The heartwood is extremely hard and historically used for archery bows by Native Americans, giving the tree its alternative name *bois d'arc or* "bow-wood tree" (Cardon, 2007). Furthermore, Osage orange was used during World War I to dye khaki colored uniforms, when supplies of synthetic dyes from Germany was cut short (Cardon, 2007).

Osage Orange in Kansas

While Osage orange is not native to Kansas it can be found running parallel to many rural gravel roads and dividing land sections. The Osage orange tree came to prominence in Kansas during the 19^{th} century as a living fence line for settler's livestock, due to its large thorns and stout nature (Rutter, 2013). These long hedge rows became obsolete as barbwire became the excepted norm for livestock containment and are now considered an obnoxious plant by some modern cattlemen, as they tend to invade range and pasture lands (Rutter, 2013).

Figure 2.3. Osage orange bark and thorns (a). Osage orange fruit (b). Osage orange fruit and leafs (c).[3]

[3] Photographs by author

Eastern Redcedar

Color from cedar (*Juniperus virginiana L.*) can vary from tan with an aluminum mordant to a warm medium brown with the addition of an iron modifier (Richards & Tyrl, 2005). Dyestuff from cedar trees is not readily available to natural dye artisans but the heartwood can be sourced from mill waste and the bark ecologically collected by harvesting from only one side tree, to allow for nutrients to flow to from the roots to the branches.

Eastern redcedar is an evergreen that grows in the mid to eastern regions of the United States and is resistant to drought, heat, and cold (Little, 1980). See Figure 2.4. Known to be a small evergreen, this species of tree is pyramidal in shape and produces small pale-blue berries (*Plant Fact Sheet*, 2002). Cedar trees grow in shelterbelts and abandoned fields, with a resiliency to environmental factors (i.e. prairie fires, drought) making it an abundant source of habitat for wildlife (Little, 1980). It is especially important in the winter, as the berries are a source of food to pheasant and whitetail deer. Settlers historically used cedar trees as a source of wood for fence post, roof rafters and windowsills, while Native Americans utilized all parts of the tree as a source of medicine for mouth sores, head colds, coughs and more (Richards & Tyrl, 2005).

Eastern Redcedar in Kansas

Redcedars are known as Kansas's only native evergreen tree and is popular in the state as a windbreak, due to its dense and compact foliage (Kansas Forest Service, 2013). However, this evergreen is considered an invasive species in Kansas, taking over undermanaged pastureland and devaluing many acres of Kansas prairie (USDA Forest Service, 2008). Dense stands of these trees will inhibit understory plant growth and can be highly problematic due to susceptibility to wildfires (USDA Forest Service, 2008). From 1965 to 2005 redcedar showed a 23,000% increase in stock volume on Kansas land (USDA Forest Service, 2008). Consequently, from 1994 to 2005 eastern redcedar had a 353% increase in timber volume of Kansas harvested trees (USDA Forest Service, 2008).

Figure 2.4. Eastern redcedar fruit (a). Eastern redcedar tree (b).[4]

[4] Photographs by author

Wool

Wool fiber from sheep is one of the most frequently used animal fibers due to its renewability, warmth and ability to except dyes (Burgess, 2011). The individual fibers can range in a variety of staple lengths (1 to 15 in.) and fiber diameters or micron counts (fine being around 15 micrometers and course fibers being around 34+). The surface of each individual fiber is made of a network of fine overlapping scales (Kadolph, 2007). This scale structure traps air, gives wool good insulating properties, and is responsible for wools ability to felt (Robson & Ekarius, 2011; Collier & Tortora, 2001). The fiber itself is comprised of a protein substance called keratin and the molecular structure of the fiber contains a high amount of amorphous material, making for a weaker yet highly absorbent textile (Collier & Tortora, 2001). Furthermore, wool is naturally fire resistant and will self-extinguish when removed from a flame (Collier & Tortora, 2001; Kadolph, 2007).

Wool is a naturally oily fiber and a fleece (wool shorn from one animal) can potentially contain around 5-50% potassium carbonate and lanolin (grease). Consequently, this fiber requires scouring before it can be spun into yarn. When commercially prepared into yarn, wool is scoured to remove lanolin, potassium carbonate and dirt from the fiber and requires several washings in a warm, soapy, and alkaline solution (Cardon, 2007; Collier & Tortora, 2001). It is then treated through carbonization, which removes any remaining vegetable matter, requiring the fiber to be dipped in sulfuric acid to eliminate cellulosic materials (Collier & Tortora, 2001). After being carded to align the fibers, wool is then prepared for spinning into yarn.

Wool Yarn

Wool yarn comes in a variety of sizes, indicated through a numbering system. The system is a display of two numbers (e.g., 20/2, 10/4) the first of which represents the general size and yardage for that yarn. For example, a 30/2 yarn is smaller diameter than a 10/2 yarn because the same amount of fiber will produce a greater amount of yardage the finer it is spun, so the 30/2 yarn will produce three times the yardage as the 10/2 yarn (Fannin, 1975). The second digit indicates the number of single plies. So a 30/2 yarn has two plies, while a 30/4 yarn has four plies.

ASTM Standards

The purpose of this research was to compare potassium aluminum sulfate (PAS) mordanted and nonmordanted wool yarns dyed with Kansas black walnut, Osage orange, and eastern redcedar mill waste on tests for light, laundering, and staining. After washfastness and lightfastness testing, the yarns were assessed for color change according to the AATCC's Gray Scale for Color Change and compared to minimum standards as set by the American Society of Testing and Materials (ASTM). Since the target audience for this research was natural dye artisans who use yarn as part of their craft, a wide selection of standards were taken into consideration. Standards for this study included Performance Specification for Woven Blouse, Dress, Dress Shirt & Sport Shirt Fabrics (D 7020 – 05), Performance Specification for Woven Necktie and Scarf Fabrics (D 3785 – 02), and Performance Specification for Knitted Necktie and Scarf Fabrics (D 4035 – 02) due to the possibility of an artisan utilizing the dyed wool for accessories or apparel, either handwoven, knitted, or crocheted. Household standards included the Performance Specification for Woven Napery and Tablecloth Fabrics: Household and Institutional (D 4111 – 02), as handweavers often create textiles for use in table setting and for kitchen use. Also considered was the Standard Performance Specification for Blanket Products for Institutional and Household Use (D 5432 – 93) for knitters, weavers, and crocheters who create coverlets for both functional and/or aesthetic reasons. Even though the end uses vary widely, the same minimum gray scale ratings exists across the standards, being a light grade of 4, laundering grade of 4, and a staining grade of 3.

Table 2.1. ASTM Gray Scale Rating Standards for Colorfastness to Light, Laundering, and Staining for Selected Textile Performance Specifications

ASTM Test Standard	Light (Gray Scale Rating)	Laundering (Gray Scale Rating)	Staining (Gray Scale Rating)
D 7020 - 05 Performance Specification for Woven Blouse, Dress, Dress Shirt & Sport Shirt Fabrics	4	4	3
D 3785 - 02 Performance Specification for Woven Necktie and Scarf Fabrics	4	4	3
D 4035 - 02 Performance Specification for Knitted Necktie and Scarf Fabrics	4	4	3
D 4111 - 02 Performance Specification for Woven Napery and Tablecloth Fabrics: Household and Institutional	4	4	3
D 5432 - 93 Performance Specification for Blanket Products for Institutional and Household Use	4	4	3

Note. Color change ratings: 5 = none, 4 = slight, 3 = noticeable, 2 = considerable, 1 = severe.

Chapter 3 - **Research Methods**

The purpose of this research was to compare the colorfastness between potassium aluminum sulfate (PAS) mordanted and nonmordanted 30/2 wool yarns dyed with Kansas black walnut (*Juglans Nigra*), Osage orange (*Maclura pomifera*), and eastern redcedar (*Juniperus virginiana*) mill waste. Pre-testing determined concentrations of these dyewoods to weight of fiber (owf) required to produce comparable depths of shade on 30/2 wool yarn. Depths of shade was visually evaluated using the American Association of Textile Colorist and Chemists (AATCC) standard depth scales, to ensure dyeings of visually comparable depth. Following pre-testing for concentration amounts, all test samples were scoured and half of the samples received the mordant treatment of 12% PAS. The test samples were dyed using the predetermined concentrations and then tested for colorfastness to laundering, light, and staining according to AATCC test methods and evaluation procedures (AATCC, 2009). Laundering tested the colorfastness of wool yarn expected to withstand five repeated hand launderings at low temperature, while the light testing determined fading after exposure to 20 AATCC fading units (AFU) or approximately 21.5 hours (AATCC, 2009). Tests for staining evaluate for possible unintended pickup of colorant by a substrate during washing (AATCC, 2009). This chapter presents the testing materials, experimental procedures, and evaluation procedures to compare mordanted and nonmordanted black walnut, Osage orange, and eastern redcedar mill waste on 30/2 wool yarn for tests to light, laundering, and staining.

Materials

Experimental Yarns

Yarn used in this study was 30/2 worsted wool yarn (style # SK 2/30 at a owf of 5 g) sourced from Testfabrics, Inc. (Testfabrics, Inc.). Size 30/2 yarn was selected due to its availability from a reliable testing company and its appropriateness for hand-weaving. The researcher used information from this study to dye yarns for use in handwoven textiles and art garments, thus the very fine 30/2 yarn was applicable for future visual research. As for fiber type, wool was selected due to its affinity towards natural dyes, allowing for richer dye colors compared to a cellulosic fiber, such as cotton (Cardon, 2007).

Scouring Agent

All yarns were scoured (pre-washed) using Orvus® paste, a surfactant paste largely made of water and sodium lauryl sulfate, obtained from Dover Saddlery (www.doversaddlery.com). Orvus® paste is pH neutral with synthetic surfactant and wetting agents and known for its excellent detergency, emulsifying, and dispersing properties (*P&G: Orvus WA Paste*, 2014). Scouring the wool fiber removes unwanted oils, soil, and chemicals allowing for a more even application of mordant and dye. In addition to its cleaning and wetting properties Orvus® paste was chosen due to its current use by fiber artists and its pH neutral properties.

Mordanting Agent

Half of the wool yarns were pre-mordanted using technical grade potassium aluminum sulfate (PAS) acquired from Carol Leigh's Hillcreek Fiber Studio (www.hillcreekfiberstudio.com), then dyed and compared to nonmordanted and dyed samples to determine if colorfastness was improved. Dyes rich in tannin, such as found in tree colorants, often don’t require a mordant (Casselman, 1942; Burgess, 2011). However, previous research by the author found PAS treated wool fabric dyed with black walnut leafs, hulls, and bark to have improved colorfastness to light ratings compared to nonmordanted fabric; yet, colorfastness to laundering and staining ratings did not improve (Doty & Haar, 2012). Thus, the researcher predicted that PAS would improve the colorfastness to light of dyes from mill waste.

Natural Dyewoods

Black walnut, Osage orange, and eastern redcedar mill waste was sourced from a privately owned sawmill located near Galva, Kansas, USA. Millings from this location contained all portions of the dye's respective tree trunk (i.e. bark, phloem, cambium, sapwood, heartwood, and pith). It cannot be determined as to the year or season when the trees were felled or milled.

Paper Teabags

To reduce the amount of sawdust particles in the dye baths, the sawdust was placed in chlorine-free paper teabags sourced from the company T-Sac and purchased through Amazon (www.amazon.com). The teabags were size 3 or 18.4 cm x 8.25 cm (7.25 in. x 3.25 in.). The teabags were sewn across the top to contain the dyestuff while allowing dye color to be extracted in hot water without sawdust particles escaping. Absorption of dye to the teabag was minimal and cleanup was efficient with the sawdust contained.

Pre-Test: Depth of Shade Determination

Dyes from different tree species have different tinctorial strengths resulting in varied color depth and colorfastness properties when used at equal concentrations. To ensure dyeings of visually comparable color depth, dye concentrations were pre-tested to find a standard depth of shade between the same dye on PAS mordanted and nonmordanted wool yarns, using AATCC's Evaluation Procedure 4-2007, Standard Depth Scales for Depth Determination (AATCC, 2009). The Depth of Shade scale consists of six designations; there is "standard depth" at 1/1, "double depth" at 2/1 that is darker than standard depth, and fractional depths with 1/3, 1/6, 1/12 and 1/25 that are lighter than standard depth (AATCC, 2009). Each chart presents the depth designations as different chromas, across different hues (AATCC, 2009). See Figure 3.1. Establishing like depths of shade between mordanted and nonmordanted yarns colored with the same dyes allowed the researcher to draw reasonable conclusions regarding colorfastness of particular dyewoods and make comparisons between PAS mordanted and nonmordanted wool yarns.

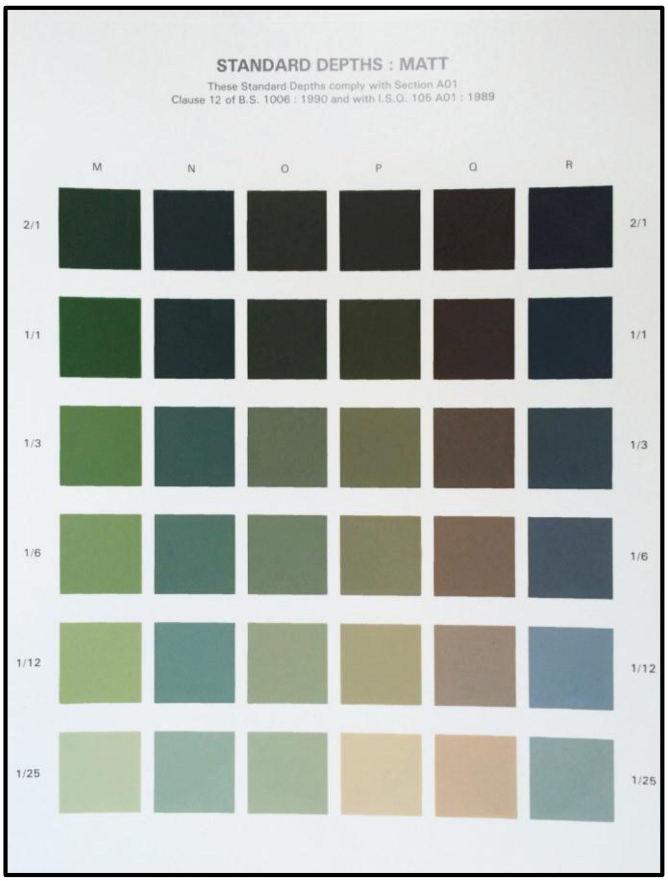

Figure 3.1. Example of Depth Matt used for AATCC Depth of Shade Scale[5]

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[5] Photograph taken by author of Standard Depths: Matt, British Standard 1006: 1990 (1989) Stockport, England. Copyright 1989, SDL Atlas Textile Testing Solutions

Dying Depth of Color Samples

Steps to scouring and pre-mordanting the wool yarn were followed according to Wipplinger's (2005) formula and Haar's (2011) procedures for treating protein fibers. Pre-testing established comparable depth of shade between mordanted and nonmordanted samples of the same dyewoods.

Scouring

All pre-test skeins were scoured using a solution of Orvus® paste and reverse osmosis (RO) water. Pre-test yarns were made into 5 ± 0.02 g, 110 meter (120 yd) skeins, with loose ends tied together and secured with three figure-eight choke ties to keep the yarn from tangling. Approximately 30 min before scouring, the sample skeins were submerged in 25 °C (77 °F) RO water to ensure a more even uptake of surfactants. Three scour baths were created and skeins were randomly assigned to a bath of 2% Orvus® paste to of fiber weight (owf) and a ratio of 80:1 (liquor-to-dry goods) of RO water (40°C± 3 or 100 °F) in beakers. The skeins were kept from tangling by suspending them from a stir stick into a beaker containing the solution. The skeins were gently hand agitated or stirred using a slotted spoon every 5 min for 20 min, then rinsed in 25 °C (77 °F) RO water and laid flat to dry on a clean towel.

Premordanting

Half of the wool yarn samples were randomly assigned to one of three premordant baths consisting of 12% potassium aluminum sulfate (PAS) to owf. The scoured wool skeins were wetted out for 30 min in 25 °C (77 °F) RO water, then added to a bath of 25 °C (77 °F) RO water at a ratio of 80:1 and 12% PAS to owf in beakers. The premordant bath was gently stirred every 10 min and the temperature raised from 25 °C (77 °F) to 90 °C (194 °F) over the span of 30 min, continued at 90 °C (194 °F) for 60 min, then left to cool overnight. Next day the skeins were rinsed in 25 °C (77 °F) RO water and laid out flat to dry on a clean towel.

Dye Extraction

Initial pre-test dyewood concentrations were determined from the researcher's prior experience extracting color from mill waste. Dye solutions were created by placing the appropriate ratio for each dyewood into individual paper teabags with pretest amounts of 100% dyewood to owf for black walnut and eastern redcedar. Osage orange was 50% dyewood to owf, due to a perceived higher dye concentration. The dyewood teabags were covered with 500 ml RO water at 60 °C (140 °F), then heated to 90 °C (176 °F) over 30 min and kept at 90 °C (176 °F) for 60 min with agitation of the teabag occurring every 5 to 10 min and then cooled and left to soak 12 hours. The teabags were then filtered out of the dye bath, squeezed gently to extract remaining moisture, and additional water was added to each dye bath to replace water lost due to evaporation.

Dyeing

The pre-test wool skeins were wetted out for 30 min in 25 °C (77 °F) RO water, then added to the respective dye baths. The dye baths were gently stirred every 10 min and the temperature raised from 25 °C (77 °F) to 90 °C (194 °F) over the span of 60 min, continued at 90 °C (194 °F) for 60 min, then left to cool overnight. Next day the skeins were removed from the dye baths, rinsed under running RO water until the rinse water was clear and laid flat to air dry on a clean towel.

Evaluation of Color Difference

The pre-test dyed samples were compared visually to the standard depth scales following AATCC Evaluation Procedure 9-2007, Visual Assessment of Color Difference of Textiles, Option C (AATCC, 2009). The dyed samples and depth scale were compared under daylight setting (D65) at a 45° angle in a light box (VeriVide®, Leicester, UK). As the mordanted and nonmordanted samples were not at equal standard depth, the dye concentrations were adjusted until both reached a comparable standard. See Table 3.2 for final concentrations and depth amounts.

Table 3.1. Final Dyewood Concentrations and Standard Depth of Shade to Achieve Comparable Depth Between PAS Mordanted and Nonmordanted Wool Yarn

Dyewood and Mordant Type	Concentration to owf	Standard Depth
Black Walnut Mordanted Yarn	100%	Q 1/12
Black Walnut Nonmordanted Yarn	200%	Q 1/12
Osage Orange Mordanted Yarn	40%	A 1/1
Osage Orange Nonmordanted Yarn	50%	A 1/1
Eastern Redcedar Mordanted Yarn	100%	P 1/25
Eastern Redcedar Nonmordanted Yarn	150%	P 1/25

Experimental Test Methods

Test yarns were made into 5 ± 0.02 g, 110 m (120 yd) skeins, with loose ends tied together and secured with three figure-eight choke ties to keep the yarn from tangling. Six separate dye baths were created for each dyewood; three baths for mordanted wool yarns and three for unmordanted wool yarns. There were 3 skeins or replications for each dyewood and treatment. Thus, there were a total of 54 skeins (3 dyewoods x 6 dyebaths x 3 skeins).

Scouring

All skeins were scoured in an aqueous solution of 2% Orvus® paste to owf and a ratio of 80:1 (liquor-to-dry goods) or 400 ml of reverse osmosis (RO) water in an Atlas Launder-Ometer. Approximately 30 min before scouring, the sample skeins were submerged in 25 °C (77 °F) RO water to ensure a more even uptake of surfactant. The wetted samples, Orvus® paste and 40°C± 3 (100 °F) RO water were then added to individual Atlas Launder-Ometer stainless steel canisters and shaken 6-7 times by hand to begin agitation. The Launder-Ometer rotates the canisters in a thermostatically controlled water bath, slowly raising the temperature of the liquid within the canisters. The canisters containing the wool skeins and aqueous scour solution began at 40 °C (170 °F) and ran for 20 min, the canisters were taken out of the Launder-Ometer, lids removed and the solution was allowed to cool down to 30 °C (86 °F) over 30 min. Reducing the temperature before rinsing the yarn under cool was important as to not shock the wool, which could result in felting. Samples were then removed from the canisters, rinsed under running RO water and laid flat on a towel to air dry.

Premordanting

Half of the wool yarn test samples were premordanted using a treatment of 12% potassium aluminum sulfate (PAS) to owf in an Atlas Launder-Ometer. The scoured wool skeins were wetted out for 30 min prior to premordanting in 25 °C (77 °F) RO water. An aqueous solution of 12% PAS to 80:1 RO water was created by dissolving the PAS in 200 ml of near boiling water, pouring the contents into the Launder-Ometer canister, then rinsing the beaker of the remaining metallic salt with 200 ml of 25 °C (77 °F) water. The wetted out samples were then randomly added to individual Atlas Launder-Ometer canisters and shaken 6-7 times by hand to begin agitation. The canisters were run in an Atlas Launder-Ometer for 90 min, the temperature raised from 40 °C (170 °F) to 90 °C (185 °F) and then cooled down to 60 °C (140 °F) over 30 min. The samples were then removed from the canisters, rinsed under running RO water for 10 seconds and laid flat on a towel to air dry.

Dyeing

Dye Solution

Six dye baths were created for each dyewood. Dye solutions were created by placing the pre-test concentration for each dyewood into individual paper tea bags, sized 18.4 cm x 8.25 cm (7.25 in. x 3.25 in.) and sewn across the top to secure the contents. Dye concentrations were: 40% Osage orange to owf on mordanted yarn, 50% Osage orange to owf on nonmordanted yarn, 100% black walnut to owf on mordanted yarn, 200% black walnut to owf on nonmordanted yarn, 100% eastern redcedar to owf on mordanted yarn, and 150% eastern redcedar to owf on nonmordanted yarn. One or two teas bag containing the appropriate amount of sawdust was suspended in 1,200 ml beakers and submerged in 1,000 ml hot RO water (approximately 60 °C). The dye bath was heated to 90 °C (176 °F) for 60 min, with agitation occurring every 5 to 10 min and then left to cool for 12 hours. The teabags were then lifted out of the dye bath and squeezed to remove remaining dye liquor. Additional water was added to each dye bath to replace water lost due to evaporation, resulting in a total of 1,000 ml of dye.

Sample Dyeing

Each dyewood produced six separate dye baths, three baths for nonmordanted skeins and three for mordanted. A single dye bath was separated into three launder-ometer canisters and a random mordanted or nonmordanted skein added to each canister. Of the three skeins, one skein was used for washfastness testing, one for lightfastness testing and one was kept as a standard. To dye each wool skein sample, 330 ml of dye solution and 70 ml of RO water was poured into individual Launder-Ometer canisters, followed by a wetted out 5g skein. Over 90 min the temperature was raised from 25 °C (77 °F) to 90 °C (176 °C) and then cooled down to 60 °C over 20 min in the Launder-Ometer. The skeins were left in their canisters to cool back down to 25 °C (77 °F) over night. Next day, samples were removed from the canisters, rinsed under running RO water for approx. 5 sec. and laid flat on a towel to air dry.

Test Specimen Preparation

Colorfastness to Light, Option 3

Yarns were prepared according to AATCC Test Method 16-2004 Colorfastness to Light, Option 3 (AATCC, 2009). Dyed yarn was wound onto frames of white card stock 63.5 mm wide and 150.0 mm long (2.5 in. x 6.0) to create 25.0 mm x 150.0 mm (1.0 in. x 6.0 in.) sections, the yarn was closely packed but not overlapping (AATCC, 2009). To evaluate for color change to light, the unmasked portion was compared to the masked or unexposed portion of the sample. There were 3 dyewoods x 2 treatments (mordant and nonmordant) x 3 replications, equaling 18 specimens for colorfastness to light testing.

Colorfastness to Laundering

Yarns were prepared according to AATCC Test Method 61-2007 Colorfastness to Laundering, Home and Commercial, Test No. 1A, Option 2 (AATCC, 2009). Dyed skeins were folded in half to create a uniform amount of yarn measuring 50 mm (2.0 in.) across the folded area (AATCC, 2009). Squares of bleached cotton test fabric equaling approximately the same weight was folded over each end of the layered yarn specimen and sewn down with a lockstitch in polyester thread (AATCC, 2009). Attached to each specimen was a No.1 multi-fiber test fabric (Testfabric, Inc.), machine-basted down with a lockstitch in polyester thread. After laundering testing was performed the samples were combed or brushed to realign the yarns before comparison to the standard sample skein. A standard sample skein was used from the same dyewood bath to compare for color change after testing. There were three replications for each of the three dyewoods and two treatments (mordant and nonmordant), equaling 18 specimens for colorfastness to laundering testing from the original 54 skeins.

Colorfastness to Staining

To test for staining, a No. 1 multi-fiber test strip (Testfabrics, Inc.) was machine-basted to each colorfastness to laundering test specimen. The multi-fiber test swatch contained six fibers (filament acetate, bleached cotton, nylon, spun silk, spun viscose, and worsted wool), which ran in individual component bands 0.8 mm (5/16 in.) in width. Each test swatch measured 5.0 cm x 5.0 cm (2 in. x 2 in.) and was attached to the folded edge of the yarn specimen, with the component bands running perpendicular to the machine-basting and the wool band on the right.

Colorfastness Testing

Test for Colorfastness to Light

Testing for colorfastness to light was performed in accordance to AATCC Test Method 16-2004 Colorfastness to Light, Option 3 (AATCC, 2009) by the Atlas Weathering Company using an Atlas Xenon Weather-Ometer. These specimens were subjected to 22 continuous AATCC fading units utilizing an Xenon-Arc light, at a temperature of 63 ± 1 °C and a relative humidity of 30 ± 5%, with specimens being rotated regularly (AATCC, 2009).

Test for Colorfastness to Laundering

Colorfastness to laundering was performed in accordance with AATCC Test Method 61-2007 Colorfastness to Laundering: Accelerated, Test No. 1A (AATCC, 2009). Test No. 1A is used to simulate five repeated hand launderings at a low temperature of 40 ± 3 °C (105 ± 5 °F) (AATCC, 2009). An Atlas Launder-Ometer was used to create systematic testing conditions by rotating stainless steel containers, with the specimen, water, and detergent in a thermostatically controlled water bath. Test No. 1A for hand laundering was selected due to the 100% wool fiber content of the yarns and the expected laundering habits of fiber artisans.

The Atlas Launder-Ometer was preheated and held at 40 ± 2 °C during the entirety of the testing procedure. Added to each stainless steel canister was 200 ml RO water at approx. 40° C (104F), .15% of total volume AATCC Standard Reference Detergent and 10 stainless steel balls. The canisters were loaded into a preheated Atlas Launder-Ometer horizontally in the adapters with the covers positioned to strike the water first. Then the temperature of the canisters was normalized without the test specimens for two min. After which, the test specimens were added by stopping the machine with a row of canisters in an upright position, removing the covers from the canisters running left to right. Then placing a crumbled test specimen into the solution, replacing the canister cover but not tightening it until the entire row is complete and then tightening the covers going left to right, continuing this procedure till all four rows were completed. After placing the specimens into the canisters, the machine ran for 45 min., continuing at 40 °C (86 °F).

After the laundering process was completed, the specimens were removed and rinsed in a succession of three beakers with 300 ml 25 °C (77 °F) RO water. The samples were rinsed by agitating (stirring and squeezing) for 1 min per bath, then placed into the next beaker of fresh RO water till all three beakers were completed. All rinsed specimens were laid flat on a towel to air dry.

Color Evaluation Procedures

Colorimetric Analysis

Test specimens subjected to AATCC Test Method 61-2007 Colorfastness to Laundering: Accelerated, Test No. 1A and AATCC Test Method 16-2004 Colorfastness to Light, Option 3 were evaluated using CIELab ratings, determining the specific color parameters lightness (L^*), greenness-redness (a^*), and blueness-yellowness (b^*) (Marcus,1998). CIE L^*, a^*, and b^* ratings inform the amount of perspective values, for L^* the higher the number the lighter the color (perfect white is 100) and the lower the number the darker the color (perfect black is 0) (Marcus, 1998). For coordinate a^*, red is +a* and green is –a* (Marcus, 1998). Lastly, for coordinate b^*, yellow is indicated by +b* and blue is indicated by –b* (see Figure 3.2) (Marcus, 1998). A RM200QC Imagining Spectrocolormeter (X-Rite, Michigan, USA) was used to obtain CIELab ratings. The device takes an image of the specimen using 8 different visible illuminations and 1 UV LED and each color measurement is a combination of 27 images taken by the device. Yarn specimens were wound onto frames of white card stock 63.5 mm wide by 150.0 mm long (2.5 in. x 6.0) to create 25.0 mm x 150.0 mm (1.0 in. x 6.0 in.) sections, the yarn was closely packed but not overlapping. All evaluated specimens were conditioned for 12 hours or longer in a room with controlled atmospheric conditions at 65 ± 2 relative humidity and 21 ± 1 °C (70 ± 2 °F). When colorimetric readings were performed the yarn was compressed together to prevent the white card stock from contaminating the reading. Three readings were taken from each specimen and averaged.

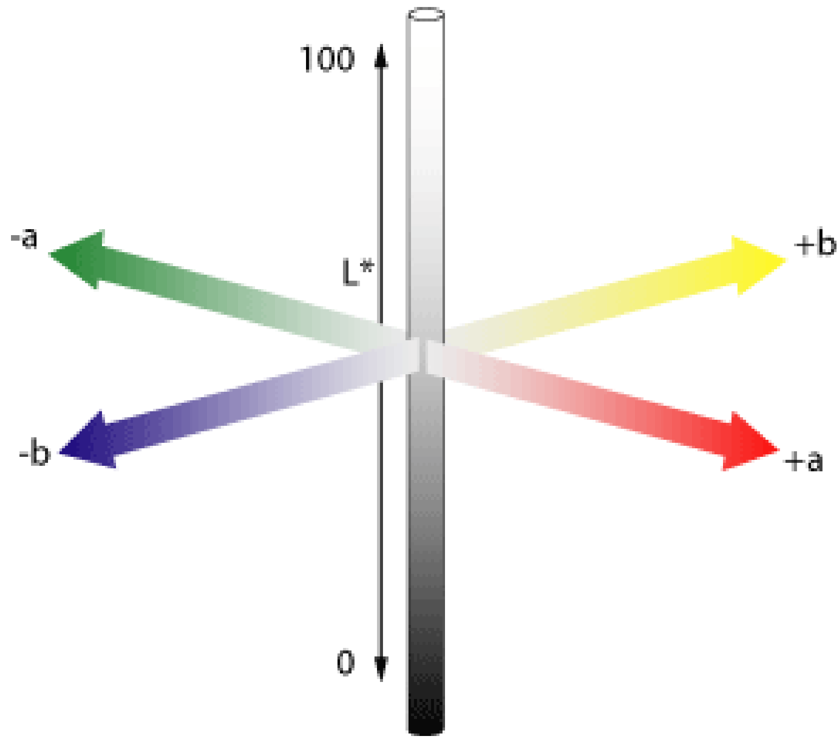

Figure 3.2. CIELab coordinates and values.[6]

[6] Retrieved from http://www.industchem.com/content/3/1/26/figure/F1

Gray Scale Rating Analysis

Test specimens subjected to AATCC Test Method 61-2007 Colorfastness to Laundering: Accelerated, Test No. 1A were evaluated for color change and staining by three in-house evaluators with normal color vision, utilizing the Gray Scale for Color Change and the Gray Scale for Staining. Gray scale ratings for color change and staining are as follows: 5= no change, 4= slight, 3= noticeable, 2= considerable, and 1= severe change. All in-house evaluated specimens were conditioned for 24 hours or longer in a room with controlled atmospheric conditions at 65 ± 2 relative humidity and 21 ± 1 °C (70 ± 2 °F). Test specimens subjected to AATCC Test Method 16-2004 Colorfastness to Light, Option 3 were evaluated by two evaluators at Atlas Weathering Company, utilizing AATCC's Gray Scale for Color Change.

Color change

Color change of dyed samples subjected to lightfastness and laundering testing were assessed utilizing AATCC Evaluation Procedure 1, Gray Scale for Color Change (AATCC, 2009). Gray Scale for Color Change is a process by which researchers visually compare the difference in color or contrast between the corresponding untreated and treated samples (AATCC, 2009). Color change is described as the change in lightness, hue and/or chroma of any tested specimen when compared to the untested specimen (AATCC, 2009). Corresponding treated and untreated specimens were placed on top of white card stock, side by side on a 45° angle with a sharp junction between the two pieces (AATCC, 2009). An AATCC gray scale was then placed along the juncture of the test specimen and a gray colored mask placed over that. This gray mask eliminated any influence of the surrounding area (AATCC, 2009). All in-house specimens were evaluated in a VeriVide® light box, with daylight lighting setting (D65) and the evaluators viewing at a 90° to the surface plane. All perceived differences were graded and recorded utilizing AATCC's Gray Scale for Color Change.

Staining

To evaluate for possible unintended pickup of colorant by a substrate during washing, AATCC Evaluation Procedure 2, Gray Scale for Staining was used. To assess the No. 1 multifiber test strips after colorfastness to laundering testing, the test strips were removed from the washed skeins and any frayed edges trimmed. Standard staining strips were also washed according to AATCC Evaluation Procedure 2, Gray Scale for Staining but without a dyed specimen. Stained strips were placed by a standard No. 1 multifiber test strip, side by side on a 45° angle (AATCC, 2009). An AATCC gray scale was then placed along the juncture of the tested and untested strips, then placing a gray colored mask over that to eliminate any influence of the surrounding area (AATCC, 2009). Each fiber type was compared to its untested counterpart, taking care to create a sharp juncture between the two (AATCC, 2009). All in-house specimens were evaluated with a light box (VeriVide®, Leicester, UK), with daylight lighting setting (D65) and the evaluators viewing at a 90° angle to the surface plane, see Table 3.1 for ratings.

Statistical Analysis

Statistical analysis was performed using Minitab®, version 17.1.0. A general linear model (GML) analysis of variance (ANOVA) was conducted to study the effect between the independent variables (mordant, exposure) on L^*, a^*, and b^* values for each dye type, at the 95% confidence level. This statistical procedure explained if there was any significant effect of mordant (no mordant and yarns mordanted with PAS) and exposure (light and laundering exposure). Descriptive statistics (mean and standard deviation) for L^*, a^*, and b^* coordinates were used to outline means and standard deviations and to examine the directional relationship within significant effects indicated by the ANOVA results. Two-sample t-test were utilized to examine if there was a significant effect of mordant treatment on colorfastness to light and laundering in overall color difference in consideration of $L*$, $a*$, and $b*$ values, calculated as the following. Δ E = $\sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2}$.

Chapter 4 - **Results**

The purpose of this research was to compare the colorfastness of PAS mordanted and nonmordanted 30/2 wool yarns dyed with Kansas black walnut, Osage orange, and eastern redcedar mill waste. Pre-testing determined concentrations of these dyewoods to owf required to produce comparable depths of shade on mordanted and nonmordanted yarns of the same dye type. Thereafter, test samples were dyed using the predetermined concentrations and then tested for colorfastness to light, laundering and staining according to AATCC test methods and evaluation procedures (AATCC, 2009). Laundering tested the colorfastness of wool yarn expected to withstand five repeated hand washings at low temperature, while the light testing determined fading after exposure to 20 AATCC fading units (AFU) or approximately 21.5 hours (AATCC, 2009). Staining was evaluated for possible unintended pickup of dye colorants by a substrate during tests for laundering (AATCC, 2009). Colorimetric readings were used to obtain CIELab measurements, determining the specific color parameters for lightness ($L*$), greenness-redness ($a*$), and blueness-yellowness ($b*$). These ratings were used to statistically analyze any significant effects of mordant type (mordanted, nonmordanted) and exposure (light, laundering). Furthermore, AATCC gray scale ratings were used to indicate color change for specimens subjected to light, laundering, and staining testing and were used to corroborate statistical data and better inform the textile testing community who are accustomed to the rating scale. Gray scale ratings were also used to assess if specimens subjected to light, laundering, and staining met ASTM Standard for Performance Specification for Woven Blouse, Dress, Dress Shirt & Sport Shirt Fabrics (D 7020 – 05), Performance Specification for Woven Necktie and Scarf Fabrics (D 3785 – 02), Performance Specification for Knitted Necktie and Scarf Fabrics (D 4035 – 02), Woven Napery and Tablecloth Fabrics: Household and Institutional (D 4111 – 02), and for

Blanket Products for Institutional and Household Use (D 5432 – 93). Data collected on tests for light, laundering, and staining informed the acceptance or rejection of the hypotheses outlined at the beginning of this paper. The research questions and hypotheses will be discussed in Chapter 5 while the results are presented in this chapter.

Dye Concentrations and Depth Amounts

To ensure dyeings of visually comparable color depth, dye concentrations were pre-tested to find a standard depth of shade between the same dye on PAS mordanted and nonmordanted wool yarns, using AATCC's Evaluation Procedure 4-2007, Standard Depth Scales for Depth Determination (AATCC, 2009). Nonmordanted yarns dyed with black walnut, Osage orange, and eastern redcedar required 10% to 100% more dye than yarns mordanted with potassium aluminum sulfate (see Table 4.1). Black Walnut had a standard depth of Q1/12 and required twice as much mill waste for nonmordanted (200% owf) compared to mordanted wool yarns (100% owf) to achieve comparable color depth. Osage orange gave a standard depth at 1/1 using 50% owf for nonmordanted and 40% owf for mordanted. Eastern redcedar had the lowest depth shade presented on the AATCC Standard Depth Scales for Depth Determination at 1/25 standard depth, with 150% owf for nonmordanted and 100% owf for mordanted yarn. The higher dyewood concentrations required to achieve comparable depth of shade between PAS mordanted and nonmordanted wool yarn indicate that a PAS mordant treatment enhanced dye absorption thus requiring much less dyewood for black walnut and eastern redcedar to obtain same depth of shade compared with nonmordanted yarns.

Table 4.1. Final Dyewood Concentrations and Standard Depth of Shade to Achieve Comparable Color Depth between PAS Mordanted and Nonmordanted Wool Yarn

Dyewood and Mordant Type	Concentration to owf	Standard Depth
Black Walnut Mordanted Yarn	100%	1/12
Black Walnut Nonmordanted Yarn	200%	1/12
Osage orange Mordanted Yarn	40%	1/1
Osage orange Nonmordanted Yarn	50%	1/1
Eastern Redcedar Mordanted Yarn	100%	1/25
Eastern Redcedar Nonmordanted Yarn	150%	1/25

Analysis of Colorimetric Data for Colorfastness to Light Results

Testing for colorfastness to light was performed in accordance to AATCC Test Method 16-2004 Colorfastness to Light, Option 3 (AATCC, 2009) by the Atlas Weathering Company using an Atlas Xenon Weather-Ometer. There were 3 dyewoods x 2 mordant types x 3 replications, equaling 18 specimens for colorfastness to light testing, which were subjected to 22 continuous AATCC fading units utilizing an Xenon-Arc light, (AATCC, 2009). Resulting color for exposed and unexposed specimens were rated using CIE *L* a* b** values. This section will assess for significant differences within the independent variables of Mordant and Exposure, evaluating the significance of a PAS mordant and the significance of lightfastness testing.

Statistical Analysis of Colorimetric Data

GLM ANOVAs were conducted separately for each CIELab coordinate, *L** (lightness), *a** (greenness-redness), *b** (blueness-yellowness), to study the effect of Mordant (mordanted, nonmordanted) and Exposure (exposed to light, not exposed to light). An ANOVA statistically informs if the change (or effect) within and between independent variables was significant. In this section we will concentrate on the effects within Mordant and Exposure. The focus on the independent variable Mordant looked at the significance of a PAS mordant on specimens before and after exposure to light. Furthermore, the focus on the independent variable Exposure reveals if there was a significant effect of lightfastness testing on nonmordanted and mordanted specimens. Descriptive statistics (mean and standard deviation) for *L* a* b** coordinates were used to examine the directional relationship within the independent variables Mordant and Exposure (Table 4.2).

Table 4.2. Descriptive Statistics for L* a* b* Coordinates of Unexposed Standards and Specimens Exposed to Colorfastness to Light Testing

Dye/Mordant	Standard / Exposed to Light	*L* Mean (SD)	*a* Mean (SD)	*b* Mean (SD)	Color Swatch
Black Walnut Mordanted	Standard	61.32 (0.96)	11.59 (0.46)	24.70 (0.71)	
Black Walnut Mordanted	Exposed to Light	65.06 (1.17)	11.66 (0.56)	25.64 (0.88)	
Black Walnut Nonmordanted	Standard	57.59 (1.43)	10.49 (0.50)	22.12 (1.25)	
Black Walnut Nonmordanted	Exposed to Light	62.54 (1.31)	10.59 (0.36)	23.43 (0.61)	
Osage Orange Mordanted	Standard	69.49 (1.52)	8.37 (0.60)	56.03 (1.79)	
Osage Orange Mordanted	Exposed to Light	63.96 (1.11)	10.12 (0.26)	45.90 (1.06)	
Osage Orange Nonmordanted	Standard	70.40 (1.67)	6.24 (0.39)	45.02 (1.76)	
Osage Orange Nonmordanted	Exposed to Light	63.66 (0.84)	10.24 (0.34)	35.29 (0.98)	

Eastern Redcedar Mordanted	Standard	73.87 (1.39)	7.98 (0.19)	24.77 (0.74)	
Eastern Redcedar Mordanted	Exposed to Light	79.21 (0.95)	6.20 (0.49)	24.46 (0.87)	
Eastern Redcedar Nonmordanted	Standard	71.06 (1.19)	9.08 (0.38)	14.93 (0.78)	
Eastern Redcedar Nonmordanted	Exposed to Light	75.93 (0.91)	6.37 (0.25)	18.22 (0.38)	

Note. Color swatches were generated with L* a* b* rounded to the nearest whole number using http://www.workwithcolor.com/color-converter-01.htm

Black Walnut

For CIELab coordinate *L** (lightness) an ANOVA found a significant effect at the $p<.001$ level for Mordant $F(1, 35)= 57.95$, as well as a significant effect for Exposure $F(1, 35)= 112.20$ for wool yarns dyed with black walnut and subjected to colorfastness to light testing (see Table 4.3). Descriptive statistics (mean and standard deviation) suggest that specimens dyed with black walnut and mordanted with PAS were lighter than nonmordanted specimens before and after lightfastness testing. Furthermore when specimens were subjected to colorfastness to light testing they were lighter for both mordanted and nonmordanted specimens when compared to the standard mordanted and nonmordanted specimens (see Table 4.2).

For CIELab coordinate *a** (greenness-redness) an ANOVA found a significant effect of Mordant at the $p<.001$ level with $F(1, 35)= 46.63$, yet no significant effect was found for Exposure $F(1, 35)= 0.28$. Descriptive statistics suggest that mordanted specimens were redder than nonmordanted before and after light exposure. For CIELab coordinate *b** (blueness-yellowness) an ANOVA found a significant effect of Mordant at the $p<.001$ level $F(1, 35)= 64.20$ and Exposure $F(1, 35)= 14.24$. Descriptive statistics suggest mordanted specimens were yellower than nonmordanted specimens before and after light exposure. Mordanted and nonmordanted specimens exposed to lightfastness testing were also bluer than unexposed specimens (see Table 4.2).

Table 4.3. ANOVA to Compare CIELab Coordinates of Mordant and Nonmordant for Colorfastness to Light Testing of Black Walnut Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	87.734	87.734	57.95***
	Exposure	1	169.868	169.868	112.20***
	Mordant x Exposure	1	3.361	3.361	2.22
*a**	Mordant	1	10.5625	10.5625	46.63***
	Exposure	1	0.0625	0.0625	0.28
	Mordant x Exposure	1	0.0025	0.0025	0.01
*b**	Mordant	1	51.6003	51.6003	64.20***
	Exposure	1	11.4469	11.4469	14.24***
	Mordant x Exposure	1	0.3025	0.3025	0.38
Total		35			

*p<.05, **p<.01, ***p<.001

Osage Orange

For coordinate *L** no significant effect was observed for Mordant $F\ (1, 35) = 0.48$ (see Table 4.4). However, a significant effect was detected at the $p<.001$ level for Exposure $F\ (1, 35) = 192.53$. Notably, descriptive statistics for *L** suggest that mordanted and nonmordanted specimens dyed with Osage orange and exposed to light were darker than the standard mordanted and nonmordanted specimens.

CIELab measurement *a** was significantly different at the $p<.001$ level for the effect for Mordant $F\ (1, 35) = 51.06$ and Exposure $F\ (1, 35) = 422.89$. Descriptive statistics suggest that mordanted standard specimens where redder than unmordanted standard specimens, however a* coordinates were similar after light exposure for mordanted and nonmordanted specimens (see Table 4.2). Furthermore, descriptive statistics suggests that mordanted and nonmordanted specimens exposed to light were redder than non-exposed specimens. For coordinate *b** significant differences were detected at the $p<.001$ level for Mordant $F\ (1, 35) = 501.78$ and for Exposure $F\ (1, 35) = 423.60$. Descriptive statistics suggest that mordanted specimens were yellower than nonmordanted specimens before and after light exposure. When looking at the effect of Exposure descriptive statistics suggest that mordanted and nonmordanted specimens were bluer when exposed to light compared to standard mordanted and nonmordanted specimens.

Table 4.4. ANOVA to Compare CIELab Coordinates of Mordant and Nonmordant for Colorfastness to Light Testing of Osage Orange Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	0.840	0.840	0.48
	Exposure	1	339.174	339.174	192.53***
	Mordant x Exposure	1	3.300	3.300	1.87
*a**	Mordant	1	9.000	9.000	51.06***
	Exposure	1	74.534	74.534	422.89***
	Mordant x Exposure	1	11.334	11.334	64.31***
*b**	Mordant	1	1051.92	1051.92	501.78***
	Exposure	1	888.04	888.04	423.60***
	Mordant x Exposure	1	0.36	0.36	0.17
Total		35			

*p<.05, **p<.01, ***p<.001

Easter Redcedar

CIELab coordinate *L** for wool yarns dyed with eastern redcedar and subjected to lightfastness testing, a significant effect was observed at the $p<.001$ level for the effect of Mordant $F(1, 35) = 65.57$ and for Exposure $F(1, 35) = 184.40$ (see Table 4.5). Descriptive statistics suggest that mordanted specimens were lighter than nonmordanted specimens before and after light exposure (see Table 4.2). When looking at the effect of Exposure, descriptive statistics suggest that light exposed mordanted and nonmordanted yarns were lighter than standard mordanted and nonmordanted specimens.

For coordinate *a** a significant effect was observed at the $p<.001$ level for the effect of Mordant $F(1, 35) = 29.54$ and for Exposure $F(1, 35) = 370.95$. Descriptive statistics suggest that mordanted standard specimens were greener than unmordanted standard specimens, however a* coordinates were similar for mordanted and nonmordanted specimens exposed to light (see Table 4.2). Furthermore, mordanted and nonmordanted specimens were both greener after light exposure compared to standard specimens. For coordinate b* a significant effect was observed at the $p<.001$ level for the effect of Mordant $F(1, 35) = 1123.85$ and for Exposure $F(1, 35) = 38.60$. Descriptive statistics suggest that overall mordanted specimens were yellower than nonmordanted specimens regardless of light exposure. Nonmordanted specimens were also yellower when exposed to light while mordanted specimens did not appear to have much change after light exposure for coordinate *b** (see Table 4.2).

Table 4.5. ANOVA to Compare CIELab Coordinates of Mordant and Nonmordant for Colorfastness to Light Testing of Eastern Redcedar Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	83.418	83.418	65.57***
	Exposure	1	235.111	235.111	184.80***
	Mordant x Exposure	1	0.490	0.490	0.39
*a**	Mordant	1	3.610	3.610	29.54***
	Exposure	1	45.388	45.388	370.95***
	Mordant x Exposure	1	1.960	1.960	16.04***
*b**	Mordant	1	580.81	580.81	1123.85***
	Exposure	1	19.95	19.95	38.60***
	Mordant x Exposure	1	29.16	29.16	56.42***
Total		35			

*p<.05, **p<.01, ***p<.001

Summary of Colorimetric Data for Colorfastness to Light

ANOVA statistical tests were used to analyze for significance of the effect of Mordant (mordanted, nonmordanted) and Exposure (exposed to light, not exposed to light) on *L* a* b** values.

Mordant

For the effect of Mordant, *L** (lightness) showed that black walnut dyed specimens were significantly different in that mordanted specimens were lighter than nonmordanted before and after lightfastness testing. Mordanted black walnut specimens were also redder (*a*)* and yellower (*b**) than nonmordanted. Osage orange dyed specimens had no significant difference for Mordant when looking at the *L** coordinate. Before light exposure mordanted Osage orange dyed yarns were redder than unmordanted standards, however *a** coordinates were similar after light exposure for mordanted and nonmordanted. Mordanted specimens were also yellower (*b**) than nonmordanted before and after lightfastness testing. Eastern redcedar dyed yarns were significantly different for *L** with mordanted specimens being lighter than nonmordanted before and after lightfastness testing. Mordanted specimens dyed with eastern redcedar were also greener (*a*)* and yellower (*b**) before and after lightfastness testing compared to nonmordanted.

Exposure

When looking at the effect of Exposure, all dyes had significant effect for *L** (lightness) when exposed to 21.5 hours of light. For black walnut the significant effect for mordanted and nonmordanted specimens was lighter than their standards, there was also a significant effect on *b** with mordanted and nonmordanted black walnut dyed yarns turning bluer when exposed to light. For Osage orange dyed yarns there was a significant effect for *L*,* in which the color was darker for mordanted and nonmordanted specimens exposed to light compared to their mordanted and nonmordanted standards. Specimens dyed with Osage orange were also redder (*a**) and bluer (*b**) after lightfastness testing. Eastern redcedar dyed mordanted and nonmordanted yarns were significantly lighter for when compared to the untreated standards. Mordanted specimens where greener (*a**) than unmordanted specimens before lightfastness testing, however *a** coordinates were similar for mordanted and nonmordanted specimens exposed to light. For coordinate *b**, mordanted specimens did not appear to have a significant effect to light exposure while nonmordanted specimens were yellower when exposed to light.

Analysis of Colorimetric Data for Colorfastness to Laundering Results

Mordanted and nonmordanted yarn dyed with black walnut, Osage orange, and eastern redcedar were tested for colorfastness to laundering in accordance with AATCC Test Method 61-2007 Colorfastness to Laundering: Accelerated, Test No. 1A (AATCC, 2009). This test was equivalent to five repeated hand launderings at a low temperature of 40 ± 3 °C (105 ± 5 °F) (AATCC, 2009). After washfastness testing, specimens were evaluated using a X-Rite RM200QC Imagining Spectrocolormeter (X-Rite, Michigan, USA) to obtain CIELab ratings, determining the specific color coordinates *L** *a** *b**. There were 3 dyewoods x 2 mordant types (mordant and nonmordant) x 3 replications, equaling 18 specimens for colorfastness to laundering testing. This section will assess for significant differences within the independent variables of Mordant and Exposure, evaluating the significance of a PAS mordant and the significance of washfastness testing.

Statistical Analysis of Colorimetric Data

ANOVAs were conducted separately for CIELab coordinates *L** *a** *b** to study the effect of Mordant (mordanted, nonmordanted) and Exposure (exposed to laundering, not exposed to laundering). An ANOVA statistically informs if the change (or effect) within and between independent variables was significant. We will again concentrate on the effects within Mordant and Exposure. The focus on the independent variable Mordant looks for a significant effect between PAS mordanted and nonmordanted specimens before and after exposure to washfastness testing. Furthermore, the focus on the independent variable Exposure reveals if there was significant effect of laundering on nonmordanted and mordanted specimens. Descriptive statistics (mean and standard deviation) for L^* a^* b^* coordinates were used to examine the directional relationship within the independent variables Mordant and Exposure (Table 4.2).

Table 4.6. Descriptive Statistics for CIELab Coordinates of Specimens Exposed to Laundering Testing and Unexposed Standards

Dye / Mordant	Standard / Exposed to Laundering	*L* Mean (SD)	*a* Mean (SD)	*b* Mean (SD)	Color Swatch
Black Walnut Mordanted	Standard	61.32 (0.96)	11.59 (0.46)	24.70 (0.71)	
Black Walnut Mordanted	Exposed to Laundering	60.01 (0.69)	10.43 (0.36)	20.50 (0.47)	
Black Walnut Nonmordanted	Standard	57.59 (1.43)	10.49 (0.50)	22.12 (1.25)	
Black Walnut Nonmordanted	Exposed to Laundering	58.30 (0.97)	9.20 (0.39)	20.20 (0.44)	
Osage Orange Mordanted	Standard	69.49 (1.52)	8.37 (0.60)	56.03 (1.79)	
Osage Orange Mordanted	Exposed to Laundering	72.06 (1.04)	7.03 (0.82)	58.70 (1.54)	
Osage Orange Nonmordanted	Standard	70.40 (1.67)	6.24 (0.39)	45.02 (1.76)	
Osage Orange Nonmordanted	Exposed to Laundering	68.88 (0.52)	7.26 (0.49)	39.81 (0.63)	

Eastern Redcedar Mordanted	Standard	73.87 (1.39)	7.98 (0.19)	24.77 (0.74)	
Eastern Redcedar Mordanted	Exposed to Laundering	71.33 (0.88)	10.04 (0.39)	23.02 (0.66)	
Eastern Redcedar Nonmordanted	Standard	71.06 (1.19)	9.08 (0.38)	14.93 (0.78)	
Eastern Redcedar Nonmordanted	Exposed to Laundering	70.82 (1.38)	9.29 (0.43)	12.99 (0.50)	

Note. Color swatches were generated with L* a* b* rounded to the nearest whole number using http://www.workwithcolor.com/color-converter-01.htm

Black Walnut

CIELab coordinate L^* for wool yarns dyed with black walnut and subjected to colorfastness to laundering, a significant effect was detected at the $p<.001$ level for Mordant $F(1, 35)=60.71$ but not for the effect of Exposure $F(1, 35)=0.74$ (see Table 4.7). Descriptive statistics suggest that mordanted specimens were lighter than nonmordanted specimens, before and after washfastness testing (see Table 4.7). However, no significance for exposure means little to no change for coordinate L^* before and after washfastness testing for both mordanted and nonmordanted specimens dyed with black walnut.

For measurement a^* significance was observed at the $p<.001$ level for Mordant $F(1, 35)=65.58$ and Exposure $F(1, 35)=71.97$. Descriptive statistics for a^* suggest that mordanted specimens were redder than nonmordanted specimens before and after exposure to laundering. Mordanted and nonmordanted specimens were also greener after washfastness testing when compared to unwashed standard specimens (See Table 4.7). For measurement b^* a significant effect was detected for Mordant $F(1, 35)=29.97$ and Exposure $F(1, 35)=135.64$. Descriptive statistics suggest that standard mordanted specimens were yellower than standard nonmordanted specimens, while mordanted and nonmordanted specimens were similar for coordinate b^* after exposure to laundering (see Table 4.7). Furthermore, both mordanted and nonmordanted specimens were bluer after washfastness testing.

Table 4.7. ANOVA to Compare Mordant and Nonmordant for Colorfastness to Laundering Testing of Black Walnut Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	66.694	66.694	60.71***
	Exposure	1	0.810	0.810	0.74
	Mordant x Exposure	1	9.201	9.201	8.38**
*a**	Mordant	1	12.250	12.250	65.58***
	Exposure	1	13.444	13.444	71.97***
	Mordant x Exposure	1	0.040	0.040	0.21
*b**	Mordant	1	18.63	18.63	29.97***
	Exposure	1	84.33	84.33	135.64***
	Mordant x Exposure	1	11.67	11.67	18.78***
Total		35			

*p<.05, **p<.01, ***p<.001

Osage Orange

CIELab measurement *L** for wool yarns dyed with Osage orange and subjected to colorfastness to laundering had a significant effect detected at the $p<.05$ level for Mordant $F\,(1, 35)=7.17$ (see Table 4.8). However, there was no significant effect from Exposure $F\,(1, 35)=1.52$. Descriptive statistics suggest that standard mordanted specimens were darker than standard nonmordanted specimens before laundering but that mordanted specimens became lighter than nonmordanted specimens after washfastness testing (see Table 4.6). No significance for Exposure means little to no change in L* before and after washfastness testing for both mordanted and nonmordanted specimens.

There was significant difference for coordinate *a** at the $p<.001$ level for Mordant $F\,(1, 35)=22.83$. However, there was no significant difference for Exposure $F\,(1, 35)= 34.76$. Descriptive statistics suggest that standard mordanted specimens were redder than standard nonmordanted specimens, however both mordanted and nonmordanted specimens had a similar rating after washfastness testing was performed. For measurement *b** significance was indicated at the $p<.001$ level for Mordant $F\,(1, 35)=887.06$. For the effect of Exposure $F\,(1, 35)=6.42$, significance was indicated at the $p<.05$ level. Descriptive statistics suggest that mordanted specimens were yellower than nonmordanted specimens before and after washfastness testing (see Table 4.6). Furthermore, washed mordanted specimens had a shift towards yellower compared to unwashed standard mordanted specimens and a shift towards bluer for washed nonmordanted compared to unwashed standard nonmordanted specimens.

Table 4.8. ANOVA to Compare Mordant and Nonmordant for Colorfastness to Laundering Testing of Osage Orange Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	11.560	11.560	7.17*
	Exposure	1	2.454	2.454	1.52
	Mordant x Exposure	1	37.618	37.618	23.23***
*a**	Mordant	1	8.123	8.123	22.83***
	Exposure	1	0.234	0.234	0.66
	Mordant x Exposure	1	12.367	12.367	34.76***
*b**	Mordant	1	2011.52	2011.52	887.06***
	Exposure	1	14.57	14.57	6.42
	Mordant x Exposure	1	139.63	139.63	61.58***
Total		35			

*p<.05, **p<.01, ***p<.001

Eastern Redcedar

For CIELab measurement L* on wool yarns dyed with eastern redcedar, a significant effect was indicated at the $p<.001$ level for Mordant $F(1, 35)=16.42$. Furthermore, significance was found at the $p<.05$ level for the effect of Exposure $F(1, 35)=11.39$ (see Table 4.9). Descriptive statistics suggest that mordanted specimens were lighter than nonmordanted specimens before and after washfastness testing. Also suggested is that washed mordanted and nonmordanted specimens were lighter than standard mordanted and nonmordanted specimens.

For measurement a*, significance was detected at the $p<.001$ level for Exposure $F(1, 35)=88.89$ but no significance was detected for Mordant $F(1, 35)=2.03$. Descriptive statistics suggest that washed specimens were redder than unwashed standard specimens, with mordanted yarn having a greater color shift than nonmordanted (see Table 4.6). Measurement b* indicated significance at the $p<.001$ level for Mordant $F(1, 35)=1909.19$ and for Exposure $F(1, 35)=65.83$. Suggestions from descriptive statistics are that mordanted specimens were yellower than nonmordanted specimens before and after washfastness testing (see Table 4.6). Furthermore mordanted and nonmordanted specimens were bluer after washfastness testing.

Table 4.9. ANOVA to Compare Mordant and Nonmordant for Colorfastness to Laundering Testing of Eastern Red Cedar Dyed Wool Yarns

CIELab	Factor	*df*	*SS*	*MS*	*F*
*L**	Mordant	1	24.83		16.42***
	Exposure	1	17.22		11.39**
	Mordant x Exposure	1	11.90		7.87**
*a**	Mordant	1	0.266		2.03
	Exposure	1	11.673		88.89***
	Mordant x Exposure	1	7.747		58.99***
*b**	Mordant	1	888.040		1909.19***
	Exposure	1	30.618		65.83***
	Mordant x Exposure	1	0.090		0.19
Total		35			

*p<.05, **p<.01, ***p<.001

Summary of Colorimetric Data for Colorfastness to Laundering

ANOVA statistical tests were performed to analyze the effect of Mordant (mordanted, nonmordanted) and Exposure (laundered, not laundered) on CIE L^* a^* b^* values.

Mordant

When reviewing the independent variable Mordant, black walnut dyed specimens with a PAS mordant were lighter (L^*) than nonmordanted specimens. Mordanted specimens were also redder (a^*) than nonmordanted specimens before and after tests for colorfastness to laundering. However mordanted specimens were also yellower (b^*) than nonmordanted specimens but only before testing occurred, after which the b^* coordinate was similar for both. For Osage orange dyed specimens looking at coordinate L^*, mordanted specimens were darker than nonmordanted before washfastness testing then became lighter after washfastness testing. Mordanted specimens dyed with Osage orange were redder (a^*) than nonmordanted before washfastness testing but become similar after laundering. For b^*, mordanted Osage orange was yellower than nonmordanted before and after washfastness testing. Eastern redcedar dyed specimens for L^*, mordanted specimens were lighter than nonmordanted specimens before and after washfastness testing. Mordanted specimens were also yellower than nonmordanted before and after tests for colorfastness to laundering.

Exposure

For the independent variable Exposure, black walnut mordanted and nonmordanted specimens had no significant difference for L^* before and after tests for colorfastness to laundering. However, mordanted and nonmordanted black walnut dyed specimens were significantly greener (a^*) and bluer (b^*) when subjected to washfastness testing. For Osage orange dyed specimens mordanted and nonmordanted had no significant difference for L^* before and after washfastness testing. Standard mordanted Osage orange specimens were redder ($a*$) than standard nonmordanted specimens but both become similar after testing occurred. Mordanted Osage orange was also yellower ($b*$) and nonmordanted Osage orange was bluer ($b*$) after wash testing (see Table 4.6). For eastern redcedar mordanted and nonmordanted specimens were lighter, redder and bluer after laundering compared to their standards.

Comparison of Overall Color Change for Mordanted and Nonmordanted Specimens When Exposed to Light or Laundering

Part of this study's purpose was to see if an additional metallic mordant could improve the colorfastness to light or laundering for wool yarns dyed with black walnut, Osage orange, and eastern redcedar. Two-sample t-tests were performed to investigate the effect of a PAS mordant versus no mordant on overall color difference between specimens exposed to light and laundering, where the overall color difference Δ E is calculated by Δ E = $\sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2}$.

L_1: $L*$ value of exposed specimen

L_2: $L*$ value of unexposed specimen

a_1: $a*$ value of exposed specimen

a_2: $a*$ value of unexposed specimen

b_1: $b*$ value of exposed specimen

b_2: $b*$ value of unexposed specimen

Gray scale ratings for lightfastness and washfastness were not statically analyzed but were used as additional visual information to better inform statistical data and as additional information for the textile testing community.

Black Walnut

For yarns dyed with black walnut and exposed to light, two-sample t-tests showed significance at the $p<0.1$ confidence level between mordanted ($M = 3.95$, $SD = 1.25$) and nonmordanted ($M = 5.17$, $SD = 1.31$) conditions; $t(16) = 2.01$, $p = 0.061$, suggesting that mordanted yarn showed less color change after exposure to light than nonmordanted yarns (see Table 4.10). Conversely, average Gray Scale ratings for lightfastness of black walnut dyed wool yarns rated nonmordanted specimens slightly better than mordanted specimens, with ratings of 4.5 (slight to no change) and 4.25 (slight change) respectively. See Table 4.12.

For yarns dyed with black walnut and laundered, two-sample t-tests show a significant effect at the $p<.001$ level between mordanted and nonmordanted for Δ E. This result suggest that nonmordanted ($M = 2.563$, $SD = 0.706$) yarns outperformed (i.e., had less color change) mordanted ($M = 4.605$, $SD = 0.500$) conditions; $t(16) = -7.09$, $p = 0.000$ (see Table 4.11). Furthermore, AATCC gray scale ratings of 4.44 (slight change) for nonmordanted and 4.06 (slight change) for mordanted yarns support the finding (see Table 4.12).

Table 4.10. Two-Sample T-Test Results of Mordant Type for Colorfastness to Light on Dyed Wool Yarn

	Nonmordanted		Mordanted		
	M	SD	M	SD	t-test
Black Walnut	5.17	1.31	3.95	1.25	2.01*
Osage Orange	12.52	1.14	11.70	1.35	1.38
Eastern Redcedar	6.51	0.72	5.72	0.95	1.99*

*p<.05, **p<.01, ***p<.001

Table 4.11. Two-Sample T-Test Results of Mordant Type for Colorfastness to Laundering on Dyed Wool Yarn

	Nonmordanted		Mordanted		
	M	SD	M	SD	t-test
Black Walnut	2.56	0.71	4.61	0.50	-7.09***
Osage Orange	5.57	0.57	4.26	1.07	3.25**
Eastern Redcedar	2.35	0.70	3.83	0.60	-4.80***

*p<.05, **p<.01, ***p<.001

Table 4.12. Gray Scale Means of Mordant Type for Colorfastness to Light and Laundering of Dyed Wool Yarn

	Lightfastness	Washfastness
Black Walnut Nonmordanted	4.5	4.44
Black Walnut Mordanted	4.25	4.06
Osage Orange Nonmordanted	2.75	3.67
Osage Orange Mordanted	2.75	4.78
Eastern Redcedar Nonmordanted	4.25	4.50
Eastern Redcedar Mordanted	4.25	3.72

Note. Color change ratings: 5 = none, 4 = slight, 3 = noticeable, 2 = considerable, 1 = severe.

Osage Orange

For Osage orange yarns exposed to light, two-sample t-tests suggest no significant effect between mordanted (M = 11.70, SD = 1.35) and nonmordanted (M = 12.52, SD = 1.14) conditions; t(16) = 1.38, p = 0.187 (see Table 4.10). AATCC gray scale ratings corroborated this finding, showing that both mordanted and nonmordanted specimens were rated at 2.75 or considerable to noticeable change (see Table 4.12). For yarns dyed with Osage orange and exposed to laundering, two-sample t-tests indicated significance at the p<.05 confidence level that mordanted (M = 4.26, SD= 1.07) yarns had less color change than nonmordanted (M = 5.569, SD = 0.565) conditions; t(16) = 3.25, p= 0.005 (see Table 4.11). Gray scale ratings show that mordanted yarns had an average rating of 4.78 or little to no change, while nonmordanted yarns had an average rating of 3.67 or noticeable to slight change (see Table 4.12).

Eastern Redcedar

For yarns exposed to light and dyed with eastern redcedar two-sample t-tests showed significance at the p<0.1 confidence level that mordanted (M= 5.72, SD= 0.95) yarns had less color change than nonmordanted (M= 6.51, SD= 0.73) conditions; t(16)= 1.99, p= 0.064 (see Table 4.10). However, AATCC gray scale ratings show that mordanted and nonmordanted yarns performed the same with a rating 4.25 (see Table 4.12). For yarns exposed to laundering two-sample t-tests showed significance at the p<.001 level that nonmordanted (M = 2.34, SD = 0.70) yarns had improved colorfastness to laundering over mordanted (M = 3.83, SD = 0.60) conditions; t(16) = -4.80, p = 0.000 (see Table 4.11). AATCC gray scale ratings of 4.25 for nonmordanted and 3.72 for mordanted yarns support this result (see Table 4.12).

Summary of Overall Color Change Between Mordanted and Nonmordanted Specimens

Two-sample t-tests were performed, investigating the effects of a PAS mordant compared to no mordant on the colorfastness to light and laundering of dyed wool yarns. Overall color difference of mordanted and nonmordanted specimens were assed using Δ E which was calculated by Δ E = $\sqrt{(L_1 - L_2)^2 + \ (a_1 - a_2)^2 + \ (b_1 - b_2)^2}$. Gray scale ratings were also used as additional visual information. Findings from Δ E indicated that a PAS mordant enhanced colorfastness to light for black walnut and eastern red cedar, while Osage orange had no color difference between mordant types. Gray scale ratings for lightfastness supported the Δ E findings for Osage orange and eastern red cedar, but found slightly less color change without the mordant for black walnut. When comparing mordanted and nonmordanted specimens exposed to laundering, Δ E findings suggest that a PAS mordant improved washfastness for Osage orange dyed yarns, while black walnut and eastern red cedar had less color change without a mordant. Gray scale ratings corroborated the findings for colorfastness to laundering.

Staining Results

Testing for staining occurred during colorfastness to laundering testing as per AATCC Test Method 61-2007 Colorfastness to Laundering, Home and Commercial, Test No. 1A, Option 2 (AATCC, 2009). The exposed to laundering multi-fiber test swatch was compared to a standard strip that had been washed according to AATCC Test Method 61-2007(AATCC, 2009). Gray scale ratings for staining are as follows: 5= no change, 4= slight, 3= noticeable, 2= considerable, and 1= severe change.

Black Walnut

Staining ratings for PAS mordanted yarns dyed black walnut ranged from 4.39 to 5 with similar ratings for nonmordanted yarns dyed with black walnut ranging from 4.28 to 4.89 (see Table 4.13). Even though overall staining was minimal across fiber types, mordanted yarns had marginally higher ratings, except for wool, which was rated the same between mordant type. Of the fiber types, nylon showed the most staining regardless of mordant type.

Osage Orange

Staining ratings for PAS mordanted yarns dyed with Osage orange ranged from 3.67 to 4.44. Nonmordanted yarns ranged from 3.89 to 4.44 (see Table 4.13). The mean gray scale for nonmordanted (4.25) was marginally higher than mordanted (4.01) and staining on the wool strip had the largest difference with nonmordanted rating 4.5 and mordanted rating 3.67.

Eastern Redcedar

Staining ratings for PAS mordanted yarns dyed with eastern redcedar ranged from 4.89 to 5 and were similar for nonmordanted yarns ranging from 4.83 to 5 (see Table 4.13). Thus, all fiber types had minimal to no color transfer from the dyed yarn.

Table 4.13. Gray Scale Means of Mordant Type for Staining Evaluation of Dyed Wool Yarn

Dye Type/ Mordant Type	Multifiber Strip						Average Rating
	Acetate	Cotton	Nylon	Silk	Viscose	Wool	
Black Walnut, Nonmordanted	4.56	4.5	4.28	4.56	4.89	4.83	4.6
Black Walnut, Mordanted	4.89	5	4.39	4.72	5	4.83	4.81
Osage Orange, Nonmordanted	4.06	4.28	3.89	4.44	4.33	4.5	4.25
Osage Orange, Mordanted	4.22	3.72	3.83	4.44	4.17	3.67	4.01
Eastern Redcedar, Nonmordanted	5	5	4.94	4.94	5	4.89	4.96
Eastern Redcedar, Mordanted	4.94	5	4.94	5	5	4.83	4.95

Note. Color change ratings: 5 = none, 4 = slight, 3 = noticeable, 2 = considerable, 1 = severe.

Comparison of Gray Scale Ratings to ASTM Standards

After testing for colorfastness to light, laundering, and staining, specimens were assessed for color change according to the AATCC's Gray Scale for Color Change and compared to minimum gray scale color ratings established by the American Society of Testing and Materials (ASTM), see Table 4.15. Since the target audience for this research was natural dye artisans who use yarn as part of their craft, a wide selection of apparel and home furnishing performance specifications were considered. Even though the end uses vary widely, the minimum gray scale ratings were the same across the standards, with a light grade of 4, a laundering grade of 4, and a staining grade of 3 (see Table 4.15).

With the exception of Osage orange lightfastness ratings, dyed yarns in this study met minimum gray scale ratings for colorfastness to light, laundering, and staining Interestingly, Osage orange dyed yarns failed due to darkening in color, instead of the more typical lightening of color. Osage orange changed from a bright yellow to a medium warm brown after exposure to light testing.

Table 4.14. Gray Scale Means of Mordant Type for Colorfastness to Light, Laundering, and Staining of Dyed Wool Yarn

	Light	Laundering	Staining
Black Walnut Nonmordanted	4.5	4.44	4.60
Black Walnut Mordanted	4.25	4.06	4.81
Osage Orange Nonmordanted	2.75	3.67	4.25
Osage Orange Mordanted	2.75	4.78	4.01
Eastern Redcedar Nonmordanted	4.25	4.50	4.96
Eastern Redcedar Mordanted	4.25	3.72	4.95

Note. Color change ratings: 5 = none, 4 = slight, 3 = noticeable, 2 = considerable, 1 = severe.

Table 4.15. ASTM Gray Scale Rating Standards for Light, Laundering, and Staining for Selected Textile Performance Specifications

ASTM Test Standard	Light	Laundering	Staining
D 7020 - 05 Performance Specification for Woven Blouse, Dress, Dress Shirt & Sport Shirt Fabrics	4	4	3
D 3785 - 02 Performance Specification for Woven Necktie and Scarf Fabrics	4	4	3
D 4035 - 02 Performance Specification for Knitted Necktie and Scarf Fabrics	4	4	3
D 4111 - 02 Performance Specification for Woven Napery and Tablecloth Fabrics: Household and Institutional	4	4	3
D 5432 - 93 Performance Specification for Blanket Products for Institutional and Household Use	4	4	3

Note. Color change ratings: 5 = none, 4 = slight, 3 = noticeable, 2 = considerable, 1 = severe.

Chapter 5 - **Summary, Discussion, and Conclusions**

The overall purpose of this study was to compare colorfastness properties between PAS mordanted and nonmordanted wool yarns. Justification for this study was to increase the knowledge of local Kansas dyewoods for use as a sustainable alternative for textile coloration by natural dye artisans who use yarn as part of their craft. Colorfastness to light, laundering, and staining tests were conducted according to AATCC test methods (AATCC, 2009). CIELab data and AATCC Gray Scale ratings were used to answer the study's research questions and hypotheses.

In this chapter, the research questions and hypotheses are answered and discussed to understand the effects of a PAS mordant on color depth and colorfastness to light, laundering, and staining, as well as in relation to ASTM standards for textiles and apparel. Implications for natural dye artisans are discussed throughout. The chapter concludes with summary, recommendations, limitations, and conclusions.

Discussion of the Findings

Question A

Does a PAS mordant impact the dye concentration needed to achieve visually comparable dye depth of shade for walnut, Osage orange, and eastern redcedar?

Hypothesis One

H_1: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will require less dye concentration to achieve visually comparable depth of shade compared to wool yarn without a premordant.

The hypothesis was supported, in that yarns premordanted with 12% PAS and dyed with black walnut, Osage orange, and eastern redcedar required less dye than nonmordanted yarns to achieve a comparable depth of shade. The effect was greater for black walnut and eastern redcedar, requiring 100% and 50% more dyestuff respectively for nonmordanted yarns to obtain similar depth of shade compared with mordanted yarns.

Black Walnut

For black walnut, nonmordanted wool yarns required 200% owf of dyewood while mordanted yarns required 100% owf to achieve a standard depth of 1/12. A depth of 1/12 is the second to lightest on the AATCC's six step scale for depth of shade (see Figure 3.1, Tables 3.1 and 4.1) (AATCC, 2009). Dye depth appeared typical when compared to walnut bark from research by Richards and Tryl (2005). Richards and Tryl (2005) reported a ratio of 1:1 black walnut bark on wool yarn achieved the colors of yellow tan for alum mordanted (potassium aluminum sulfate at 25% owf) and light salmon tan for nonmordanted yarns (see Table 5.1). However, dye depth results did not match a previous study by the author that showed wool fabric mordanted with 12% PAS and dyed with black walnut bark was only slightly (with an AATCC color change rating of 4) darker when compared to nonmordanted wool fabric dyed with the same owf, suggesting that mordanted and nonmordanted had similar depth of shade (Doty & Haar, 2013). Mordanted wool yarn used in this study may have needed less owf for black walnut due to an improved bridge created between fiber and dye by the PAS mordant.

Osage Orange

Osage orange had more similar dyewood concentrations between mordant types with nonmordanted yarns at 50% owf and mordanted wool yarns at 40% owf for a standard depth of 1/1. A 1/1 depth is designated as the "standard depth" by AATCC's Evaluation Procedure 4-

2007 and is the second darkest hue on the six step scale (see Figure 3.1, Tables 3.1 and 4.1). These findings are refuted by research performed by Richards and Tryl (2005) which reported less saturated shades of yellow obtained by Osage orange bark at a ratio of 1:1 owf on wool yarn mordanted with 25% alum (potassium aluminum sulfate) as well as on nonmordanted yarn (see Table 5.1). The difference in depth findings could be due to the yarn structure used by Richards and Tryl (2005) or the dye obtained from the bark.

Overall, it appears that it takes much less Osage orange to achieve better saturation than either black walnut or eastern redcedar. Furthermore, it could be theorized that the lower and more similar concentrations for Osage orange between mordanted (40% owf) and nonmordanted (50% owf) yarns may be due to a high amount of tannin. While this study did not measure tannin amounts of the dyewoods, Kressman (1916) reported 10-11% tannin content in Osage orange sawmill waste from Texas. The higher amount of tannin may have allowed for improved absorption regardless of treatment.

Eastern Redcedar

For eastern redcedar, nonmordanted yarns required 150% owf of dyewood while mordanted yarns required 100% owf to reach a 1/25 depth of shade. A 1/25 depth is the lightest shade on AATCC's depth scale (see Figure 3.1, Tables 3.1 and 4.1). Eastern redcedar sawmill waste yielded a light warm tan color for mordanted wool yarns and a light mauve pink on nonmordanted. These colors appear to refute Richards and Tyrl (2005), which report both mordanted and nonmordanted wool yarns dyed at a ratio of 1:1 to be the same light peach color (see Table 5.1). Richards and Tyrl (2005) findings suggest that nonmordanted wool yarns did not need more dyestuff to achieve a similar depth of shade. Nevertheless, both the current study and the one performed by Richards and Tryl (2005) resulted in overall light color depths for eastern redcedar.

Table 5.1. Color Names and Munsell Color Notations from Richards and Tyrl (2005) for Mordanted and Nonmordanted Wool Yarn

Dye Type	Mordant Type	Color Names	Munsell Color Notations		
Black Walnut	Alum Mordant	Yellow Tan	10YR 6/6	10YR 7/6	2.5Y 8/6
	No Mordant	Light Salmon-Tan	7.5YR 7/6		
Osage Orange	Alum Mordant	Golden Yellow	2.5Y 8/8	2.5Y 8/10	
	No Mordant	Yellow Tan	2.5Y 8/6	10YR 6/6	10YR 7/6
Eastern Redcedar	Alum Mordant	Peach	7.5YR 8/4		
	No Mordant	Peach	7.5YR 8/4		

Note. Alum was identified as potassium aluminum sulfate and applied at 25% owf. It is not known why more than one color notation were included for black walnut mordanted and Osage orange.

Summary

The amount of dyewood it takes to achieve these color depths may be important to natural dye artisans who are looking for the benefit of the darkest color for owf. Timber mills, farmers, and ranchers may also find this dye concentration information useful when pricing and packaging mill waste to sell to artisans, making the dyewoods that produce dark color from low concentrations, such as Osage orange, more expensive than lesser yielding dyewoods. It is recommended that natural dyers looking to use less dyewood per owf for black walnut and eastern redcedar on wool yarn to use a PAS mordant. To summarize, a PAS mordant enhanced dye absorption requiring less dyestuff to achieve comparable color depth between mordant types.

Question B

How does a PAS mordant effect the overall color parameters of yarns dyed with black walnut, Osage orange, and eastern redcedar and subjected to tests of colorfastness to light and laundering?

Hypothesis Two

H_2: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly warmer (yellower or redder) when compared to wool yarn without a premordant before and after colorfastness to light testing.

Black Walnut

For yarns mordanted with PAS and dyed with black walnut the hypothesis was supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on $a*$ (greenness – redness) and $b*$ (blueness – yellowness). Descriptive statistics showed a trend towards redder and yellower for mordanted specimens compared to nonmordanted before and after colorfastness to light testing. The difference between mordanted and nonmordanted dye colors preceding lightfastness testing is supported by prior research conducted by the author that showed aluminum premordanted worsted wool gabardine fabric dyed with black walnut bark yellower and brighter when compared to nonmordanted wool (Doty & Haar, 2013). Mordanted yarns may be redder and yellower due to the PAS mordant, known for shifting natural dye colors towards warmer and brighter (Haar, et al., 2013; Doty & Haar, 2013; Cardon, 2007). Conversely, Richards and Tryl (2005) reported that mordanted wool yarn was a yellow tan while nonmordanted wool was a redder light salmon tan color (see Table 5.1). It should be noted that color change following exposure to light was not analyzed for either study, so it is not known how the colors changed after testing.

Even though black walnut mordanted specimens were statistically redder and yellower before and after colorfastness to light testing, the actual visual color change between mordanted and nonmordanted may not be enough to warrant the use of a mordant when considering for dye color, see Table 4.2 for color examples. When visually assessing the dye color both mordanted and nonmordanted yarns were a light brown color, with mordanted yarns appearing slightly brighter and warmer.

Osage Orange

For yarns mordanted with PAS and dyed with Osage orange the hypothesis was partially supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on a^* and b^*. For a^* descriptive statistics suggest that mordanted specimens were redder before colorfastness to light testing but became similar to nonmordanted specimens after exposure to light. For b^* descriptive statistics showed a trend towards yellower for mordanted specimens compared to nonmordanted before and after colorfastness to light testing. These color results appeared similar to research by Richards and Tryl (2005) that showed Osage orange bark dyed on alum mordanted wool yarn to be a golden yellow color and nonmordanted yarns to be a yellow tan color (see Table 5.1). Color change following lightfastness was not analyzed for Richards and Tryl (2005) study, so it is not known how the colors would behave after testing. When looking at the specimens there may not be enough change between mordanted and nonmordanted Osage orange dyed yarns before and after exposure to light to warrant the use of a mordant (see Table 4.2).

Eastern Redcedar

For yarns mordanted with PAS and dyed with eastern redcedar the hypothesis was partially supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on $a*$ and $b*$. For $a*$ descriptive statistics suggest that mordanted specimens were greener but became similar to nonmordanted specimens after lightfastness testing. Since nonmordanted yarns were pink in color they did not appear green and the statistical rating for $a*$ shows an absence of red for mordanted yarns along the red-green axes when compared to the light tan color of mordanted yarns. For $b*$ descriptive statistics showed a trend towards yellower for mordanted specimens compared to nonmordanted before and after colorfastness to light testing. These results differ from research by Richards and Tryl (2005) that showed eastern redcedar bark dyed on wool yarn to be a peach color for both mordanted and nonmordanted yarn, prior to any lightfastness testing (see Table 5.1). It is known that a PAS mordant may produce redder and yellower natural dye colors and the PAS mordant in association with the eastern redcedar millwaste may have caused the dye to turn towards a more yellow color (Haar,et al., 2013; Doty & Haar, 2013; Cardon, 2007).

Natural dyers should note that there was a clear visual difference between mordanted and nonmordanted specimens before exposure to light, with the mordanted yarns colored a light tan and nonmordanted yarns a light pink mauve (see Table 4.2); after exposure to light both specimens appeared to be a similar pale tan color (see Table 4.2). It is recommended for natural dyers to use a PAS mordant on wool yarns to shift eastern redcedar dye from light mauve for nonmordanted to a light tan for mordanted.

Summary

The PAS mordant influenced the color cast of wool yarns dyed with black walnut, Osage orange, and eastern redcedar prior to and following exposure to light testing. While all three mordanted dyewood yarns had warmer (yellower and/or redder) tones prior to light exposure compared with nonmordanted yarns, eastern redcedar was also cooler or greener prior to exposure. The warmer color cast supports previous research that use of PAS as a textile mordant changes the color of natural dyes towards warmer on wool and cotton with a variety of dyestuffs (Doty & Haar, 2013; Haar, et al., 2013). Understanding the influence of PAS on color cast following dyeing and exposure to light may be important to natural dyers looking for long term color results. Due to information from this study and previous research it is recommended for natural dyers wanting to achieve warmer color variations of these dyewoods on wool yarn to use PAS when dyeing with eastern redcedar and black walnut.

Hypothesis Three

H_3: Wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly warmer (yellower or redder) when compared to wool yarn without a premordant before and after tests for colorfastness to laundering.

Black Walnut

For yarns mordanted with PAS and dyed with black walnut the hypothesis was partially supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on *a** and b^*. For a^* descriptive statistics showed a trend towards redder for mordanted specimens compared to nonmordanted before and after tests for colorfastness to laundering. For b^* descriptive statistics suggested that mordanted specimens were yellower than nonmordanted specimens before tests for colorfastness to laundering, however the b^* coordinate became similar after washfastness testing occurred. This may be due to the PAS mordanted yarns retaining red hues during dyeing, which are later lost due to laundering. The color results obtained before washfastness testing are supported by prior research conducted by the author showing PAS premordanted wool fabric dyed with black walnut bark was yellower and brighter compared to nonmordanted wool (Doty & Haar, 2013). However these results are refuted by Richards and Tryl (2005) which reported that mordanted wool yarn was a yellow tan while nonmordanted wool was a redder light salmon tan color (see Table 5.1). Color change following washfastness was not analyzed for either study, so it is not known how the colors changed after laundering.

When reviewing the data for the use of these dyewoods for natural dyers, black walnut mordanted specimens were statistically redder and yellower before tests for laundering but lost some of its red hues during laundering. The actual visual color change between mordanted and nonmordanted may not be enough to warrant the use of a PAS mordant when considering for dye color, see Table 4.6.

Osage Orange

For yarns mordanted with PAS and dyed with Osage orange the hypothesis was partially supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on a^* and b^*. For a^* descriptive statistics suggested that mordanted specimens were redder than nonmordanted specimens before washfastness testing, however the a^* coordinates for mordanted and nonmordanted became similar after washfastness testing occurred. Possibly the PAS mordanted yarns retained red hues during dyeing, which were later lost during laundering. For b^* descriptive statistics showed a trend towards yellower for mordanted specimens compared to nonmordanted before and after washfastness testing. Color results prior to laundering appear similar to research by Richards and Tryl (2005) that showed Osage orange bark dyed on alum mordanted wool yarn to be a golden yellow color and nonmordanted yarns to be a yellow tan color (see Table 5.1). When reviewing this data for natural dyers, Osage orange mordanted specimens had a statistically significant amount of red and yellow compared to nonmordanted before laundering but some red hues were lost during laundering. Actual visual color change between mordanted and nonmordanted may again not be enough to warrant the use of a mordant when considering for dye color, see Table 4.6.

Eastern Redcedar

For yarns mordanted with PAS and dyed with eastern redcedar the hypothesis was partially supported. ANOVA tests suggested significance at the $p<.001$ for the effect of a mordant on b^* and no significance was detected for a^*. For b^* mordanted specimens were yellower than nonmordanted specimens before and after washfastness testing. The eastern redcedar dyed yarns resulted in a light tan color for mordanted and a light mauve pink for nonmordanted before and after washfastness testing. This differs from research by Richards and Tryl (2005) that showed eastern redcedar bark dyed on wool yarn to be a peach color for both mordanted and nonmordanted prior to washfastness testing (see Table 5.1). When accessing for natural dyers, eastern redcedar mordanted yarns had a statistically significant amount of yellow when compared to nonmordanted before and after washfastness testing. It should be noted that there was a definite color difference between mordanted and nonmordanted specimens before laundering and after laundering, with the mordanted a light tan color and the nonmordanted yarns with a light pink mauve color (see Table 4.2). Both colors remained similar to their prewashed shades after laundering.

Summary

The color composition for PAS mordanted yarns dyed with black walnut, Osage orange, and eastern redcedar were warmer (redder/ yellower) than nonmordanted yarns, confirming previous research that the use of PAS mordant changes the color of natural dyes towards warmer on wool and cotton with a variety of dyestuffs (Doty & Haar, 2013; Haar, et al., 2013). These findings may be important to natural dyers who want to know if a PAS mordant will necessarily maintain an intended color after washing. To achieve warmer colored variations of these dyes on wool, especially eastern redcedar, the researcher recommends for natural dyers to use a PAS mordant.

Summary for Question B

When comparing mordanted and nonmordanted wool yarns dyed with black walnut, Osage orange, and eastern redcedar, exposure to light influenced color differently than laundering. Statistically, mordanted black walnut was redder and yellower than nonmordanted before and after exposure to light; mordanted yarns were also redder and yellower than nonmordanted before exposure to laundering, however they became similar for b^* (blue-yellow) after laundering. When looking at the specimens a PAS mordant did not appear to make much of a visual difference between the standards and exposed specimens. Thus, even though there were significant effects, the subtle visual color difference may not warrant the use of a PAS mordant.

For Osage orange, statistics showed mordanted specimens were redder and yellower than nonmordanted specimens before exposure to light and laundering, however mordanted and nonmordanted yarns became similar after exposure to light and laundering for a^* (red-green).

When visually inspecting the specimens, mordanted and laundered Osage orange was a brighter and clearer yellow when compared to nonmordanted. It is recommended for natural dyers wanting to maintain a bright yellow to use a PAS mordant on wool yarns.

For eastern redcedar, the color changes between mordanted and nonmordanted were more complex. Statistics showed that mordanted yarns had less red and more yellow than nonmordanted yarns, however mordanted and nonmordanted yarns became similar for *a** (redder-greener) after lightfastness testing. For laundering, mordanted yarns were yellower than nonmordanted yarns before and after testing. Visual inspection supports the statistics as mordanted yarns have a slight yellow cast (light tan color) and the nonmordanted yarns a slight red cast (light pink mauve color). It is recommended for natural dyers to use a PAS mordant if they want to shift the color of eastern redcedar from pink to tan. However the pink color may fade towards tan due to exposure to light, so dyers should protect any project they want to remain pink from exposure.

Question C

Does a PAS mordant improve colorfastness properties of black walnut, Osage orange, and eastern redcedar on wool yarn when subjected to tests of colorfastness to light, laundering, and staining?

Hypothesis Four

H_4: On tests for colorfastness to light, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly different for ΔE and have less color change compared to wool yarn without a premordant.

Black Walnut

For yarns mordanted with PAS and dyed with black walnut the hypothesis was only partially supported. Two-sample t-tests performed on Δ E showed significance at a low level ($p<0.1$) that mordanted yarn exposed to light had less color change than exposed nonmordanted yarn. Conversely, average AATCC Gray Scale for Color Change ratings indicated nonmordanted specimens had less color change than mordanted ones (4.5 and 4.25 respectively). This difference between the colorimeter data and the gray scale rating is most likely due to the colorimeter reading color difference not noticeable by the human eye in gray scale assessment.

The finding that a PAS mordant did not have a highly significant effect on colorfastness to light was unexpected and contradicts a previous study that found black walnut bark dyed on aluminum mordanted wool fabric had a marked improvement over nonmordanted wool fabric with Gray Scale ratings of 4-5 for mordanted and 3-4 for nonmordanted (Doty & Haar, 2013).

Overall, black walnut had good lightfastness, this may be due to chemical compounds that are only found in the Juglandaceae family; specifically the quinone compound juglone and the hydrolysoble tannin compound juglandaceae (Cardon, 2007; Baker, 1958). These walnut specific compounds may be in part responsible for black walnut's colorfastness to light, regardless of mordant treatment. When reviewing the data for natural dye artisans, black walnut dyed mordanted yarns had statistical significance towards less color change than nonmordanted. This however was at a low significance level ($p<0.1$) and natural dyers can be advised that visually there was little color difference between mordanted and nonmordanted.

Osage Orange

For yarns mordanted with PAS and dyed with Osage orange the hypothesis was not supported. Two-sample t-tests suggested no significant effect between the Δ E of mordanted and nonmordanted specimens subjected to lightfastness tests. AATCC Gray Scale for Color Change ratings corroborated this finding with evaluators finding no difference between mordant types and assigning both a rating of 2.75 (considerable to noticeable change). It is important to note that flavonoid compounds found in Osage orange dyes are claimed to have poor lightfastness and the research findings contradicts the suggestion that the colorfastness of flavonoid dyes may be enhanced with the use of a metallic mordant (Richards & Tyrl, 2005; Cardon, 2007; Cristea & Vilarem, 2006).

Furthermore, while mordanted and nonmordanted Osage orange dyed specimens had similar change (not significant) compared to each other, they also had considerable to noticeable color change that made the dyed color darker when exposed to light, instead of fading, which often occurs. This is a meaningful finding since most color change from exposure to light results in color loss or fading, however for Osage orange the reverse was true. This is inconsistent with Hasam's (2013) findings that Osage orange extract became lighter, rather than darker, on wool and silk fabric exposed to light. Many variables differ between the previous study and this one, such as textile structure and premade extract versus the dye being extracted from mill waste. However, tannin is known to darken when exposed to light and the turning of Osage orange to a darker brown color seems to closely parallel the color change associated with oxidizing or the shifting process that turns solid Osage orange wood products from bright golden yellow to brown (Meier, 2014; Richards & Tyrl, 2005; Tull, 1999). For natural dyers the finding that Osage orange mill waste on wool yarn may darken when exposed to light is important and dyers may want the darker caramel brown. Instead of discouraging the exposure of Osage orange dyed textile to sunlight, artisans could instead expose parts or the entire textile to the sun to achieve darker tones. It is also important to note that Osage orange dyed yarns did not indicate a significant effect between mordanted and nonmordanted and visually the sample appeared similar after colorfastness to light testing.

Eastern Redcedar

For yarns mordanted with PAS and dyed with eastern redcedar the hypothesis was partially supported. Two-sample t-tests showed significance at a low level ($p<0.1$) that mordanted yarns showed less color change after exposure to light than nonmordanted yarns. However, AATCC Gray Scale for Color Change ratings showed no difference between mordanted and nonmordanted, with a rating 4.25 or slight change for both. As with black walnut, perhaps the colorimeter reading was able to detect color differences that the human eye was not able to distinguish. The overall minimal loss of color after exposure to light was expected for eastern red cedar as light colored dyes often have high ratings, since there is less color to be lost. A visual assessment of the samples showed that mordanted and nonmordanted samples exposed to light resulted in similar colors and the difference between them would mean little to natural dye artisans.

Summary

Overall, the data suggests that PAS mordanted wool yarn dyed with black walnut, Osage orange, and eastern redcedar may minimally assist in lightfastness when comparing mordanted and nonmordanted specimens dyed at a comparable depth of shade. These findings are important for natural dyers looking to avoid a metallic mordant, in that lightfastness is only slightly improved using PAS at 12% owf for wool yarns dyed with black walnut and eastern redcedar, with no improvement for Osage orange.

Hypothesis Five

H_5: On tests for colorfastness to laundering, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will be significantly different for ΔE and have less color change compared to wool yarn without a premordant.

Black Walnut

For yarns premordanted with PAS and dyed with black walnut, the hypothesis was not supported. Two-sample t-test for ΔE suggest that nonmordanted specimens had less color change than mordanted at the $p<.001$ level after exposure to laundering. Gray Scale ratings also indicate nonmordanted yarns had less color change than mordanted (4.44 and 4.06 respectively), however the difference was minimal. It was unexpected for nonmordanted yarn to perform better than mordanted, however the mordanted yarns dyed with black walnut may have allowed more yellow hues (located in the b^* coordinate) to adhere to the yarns, later washing away during testing and affecting overall color change (see Table 4.3). Notably, the findings that nonmordanted specimens had less color change than mordanted contradicts a previous study that black walnut bark on nonmordanted wool fabric had an average AATCC Gray Scale rating of 3-4 when compared to its standard, while mordanted had an average of 4-5 when compared to its standard (Doty & Haar, 2013).

While the findings are statistically significant for nonmordanted black walnut dyed yarn to have improved colorfastness to laundering compared to mordanted yarns, the visual difference between mordant types was similar with ratings of only a slight change. The overall good washfastness ratings for mordanted and nonmordanted yarns dyed with black walnut may be due to special types of hydrolysoble tannin and quinone compounds found in the walnut tree family, allowing the dye to be fast regardless of mordant treatment. When reviewing the data for natural dye artisans, black walnut dyed nonmordanted yarns had statistical significance towards less color change than mordanted. However, when looking at the specimens there was most likely not enough overall color difference between mordanted and nonmordanted for a natural dyer to notice.

Osage Orange

For yarns premordanted with PAS and dyed with Osage orange the hypothesis was accepted. Two-sample t-test for Δ E showed significance at the $p<.05$ confidence level that mordanted specimens had less overall color change than nonmordanted after exposure to laundering. Gray scale ratings support this finding with mordanted yarns average rating of 4.78 or no change, while nonmordanted yarns had an average rating of 3.67 or noticeable to slight change. Notable is the suggestion that flavonoid compounds found in Osage orange dyes may have poor colorfastness, however they may be enhanced with the use of a metallic mordant (Richards & Tyrl, 2005; Cardon, 2007; Cristea & Vilarem, 2006). In a previous study on the colorfastness to laundering of Osage orange on alum premordanted flax, the AATCC average gray scale rating was 1-2 or considerable to severe change after washfastness testing (Sarkar & Seal, 2003). Since the Osage orange dyed wool yarn in this study performed considerably well with and without the PAS mordant, it could attest to wool's affinity towards natural dye. The results of the study at hand suggest that a PAS mordant improves colorfastness to laundering for Osage orange dyed wool yarn. When visually assessing the specimens for natural dyers, mordanted yarns did appear brighter and more yellow than nonmordanted and it is advised for natural dyers to use a mordant with Osage orange to maintain a consistent color after washing.

Eastern Redcedar

For yarns premordanted with PAS and dyed with eastern redcedar the hypothesis was not supported. Two-sample t-tests performed on Δ E found significance at a low level ($p<0.1$) that nonmordanted yarns had less color change than mordanted after exposure to laundering. AATCC Gray Scale for Color Change had similar ratings of 4.25 for nonmordanted and 3.72 for mordanted yarns, supporting the lower significance level of the two-sample t-tests. While the difference between mordanted and nonmordanted is slight it might seem counter intuitive that a fiber with both a tannin and aluminum mordant would have more color change than a yarn with just the tannin found in eastern redcedar. However, the PAS mordanted yarns dyed with eastern redcedar may have allowed more green hues (found in the a^* coordinate) to adhere to the yarns, later washing away during testing and affecting overall color change (see Table 4.3). When looking at the specimens mordanted and nonmordanted yarns appeared to retain their original colors of light tan for mordanted and light mauve pink for nonmordanted. Natural dyers are advised to use a PAS mordant to sift the color of mordanted eastern redcedar towards tan and that it will remain after laundering. However, remember that nonmordanted eastern redcedar dyed wool yarns will shift towards tan if exposed to sunlight.

Summary

Overall, the data suggests that a PAS mordant at 12% owf improves colorfastness to laundering of wool yarn dyed with Osage orange. Conversely, black walnut and eastern redcedar nonmordanted yarns had less color change compared to mordanted yarns. This may be due to the mordant allowing some color hues to adhere to the yarn during dyeing which were subsequently washed away during testing. For natural dyers looking to avoid a metallic mordant these findings are noteworthy, in that washfastness may remain similar for wool yarns dyed with black walnut and eastern redcedar regardless of a PAS mordant. It is recommended to use a PAS mordant to improve the colorfastness to laundering of Osage orange, however a PAS mordant may not be needed for black walnut and eastern redcedar on wool yarn.

Hypothesis Six

H_6: On tests for colorfastness to staining, wool yarn premordanted with PAS and dyed with black walnut, Osage orange, and eastern redcedar dyewoods will have similar color change ratings compared to wool yarn without a premordant.

For yarns dyed with black walnut, Osage orange, and eastern redcedar the hypothesis was supported. Average gray scale for staining ratings were similar between mordant types for all three dyes. Mordanted and nonmordanted black walnut dyed specimens had comparable mean gray scale staining ratings of 4.81 and 4.6 respectively. The wool strip was rated the same (4.83) for both mordant types. While Osage orange dyed specimens were also similar, the mean gray scale for nonmordanted was marginally higher than mordanted (4.25 and 4.01 respectively). Of note, Osage orange staining on the wool strip had the most staining variation with nonmordanted rating 4.5 and mordanted rating 3.67. Eastern red cedar had minimal to no staining across the fiber strips for both mordant types with mean gray scale for staining ratings of 4.96 (nonmordanted) and 4.95 (mordanted). The lack of staining was expected of such a light colored dye. These findings are similar to previous studies that found black walnut bark dyed wool fabric and Osage orange dyed flax fabric had little to no staining when washed (Doty & Haar, 2013; Sarkar & Seal, 2003). This could be important for dyers wanting to wash their dyed yarns with other textiles, however it is recommended to hand wash wool yarns separately, regardless of dye type, in lukewarm water to avoid agitation and felting.

It is important to note that the researchers formulated the hypothesis that mordanted and nonmordanted specimens would have similar color change ratings when testing for staining around assumptions from prior research experience and from previous research studies showing natural dyes from trees to have minimal staining (Doty & Haar, 2013; Haar, et al., 2013; Sharma & Grover, 2011). This expectation of similarly high staining results regardless of mordant treatment may not be due to the dye's ability to stay on the intended yarn but because of dye's inherently limited ability to adhere to textiles.

Question C

Will gray scale ratings for wool yarns dyed with black walnut, Osage orange, and eastern redcedar meet minimum American Society of Testing and Materials (ASTM) standards, requiring a light grade of 4, a laundering grade of 4, and a staining grade of 3?

Hypothesis Seven

H_7: Minimum ASTM standards for colorfastness to light, laundering, and staining for apparel and home furnishing use will be met with wool yarns both premordanted with PAS and unmordanted and dyed with black walnut, Osage orange, and eastern redcedar dyewoods.

This hypothesis was partially accepted when assessing for a light grade of 4, a laundering grade of 3, and a staining grade of 3 (see Table 4.15). All mordanted and nonmordanted yarns in this study, except Osage orange exposed to light, passed minimum Gray Scale ratings for light, laundering, and staining. For Osage orange, both mordanted and nonmordanted yarns exposed to colorfastness to light testing failed to meet the ASTM standard. Interestingly, Osage orange dyed yarns did not fail due to a lightening of color, as many synthetically and naturally dyed yarns do, instead failing due to a significant darkening of color, from a bright yellow to a medium warm brown. Regardless of mordant type, wool yarn dyed with the selected dyewoods can be used in a wide selection of woven or knitted apparel and home furnishings (see Table 4.15).

Summary and Conclusion

The overarching aims of this research was to gain knowledge of black walnut, Osage orange, and eastern redcedar dyes for incorporation into handwoven textiles as part of an art garment display and to increase the knowledge of the application and colorfastness properties of local Kansas dyewoods for use as a sustainable alternative for textile coloration by natural dye artisans who use yarn as part of their craft. Specifically, this study examined the effect of PAS on color depth and color change. The following are summaries for each dyewood.

Black Walnut

The absorption of black walnut dye was improved with the use of a PAS mordant, from 200% owf for nonmordanted wool yarn compared to 100% owf for mordanted to achieve a similar standard depth. When comparing the nonmordanted to mordanted wool yarn for color differences the mordanted yarns were statistically significant towards yellower and/or redder compared to nonmordanted yarns before and after colorfastness to light and laundering tests. When exposed to light, mordanted black walnut dyed yarns had less color change according to two sample t-tests; conversely, when exposed to laundering, nonmordanted yarns had less color change. For staining tests there was little change between nonmordanted and mordanted and staining was minimal. Finally, black walnut dyed yarns met the ASTM Standards for colorfastness to light, laundering, and staining for selected woven and knitted apparel and home furnishings.

Overall, it is recommended to use a PAS mordant with black walnut dyed yarn due to its improved dye absorption requiring less dyewood owf and some improvement to colorfastness to light. A PAS mordant will shift the color towards redder and yellower, however the slight change may not be noticeable to the natural dyer. A PAS mordant did not enhance colorfastness to laundering or staining.

Osage Orange

The absorption of Osage orange was slightly improved with the use of a PAS mordant, from 50% owf for nonmordanted wool yarn compared to 40% owf for mordanted to achieve a similar standard depth. When comparing the nonmordanted to mordanted wool yarn for color differences the mordanted yarns were statistically significant towards redder before and after colorfastness to light and laundering tests. However, when comparing between mordanted and nonmordanted for visual difference before and after exposure to light, there was minimal difference between the two. For laundering the mordanted samples appeared to be a brighter and clearer yellow following testing.

When exposed to light, Osage orange dyed yarns preformed similarly between mordanted and nonmordanted specimens. However, both nonmordanted and mordanted yarns went from a bright golden yellow to a warm brown after exposure to light. When exposed to laundering, mordanted Osage orange dyed yarns had less color change over nonmordanted. For tests for staining there was little difference between nonmordanted and mordanted. Osage orange mordanted and nonmordanted yarns met ASTM apparel and home furnishing standards for colorfastness to laundering and staining, while barely missing the expectation for lightfastness

Overall, a PAS mordant improved dye absorption and colorfastness to laundering, and maintained a bright yellow color, but had no impact on lightfastness or staining when using Osage orange millwaste on wool yarn. The use of a PAS mordant shifted the color towards red, however this shift may be too subtle for a dyer to notice. Regardless of mordant type, exposure to light darkens the color.

Eastern Redcedar

The absorption of eastern redcedar was improved with the use of a PAS mordant, from 150% owf for nonmordanted to 100% owf for yarn with a mordant. When looking at color differences mordanted yarns were statistically significant towards greener and/ or yellower for mordanted yarns compared to nonmordanted. The visual difference between nonmordanted and mordanted dyed yarns was noticeable, with nonmordanted yarns resulting in light pink mauve color and mordanted yarns colored a light tan.

When exposed to light, two sample t-test indicated mordanted eastern redcedar dyed yarns had a slight improvement to color change compared to nonmordanted. In contrast, when exposed to laundering, nonmordanted had less color change. On tests for staining there was little difference between mordanted and nonmordanted yarns dyed with eastern redcedar. When comparing to ASTM Standards both mordanted and nonmordanted yarns met standards for colorfastness to light, laundering, and staining for selected apparel and home furnishings.

Overall, a PAS mordant is recommended for eastern red cedar to reduce overall amount of dyestuff needed and improve colorfastness to light. A PAS mordant will shift the color towards yellow. A PAS mordant did not improve colorfastness to laundering or staining; however, color change was minimal regardless of mordant type in regards to laundering.

Selling Mill Waste for Profit.

For mill owners, farmers, or ranchers it is advised that the mill waste of all three trees may make a viable, value added commodity that may otherwise be disposed. While eastern redcedar does give an attractive tan or mauve color, it is a light color that dyers may not have as much interest in using. Black walnut and Osage orange give darker values for owf and are familiar to artisans as both are already available on the market; thus competitive pricing would need to be established. However if mill waste is already being discarded, mill owners may find an advantage in recuperating some of their potential gains.

Limitations

Black walnut, Osage orange, and eastern redcedar mill waste was sourced from a privately owned sawmill and it cannot be determined as to the year or season when the trees were felled or milled; factors that could influence dye properties of the mill waste. Tannin content for the dyewoods was not determined in this study. As tannin influences dye properties, knowing the tannin concentration of each dyewood may have aided interpretation of the results. In addition, documenting alkalinity and acidity by taking pH readings of the dye solutions may have assisted with understanding the results and with the controlling the absorption of the PAS mordant. Furthermore, mordant and dye baths were not tested for amount of PAS and dyestuff that may have been left after processing and not bonded to the wool fibers. The amount of remaining alum found in the solutions could be important for environmental reasons, as one of the main functions of this study is to limit the amount of effluents into the environment, even seemingly harmless ones. Knowing the amount of dyestuff remaining would aid in the understanding of dye absorption and recommended amounts of dye.

Recommendations for Further Study

The overall purpose of this study was to compare colorfastness properties between PAS mordanted and nonmordanted wool yarns, dyed with Kansas black walnut, Osage orange, and eastern redcedar to create a better understanding of sawdust waste as a colorant. For this study colorfastness testing included light, laundering, and staining. However, a better context could be built around black walnut, Osage orange, and eastern redcedar dye by also testing for perspiration and crocking. Taking pH readings is recommended to document the acidity and alkalinity of the dyebaths.

Due to a lack of knowledge of tannin amounts found in mill waste, future research could include surveying the tannin content of different sources of the same dye and then evaluating colorfastness compared to tannin amounts. Future research could also include the effects of tannin rich dyes on cellulosic fibers, since tannins have a natural affinity to plant fibers (Cardon, 2007). Furthermore, since many natural dye artisans dye with fabric, further studies could be conducted with wool, cotton, or silk in various fabric structures. Lastly, this study did not use cream of tartar during premordanting but it has been suggested that tartar could improve the brightness of dyes on wool and further exploration may yield improved dye results (Hasam, 2013).

Conclusions

In summary, this research compared 12% owf PAS mordanted and nonmordanted wool yarns dyed with black walnut, Osage orange, and eastern redcedar and added to the knowledge of Kansas mill waste as a possible substantive dye option for home artisans. Findings indicated that dye absorption was improved with the use of a PAS mordant, especially for black walnut and eastern redcedar. Thus, much less dye concentration is needed when using a PAS mordant. For yarns premordanted with PAS the dyewoods became yellower. A PAS mordant slightly improved colorfastness to light for black walnut and eastern redcedar, but did not influence Osage orange. Colorfastness to laundering was slightly improved with PAS for Osage orange, while black walnut and eastern red cedar had slightly less color change without the mordant.

In practice, it is recommended for natural dye artisans to use a PAS mordant to improve dye absorption of black walnut and eastern redcedar. For dyers wanting to manipulate the resulting color of a dye with an aluminum mordant, eastern redcedar had the most noticeable color change from a red cast (light pink mauve) to a yellow cast (light tan) with the mordant. However, the color difference was less noticeable after exposure to light. For dyers wanting to manipulate color with exposure to light, Osage orange went from a bright golden yellow to a warm brown after exposure. If a natural dyer is looking for a dye that will maintain a constant color over time, black walnut performed well and did not drastically change color when exposed to light or laundering. It can be concluded that natural dyers can be confident that these dyes on wool yarn will hold up to ASTM standard for light, laundering, and staining, with the exception of Osage orange exposed to light. Regardless of mordant, Osage orange turned darker when exposed to light testing, changing from bright golden yellow to medium caramel brown.

It is the intension of this information that increased knowledge of natural dyes from sawmill waste will make them more desirable to natural dyers and in turn a valuable commodity to farmers and ranchers who would otherwise find little use for their mill waste.

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Appendix A- Budget

Table A.1

Items for the Kansas State University Graduate School

Item	Price
ETDR Submission Fee	$100.00
Copyright Fee	$55.00
Total	$155.00

Table A.2

Items Covered by the Agriculture Experiment Station

Category	Item	Description	Source	Price	Quantity	Total Estimate
Aid	Potassium Aluminum Sulfate	16 oz	Hill Creek Fiber Studio	$6.40	1	$6.40
Aid	Orvus Paste	7.5 lbs	Dover Saddlery	$24.99	1	$24.99
Yarn	2/30 Worsted Wool yarn	10 g each	Testfabrics, Inc	$0.61	100	$61.00
Colorfast-ness	Lightfast-ness testing	Dyed specimens	Professional Testing Labs	$10 per sample	18	$569.00
					Total	$661.39

Appendix B- Timeline

- May 9th 2014
    - Presentation of Thesis Proposal
- June – August 2014
    - Perform scour, pre-mordant, and dye samples
    - Conduct colorfastness to washing testing
    - Send out sample for colorfastness to light testing
- March 15th –May 1st
    - Gather and interpret color results
    - Write-up results
- May 2nd –July 1st
    - Revise and edit thesis
- July 25th 2014
    - Thesis to committee members
- July 1st
    - Complete KSIS Graduation Application
- August 10th
    - Defense of Thesis
- August 14th
    - Submit Final Examination Ballot
    - ETDR Submission to KREX
    - Exit Survey

Appendix C- Manuscript of Research Appropriate for Publication

- Clothing and Textile Research Journal
- Textile Research Journal
- Textile Review
- Turkey Red Journal
- The Indian Textile Journal
- Indian Journal of Natural Products and Resources
- Clourage
- Dyes and Pigments
- International Journal of Agricultural Sustainability