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Research Article

Extraction of Natural Dye using Peels of Citrus Fruits for Enhancing Color Fastness of Fabrics

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Abstract: The current study investigates the extraction of dyestuff using citrus fruits and assesses its color, light, rubbing, and perspiration fastness. For the experimental study, three types of citrus fruits—orange, lemon, and grapefruit—were chosen to extract dye from their peels. The dye was applied to two types of fabrics (100% cotton and a blended fabric made of polyester-cotton (PC) with a ratio of 50:50) using the conventional aqueous method. The results revealed the remarkable efficacy of orange dye on both fabric types, demonstrating excellent color fastness attributes, with a minor preference for PC fabric in washing fastness. In contrast, lemon dye displayed better washing fastness properties on the tested materials as well as considerable staining potential. Grapefruit dye performed exceptionally well in terms of water and perspiration fastness. Future research could focus on improving dye extraction techniques for citrus fruits to increase color absorption and penetration. Determining various solvents, time duration for dyeing, and temperature settings could enhance the dyeing performance and effectiveness of natural dyes in different industrial applications. Further studies could also conduct life cycle assessments to measure the environmental impact of using citrus peels as a natural dye source compared to synthetic dyes in the textile industry.

Keywords: Color Fastness, Staining Tests, Agricultural Waste, Citrus Fruits, Natural Dyeing, Textile Sustainability.

1. INTRODUCTION

Nature, without which life would be dull, is completely charming and attractive with its beautiful colors. Color provides us with a unique perception and understanding of how a particular object exhibits or reflects light. The use of these colors as dye is an ancient art form that predates written records. It was used as far back as the Bronze Age, where the methods of dyeing involved placing flowers on cloth and rubbing the pigments onto the surface of fabrics [1]. Some historical dyes included madder, a red-colored dye obtained from Rubia tinctorum, indigo derived from indigo leaves, and yellow extracted from saffron [2]. However, with the development of artificial dyes, the tradition of using natural materials to extract dye significantly decreased, especially after the discovery of the first synthetic dye, mauveine, by William Perkin in 1856. Synthetic dyes were cheaper to produce and available in a wider range of shades. As a result, at the start of the 20th century, natural dyes had been practically removed because of the low-cost artificial dyes and their availability [3]. Synthetic dyes are predominantly used in the fabric industry for silk, cotton, wool, nylon, and leather. However, due to the toxic effects of chemical dyes, their unsafe behavior for humans as well as for the environment revived interest in the use of natural dyestuff [4]. In contrast, natural dyes are considered an environment-friendly source compared to synthetic dyestuff, as these are extracted from renewable means such as plants, insects, herbs, and minerals instead of petrochemicals. This encourages the preservation of natural resources and lowers the carbon footprint connected to the manufacture of textiles [5].

Natural dyes extracted from plants and minerals have long been used by rural communities in textile and clothing production. This practice not only helps them to produce high-quality products but also ensures the safety of the environment [6, 7]. However, it was a challenging task to obtain bright and durable colors. The manufacturing of synthetic dyes in textile and garment industries

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has greatly affected the surrounding environment. In contrast, natural dyes are considered as safer for our surrounding atmosphere and benefit the physical and mental health of all those involved in its production process [8]. Natural dyes are primarily obtained from plants such as madder, indigo, turmeric, henna, and saffron; insects like sepia, kermes, cochineal beetles, and lac; and minerals such as malachite, cinnabar, ferrous sulfate, ochre, lapis lazuli, and clay, without any chemical treatment [9]. In addition to these, many fruits are also used for the extraction of textile dyes that have permanent colorfastness properties, such as pomegranate, berries, most citrus fruits, and stone fruits.

Citrus fruits belong to the family Rutaceous with different forms and sizes such as oranges, citrons, grapefruits, mandarins, lemons, and limes. Furthermore, they have been used as a conventional herbal medicine in various Asian countries like China, Japan, and Korea [10]. Additionally, citrus fruits are a great source of natural dyes from which the dye is extracted and applied to fabrics thus protecting the environment from harmful chemicals and other substances. Oranges are a highly favored fruit and are harvested by more than one hundred million each year worldwide. The orange peel constitutes around 20-30% of the overall weight of the orange, making it a plentiful, affordable, and easily accessible source of biomass [11]. The peel of the orange comprises cellulose, hemicellulose, volatile essential oils, carotenoids, lignin, and phenolic components [12]. The dye extracted from the peels of the orange was used for dyeing many fabrics [13]. Lemons hold agronomic significance due to their versatile use as an ingredient in cooking. To make maximum use of its peel as a plentiful agricultural waste can be extracted as a great source of natural dyes used for textiles [14]. Grapefruit, in all tropical and subtropical regions of the world, is planted with almost 4 million metric tons of annual production [15]. It comprises many water-soluble and insoluble polymers and monomers [16, 17]. Additionally, waste peels of grapefruits are also useful for making sustainable dyestuff for coloring fabrics.

In the present study, natural dyes were extracted from peels of citrus fruits to improve color fastness, brightness, and dyeing effectiveness. The purpose is to develop an eco-friendly dyestuff that helps to reduce the negative impact of synthetic dyes used in the textile industry. Although some previous studies [18] have investigated multiple benefits of using natural dyes, there are certain challenges in improving their color fastness, efficiency, and performance. This study intends to fill this gap by identifying the unexplored potential of citrus fruits as a sustainable natural dying ingredient that can help reduce the environmental footprint.

2. MATERIALS AND METHODS

The experimental study was conducted at the Dyeing and Finishing Unit of Nishat Mills Pvt. Ltd. Three citrus fruits such as orange, lemon, and grapefruit were taken to extract the dye from their peels. The concentration of mordant, dyeing temperature, and time were determined using the conventional aqueous method.

The citrus fruits were collected from the local market. These were then washed gently using tap water and their peels were removed carefully. The peels were then sundried for 3 to 4 days. After complete drying, the peels were ground into powder using an electric beater. Subsequently, 100 g peels of orange, lemon, and grapefruit were taken in separate containers with 500 ml water. The extraction of dye was achieved by boiling the mixture at 80 °C for 1 hour. Then the mixture was cooled down and filtered; the dye extraction process is shown in Figure 1. Fabric samples for dye application were also collected from Nishat Mills. The specifications of these samples are given in Table 1.

Table 1. Construction parameters of samples.

Sample code	Fabric type	Thread count	Weave type	Mass (gm)
A	Cotton -100%	110 × 90	Plain	125
B	Polyester / Cotton Blend (50:50)	76 × 68	Plain	190

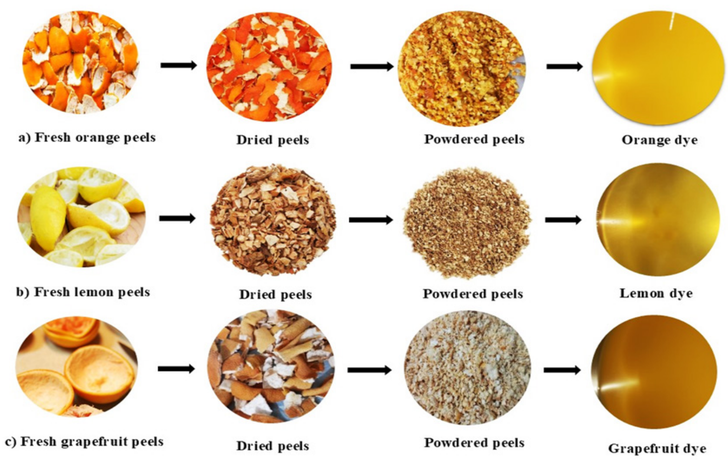

Fig. 1. Process of dye extraction using fruit peels.

The mordant used in this research was copper sulfate. It has been extensively used as a mordant during the dyeing process of cellulosic-based fibers such as cotton and cotton-polyester blends [19, 20]. It can significantly enhance the bonding of dye with the fiber thus increasing the color fastness to water, light, and washing. Moreover, copper sulfate also induces luster and depth to the dyed fabric to develop vibrant shades, as natural fibers tend to fade quickly. An adequate wastewater system was implemented to ensure its proper disposal to prevent environmental degradation. Both types of fabrics were pretreated using a mild detergent to remove the impurities that may affect dye penetration. 20 grams of $CuSO_4$ per liter was used. The pretreated fabric was immersed in the prepared mordant solution and simmered at 80 °C for approximately 45 minutes. A ratio of 1:20 was maintained between the fabric and the dye to ensure even dyeing.

The fabric samples were passed through the padding machine where the machine parameters were carefully controlled. The pressure was set to 1.5 bar which ensured maximum dye penetration. The speed of the machine was set at 7 rounds per minute. The pick-up rate of 80% was kept throughout the whole process to ensure complete saturation of each sample in a dye bath. The extra dye liquor was squeezed off and each sample was dried separately in a drying chamber at 130 °C for 2 minutes. The samples were then washed thoroughly to remove any excessive dye or other impurities from the surface.

Before testing the fabric, samples were conditioned at 65% ± 5 relative humidity and a standard temperature of 20 °C for approximately 24 hours following the guidelines of ISO 139:2005 [21] The fastness of the fabrics against acid perspiration (sweat) was tested using the AATCC 15: 2002 [22]. Specimen with the dimension of 60.3 mm × 60.3 mm was cut from each type of fabric. It was weighed on a weighing scale to record the dry weight of the specimen. A specimen was soaked in the test solution for 30 minutes and occasionally squeezed. After half an hour, it was removed from the solution, passed through the wringer, and reweighed. To start the test, the plexiglass plates were placed in the perspiration tester along with the specimen/multi-fiber strips uniformly spread between 21 plates. 8.0 lb. weight was applied on top making a total of 10.0 lb. under the pressure plate. It was then locked, and the weight was removed. Specimens in the oven were preheated at 37 °C for 6 hours. The multifiber strip from the fabric was separated and placed in a conditioning atmosphere overnight. The color change grade was noted against each grey scale for each of the tested specimens.

The fastness test against alkaline perspiration was conducted according to the ISO-E04:2013 [23]. A specimen measuring 40 mm × 100 mm is sewn with the shorter side of the multifiber adjacent fabric also measuring 40 mm × 100 mm. Then the specimen was soaked in an alkaline solution of pH 8 (±0.2) with a liquor ratio of 50:1 at room temperature for half an hour. It was pressed from time to time to remove excess water. The specimen was placed between two glass plates under a nominal pressure of 12 kPa and placed in a tester to preheat. Using the same method, the test specimen was wet in the acid-based solution at a pH of 5.5 and then tested in a pre-heated testing oven. Specimen was placed in it for 4 hours at 37 °C, aligning it so that the specimens were either in vertical or horizontal direction. A specimen was opened out removing the stitching except at one of the short sides and dried it at a temperature of 60 °C, with only two parts in contact at the line of stitching. The change in color and shade of each of the tested specimen and the staining on the adjacent fabric swatches were assessed against grey scales.

The light fastness test was performed by AATCC Lightfastness Standard 16: 2004 [24]. The fabric was cut at least 70.0 mm × 120.0 mm while the exposing area was only 30 mm × 30 mm. The fabric was then exposed for 20 operating hours at the standardized temperature and humidity. Measured the exposed standard specimen, either visual comparison (if the changed color equaled the Xenon Reference Fabric of Fade in 20 continuous light-on operating hours then the test equipment was maintaining the correct temperature) or Instrumental Color Measurement (If the exposed standard specimen equaled 20 ± 1.7 CIELAB units of color change in 20 ± 2 continuous light-on operating hours, the test machine was providing the correct temperature). If the exposed Reference Fabric differed through visual inspection or instrument testing, after 20 light-on operating hours, it was a sign that temperature sensing units within the test chamber were not responding accurately and need readjustments.

Two fabric pieces of 50 mm × 140 mm were used for rubbing in dry and wet conditions. The specimen was tested according to ISO 105-X12:2016 [25]. The conditioned rubbing cloth was placed flat over the end of the finger with the weave parallel to the direction of the rubbing finger. At a rate of one cycle per second, rubbing was made in to and fro in a straight line for 20 times, 10 times to and 10 times fro, along a track of 104 mm long on dry specimen, with the downwards force of 9 N. Any unnecessary fibrous or other material was detached before evaluation, that might impact the results. Rubbing cloth was conditioned and thoroughly soaked in distilled water and reweighed to ensure take-up of 95% to 100%. The procedure was similar to that used for dry rubbing. The specimen was then air dried. Each test cloth was backed with three layers of rubbing cloth in white color while measuring the fastness. The gray scale was used to assess the staining of cotton. The numerical rating of each of the tested specimens was made against a grey scale.

The fabrics were evaluated for their color against water [26]. A specimen of (60 ± 2) mm × (60 ± 2) was cut using a template. It was then immersed in a prepared test solution for an hour and squeezed between the rollers to remove excess liquor. The process was repeated to achieve thorough wetting. The specimen was placed in a perspiration tester with a pressure of 9.9 lb. The heat was provided at 38 °C for 18 hours in an oven. It was then removed and dried at room temperature. It was evaluated for its color change against the grayscale.

The fabrics were evaluated for their color against washing [27]. A specimen (50 × 100 ± 2 mm) was cut using a template. The laundering machine was adjusted according to the instructions given in the test procedure. The specimen was immersed in a standardized washing solution. It was placed in a canister that was sealed and agitated in the washing solution for half an hour at 40 °C. After thorough agitation, the specimen was rinsed to remove any excess liquor and dried at room temperature. It was also evaluated for its color change against the grayscale.

3. RESULTS AND DISCUSSION

Three types of dyestuff extracted from orange, lemon and grapefruits were tested on cotton and a blend of Polyester and Cotton (PC) using copper sulfate as a pre-mordanting technique. The resulting color of the dyes is given in Figure 2. The tests for color fastness by washing, perspiration, rubbing (wet and dry), and light fastness, were performed on cotton (A) and PC (B) fabrics. The results are shown in Table 2.

Table 2 depicts that the orange dye exhibited poor washing fastness properties on cotton fabric, but its performance was slightly improved on PC fabric. One of the possible reasons is that cotton, being a natural fabric, tends to retain water, potentially leading to dye bleeding due to weaker chemical bonds. In contrast, PC fabric, being synthetic, is less absorbent and possesses stronger chemical bonds, contributing to its resistance to fading. A study showed that a blend of cotton

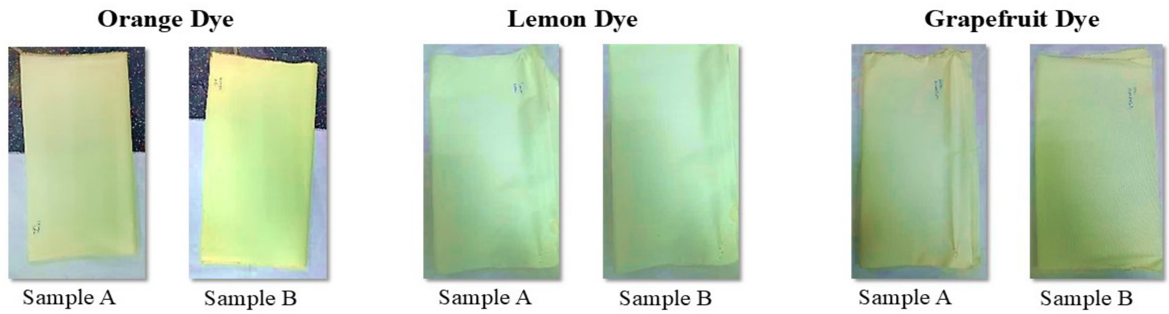

Fig. 2. Fabric dyed with orange, lemon, and grapefruit.

Table 2. Colorfastness of fabrics for each dye.

Sample	Dyestuff	Washing fastness	Water	Light fastness	Rubbing fastness		Perspiration fastness	
					Dry	Wet	Acid	Alkali
A	Orange	1-2	4	4	4	4	4	4
B	Orange	1	4	4	4	3-4	4	4
A	Lemon	0	4	4	4	3-4	4	4
B	Lemon	0	4	4	4	3	4	4
A	Grapefruit	0	4	3	4	3	4	4
B	Grapefruit	0	4	4	4	3	4	4

and polyester had better dyeability compared with 100% cotton fabrics when dyed using henna and onion against wash fastness, light fastness, acid, and alkali perspiration [28]. Notably, other fastness properties such as light fastness, rubbing fastness, and perspiration showed good results for the orange dye on both tested fabrics.

On the other hand, the results of lemon dye on both cotton and PC fabric showed good color fastnesses against rubbing, light fastness, and perspiration tests except for washing fastness. This was attributed to the dye's inability to form a strong bond with the fibers or its poor affinity for the cloth. Conversely, the study investigated the fastness and staining properties of grapefruit dye on both fabrics. The results showed that this dye had good color fastness properties against perspiration and water tests but had poor washing fastness properties for both fabrics. The obtained data showed that the orange dye had excellent results than dyes extracted from lemon and grapefruit. Furthermore, PC fabric presented better dye intake than cotton fabric due to its stronger chemical bond.

Figure 3 elucidates the comparison between samples A and B with the contrast between orange, lemon, and grapefruit dyes for the fastness tests of washing, light, rubbing, and perspiration. A descriptive analysis was made among different fruit peels used to extract dye for their colorfastness. The maximum and minimum range for each of the scores was determined and the results are depicted in Table 3.

Table 4 shows that the staining tests for the orange dye had good to excellent properties and can be used in a wide range of fabrics and other textile applications. The staining results, on the other hand, showed that the lemon dye could be applied to many fabrics with minimal variations in staining capability. The staining potential of grapefruit was moderate for all samples except for cotton in acid perspiration. Figure 4 elucidates the comparison between samples A and B with the contrast between orange, lemon, and grapefruit dyes for the staining tests along with standardized fabrics.

Fig. 3. Comparison of colorfastness of dye.

Table 3. Statistical analysis of colorfast test.

Peels	Orange		Lemon		Grapefruit	
Range	Max	Min	Max	Min	Max	Min
Fabrics	A	B	A	B	A	B
Washing	1-2	1	0	0	0	0
Water	4	4	4	4	4	4
Light	4	4	4	4	4	3
Rubbing (dry condition)	4	4	4	4	4	4
Rubbing (wet condition)	4	3-4	3-4	3	3	3
Perspiration (in acid)	4	4	4	4	4	4
Perspiration (in alkali)	4	4	4	4	4	4

A previous study by Li et al. [29] has investigated that the cotton fabric dyed with an orange peel has low rubbing fastness properties both in dry and wet conditions against tannic acid used as a mordant. Another research by Taura et al. [8], in which cotton was dyed with an orange peel dye using NaOH mordant indicated that rubbing and sunlight had no significant effect on cotton fabric, but washing had increased the paleness in the overall color of the fabric. To achieve good fastness properties especially wash fastness future studies can be conducted by using NaOH and Tannic acid as mordant as they proved good mordant in the fixation of dye. A descriptive analysis was made among different fruit peels used to extract dye for their staining test. The maximum and minimum range for each of the peel are presented in Table 5. Compared to the synthetic dyes, natural dyes are eco-friendly, sustainable, non-toxic, biodegradable, and non-allergic when treated with turmeric and neem dye solutions [30]. Suri et al. [31] investigated the benefits of citrus fruit waste, highlighting their potential for recovering bioactive compounds, pectin, biofuels, essential oils, dyes, and micro and macro-nutrients. Devi and Saini [32] have also highlighted the application of an orange peel in textile industry, using it as a source of natural dye, waste absorbent, antimicrobial agent, mosquito repellent, sustainable fiber, and perfume retention agent. Taura *et al.* [8] have reported that orange

Table 4. Staining test of each dye and fabric.

	Orange								Lemon								Grapefruit							
Fabrics	Wash		Water		*ALKP		**ACIP		Wash		Water		ALKP		ACIP		Wash		Water		ALKP		ACIP	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Acetate	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Cotton	4	4	4	3-4	4	4	4	4	4	4	4-5	4-5	4	4-5	4	4-5	4	4	4	4	4	4	3-4	4
Nylon	4-5	4-5	4-5	4	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Polyester	4-5	4-5	4-5	4	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Acrylic	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Wool	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5

Note: *ALKP = Alkaline Perspiration, **ACIP = Acidic Perspiration

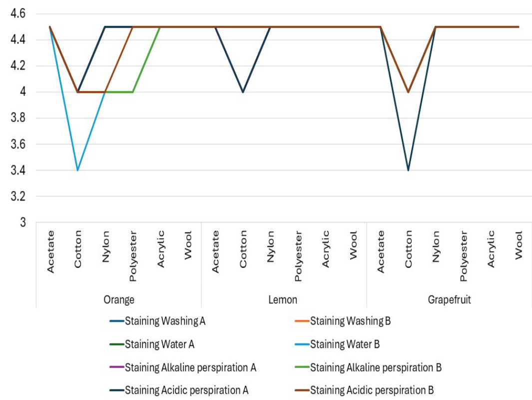

Fig. 4. Comparison of staining test.

peel is the most effective part of a plant for color extraction. It also promotes the use of sustainable and eco-friendly materials in the textile industry for various applications.

4. CONCLUSIONS

From the results of the present study, we can conclude that orange dye is more effective on both types of fabric and exhibited excellent color fastness characteristics, except for washing fastness, which slightly favored PC fabric. It demonstrated adaptability across various fabrics. On the other hand, lemon dye showed better overall fastness qualities and considerable staining potential, although it had slightly weaker washing fastness on the tested fabrics. Grapefruit dye performed poorly in washing tests but showed good results for water and perspiration fastness. These collective results highlight the potential of citrus fruits as a viable and natural source of dyes for fabrics. The findings of the study can be attributed to the high concentration of natural colorants in citrus fruits, such as flavonoids and carotenoids, which aid in the quick adherence of dyestuff to textile fibers, making them useful for various applications. The use of fruit peels as coloring agents in the textile industry holds significant potential due to their abundance, cost-effectiveness, and eco-friendly nature. Their application in other industries, such as cosmetics, food packaging, and the leather sector, could help reduce industrial waste and promote a circular economy.

5. CONFLICT OF INTEREST

The authors declare no conflict of interest.

Table 5. Statistical analysis of staining test.

Peels	Orange		Lemon		Grapefruit	
Range	Max	Min	Max	Min	Max	Min
Fabrics	A	B	A	B	A	B
Acetate	4-5	4-5	4-5	4-5	4-5	4-5
Cotton	4	3	4-5	4	4-5	3
Nylon	4-5	4	4-5	4-5	4-5	4-5
Polyester	4-5	4	4-5	4-5	4-5	4-5
Acrylic	4-5	4-5	4-5	4-5	4-5	4-5
Wool	4-5	4-5	4-5	4-5	4-5	4-5

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