THESIS

COLORFASTNESS PROPERTIES OF PERSIMMON DYE ON COTTON AND WOOL SUBSTRATES

Submitted by

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ABSTRACT

COMPARING PERSIMMON DYE’S COLORFASTNESS WHEN APPLIED TO WOOL AND COTTON

Persimmon dye is a natural dye that imparts unique properties, including fungal resistance and water resistance. This study investigated persimmon dye’s performance on cotton and wool fabric. Color strength and appearance of dyed cotton and wool fabrics at various dyeing conditions (mordanting order, dye concentration, and dyeing time length) were evaluated. Dyed fabrics had high color strength when using a mordant. Color strength on dyed cotton and wool increased with increased dye concentration and dyeing time. In this study, post-mordanting, 200% dye concentration, and 60 minutes dyeing length yielded the highest color strength for cotton and wool. These samples were then assessed using AATCC laundering, perspiration, and crocking colorfastness tests. Cotton samples received a 2-3 to 4 shade change rating, while wool received a 3 to 4 depending on the test. Cotton and wool samples received a 4 to 5 staining rating except in crocking, where they received a 2-3 and 3, respectively. FTIR analysis showed that the persimmon dye formed weak bonds on both fabrics, resulting in minimal chemical changes. The results suggest that natural persimmon dye can provide good colorfastness and minimal chemical changes on wool and cotton. The comparison between dyed cotton and wool suggests wool fabric is better suited for persimmon dye application than cotton because of its slightly better colorfastness ratings and significantly higher color strength.

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Chapter 1: Introduction

Natural dyes were the primary textile dyeing materials before the advent of synthetic dyes and pigments. Natural dye use has been widely reported throughout history, such as humans living in present-day Iraq in 2000 BC using indigo dye to paint and dye clothing (Mansour, Ezzili, & Farouk, 2013). Natural dyes are usually extracted from roots, stems, berries, leaves, and flowers of a variety of plants. In addition to plant parts, some insects, like cochineal, are used (Arora, Rastogi, Gupta, & Gulrajani, 2012). Less commonly used natural dyes include fungi, mold, or algae. Interest in these less commonly used natural dyes began because of plant-based natural dyes' high cost (Parthiban & Thilagavathi, 2012). Natural dyes are almost exclusively applied to natural fibers in the literature, with cotton being the most commonly tested fiber. Wool, silk, and flax are the other most commonly used fibers in the surveyed literature (Arora et al., 2012; Deo & Desai, 1999; Mansour et al., 2013; Parthiban & Thilagavathi, 2012; Sarkar & Seal, 2003; Yi & Cho, 2007). It is well known that natural dyes are a safe, eco-friendly alternative to synthetic dyes (Hill, 1997; Mirjalili, Nazarpoor, & Karimi, 2011; Sarkar & Seal, 2003). Despite their relative safety, there are drawbacks to using natural dyes. Dawson (2008) suggests natural dyes are less viable than synthetic dyes because natural dyes are generally less colorfast than their synthetic counterparts, reducing their demand in a marketplace that desires excellent colorfastness. Despite the relative safety of natural dyes, not all natural dyes are completely safe. Elderberry and safflower may have some detectable mutagenic effects, while carmine dust can cause asthma in some rare cases (Hill, 1997). Additionally, it is suggested that combining natural dyes with mordants can be very damaging to the environment due to excess metal ion production (Asplund, 1998). However, natural dyes' current drawbacks could indicate that more research is needed to provide a good understanding of natural dyes and improve natural dyes' performance. Research in this area is therefore warranted and could lend support for future natural dye use.

Persimmon dye is a naturally-occurring dye made from persimmon juice. Based on initial information gathering, relatively little research has been completed on persimmon dye. The literature review synthesizes and assesses knowledge on persimmon dye's characteristics, standard dyeing procedures using persimmon dye, and dyeing properties that are known. Because relatively little research has been conducted, general natural dyeing procedures were reviewed as well. Persimmon dye properties and characteristics were also compared to knowledge about other common natural dyes. As natural dyes are used on a wide range of natural fibers, persimmon dye's use and performance on various fibers in prior research was investigated. Persimmon dye is specifically important to current research because preliminary research indicates that it possesses unique traits, like water- and UV-resistance, that make its performance on fabrics superior to other natural dyes for some applications (Yi, Kim, and Park, 2007).

The literature review chapter discusses dyeing procedures, quantitative evaluation, experimental methods, and persimmon dye performance compared to other natural dyes. Previous research indicates variation within natural dyeing techniques. The quantitative evaluation section discusses the methods used to evaluate dye coloration and test results quantitatively. The experimental methods section focuses on the American Association of Textile Chemists and Colorists (AATCC) standard tests used in current research. Dyeing performance is discussed as functions of coloration, dyeing procedure, and standard testing results.

The materials and methods chapter discusses the materials used and experiments performed in the current study. The materials section focuses on fabrics chosen to dye, persimmon dye sourcing, and mordant selection. Dyeing procedure is then discussed, followed by a discussion of data collection and analysis methods.

Chapter 2: Literature Review

The following chapter discusses various natural dyeing parameters, including dye concentration, mordant, treatment time, and pH. Surprisingly, these variables vary widely in literature. General natural dyeing studies were reviewed instead of only studies using persimmon dye because very few studies investigated persimmon dye.

Dye concentration. Overall, natural dye concentration varied widely depending on dye and fabric used. Deo and Desai (1999) and Yi and Cho (2007) added dye to the dye bath based on their fabric samples' weight. Deo and Desai (1999) tested black tea-dyed samples using 2% and 5% dye concentrations, meaning dye equal to 2% or 5% of the fabrics' weight was added to the dye bath. Yi and Cho (2007) tested 17 dyes, including persimmon dye, arecae semen, and cochineal, and varied their dyeing concentration from 75% to 300%. Based on these two studies, black tea was exceptionally effective because it only needed 5% concentration to produce color strength comparable to other natural dyes. Yi and Cho (2007)'s wide range of dye concentrations suggests natural dye concentration varies substantially between dyes. When testing persimmon dye, Yi and Cho (2007) did not use a dye bath with a specific dye concentration but instead used sunlight exposure to strengthen color, reinforcing the idea that optimal dyeing techniques for natural dyes greatly vary. Yi and Cho (2007) did not detail how the persimmon samples were initially dyed. In Yi, Kim, and Park's (2007) paper, cotton samples were dyed by dipping them into a bath consisting only of persimmon juice and letting them dry.

Mordant. Mordants used for textile dyeing are substances used to increase natural dyes' affinity for fabrics and colorfastness by chemically binding together (Tiedemann & Yang, 1995). Mordants are generally inorganic elements, like iron or aluminum (Popoola, 2000). Most experimental papers dealing with natural dyes employed aluminum, iron, or copper as mordants (Deo & Desai, 1999; Popoola, 2000; Sarkar & Seal, 2003; Yi & Cho, 2007). Wide variation in mordant concentration occurred within the studies identified. Mordant concentration was as low as 1% on weight of fabric (Yi & Cho, 2007) and as high as 10% on weight of fabric (Sarkar & Seal, 2003). Reasoning behind choosing specific mordant concentrations was not explained in any of the four papers mentioned above. The dyeing stage when the mordants are added also affects the dye's color strength. Sarkar and Seal (2003) found statistically significant differences in color strength and color appearance between pre-mordanted and meta-mordanted fabric samples. In the pre-mordanting process, mordant and sample fabric were added to water and then the solution was heated and kept boiling for 45 minutes, whereas in the meta-mordanting process, the mordant was simply added to the dye bath without boiling treatment. On the other hand, despite mordant use being common for natural dyes, it appears some natural dyes, including persimmon dye, do not require mordanting. Yi and Cho (2007) and Yi et al. (2007) did not use a mordant but instead used sun exposure to enhance persimmon dye's color. Persimmons naturally contain tannin (Park, C.H. Kim, Suh, D.S. Kim, & Hwang, 2005), and Bechtold, Mahmud-Ali, and Mussak (2007) used tannin as a mordant when dyeing fabric with grapes, further suggesting that mordants are not required for adequate color strength when using persimmon dye. Because the dyeing method was not well-established for persimmon dye, there is a strong need to investigate optimal dyeing procedures for persimmon dye use on textiles.

Dyeing length. In previous natural dye studies, dyeing length stayed consistent for all natural dyes except persimmon dye. Deo and Desai (1999), Sarkar and Seal (2003), and Yi and Cho (2007) left test samples in the dye bath for 60 minutes for natural dyes other than persimmon dye. For persimmon dye, Yi et al. (2007) and Yi and Cho (2007) simply immersed test fabric in persimmon juice and removed it shortly thereafter.

Dyeing temperature. In previous studies, dyeing temperature varied within a small range. Dyeing with dyes other than persimmon dye was carried out at 100 °C by Deo and Desai (1999) and Sarkar & Seal (2003). Bechtold, et al. (2007) dyed their fabric samples at 95 °C. Yi and Cho (2007) dyed their fabric samples at 60 °C for all colorants except persimmon dye. In Popoola (2000)'s study, dyeing was carried out at 80 °C using distilled alcohol instead of water in the dye bath. In Tiedemann and Yang (1995)'s study, dyeing using cochineal and madder roots were both carried out at 90 °C. Arora et al. (2012) discovered that a dye bath created from Ratanjot root contained the most color between 70 °C and 80 °C. Above that temperature range, the dye bath lost up to 100% of its color at 130 °C depending on pH. Yi and Cho (2007)'s study was the only natural dyeing study reviewed that did not carry out dyeing at or near 100 °C (boiling) when using water in the dye bath. For persimmon dye, Yi et al. (2007) and Yi and Cho (2007) did not report a specific dyeing temperature. Similar to mordanting, none of the papers referenced explained their choice of dyeing temperature.

Dye bath pH. Dye bath pH can have significant effects on color strength (Deo & Desai, 1999). Of the literature reviewed, five studies investigated pH's impact on color strength. All studies used similar procedures to modify the dye bath pH, although some variation in acids/alkalis added occurred. In Deo and Desai (1999)'s study, dyeing was carried out at five different pH levels. The black tea dye used yielded roughly double color strength at pH 3 compared to pH 8 for cotton. For jute, the black tea dye yielded roughly four times the color strength at pH 3 compared to pH 8 (Figure 1)(Deo & Desai, 1999). In Parthiban and Thilagavathi (2012)'s study, it was discovered that a lower pH was optimal when dyeing with Thermomyces

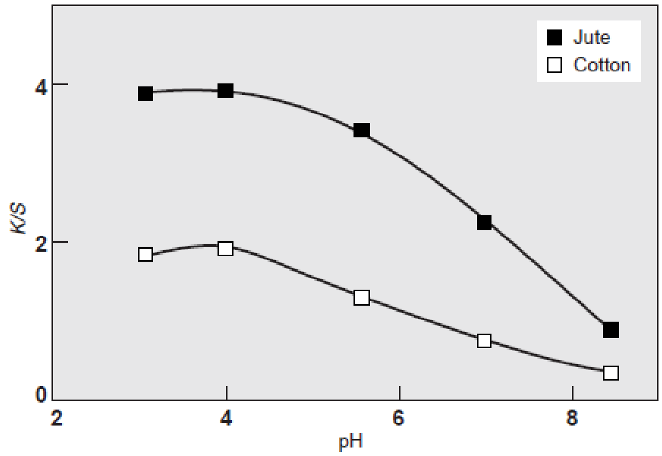


Figure 1. Dye bath pH's influence on cotton and jute dyeing using black tea dye (Deo & Desai, 1999).

(a fungus) and color strength peaked at pH 3. Similarly to the previous studies mentioned, Mansour et al. (2013) found that low pH increased color strength when using Black Grenache leaf dye. At pH 1 and 2, color strength was relatively close. Once pH increased above 2, however, color strength rapidly decreased to up to one-fifth at pH 8 (Figure 2)(Mansour et al., 2013).

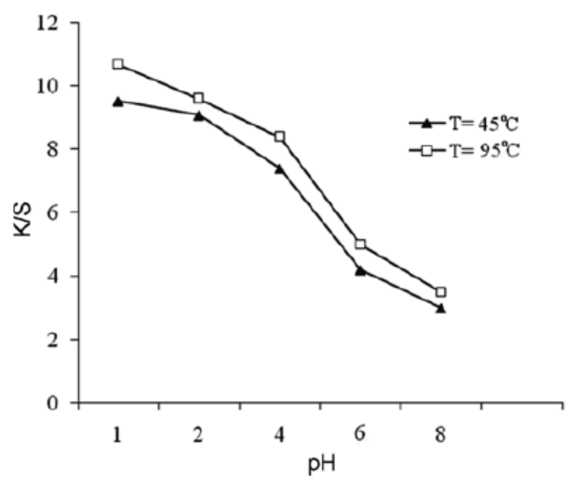


Figure 2. Dye bath pH’s effect on wool fabric dyed with Black Grenache leaf extract at 45 °C and 95 °C (Mansour et al., 2013).

Mishra, Singh, Gupta, Tiwari, and Srivastava (2012)'s study discovered a pH of 2 yielded the highest dye extraction from Dahlia flower petals. It was also noted that at pH 2 the dye solution appeared deep red, while at pH 8 the dye solution appeared blue-green (Mishra et al., 2012). Although the study did not alter pH when applying their extracted dye to fabrics, it is important to note that pH can affect dye color during extraction as many natural dyes require some extraction method (Mansour et al., 2013; Mishra et al., 2012; Pathiban & Thilagavathi, 2012; Sarkar & Seal, 2003). Unlike previous studies mentioned, Arora et al. (2012)'s study showed decreased color strength across six different fibers as pH increased or became more alkaline for Ratanjot dye. Fibers were dyed at 4.5, 7, and 10 pH. Depending on the fiber used, color strength decreased up to three and a half times when dyed at pH 10 compared to pH 4.5 (Arora, et al., 2012). All studies reviewed, with the exception of one, used similar pH modifiers and one study did not mention the modifiers used. Acetic acid was the modifier used to decrease pH and sodium hydroxide was the modifier used to increase pH (Arora et al., 2012; Deo & Desai, 1999; Mansour et al., 2013). Mishra et al. (2012)'s study used hydrochloric acid to decrease pH and sodium carbonate to increase pH. Parthiban and Thilagavathi did not mention the modifiers used in their study.

Quantitative Evaluation Methods

Three common quantitative methods to evaluate dyeing results were examined in previous studies: color strength, color appearance, and statistical tests.

Color strength. Sarkar and Seal (2003) and Deo and Desai (1999) used the Kubelka and Munk equation (K/S) to examine color strength obtained during dyeing. K/S is a function of color depth; higher numbers mean deeper coloration (Etters & Hurwitz, 1986). The K/S equation is as follows: $K/S = (1-R)^2/2R$. R is the reflectance of the dyed fabric, K is the sorption coefficient, and S is the scattering coefficient (Sarkar & Seal, 2003). Spectrophotometers are generally used to calculate this number (Sarkar and Seal (2003) and Deo and Desai (1999)). Munsell color notation also measures color strength, but expresses color values differently (Sproull, 1973). Munsell color notation is a color space that specifies colors based on three color dimensions: hue (the color's appearance), value (lightness/darkness), and chroma (intensity). Colors are reported as a letter followed by numbers separated by a backslash (Sproull, 1973). Of the literature reviewed that discussed color depth, K/S was more common. Only Yi and Cho (2007) used Munsell color notation.

CIELAB L*, a*, b* values. CIELAB is an internationally recognized method for quantifying color appearance and was developed by the Commission International D'Eclairage (Weatherall & Coombs, 1992). CIELAB lays color out in a three dimensional space, with the vertical axis, L*, ranging from 0 (black) to 100 (white). One horizontal axis ranges from green (negative a* values) to red (positive a* values). The second horizontal axis ranges from blue (negative b* values) to yellow (positive b* values) (Figure 3)(Weatherall & Coombs, 1992). The purpose for using this system is to increase precision when discussing color and to create

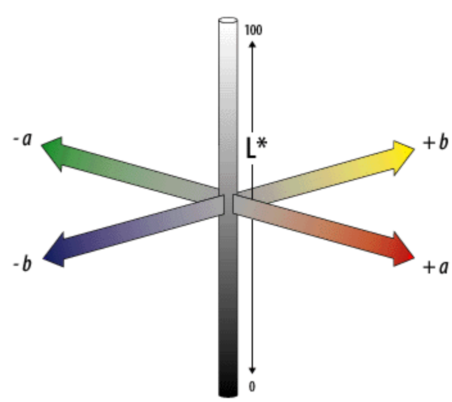


Figure 3. Graphical representation of the CIELAB L*,a*,b* value three dimensional space (Adobe Systems Incorporated, 2000).

consistent color interpretation regardless of language or time (Weatherall & Coombs, 1992). It has not been commonly used in previous dyeing studies. However, CIELAB provides a simple method to quantitatively describe color, so it is worthwhile to be adopted in natural dye research.

Statistical tests. The relevant statistical tests for comparing dyeing results (color appearance and color strength, mainly) are the two sample t-test and Wilcoxon Rank Sum Test. These tests compare two treatments and determine whether there is a statistically significant difference (i.e., a difference not due to chance) between them (Ott & Longnecker, 2010). The two sample t-test is used when data are approximately normal and the Wilcoxon Rank Sum Test is used when data are not normally distributed. Normality can be determined by constructing a histogram with the data and examining it for a bell-shaped curve (Ott & Longnecker, 2010) or by using the Wilks-Shapiro or Kolmogorov-Smirnoff tests in statistics software like R (A.M. Hess, personal communication, September 11, 2012). When comparing more than two groups, a one-way ANOVA test can be used. This test completes a similar function as a two sample t-test but completes the comparisons simultaneously. This removes the need to conduct multiple two sample t-tests (Ott & Longnecker, 2010).

Characterization

Characterization, or examining the structure or properties of a material, is commonly included in textile dyeing studies to examine dyes and fabrics. Common characterization tests include Fourier-Transform Infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and ultraviolet-visible (UV-VIS). Of these tests, FTIR was the most commonly used test in the literature reviewed (Arora et al., 2012; Chung, Lee, & Choe, 2004; Mishra et al., 2012; Parthiban & Thilagavathi, 2011). FTIR analysis determines the chemical groups present on dye, textiles, or other materials (Hesse, Meier, & Zeeh, 1995). This can help determine whether persimmon dye is a satisfactory dye or not. Analyzing the chemical structures present on the dye could give insight into which color-imparting chemicals are present in persimmon dye. Knowledge of these chemical groups could further strengthen the ability to determine what fabrics are more suitable to be used with persimmon dye and what dyeing parameters are most optimal (Arora et al., 2012; Chung, Lee, & Choe, 2004; Mishra et al., 2012; Parthiban & Thilagavathi, 2011).

Relevant Standard Tests

Previous natural dyeing studies use a select few AATCC tests. The studies have focused on analyzing how well different natural dyes perform in regard to colorfastness when applied to various fibers. This is likely due to natural dyes generally being regarded as having inferior colorfastness to synthetic dyes (Deo and Desai, 1999). Standard tests relevant to natural dyeing study are: AATCC 21, 61-2003, 116-2005, and 15-2002.

AATCC 21. Yi et al. (2007) used a modified version of AATCC 21 to evaluate the water absorption rate of persimmon-dyed t-shirts. Test 21 originally tested static water absorption, but was modified to simulate and test dynamic wetting (i.e., sweating) a user would experience during wear and dynamic water absorption for a dyed fabric (Yi et al. 2007). It is also important to note that this test was discontinued in 2006 or earlier (AATCC, 2006).

AATCC 61-2003. Test 61-2003 tests dyed fabric for colorfastness to laundering (AATCC, 2006). It was used by Sarkar and Seal (2003) without modifications. The test simulates five typical home or commercial launderings in one 45 minute test using an Atlas brand Launder-Ometer (AATCC, 2006). This test is extremely important for persimmon dye in the current study because Yi et al. (2007)'s results suggest persimmon dye is an adequate dye for garments close to the skin. In general, garments worn against the skin are washed more due to contact with perspiration. If persimmon dye loses color significantly when laundered, its marketplace use will be severely limited.

AATCC 116-2005. Test 116-2005 tests dyed fabric for colorfastness to crocking (AATCC, 2006). Crocking is when color is transferred from the textile's surface to other surfaces due to rubbing (AATCC, 2006). This test is fairly important to pass for most worn garments because garments brush against other surfaces commonly during daily activities. Little research was found that specifically tested persimmon dye's crocking colorfastness. In the current research, crocking testing for persimmon dyes is meaningful in the commercial application point of view.

AATCC 15-2002. Test 15-2002 tests dyed fabric for colorfastness to perspiration (AATCC, 2006). The test calls for sample fabric to be immersed in a synthetic sweat solution, placed in an AATCC device that exerts 10 pounds of pressure on the samples, and then placed in an oven at $38 \pm 1^{\circ}$ C for six hours (AATCC, 2006). Park et al. (2005) mentioned that persimmon dyed garments are commonly used for summer clothing on Jeju Island, Korea. Although more laboratory testing needs to be done to evaluate persimmon dye's perspiration fastness, it is reasonable to assume persimmon dye has good perspiration colorfastness based on Park et al. (2005)'s statements. This test is extremely important to conduct on persimmon dye because Yi et al. (2007)'s results suggest persimmon dye is an adequate dye for garments close to the skin and will therefore be exposed to significant perspiration.

Persimmon Dye Performance

Coloration compared to other natural dyes. Previous research shows persimmon dyed fabrics yield color results competitive with common natural dyes. Yi and Cho (2007)'s findings indicate arecae semen dye at 300% concentration yields a darker color than persimmon dye, but the two dyes have similar hue and intensity after persimmon dye undergoes six sunlight exposures. No specific concentration was given for persimmon dye in Yi and Cho (2007); persimmon dyed fabric was dipped in persimmon juice and exposed to sunlight. Unfortunately, Yi and Cho (2007) do not indicate how long each sun exposure lasts. The ability to strengthen persimmon dyed fabric's coloration simply through sun exposure gives it an advantage over other natural dyes in needing fewer physical resources to achieve good results. However, the persimmon dyeing process may take much longer than other dyeing processes due to needing multiple sun exposures. Using fewer resources in return for taking more time can be a strength or weakness depending on the application.

Color appearance. Yi and Cho (2007) indicate that fabric samples dyed with persimmon dye have a yellow-red hue initially. However, as sun exposure frequencies increased, it was found that the samples' color was likely to become more red (Yi and Cho, 2007). No time lengths were given for the sun exposures. Similarly to persimmon dye's dyeing procedure, fabric changing color over time could be regarded as a strength or weakness depending on the application and consumer attitudes.

Comfort properties. Yi et al. (2007)'s study focused on the comfort of persimmon-dyed t-shirts compared to un-dyed t-shirts. The authors conducted an experiment where participants walked on a treadmill for 30 minutes (Yi et al., 2007). Each participant completed the experiment twice, once wearing a persimmon-dyed t-shirt and once wearing an un-dyed t-shirt (Yi et al., 2007). Factors including moisture regain, air permeability, water absorption, and wickability were evaluated using objective measures. Thermal sensation, humidity sensation, and overall comfort were tested subjectively by the participants. Participants rated the t-shirt's performance in each category every five minutes during the experiment (Yi et al., 2007). The study found improved air permeability and reduced wickability in persimmon-dyed t-shirts. More air permeability increases comfort, while less wickability decreases comfort (Yi et al., 2007). This makes persimmon-dyed fabric's comfort properties fairly ambiguous. However, the fact that persimmon-dyed clothing is popular for summer clothing on Jeju Island, Korea (Park et al., 2005) may indicate persimmon-dyed clothing is adequately comfortable for the average consumer.

Persimmon dye's added benefits. Persimmon dye imparts various important characteristics on fabric according to Park et al. (2005). The authors found that fabrics dyed with tannin (like persimmon dye) can block ultraviolet rays, are water-resistant, and have anti-fungal properties (Park et al., 2005). Additionally, fabrics dyed with persimmon juice were found to have high tensile strength and flexibility (Park et al., 2005). These characteristics, if found to be present in future research, make persimmon dye applicable for a wide range of end uses within and outside the apparel industry.

Crocking colorfastness performance. Although there are no papers that directly test persimmon dye's crocking colorfastness, some conjectures can be made on how persimmon dye will perform. Tiedemann and Yang (1995) state that mordanting may increase crocking fastness due to the mordant-dye complex's large size after combining. The complex's large size may lead to dye being physically stuck in the fabric's fibers and being less able to be removed (Tiedemann and Yang, 1995). If persimmon dye's crocking colorfastness is not adequate when using sun exposure instead of a mordant like in Yi and Cho (2007)'s research, the dyeing procedure could be altered to include a mordant to remedy this based on Tiedemann and Yang (1995)'s suggestion.

Persimmon Dye in Fiber Arts

Literature reviewed specifically discussing persimmon dye's dyeing technique was vague, thus, dyeing techniques for persimmon dye in art applications were investigated. Hyde (2009) described a method for persimmon dyeing on fabric that was used to construct apparel in Korea. Unripe persimmon fruits were acquired and crushed or juiced to start the process. Fabric was then soaked in the juice and the fabric was agitated in the juice by hand or foot for roughly five minutes. Soaked fabric was laid out in the sun between one and seven days depending on desired color. One day of sun exposure yielded a pink-yellow color, while five to seven days yielded a strong orange color (Hyde, 2009). Dye concentration and dyeing temperature, among other dyeing variables, were not specified by Hyde (2009), creating difficulties in replicating the technique. A similar, more detailed process was outlined by Conrad (2008). Wetted fabric was dipped into a bath of persimmon dye and water at a 1:2 ratio (two parts water to one part persimmon dye). This ratio can be modified from 1:1 to 1:10 parts persimmon dye to water. Similarly to Hyde (2009)'s technique, fabric was agitated by hand for around five minutes. Dye was then drained from the fabric using gravity instead of squeezing and sun-dried (Conrad, 2008). No sun exposure length was given with this method. Interestingly, Conrad (2008) notes treating dyed fabrics with bicarbonate of soda or iron as modifiers after dyeing changed the dye's coloration. Bicarbonate of soda yielded a brown color, while iron yielded varying grey shades compared to persimmon dye's usual orange-brown coloration (Figure 4)(Conrad, 2008). Dye concentration, dyeing temperature, and sun exposure, among other dyeing variables, were not mentioned by Conrad (2008).

Figure 4. Cotton yarns dyed using various persimmon dye-to-water ratios and modifiers (Conrad, 2008).

Persimmon dye's dyeing procedure in fiber arts seem to be related to the traditional method that Yi and Cho (2007) and Yi et al. (2007) used. However, the outlined dyeing procedures are still missing information on some key dyeing parameters, like dyeing temperature and dye concentration. These absences increase the difficulty of obtaining consistent results and replicating the dyeing procedure for future use.

Conclusions

Most research conducted on natural dyeing has used cotton as its test fabric. Few studies were found that applied natural dye to non-cotton natural fabrics or synthetic fabrics. This narrows the research's applicability. Most dyeing variables investigated regarding natural dyeing in the literature were fairly similar, but there was little to no explanation for changes between mordant concentration and dye bath temperature. For example, Yi et al. (2007) and Yi and Cho (2007) used a traditional dyeing technique to dye using persimmon dye as discussed earlier in the paper; however, these researchers did not explain this method in detail. If the authors had done so, replicating their experiments and building off traditional methods would be more feasible. Explaining the reasoning behind using the traditional dyeing technique is also extremely important to study persimmon dye because persimmon dye was the only dye studied that had a vastly different dyeing technique. While two dyeing procedures were examined that greatly expanded on the traditional method, these studies still failed to mention key dyeing parameters. No studies were found that tested persimmon dye's colorfastness performance after dyeing, which, based on Dawson (2008)'s study, should be completed to accurately assess viability in the apparel marketplace. Finally, little subjective research (research using data created from participants' opinions) was found except some examination of subjective performance measures in Yi et al. (2007)'s paper.

Based on these gaps, there is need for additional research that addresses persimmon dye specifically. The current research seeks to discover an optimal dyeing procedure for persimmon dye that is similar to other natural dyes' procedures and can be replicated easily. Additionally, because colorfastness is an important test for natural dyes to undergo, persimmon dye's colorfastness performance will be investigated. The study also seeks to compare persimmon dye's colorfastness performance on cotton fabric versus wool fabric. This will increase the study's applicability to more than one fiber type and add to the body of research regarding using natural dyes on fibers other than cotton.

Chapter 3: Materials and Methods

Materials

Bleached, desized, and mercerized 100% cotton print cloth (style 400M) and carbonized 100% wool flannel (style 527) obtained from Testfabrics, Inc. were used in the study. The cotton's characteristics were: 107 g/m^2 weight, 150 count, 0.356 mm thickness. The wool's characteristics were: 255 g/m^2 weight, 57 count, 0.965 mm thickness. Persimmon dye was 100% fermented persimmon juice powder acquired from Bauhaus of Forest (Figure 5).

Figure 5. Persimmon dye powder.

The mordant used was reagent-grade crystalline ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) from Fisher Scientific, Inc. AATCC laundering and perspiration testing required standard multi-fiber fabric (style MFF1) from Test Fabrics, Inc. The standard multi-fiber fabric contains acetate, cotton, nylon, silk, viscose, wool, and polyester fibers and was used to evaluate staining performance. The detergent used during laundering testing was AATCC Standard Reference Detergent. AATCC crocking testing utilized 2"x2" standard crocking squares made from mercerized, bleached cotton fabric from Atlas SDL. De-ionized water was used for all dyeing and experiments to reduce the chance of contaminants affecting the experiments.

Dyeing Procedure

Cotton and wool fabrics were dyed using a Datacolor Ahiba Nuance ECO-B dyeing machine. The dyeing procedure was adapted from Sarkar and Seal (2003)'s method. The dye bath's liquor ratio, calculated by the weight of fabric to water, was 1:50 (1 g fabric per 50 ml water) for all samples. The persimmon dye concentration used was 50%, 100%, and 200% on weight of fabric (OWF). OWF means the amount of dye or mordant added is directly proportional to the fabric sample's weight. At each dye concentration, the dyeing time length was carried out at 30, 45, and 60 minutes. Mordanting treatment ($FeSO_4 \cdot 7H_2O$) was carried out before dyeing, during dyeing, or after dyeing, which are called pre-, meta-, and post-mordanting, respectively. Mordant concentrations in the literature ranged from 1% to 10% OWF. A 5% OWF was chosen for this study. Cotton and wool samples were also dyed without mordant to provide control samples. The mordanting procedure, dye concentration, and dyeing time combination that yielded the highest color strength was then used to dye samples for further tests.

Pre-mordanted samples were treated in a mordant bath before dyeing. Mordanting was carried out using the dyeing machine. Fabric samples were introduced to the mordanting solution (water and iron) in stainless steel canisters at room temperature and loaded into the dyeing machine. Temperature was raised to boiling (100 °C) using a 0 °C temperature gradient setting (temperature raised as quickly as the machine is able to). The canisters were rotated by the machine at a rate of 30 rpm. Mordanting continued at boiling for 60 minutes. After mordanting, samples were thoroughly squeezed and placed into a persimmon dye bath containing water and dye powder. Dyeing was carried out at boiling for 30, 45, and 60 minutes. Dye concentration OWF was varied within each dyeing length. The concentrations used were 50%, 100%, and 200% OWF. After dyeing, samples were rinsed with de-ionized water and air-dried.

Post-mordanted samples were dyed in the persimmon dye bath before being treated in the mordant bath. Samples were dyed in a dye bath (containing water and dye) in the dyeing machine. The samples were dyed for 30, 45, and 60 minutes. Dyeing concentrations were varied at 50%, 100%, and 200% OWF. After dyeing, samples were thoroughly squeezed and placed into the mordanting bath (water and iron) for 60 minutes. Samples were rinsed with de-ionized water and air-dried after mordanting.

Meta-mordanted samples were concurrently dyed and mordanted in one bath. Samples were placed in a bath containing water, iron, and dye powder for 30, 45, and 60 minutes. Dyeing concentrations were varied at 50%, 100%, and 200% OWF. After being removed from the solution, the samples were rinsed with de-ionized water and air-dried.

Non-mordanted samples were dyed without mordanting treatment. Fabric samples were dyed for 30, 45, and 60 minutes. Dyeing concentrations were varied at 50%, 100%, and 200% OWF. The samples were rinsed with de-ionized water and air-dried after dyeing was completed.

After rinsing off the excess dye, samples were air dried overnight (around eight hours) before their color strength was evaluated. Each dyeing, mordanting, and time length procedure was replicated three times.

Color Strength and Appearance

Color strength and appearance was evaluated using a HunterLab ColorQuest XE® diffuse/8^0 spectrophotometer. The spectrophotometer was operated with a one-inch diameter specimen viewing aperture in reflectance – specular included mode. Fabric samples were held in place using a spring-loaded sample clamp. Each sample was scanned three times and rotated 90 degrees between each scan to minimize error and increase accuracy. The data recorded from the scans consisted of K/S values and CIELAB L*, a*, b* values. Dyed samples from the pilot dyeing experiment were scanned after drying overnight. Colorfastness-tested samples were evaluated using the spectrophotometer immediately after AATCC Gray Scale evaluation so there was no significant change in the samples' temperature and humidity. This ensured the measurements corresponded closely.

AATCC Colorfastness Testing

The following AATCC standard colorfastness tests were used to evaluate persimmon dye's performance on cotton and wool fabrics: AATCC 61-2003, AATCC 116-2005, and AATCC 15-2002. These tests were selected because they simulate common textile colorfastness situations. Because natural dyes' colorfastness is generally poor (Dawson, 2008), these tests can provide preliminary evaluation on persimmon dye's viability in the consumer marketplace.

AATCC 61-2003. AATCC 61-2003 tests colorfastness to laundering. The test was performed using an Atlas Launderometer according to method 1A. A 2"x2" multi-fiber fabric evaluation swatch was attached to a 4"x2" laundering sample by stapling. The stapled samples were placed in an Atlas Launder-Ometer test canister with 200 ml total liquor volume (.37% detergent of total volume) and 10 steel balls. Sealed canisters were treated in the Launder-

Ometer at 40 °C and 40 revolutions/minute for 45 minutes. Afterward, samples were removed from the canister and rinsed in a beaker of 40 °C water three times (one minute periods) and then placed in an air circulating oven at 70 °C until dry. The samples were then removed from the oven and immediately conditioned at 21±1 °C and 65±2% relative humidity for one hour. After conditioning, the samples were evaluated using the AATCC Gray Scale for Color Change and AATCC Gray Scale for Staining (AATCC, 2006) and further evaluated for color strength using the spectrophotometer.

AATCC 116-2005. AATCC 116-2005 tests colorfastness to crocking. The test was carried out using an Atlas Rotary Vertical Crockmeter. Before crocking, each dyed fabric specimen and its corresponding crocking square were conditioned at 21±1 °C and 65±2% relative humidity for four hours. After conditioning, dry crocking samples and corresponding crocking squares were placed in the Atlas Rotary Vertical Crockmeter and exposed to 40 reciprocal turns of the vertical shaft. For wet crocking, crocking squares were treated with de-ionized water equal to .65 times their weight before being placed in the Atlas Rotary Vertical Crockmeter. Crocking squares were wetted individually to avoid excess evaporation. The dyed samples and wet crocking squares were then exposed to 40 reciprocal turns of the vertical shaft. After crocking, the tested squares and samples were evaluated by the AATCC Gray Scale for Staining (AATCC, 2006). Unlike the other two colorfastness tests, samples' color strength was not evaluated using a spectrophotometer. Accurate readings were not available for crocking samples because the area affected by crocking (.5" in diameter) was 50% smaller than the fabric size required (greater than 1" diameter) for accurate readings by the spectrophotometer.

AATCC 15-2002. AATCC 15-2002 tests colorfastness to perspiration. The test was performed using the standard vertical perspiration testing method. A perspiration solution (10g

NaCl, 1g lactic acid USP 85%, 1g Na_2HPO_4, and .25g ℓ-histidine monohydrochloride) was prepared and its pH evaluated to ensure it was 4.3±.02. Dyed fabrics were cut into 6x6 cm squares and paired with a multi-fiber fabric swatch of equal size. Test samples were soaked in the perspiration solution for 30±2 minutes with periodic squeezing and stirring. Then the samples were passed through a wringer until the desired weight was achieved for each sample (2.25±.05 times its original weight). Dyed samples and their corresponding multi-fiber swatches were placed on the vertical AATCC Perspiration Tester's Plexiglas plates at even intervals. Ten pounds of force were applied to the plates and the entire assembly placed in an oven at 38±1 °C for six hours. After removal from the oven and Perspiration Tester, the fabric samples and their multi-fiber swatches were conditioned at 21±1 °C and 65±2% relative humidity overnight. The samples and multi-fiber swatches were then evaluated using the AATCC Gray Scale for Color Change and Gray Scale for Staining (AATCC, 2006). Samples' color strength was also evaluated using a spectrophotometer.

Color change and staining. All samples were evaluated after colorfastness testing using the AATCC Gray Scale for Color Change and AATCC Gray Scale for Staining according to AATCC standards (AATCC, 2006). Gray scale evaluations were completed by visually comparing a dyed, untested sample with a tested sample under standard conditions and assigning a rating between 1 and 5. For Gray Scale for Color Change evaluation, a grade of 1 indicates most color was lost and a grade of 5 indicates no color was lost. A rating of less than 3 indicates extensive color alteration after testing (AATCC, 2006). For Gray Scale for Staining evaluation, a grade of 1 indicates very heavy staining of the multi-fiber fabric and a grade of 5 indicates no staining of the multi-fiber fabric. A rating of less than 3 indicates extensive staining after testing (AATCC, 2006).

Statistical Analysis

Dyed cotton and wool samples' color strength and color appearance were compared quantitatively before and after undergoing AATCC standard testing. This was done to determine whether the tests significantly affected the samples. Each sample served as the control and treatment sample, so a paired t-test was used (Ott & Longnecker, 2010). All statistical tests were conducted using the statistics software R at a significance level of $p \leq .05$.

Chemical Composition Analysis

Dye performance on cotton and wool was further analyzed using FTIR with advanced attenuated total reflectance. Fabric samples were tested using a Nicolet iS50 FT-IR Spectrometer. All samples were evaluated by placing the sample directly on a crystal (diamond/ZnSe) and exerting pressure on the sample with a sample holder. Scans were taken using a 400-4000 cm^{-1} spectrum, 16 co-addition scans, and 4 cm^{-1} resolution. Data were generated and analyzed using OMNIC Specta software.

Chapter 4: Results and Discussion

The current work consisted of a pilot study on dyeing cotton and wool fabrics using persimmon dye and a comparison study of dyeing performance on cotton and wool fabric using AATCC standard tests. The pilot study investigated the impact of dye concentration, dyeing length, and mordanting order on color strength and appearance, suggesting the optimal dyeing procedures used in the current work. Dyeing performance on the dyed fabrics with highest color strength was further evaluated using AATCC standard colorfastness tests. Chemical composition analysis was carried out on the persimmon dye and dyed fabrics using FTIR.

Pilot Dyeing Results

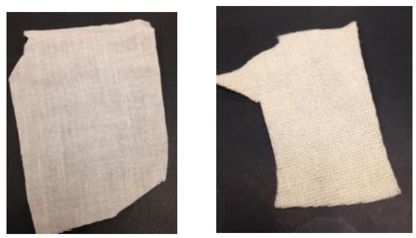
Un-dyed fabric analysis. Un-dyed cotton and wool fabrics (Figure 6) were evaluated for their color strength and appearance indicated by K/S and L*, a*, b* values (Table 1). Un-dyed wool fabric had a much more yellow (+b*) appearance, slightly greener (-a*) appearance, and higher initial K/S than un-dyed cotton fabric. Wool's slight coloration is typical because the only treatment done to the wool used in this work was carbonization. Carbonization only removes organic matter that may have been trapped in the fibers during production (Halliday, 2002), so some natural color was still present. Un-dyed cotton fabric had much less initial coloration because it was bleached, which removes natural pigmentation (Wong, Lam, Kan, & Postle, 2013). The cotton was also mercerized and desized. Mercerization increases dye uptake by opening the fiber structure at the molecular level (Tóth, Borsa, Reicher, Sallay, Sajó, & Tanczos, 2003). Desizing removes sizing starch commonly used in cotton production (Schwarz, Kovacevic, & Dimitrovski, 2011). These treatments, along with bleaching, increase dye uptake and decrease the influence of chemicals and natural pigment on the cotton's dyeing results.

Table 1

Un-Dyed Cotton and Wool K/S and L, a*, b* Values*

Fabric Composition	Treatment	K/S	L*	a*	b*
Cotton	No treatment	0.13	82.257	-0.051	-0.196
Wool	No treatment	0.44	82.704	-1.532	7.547

Note. Cotton K/S and L*, a*, b* values taken at 670 nm



(a) (b)

Figure 6. Un-dyed cotton (a) and wool (b) samples.

Dyed fabric analysis. Cotton and wool samples' color strength and appearance were measured after dyeing was completed. Figures 7-10 show the K/S values of dyed cotton samples and Figures 11-14 show the K/S values of dyed wool samples. Tables containing exact K/S values for all samples are located in Appendix 1. All K/S and L*, a*, b* readings were taken at the wavelength yielding the highest values. This wavelength was 400 nm for all samples unless otherwise noted. Wool samples' K/S was significantly higher than cotton samples' under all dyeing conditions. Several dye performance patterns were indicated by the color strength evaluations in Figures 7-14. Color strength on the cotton and wool fabrics increased significantly when a mordant was used. This agrees with Tiedemann and Yang's (1995) research. Iron's good performance as a mordant also agrees with Deo and Desai (1999)'s research. The tea dye used in their study contained tannins, which are also found in persimmon dye (Nakajima & Sakaguchi, 2000). Deo and Desai (1999) obtained significant K/S increases after using an iron mordant, like the current study. Additionally, the fabrics' color strength generally increased when dye concentration increased at constant dyeing time length. Similar results were demonstrated on multiple natural dyes in Yi and Cho's (2007) study. The fabrics' color strength also generally increased with longer dyeing times and more dye concentration, with dye concentration usually affecting K/S more overall. However, the impact of those variables varied depending on the mordanting procedure. Meta-mordanted samples' K/S was affected more by time increase, but pre-mordanted samples' K/S was affected more by concentration increase. Another important result to note was that for all dyeing conditions, meta-mordanted wool and cotton samples became much stiffer with a rougher hand than any other samples. This effect was especially severe on the wool samples, with a 2"x2" size sample not bending when held by one edge. Dyeing using meta-mordanting also resulted in the presence of clumps of black gel residue on the fabric and dye container surface. This was also discovered in previous studies and was suggested to be a reaction between the tannin in the dye and the mordant (Rahim & Kassim, 2008; Nakajima & Sakaguchi, 2000). Much of the gel was removed during rinsing, but some stayed attached to the fabric (appearing as black coloration) as seen in Figure 15 (c) and (g). Therefore, meta-mordanting, especially when dyeing wool fabric, is not recommended unless a particular end-use requires an unevenly dyed or extremely stiff fabric. It is important to note that the mordanting procedure impacted the samples' color appearance greatly (shown in Figure 15), which corresponds to Yi and Cho's (2007) findings.

Cotton and wool samples dyed using post-mordanting, 200% dye concentration, and 60 minutes dyeing length exhibited the highest K/S values (6.9 and 20.9, respectively). Therefore, that dyeing procedure was used to dye samples for all AATCC colorfastness tests.

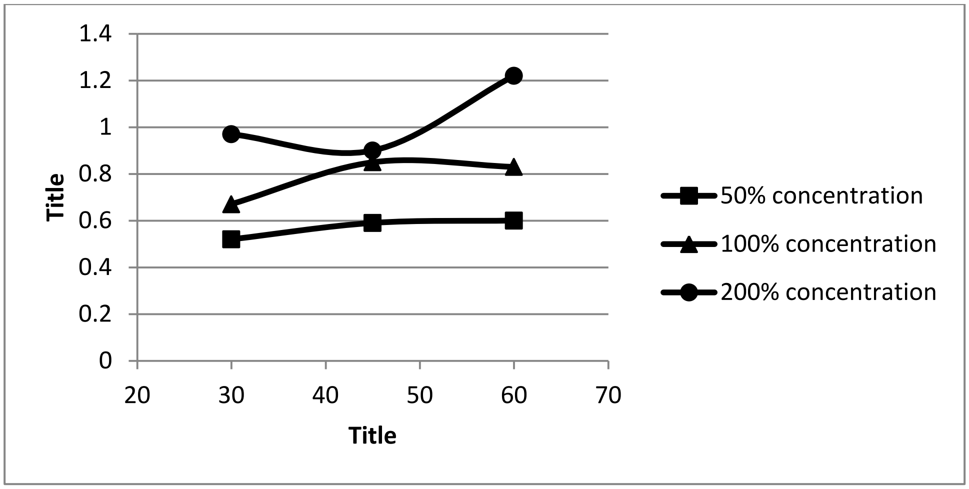


Figure 7. Non-mordanted cotton samples' K/S values.

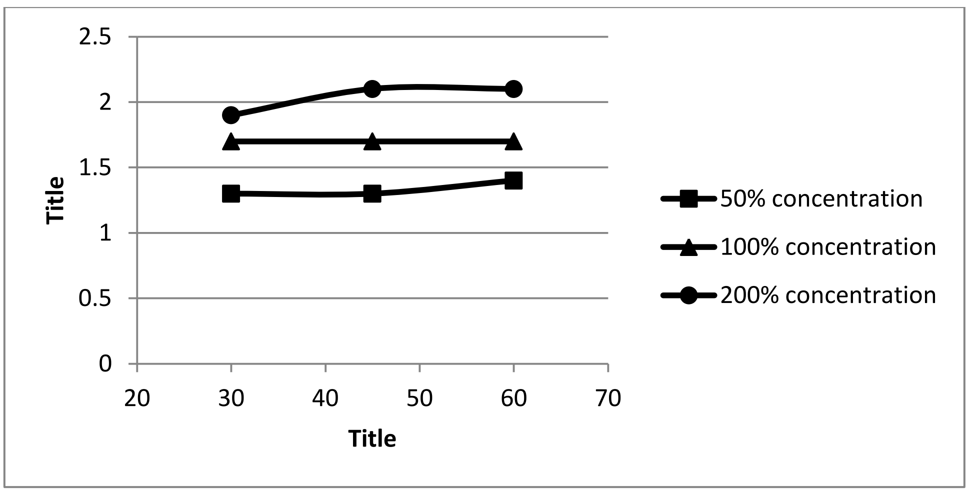


Figure 8. Pre-mordanted cotton samples' K/S values.

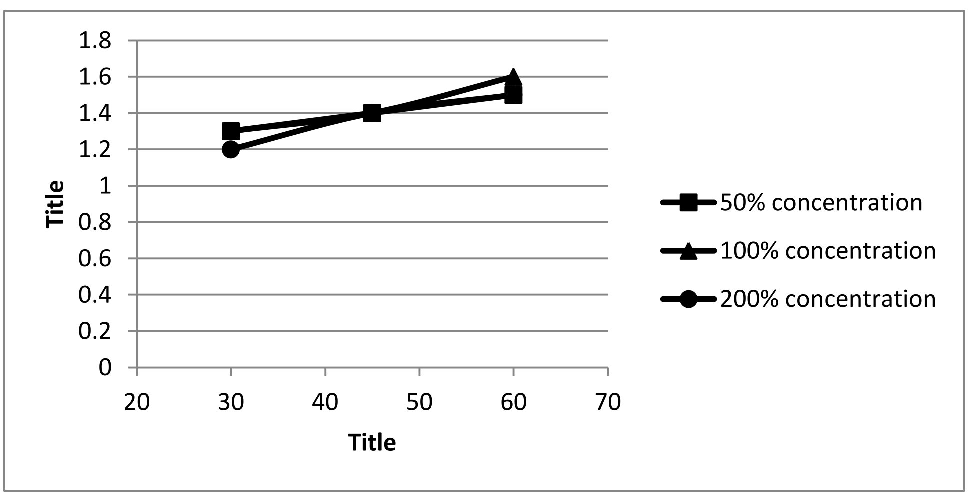


Figure 9. Meta-mordanted cotton samples' K/S values.

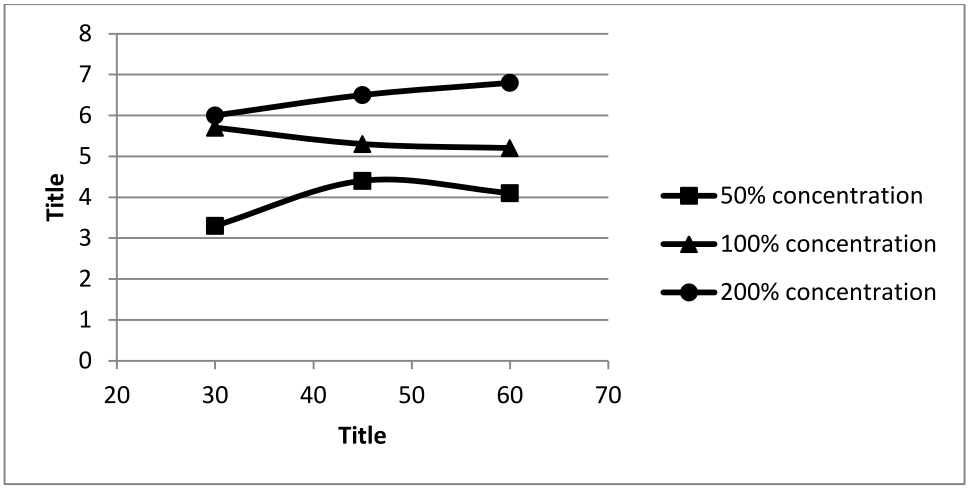


Figure 10. Post-mordanted cotton samples' K/S values.

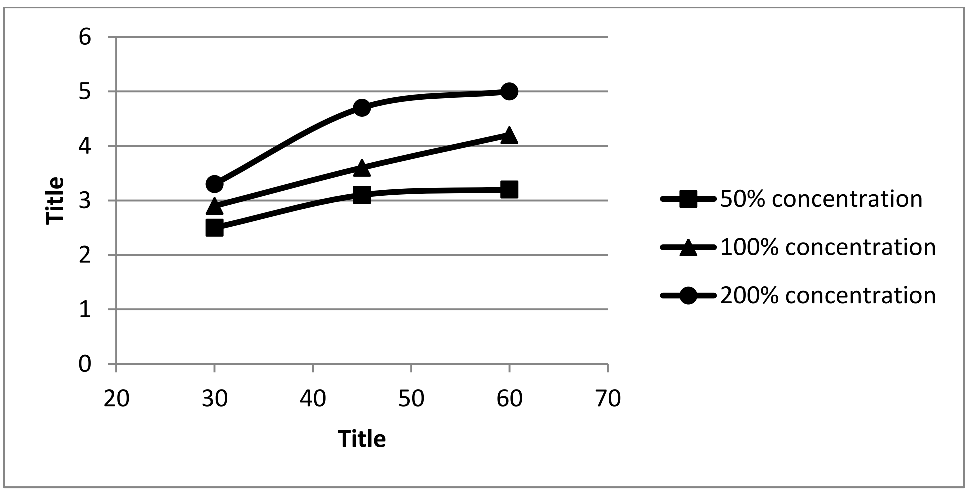


Figure 11. Non-mordanted wool samples' K/S values.

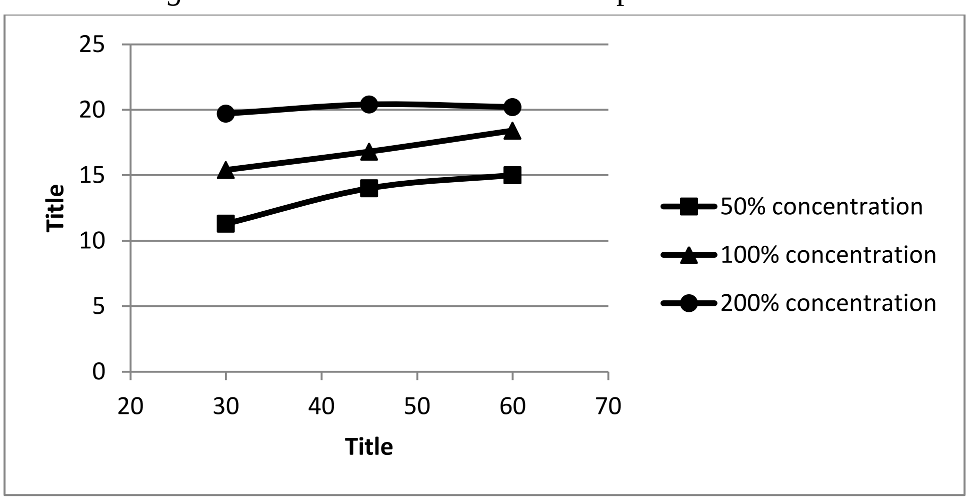


Figure 12. Pre-mordanted wool samples' K/S values.

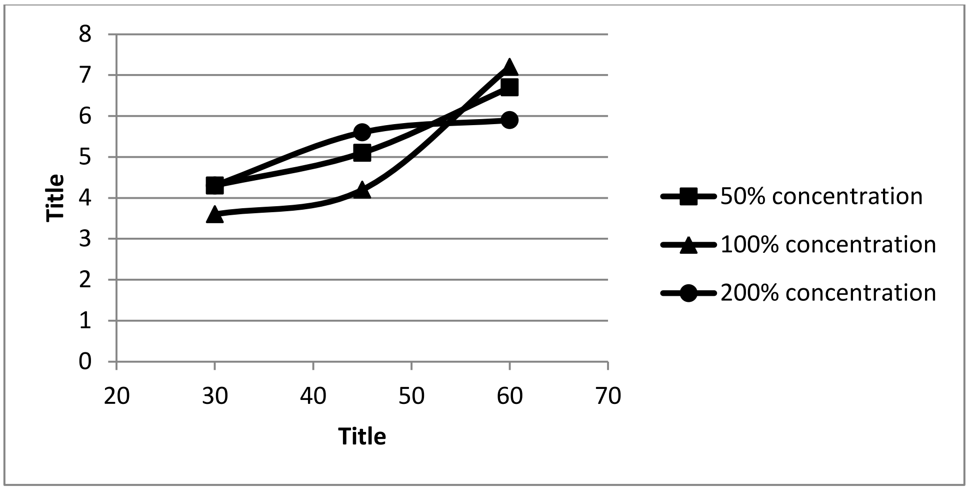


Figure 13. Meta-mordanted wool samples' K/S values.

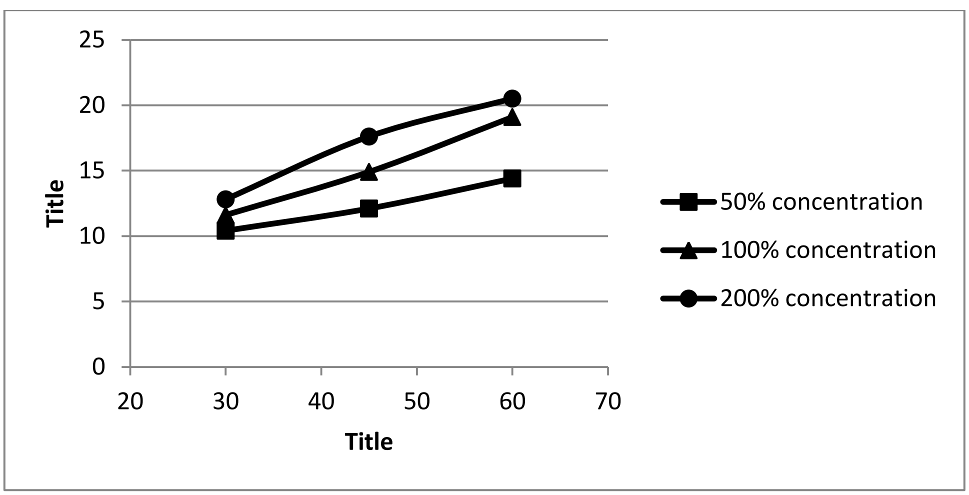


Figure 14. Post-mordanted wool samples' K/S values.

(a) Non-mordanted cotton

(b) Pre-mordanted cotton

(c) Meta-mordanted cotton

(d) Post-mordanted cotton

(e) Non-mordanted wool

(f) Pre-mordanted wool

(g) Meta-mordanted wool

(h) Post-mordanted wool

Figure 15. Cotton and wool samples dyed using various mordanting methods. All samples pictured were dyed at 200% concentration for 60 minutes.

Table 2 shows the average CIELAB L*, a*, b* values for the samples with the highest K/S per mordanting procedure per fabric. The L*,a*,b* values show how the samples' appearances were altered based on mordanting procedure and fabric used. All non-mordanted samples yielded a light brown-orange coloration (highest L*, a*, and b* values). Post-mordanted samples yielded medium gray (L*, a*, b* values closer to zero) on cotton samples and dark purple-gray (lowest L* and b* values) on wool samples. Meta-mordanted wool and cotton yielded light gray on cotton samples and medium gray on wool samples, which corresponded with their L* values being second-highest out of all mordanting methods. Interestingly, wool samples yielded darker colors (lower L* value) than cotton samples dyed when using the same dyeing procedure for both fabrics. Because the dye changed color or became stiffer depending on whether a mordant was used or mordanting order, it was hypothesized that a chemical reaction could be occurring between the dye and mordant or dye and fabric. Based on that hypothesis, chemical composition analysis using FTIR was conducted to characterize the dyed fabrics and dye and determine whether the dye was chemically bonding to the fabric.

Table 2

CIELAB L,a*,b* Values for Persimmon-Dyed Cotton and Wool Fabrics*

Fabric composition	Mordanting procedure	L*	a*	b*
Cotton	None	62.336	11.109	13.066
	Pre	54.363	9.178	11.558
	Meta	58.024	4.390	8.827
	Post	39.637	4.642	10.680
Wool	None	44.748	14.873	14.308
	Pre	20.834	2.739	3.267
	Meta	33.264	3.197	4.454
	Post	18.510	1.971	.921

Colorfastness

Colorfastness fabric samples were prepared using post-mordanting, 200% dye concentration, and 60 minutes dyeing length because those conditions resulted in the highest color strength for both fabrics. Table 3 shows the K/S and L*,a*,b* values of the dyed samples before laundering and perspiration colorfastness testing. For K/S and L*,a*,b* values of the dyed samples before and after testing, see Appendix 2. Crocking K/S and L*,a*,b* values were not recorded because the tested fabric area was smaller than the spectrophotometer's viewing port and could not be accurately scanned. The K/S and L*,a*,b* values were different than the results obtained from pilot dyeing because the dye was from a different manufacturing batch.

Table 3

CIELAB L,a*,b* and K/S Values for Persimmon-Dyed Cotton and Wool Samples Before Undergoing Colorfastness Testing*

Fabric	Testing procedure	K/S value	L*	a*	b*
Cotton	Laundering	6.2	39.931	4.338	9.533
	Perspiration	6.6	39.183	4.392	9.543
Wool	Laundering	16.8	20.032	2.023	0.634
	Perspiration	14.7	21.781	2.252	0.987

Cotton colorfastness performance. Table 4 shows the AATCC Gray Scale ratings for cotton's colorfastness testing. Dyed cotton samples' colorfastness performance varied depending on the test performed. Tested cotton samples performed below-average for laundering color change (2-3), slightly above-average for crocking color change (4 dry, 3 wet), and average for perspiration color change (4). For staining, cotton performed excellently for laundering (4-5), above average for dry crocking (4), below average for wet crocking (2-3), and excellently for perspiration (4-5).

It is important to note that cotton's color strength and color appearance changed significantly during laundering. Figure 16 shows these changes in K/S and L*, a*, b* after laundering testing. Samples' redness (+a*), yellowness (+b*), and darkness (decrease in L*)

Table 4

AATCC Gray Scale Evaluation for Colorfastness of Persimmon-Dyed Cotton Samples

AATCC test	Color change (gray scale grade)	Staining (gray scale grade)
Laundering	2-3	4-5
Perspiration	3	4-5
Crocking (dry)	4	4
Crocking (wet)	3	2-3

increased at a statistically significant level. Another observation was that cotton's color strength increased significantly after laundering. Yi and Cho (2007) increased persimmon-dyed fabrics' color strength using sunlight, so it is possible that the increase in the current study was due to the 40 °C laundering temperature. Another possible cause is the detergent, as detergent can affect K/S (Soliman, Carr, Jones, & Rigout, 2013).

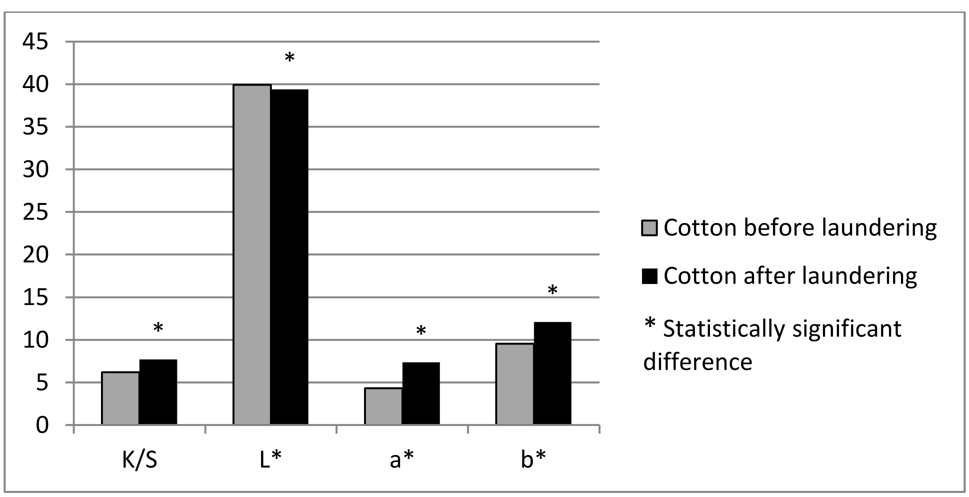


Figure 16. Cotton samples' K/S and L*,a*,b* before and after laundering testing.

Figure 17 shows the changes in the cotton samples' K/S and L*, a*, b* values after they underwent perspiration testing. Perspiration testing yielded a significant decrease in samples' K/S and significant changes in color appearance. Samples’ redness (+a), yellowness (+b) and lightness (increased L*) increased at statistically significant levels.

Overall, persimmon dye performed acceptably on cotton fabric. It received average or above average ratings except in laundering color change (2-3) and wet crocking staining (2-3). However, Dawson (2008) suggested consumers demand excellent colorfastness. Because of the dye's significant color change after laundering, applications with minimal laundering would be ideal. This drastically reduces persimmon dye's viability on cotton fabric.

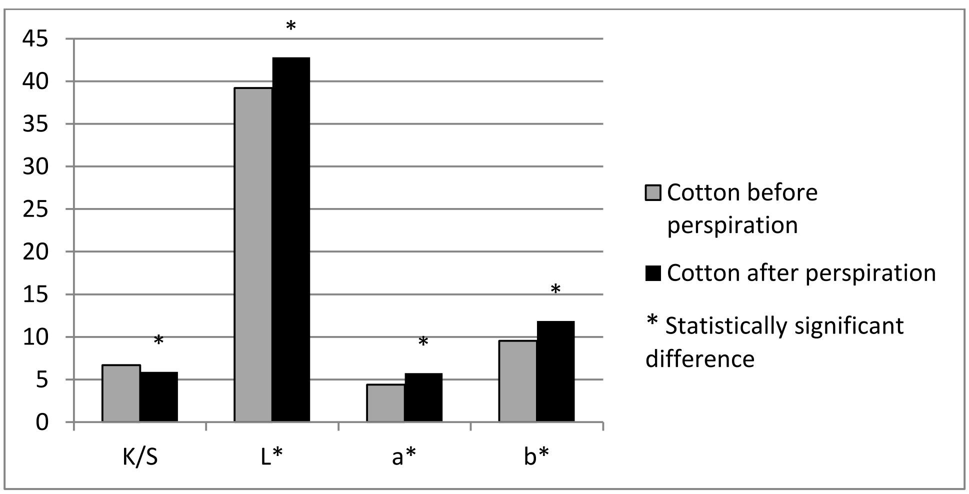


Figure 17. Cotton samples' K/S and L*,a*,b* before and after perspiration testing.

Wool colorfastness performance. Dyed wool’s colorfastness performance varied depending on the test performed. Table 7 shows the AATCC Gray Scale ratings for wool's colorfastness testing. Tested wool samples performed average for laundering color change (3), above-average for crocking color change (4 wet and dry), and above-average for perspiration color change (4). For staining, wool performed excellently for laundering (4-5), above-average to average for crocking (4-5 dry, 3 wet), and excellently for perspiration (5).

Table 5

AATCC Gray Scale Evaluation for Colorfastness of Persimmon-Dyed Wool Samples

AATCC test	Color change (gray scale grade)	Staining (gray scale grade)
Laundering	3	4-5
Perspiration	4	5
Crocking (dry)	4	4-5
Crocking (wet)	4	3

Like the cotton samples, the wool samples’ color appearance changed significantly after laundering. Figure 18 shows these changes in the samples' K/S and L*, a*, b* values after they underwent laundering testing. Wool samples’ redness (+a*) and yellowness (+b*) increased at statistically significant levels, as found in the cotton samples. However, unlike the treated cotton, wool samples’ L* values did not change at a statistically significant level. The wool samples performed similarly to cotton samples in that the color strength increased significantly after laundering. Wool samples' K/S increased slightly more than cotton samples' K/S (13% more)

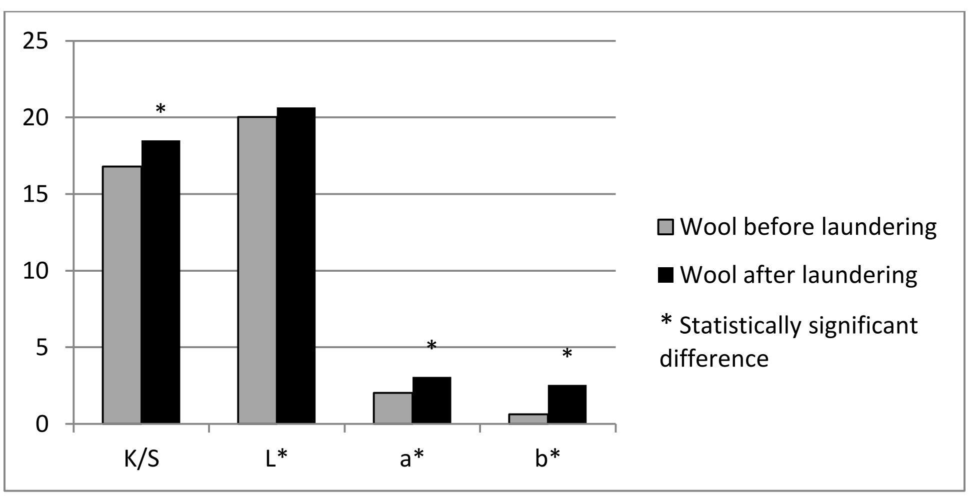


Figure 18. Wool samples' K/S and L*,a*,b* before and after laundering testing.

after the samples underwent laundering. However, the wool samples' L*, a*, b* value change was 45% less on average than the cotton fabric samples'.

Figure 19 shows the changes in K/S and L*, a*, b* values after the wool samples underwent perspiration testing. Unlike the cotton samples, the wool samples changed little after perspiration testing, and only the wool samples' yellowness (+b*) increased at a statistically significant level.

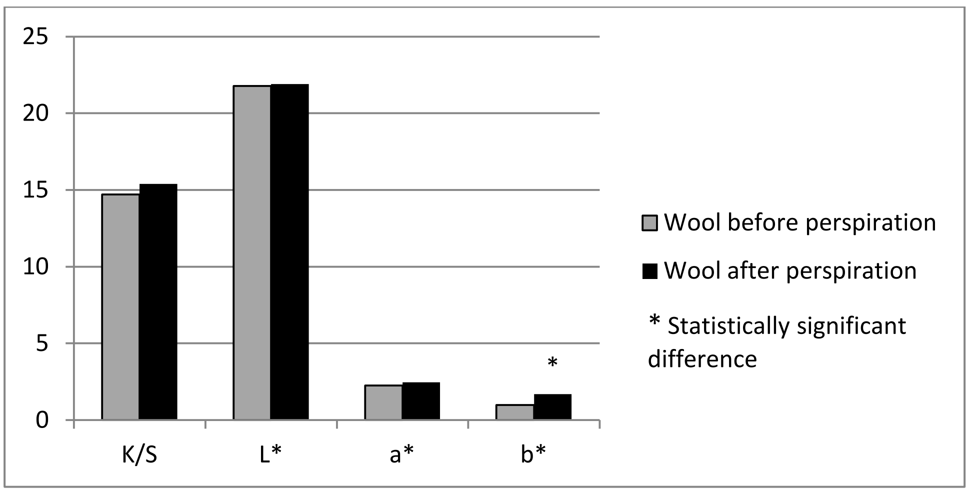


Figure 19. Wool samples' K/S and L*,a*,b* before and after perspiration testing.

Overall, persimmon dye performed well on wool fabric. It received above-average to excellent ratings except in laundering (3) and wet crocking (3). The wool exhibited more K/S increase compared to the cotton fabric but color appearance changed substantially less (45%, as mentioned above). This suggests persimmon-dyed wool fabric exhibits better washability than dyed cotton fabric in regard to colorfastness, but exposure to laundering should still be limited.

Comparison between wool and cotton. The color strength and color appearance of the tested cotton and wool samples was compared after colorfastness testing. Wool samples yielded a much higher K/S than cotton for all tested dyeing procedures. The biggest difference occurred when wool generated an 822% higher K/S than cotton using pre-mordanting, 200% concentration, and 45 minutes length for each fabric. Post-mordanting, 200% concentration, and 60 minutes dyeing length yielded a 201% higher K/S on wool compared to cotton. Some of wool’s higher color strength could be attributed to surface roughness. Fabrics with rougher surfaces (the wool in this study) produce a higher K/S value compared to smooth fabrics (the cotton in this study) (Glogar et al., 2011).

Dye performance to laundering was fairly similar for the wool and cotton fabrics. Fabric samples became redder (increase in a*) for both fabrics, but the cotton samples’ redness increased much more than wool’s (1.97 more a*). Additionally, cotton samples appeared darker (decreased L*), while wool’s L* did not change significantly. The wool fabric's color strength and color appearance changed 15% less on average compared to the cotton fabric after laundering testing. Both fabrics performed similarly during laundering testing overall, though, with wool performing only slightly better in regard to AATCC ratings.

Persimmon-dyed wool received ratings one rank higher than cotton for dry and wet crocking, suggesting the color imparted on wool from persimmon dye is preserved better in situations where fabric-on-fabric rubbing occurs.

Dye performance to perspiration testing varied significantly between wool and cotton. Cotton samples' K/S and L*, a*, b* values changed at significant levels, whereas only wool samples' b* value was significantly different. Cotton samples became significantly lighter (-K/S, -L*) and appeared more orange (+a*, +b*). Additionally, wool received an AATCC Gray Scale rating of 4 compared to cotton’s rating of 3. Color strength and color appearance changed 68% less on average on the wool fabric than on the cotton fabric after perspiration treatment. These differences suggest that persimmon dye's colorfastness to perspiration is better on wool than on cotton. It may be because wool fibers usually show better resistant to acids than cotton (Kadolph, 2007). The change of cotton fibers due to acids may cause persimmon dye's poor colorfastness on cotton.

Chemical Composition Analysis

Persimmon dye powder. Persimmon dye powder FTIR spectra (Figure 20) showed peaks similar to other materials containing tannins (Fernandez & Agosin, 2007; Kim, S. & Kim, H.-J., 2003; Oo, Kassim, & Pizzi, 2009). Peaks at 1608.28 cm^{-1}, 1535.64 cm^{-1}, and 1444.70 cm^{-1} likely indicate the presence of aromatic rings. The high-intensity peak at 1608.28 cm^{-1} could indicate a C-C stretch corresponding with interflavonoid linkages. The single peak at 1535.64 cm^{-1} suggests that the persimmon tannin consists mainly of procyanidin (Oo, Kassim, & Pizzi, 2009). The single 1206.13 cm^{-1} peak is likely due to –CH stretching and the peaks at 828.42 cm^{-1} and 763.85 cm^{-1} are likely from C-H bond vibrations in the aromatic rings (Kim, S. & Kim, H.-J., 2003). Finally, peaks at 1146.151 cm^{-1}, 1104.53 cm^{-1}, and 1032.99 cm^{-1} are suggested to be due to aromatic C-H bending by Fernandez and Agosin (2007).

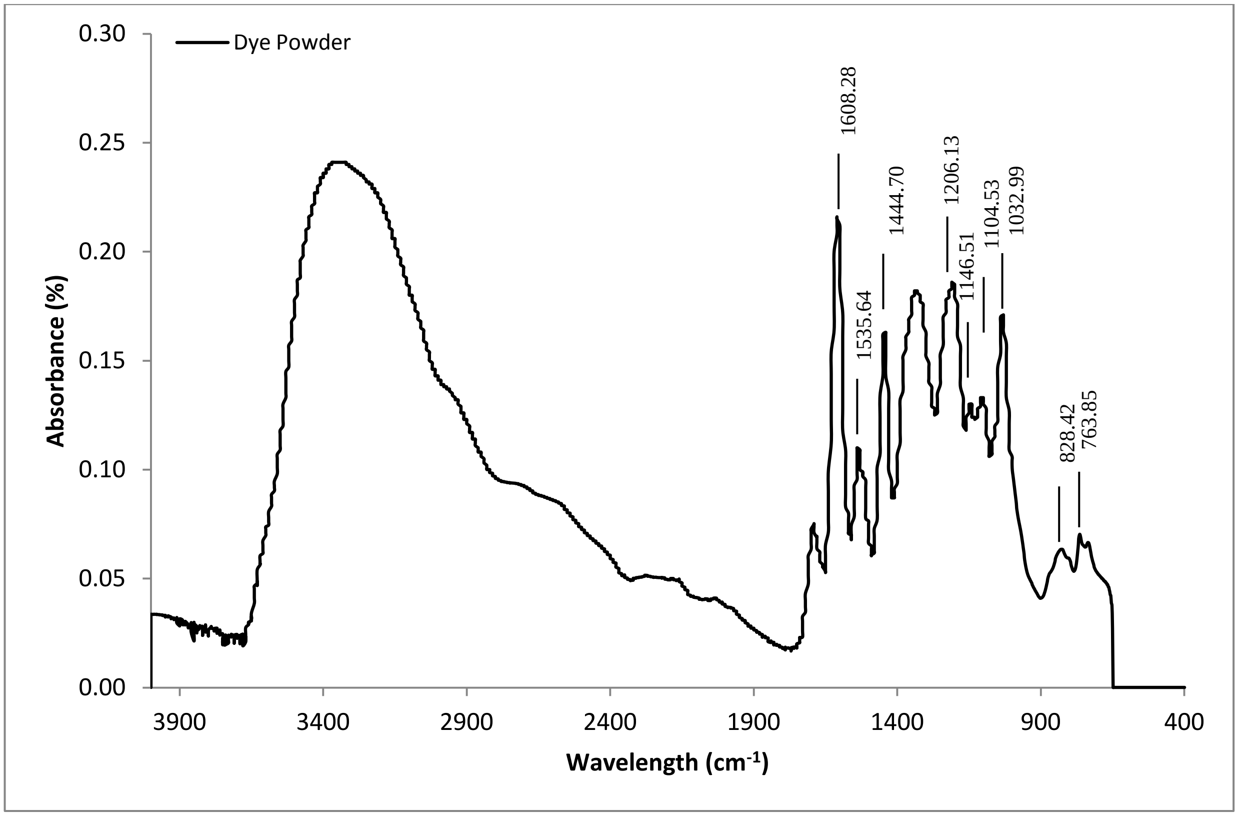


Figure 20. Persimmon dye FTIR spectra.

Cotton. Figure 21 shows the FTIR spectrum for un-dyed cotton. It appeared similar to results obtained in previous studies as expected (Chung, Lee, & Choe, 2004; Garside & Wyeth, 2003). The spectrum showed characteristic water adsorption (1641.83 cm^{-1}), C-H groups (2894.28 cm^{-1}, 1427.57 cm^{-1}, and 1314.66 cm^{-1}), CH_2 wagging (1335.31 cm^{-1}), CH_2 twist (1278.30 cm^{-1}), C-OH or C-CH (1200.92 cm^{-1}), C-C ring (1159.24 cm^{-1}), C-OH (1055.89 cm^{-1} and 1031.08 cm^{-1}), and C-O-C (895.31 cm^{-1}) (Garside & Wyeth, 2003).

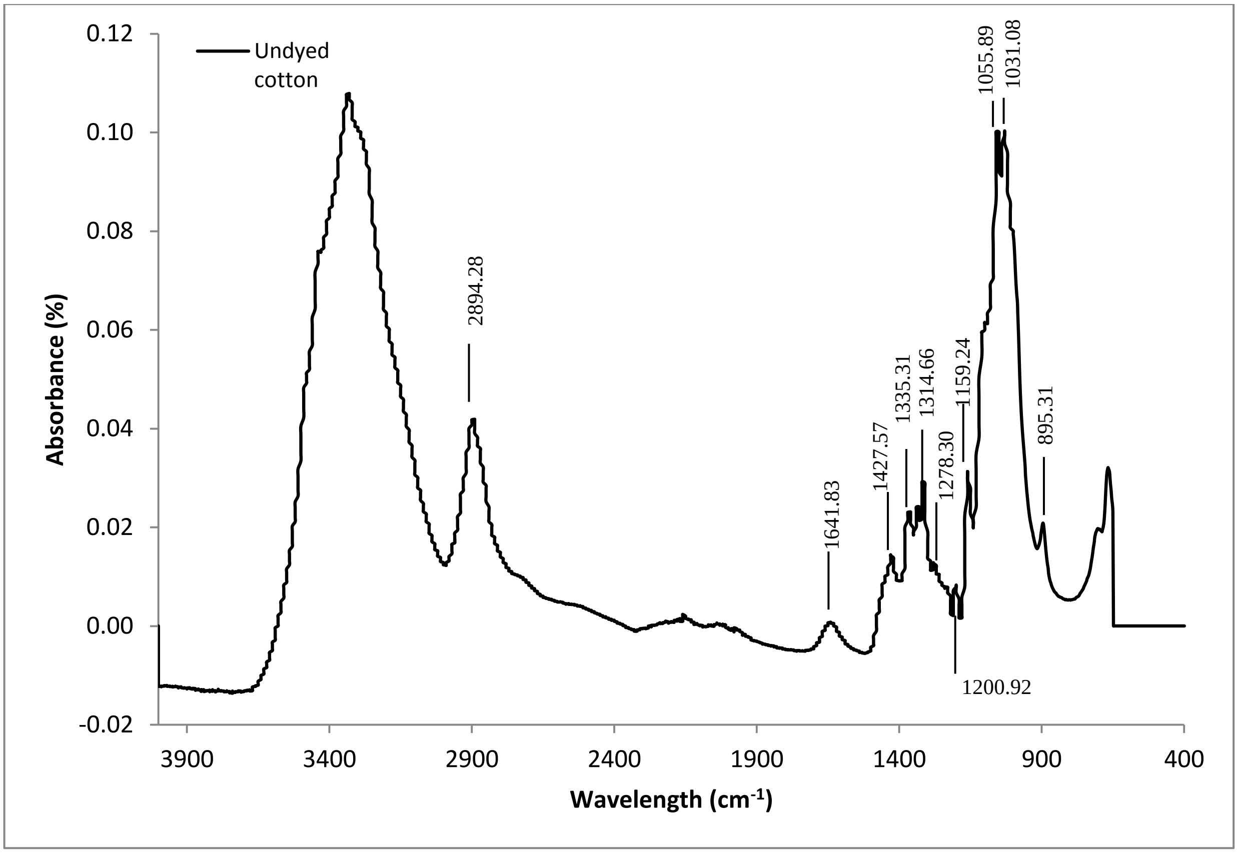


Figure 21. Un-dyed cotton FTIR spectrum.

Compared to un-dyed cotton's FTIR spectrum, dyed cotton FTIR spectra showed a peak shift from 1641.83 cm^{-1} to between 1630.67 cm^{-1} and 1617.97 cm^{-1} depending on mordanting procedure (Figure 22). This shift brings the peak more in line to the 1608.28 cm^{-1} peak in the dye powder. The 1630-1640 cm^{-1} range corresponds with water in cellulose (Garside & Wyeth, 2003) and the peak's intensity increased, suggesting an increase of water absorbed in the cotton. This may imply cotton’s hydrophilicity increases due to the persimmon dye. However, Park et al. (2005) suggest persimmon dye decreases hydrophilicity. The discrepancy of hydrophilicity is not clearly understood yet. Further analysis, including contact angle and surface tension measurements, is warranted for future work to assess persimmon dye’s impact on cotton hydrophilicity. This peak shift was unaffected by laundering and perspiration testing, suggesting

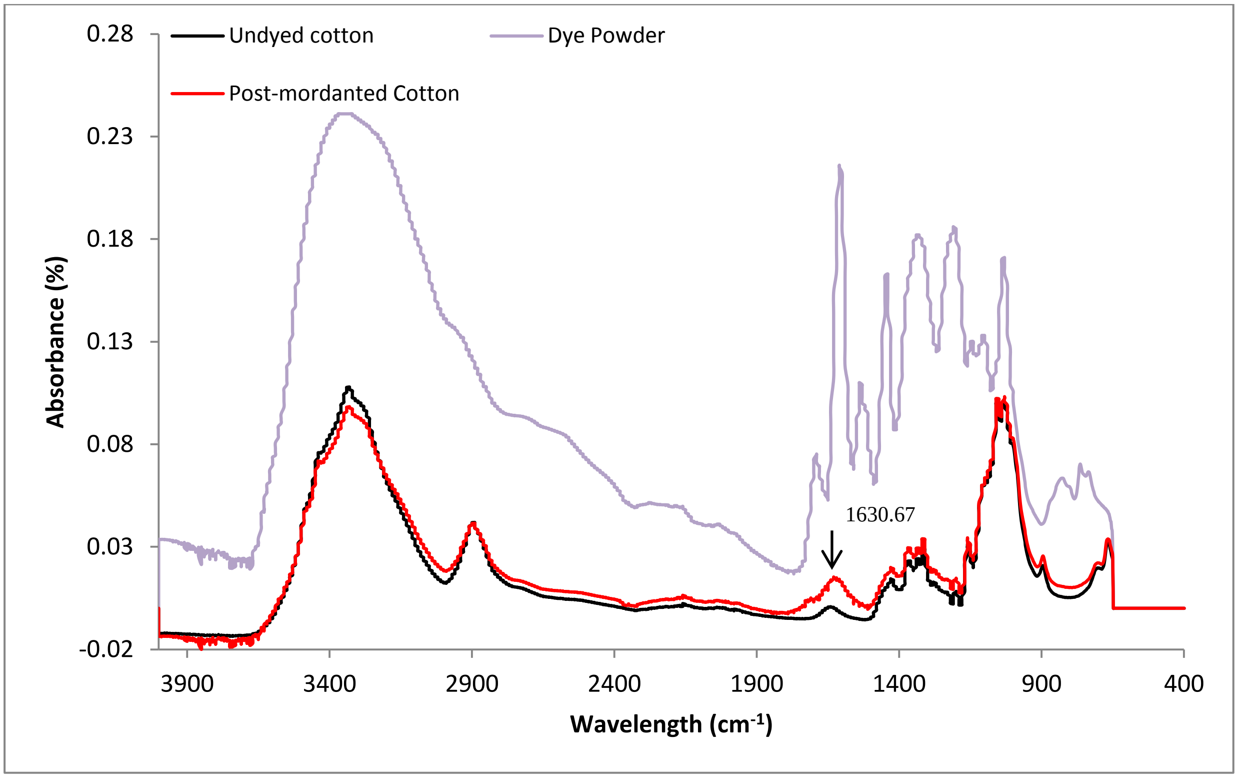


Figure 22. Un-dyed cotton, post-mordanted cotton, and dye powder FTIR spectra.

the change was resistant to those conditions (Figures 23 and 24). Other than the peak shift at 1641.83 cm^{-1}, little chemical structure change took place in cotton (represented by new peaks or peak shifts) during any dyeing procedure, suggesting persimmon dye forms minimal chemical bonds with cotton fabric.

Meta-mordanted cotton's FTIR spectrum is not shown because no significant changes in peak intensity, new peaks, or peak shifts occurred.

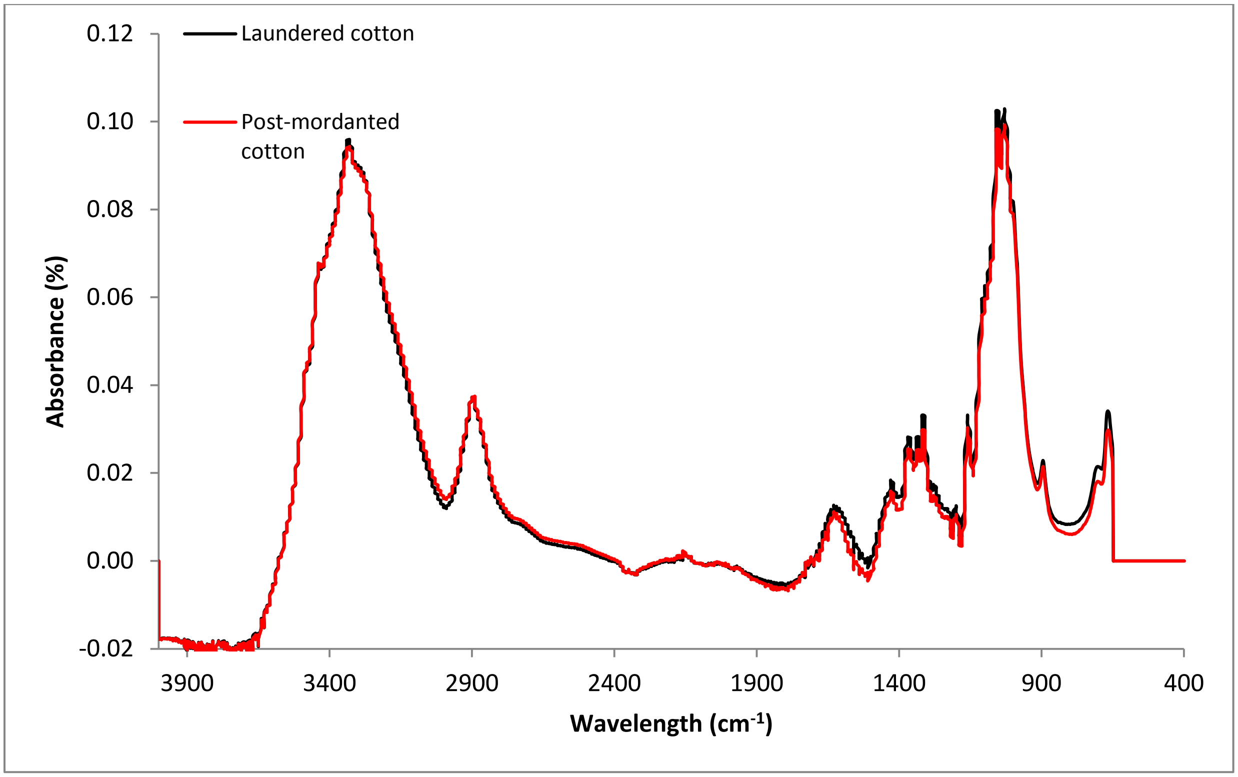


Figure 23. Post-mordanted cotton before and after laundering FTIR spectra.

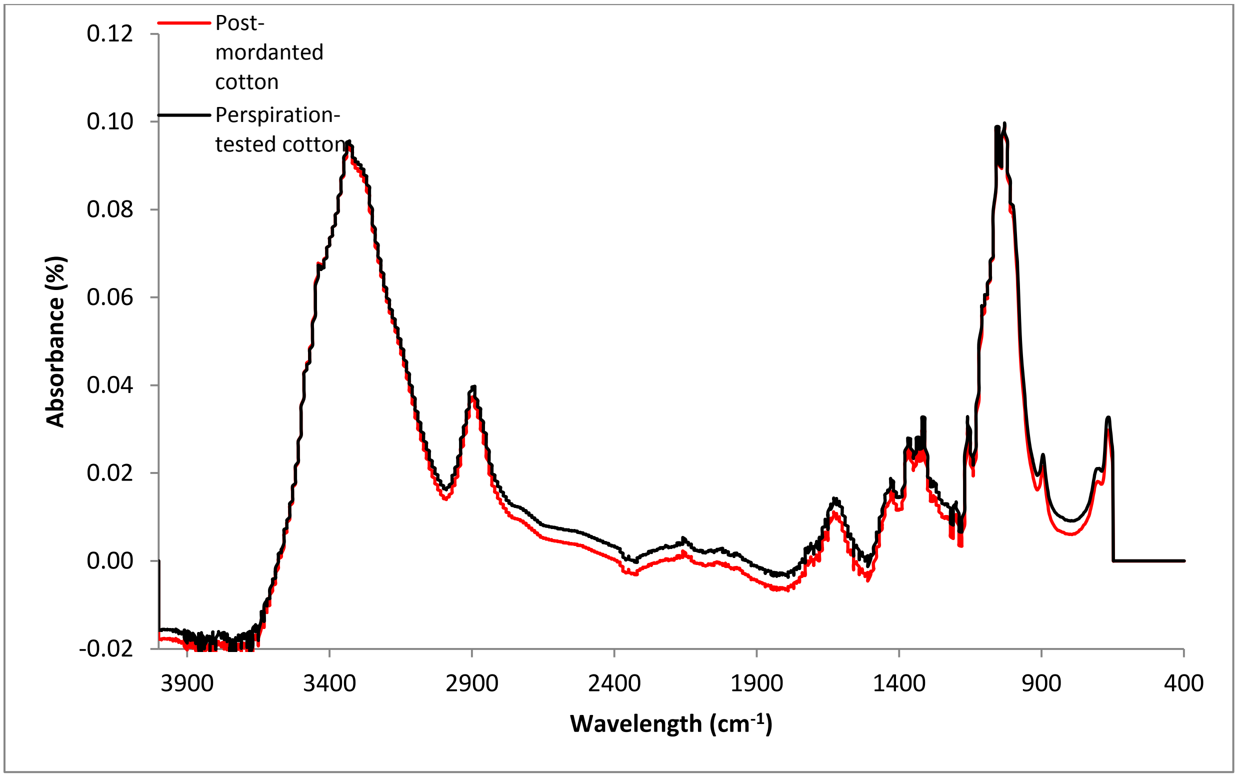


Figure 24. Post-mordanted cotton before and after perspiration testing FTIR spectra.

Wool. Un-dyed wool is shown in Figure 25 and appeared similar to results obtained in previous work as expected (Khan S., Ahmad, Khan M., Yusuf, Shahid, Manzoor, & Mohammad, 2012). The spectrum showed characteristic amide I, II, and III peaks at 1641.82 cm^{-1} (C=O stretching), 1529.81 cm^{-1} (N-H bending, C-N stretching), and 1077.23 cm^{-1} (C-N stretching, N-H bending), respectively (Khan, et al., 2012; Odlyha, Theodorakopoulos, & Campana, 2007). A peak at 1237.19 cm^{-1} is suggested to be CH_2 and CH_3 bending (Li, Hurren, & Wang, 2012). The peak at 1390.84 cm^{-1} is suggested to be from the presence of an amino acid (COO-) (Lipp-Symonowicz, Sztajnowski, & Kulak, 2012).

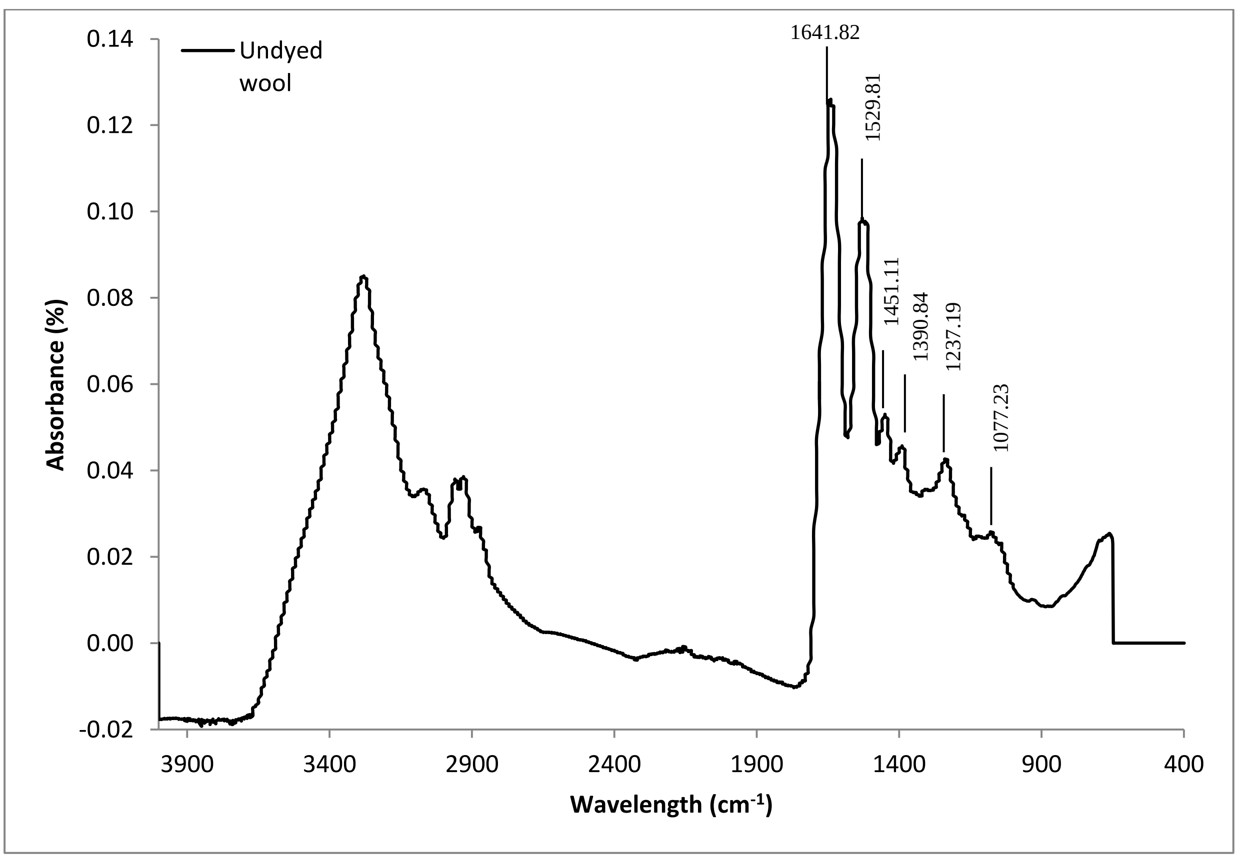


Figure 25. Un-dyed wool FTIR spectrum.

The peaks at 1641.82 cm^{-1} and 1529.81 cm-1 associated with Amide I and II bands decreased in intensity after dyeing as shown in Figures 26 and 27. Interestingly, the peak at 1390.84 cm^{-1} changed intensity depending on the mordanting procedure, also shown in Figures 26, 27, and 28. The peak's intensity on un-dyed wool was .0457 versus .0291 on post-mordanted wool and .0501 on meta-mordanted wool. The decrease in peak intensity on post-mordanted wool is likely due to persimmon dye covering the wool, indicating a persimmon dye layer is present on the fiber surface. A similar intensity decrease in the 1390.84 cm^{-1} peak also occurred when Odlyha et al. (2007) treated wool tapestries with extensive sunlight. This may suggest that this amino acid group is easily covered by the dye. However, the intensity changes did not

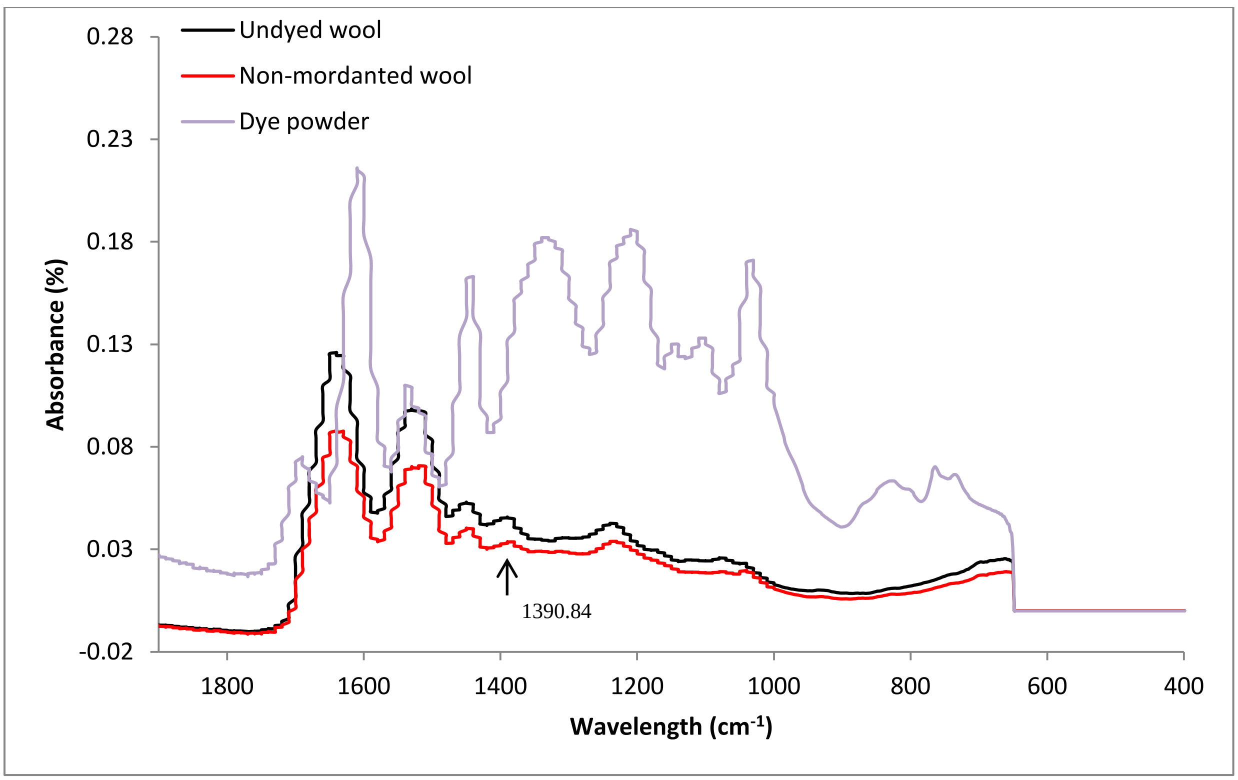


Figure 26. Un-dyed wool, non-mordanted wool, and dye powder FTIR spectra.

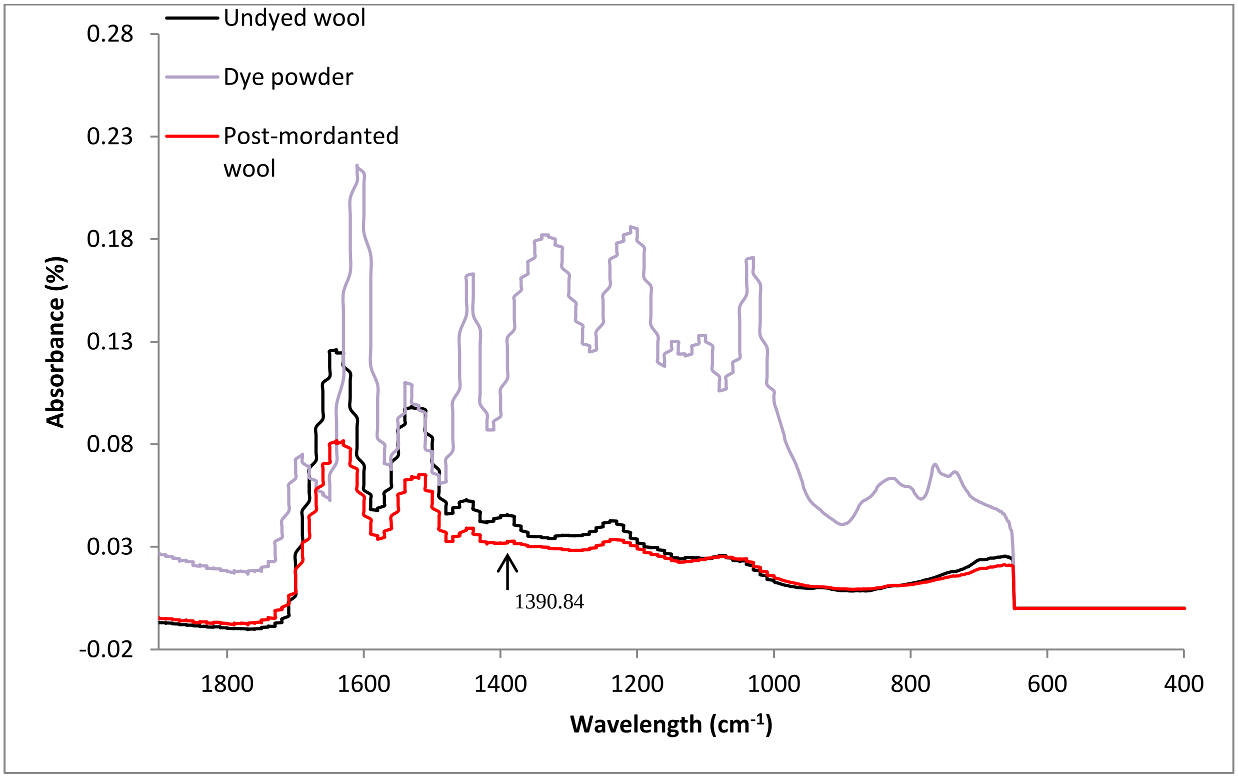


Figure 27. Un-dyed wool, post-mordanted wool, and dye powder FTIR spectra.

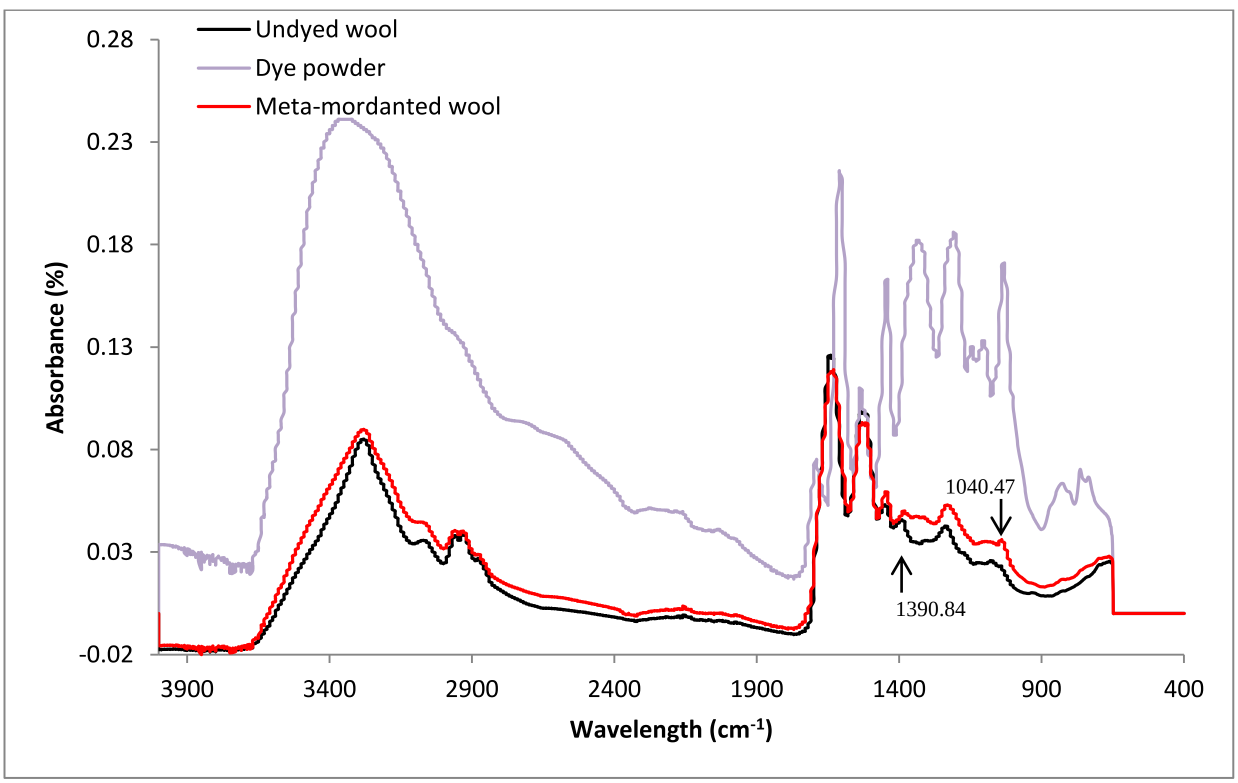


Figure 28. Un-dyed wool, meta-mordanted wool, and dye powder FTIR spectra.

appear to be permanent in this case. After undergoing laundering testing, the peak at 1390.84 cm^{-1}'s intensity returned to .0401 compared to its original .0457 shown in Figure 29. The impermanent intensity decrease at 1390.84 cm^{-1} suggests a weak interaction (Yu & Zhang, 2013). It is important to note that Kourkoumelis, El-Gaoudy, Varella, & Kovala-Demertzi (2013) suggested strong bonds can form when a dye and a fabric show similar peaks on FTIR spectra. Wool has peaks at 1529.81 cm^{-1} and 1451.11 cm^{-1}, which correspond closely to persimmon dye's peaks at 1535.64 cm^{-1} and 1444.70 cm^{-1}. This could help explain wool's greater affinity to persimmon dye compared to cotton, as cotton shared no similar peaks with the persimmon dye.

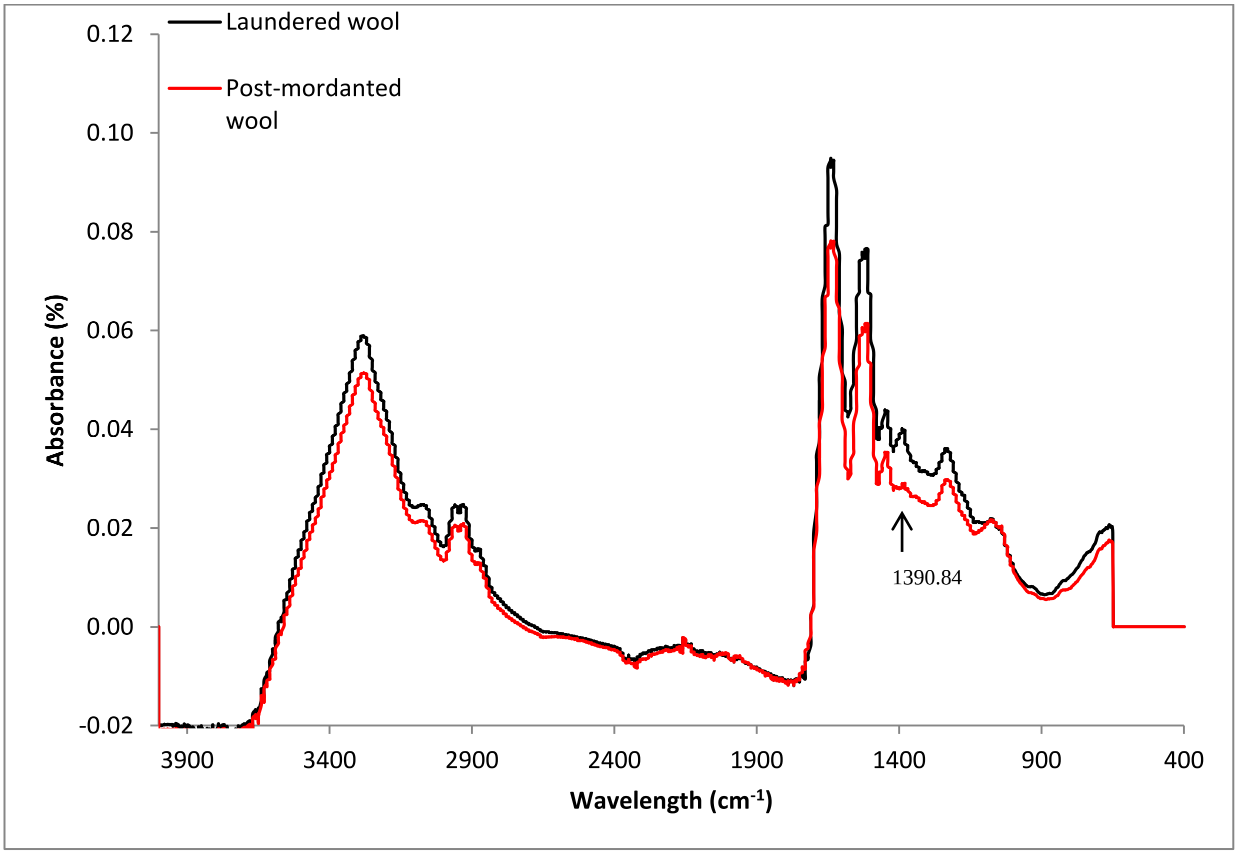


Figure 29. Laundering-tested and post-mordanted wool FTIR spectra.

Meta-mordanted wool (Figure 28) exhibited a peak shift from 1077.23 to 1040.47. A peak near 1040.47 is suggested to be due to the bonding of the ferrous sulfate mordant (Khan et al., 2012). This could explain the appearance of dark spots and change in flexibility as discussed in the previous section and could suggest the ferrous sulfate has chemically bonded to the fabric. As mentioned before, meta-mordanted wool shows a peak intensity increase at 1390.84 from .0457 to .0501. This might be due to the reaction between the persimmon dye and ferrous sulfate.

Perspiration-tested FTIR spectrum is shown in Figure 30. Little difference appeared between post-mordanted and perspiration-tested samples, suggesting perspiration has little chemical effect on the wool samples.

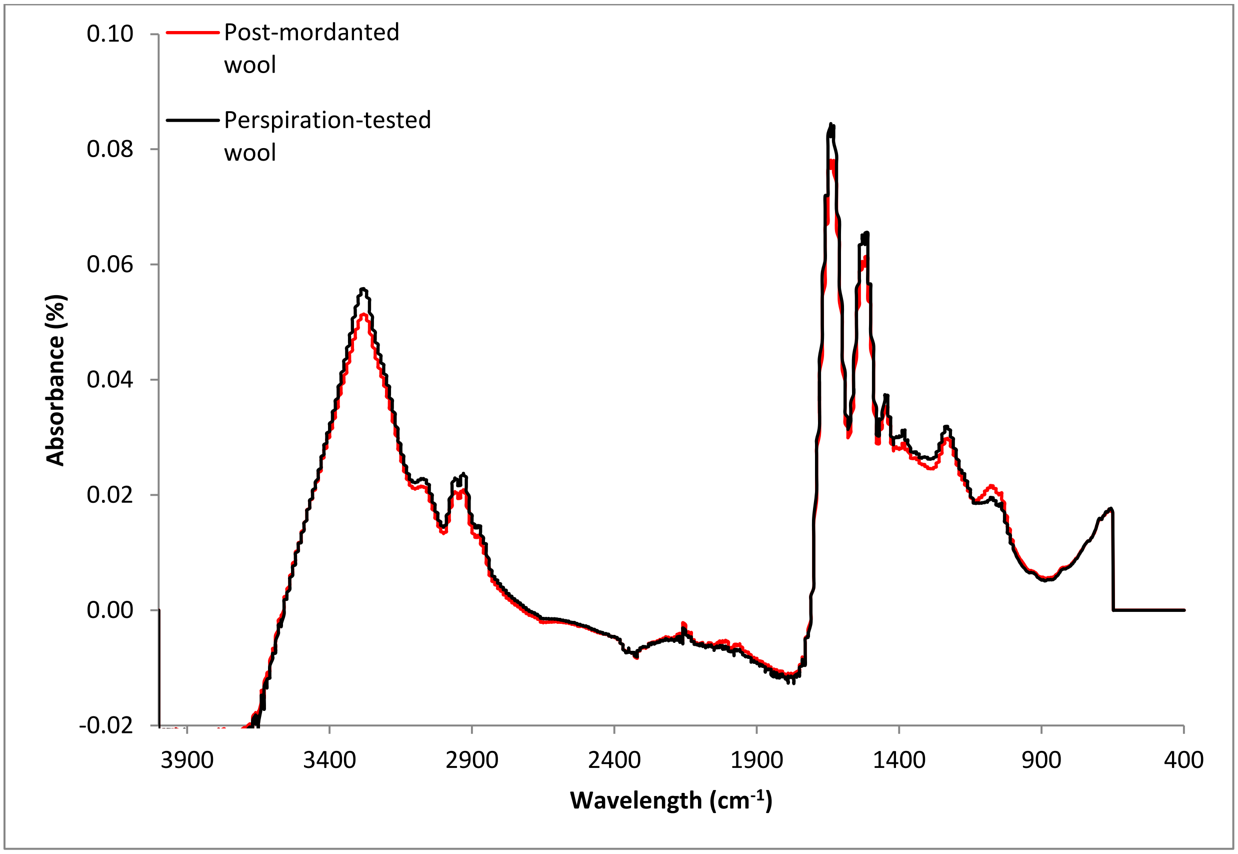


Figure 30. Perspiration-tested and post-mordanted wool FTIR spectra.

Chapter 5: Conclusions

Persimmon dye was applied to cotton and wool fabrics. Mordanting, dye concentration, dyeing length, and colorfastness were studied regarding color strength and chemical changes during dyeing. In comparison to other natural dyes, the study revealed that good color strength can be imparted when using persimmon dye on wool and cotton fabrics, and that persimmon dye has relatively acceptable colorfastness performance (Deo & Desai, 1999; Sarkar & Seal, 2003; Yi & Cho, 2007). Mordanting increased cotton and wool's color strength. Post mordanting produced better dye performance on both fabrics than pre-mordanting and meta-mordanting. Color strength and appearance increased with increased dye concentration and time length for both fabrics. Cotton and wool fabrics dyed using post-mordanting, 200% concentration, and 60 minutes dyeing length yielded the highest K/S for both fabrics (6.9 for cotton and 20.9 for wool) in this study. It is important to note that some mordanting procedures still yielded good color strength at concentrations lower than 200%. Using lower dye concentration may be more desirable in a manufacturing setting as 200% concentration is very high. FTIR analysis on dyed and tested samples showed that the persimmon dye formed weak bonds with both fabrics' surfaces, meaning there was minimal chemical change on the fabrics. Meta-mordanted wool and cotton samples became much stiffer with a rougher hand due to black residue on the fabric surface. This effect was especially severe on the wool. FTIR analysis indicated the stiff hand could be attributed to the iron mordant bonding to the surface of the fabric.

Colorfastness-tested cotton samples exhibited average to above-average color change except in laundering testing where they performed below average. Cotton samples also had above-average to excellent staining performance except in wet crocking where they performed below-average. Wool samples achieved average to excellent color change ratings except in laundering testing where they performed average. Wool samples also had above-average to excellent staining ratings except in wet crocking where they performed average. Color strength and appearance measurements showed that wool samples changed 45% less on average after colorfastness testing than cotton, suggesting wool achieved greater dye performance than cotton. When the fabrics were dyed using post-mordanting, 200% dye concentration, and 60 minutes dyeing length, wool achieved a 201% higher K/S than cotton. This suggests wool is a more suitable fabric to use than cotton when dyeing with persimmon dye.

The current study investigated three dyeing variables' impact on color strength and appearance, whether the dye affected the fabrics chemically, and the dye's colorfastness to laundering, perspiration, and crocking. Through these experiments, the current study met its goal of expanding the knowledge base on persimmon dye and providing a foundational dyeing technique. Future work is suggested as follows:

1. Further studies could be conducted examining dye performance on other cellulosic or protein fibers, like flax, ramie, or silk.
2. Future studies could include more chemical structure characterizations, such as X-ray diffraction, to expand the knowledge of dye adsorption on fibers.
3. Future research could be conducted fermenting persimmon juice to use as dye from fruits at various stages of ripeness to determine which ripeness yields the best color strength, color appearance, and colorfastness properties.
4. Dyeing could be carried out at varied pH levels to determine pH's effect on color strength, color appearance, and colorfastness.
5. A cost and economic feasibility study would help determine whether persimmon dye would be a viable dye for textile production.

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Appendix 1: K/S Values for Pilot Dyeing Samples

Table 6

Non-Mordanted Cotton Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	.52	.67	.97
45	.59	.85	.90
60	.60	.83	1.22[a]

[a]Highest K/S for mordanting procedure

Table 7

Pre-Mordanted Cotton Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	1.3	1.7	1.9
45	1.3	1.7	2.1[a]
60	1.4	1.7	2.1

[a]Highest K/S for mordanting procedure

Table 8

Meta-Mordanted Cotton Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	1.3	1.3	1.2
45	1.4	1.4	1.4
60	1.5	1.6[a]	1.5

[a]Highest K/S for mordanting procedure

Table 9

Post-Mordanted Cotton Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	3.3	5.7	6.0
45	4.4	5.3	6.5
60	4.1	5.2	6.8[a b]

[a]Highest K/S for mordanting procedure
[b]Highest K/S for fabric

Table 10

Non-Mordanted Wool Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	2.5	2.9	3.3
45	3.1	3.6	4.7
60	3.2	4.2	5.0[a]

[a]Highest K/S for mordanting procedure

Table 11

Pre-Mordanted Wool Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	11.3	15.4	19.7
45	14.0	16.8	20.4[a]
60	15.0	18.4	20.2

[a]Highest K/S for mordanting procedure

Table 12

Meta-Mordanted Wool Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	4.3	3.6	4.3
45	5.1	4.2	5.6
60	6.7	7.2[a]	5.9

[a]Highest K/S for mordanting procedure

Table 13

Post-Mordanted Wool Samples' Color Strength in K/S

Dyeing time (in minutes)	Persimmon dye concentration		
	50%	100%	200%
30	10.4	11.6	12.8
45	12.1	14.9	17.6
60	14.4	19.1	20.5[a b]

[a]Highest K/S for mordanting procedure
[b]Highest K/S for fabric

Appendix 2: K/S and L*,a*,b* Values for Colorfastness Testing Samples

Table 14

CIELAB L,a*,b* and K/S Values for Persimmon-Dyed Cotton Before and After Laundering Testing*

	K/S	L*	a*	b*
Cotton before laundering	6.2	39.931	4.338	9.533
Cotton after laundering	7.7	39.403	7.361	12.108
Difference	+1.5[a]	-0.528[a]	+3.023[a]	+2.575[a]

[a]Statistically significant.

Table 15

CIELAB L,a*,b* and K/S Values for Persimmon-Dyed Cotton Before and After Perspiration Testing*

	K/S	L*	a*	b*
Cotton before perspiration	6.7	39.183	4.392	9.543
Cotton after perspiration	5.9	42.793	5.739	11.888
Difference	-0.8[a]	+3.61[a]	+1.347[a]	+2.345[a]

[a]Statistically significant.

Table 16

CIELAB L,a*,b* and K/S Values for Persimmon-Dyed Wool Before and After Laundering Testing*

	K/S	L*	a*	b*
Wool before laundering	16.8	20.032	2.023	0.634
Wool after laundering	18.5	20.644	3.072	2.546
Difference	+1.7[a]	+0.612	+1.049[a]	+1.912[a]

[a]Statistically significant.

Table 17

CIELAB L,a*,b* and K/S Values for Persimmon-Dyed Wool Before and After Perspiration Testing*

	K/S	L*	a*	b*
Wool before perspiration	14.7	21.781	2.252	0.987
Wool after perspiration	15.4	21.896	2.458	1.699
Difference	+0.7	+0.115	+0.062	+0.712[a]

[a]Statistically significant.