## Dyeing of Egyptian Cotton Fabrics with Orange Peel Using Padding Technique

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#### Abstract:

The extraction from orange peel using the pad-dry-technique has been used as natural dye for cotton fabrics. In the current study, fabrics made from extra long Egyptian cotton of Giza 93 were dyed with the color extracted. The study was conducted in progressive stages to obtain how orange peel was to be used as the selected natural dyes. The factors affecting the fixation of the extracted dye with cellulose was studied. The treated and untreated fabrics samples were tested for their mechanical properties expressed as tensile strength (kg/force) and elongation%. Dyeing performance in terms of color parameters (K/S, L\*, a\*, b\*, Chroma, hue and  $\Delta E$ ), and fastness properties (wash, perspiration, and light fastness) were studied. The samples show high tensile strength, high color strength, and high fastness properties. These results are very important for industrial application with the production of a natural dye as an inexpensive source from orange peel as a by-product. The ideal padding parameters for a given dye concentration, improving the dye uptake may be employed, potential for additional benefits from such improvement techniques and the feasibility of a coloring process that uses chemicals with a lower environmental impact as compared to current practices.

Keywords:

- Orange Peel By-Product,
- Egyptian Cotton,
- Padding Technique.
- Color Components,
- Fastness Properties,
- Mechanical
- **Properties**

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#### Introduction

Natural dyes are known for their use in coloring of food substrate, leather, wood as well as natural fibers like wool, silk, cotton and flax as major areas of application since ancient times <sup>1</sup>. Natural dyes may have a wide range of shades, and can be obtained from various parts of plants including roots, bark, leaves, flowers, and fruit <sup>2</sup>. The use of natural dyes having poor to moderate wash and light fastness has declined to a great extent. However, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to worldwide environmental consciousness <sup>3</sup>.

Calls for the use of natural dyes on textiles have been just one of the consequences of increased environmental awareness <sup>4</sup>. The use of natural dyes for the coloration of textiles has mainly been confined to craft dyes and printers. Recently, more interests have been devoted to the use of these dyes and a limited number of commercial dyes and small business have started to look at the possibilities of using natural dyes for dyeing and printing of textiles. Natural dyes are less toxic, non pollutant, less health hazard, very brilliant, rare color idea, and allergic reactions <sup>5</sup>.

The use of non-toxic and eco-friendly natural dyes on textiles has become a matter of significant importance because the of increased environmental awareness in order to some hazardous synthetic avoid dves. However, worldwide the use of natural dyes for the coloration of textiles has mainly been confined to craftsman, small scale dyers and printers as well as small scale exporters and producers dealing with high valued ecofriendly textile production and sales <sup>6</sup>.

Carotenes are found in, for example, carrots, red peppers, oranges and tomatoes. They are also responsible for the color of most yellow and orange flowers. Most carotenes are hydrocarbons though some also contain oxygen atoms. The most well-known is often simply called carotene and a group of compounds called carotenes <sup>7</sup>. All carotenoids are based on this carotene structure. The central carbon chain remains the same, but the groups at either end differ slightly. The central chain consists of 18 carbon atoms bonded alternatively with single and double covalent

bonds with four methyl groups attached (always in the same positions).

The sequence of alternative single and double covalent bonds is called a conjugated system. The lack of hydroxyl groups makes  $\beta$ -carotene hydrophobic. Due to its two cyclic rings at each end of the molecule chain,  $\beta$ -carotene is a dicyclic compound, composed of 8 isopreneunits (C<sub>5</sub>H<sub>8</sub>). The high amount of conjugated double bonds is called chromophor and is responsible for the color impression <sup>8</sup>. Bcarotene absorbs light of the wavelength 450 nm of the visible part of the spectrum <sup>9</sup>.

Main compounds in orange peel are  $\beta$ carotene,  $\alpha$ -carotene, lutein and zeaxanthin <sup>10</sup>. Additionally orange peel contains flavonoids, phenolic acids, pectin and waxes<sup>11</sup>. The compounds in orange peels extracts are by some researchers tested on their UVprotective properties and antimicrobial activity <sup>12</sup>. Different extraction methods for  $\beta$ carotene (carotenes) from orange peel, carrots and other fruits, vegetables and plants are described. The most common method is the organic solvent extraction. It was described by several different researches for the extraction of  $\beta$ -carotene from orange peel<sup>13</sup>.

The padding technologies are particularly advantageous to dyeing with the low-affinity products, because the dye affinity to fiber by padding is unnecessary (in phase of the dye deposition on the fabric)<sup>14</sup>. The dye bath is cloth "padded": mechanically applied by the rapid passage through the small padding trough, the intensive squeezing between expression rollers follows immediately. The process of padding is continuous and very rapid. It depends on the arrangement of the following dye fixation if the total procedure is continuous or semi continuous. The dye bath by padding is about one order higher than by the common dyeing from the "long bath" (the so-called exhaustion methods), in which the dyestuff exhausts on the fiber in consequence to its affinity to the fiber. The higher padding bath concentration results in more rapid dye diffusion in fiber during the next fixation operation. Much smaller bath volume (related to the fiber unit) causes the higher dye exploitation<sup>15</sup>.

The application of padding technique on cotton was achieved by Saminatahn  $R^{16}$ . The pad-batch dyeing process is the most economical of all pad dyeing processes for the reactive dyeing of cotton. It is claimed that this process is more economical than exhaust dyeing mainly due to minimal energy requirements <sup>17.</sup>

The main target of this investigation was to dye the Egyptian cotton fabric made from the Extra long stable (ELS) Giza 93 with Orange byproduct using padding technique.

#### Materials And Methods Materials:

The residue of orange peels was from the local juice company Juhayna Company, 6th of October, Egypt. Bleached cotton fabrics with the same structure of ELS Egyptian cotton variety Giza 86 were purchased from El-Nasr for weaving and spinning Company- El-Mehala, Egypt, and used throughout this study. The properties of Giza 93 were measured in Cotton Research Institute (CRI) labs by HVI spectrum instrument with the following specification: Color: Creamy, RD% 67.5, Yellowness (+b) 11.6, Micronaire value 3, maturity ratio (MR) 0.89, Fiber length (UHM) 36.8mm, Uniformity index (%)8 7.4, fiber strength (g/tex) 47.6, elongation (%) 6.7, fiber perimeter( µ) 42.4, cross section area  $(\mu^2)$  135 and total reducing sugars (%) 014.

# Padding mangle

Padding was carried out on a laboratory-scale two-bowl padding mangle, with horizontal squeeze-roller geometry, manufactured by Werner-Mathis.

## Steamer

A laboratory-scale steamer manufactured by Werner Mathis was employed for the steaming of samples. The equipment featured a fully-contained steam atmosphere with temperature and humidity controls. The period of sample exposure was controlled using an integrated timer. In addition, this equipment could be used as a 44 curing chamber by excluding steam. Samples were mounted on pins set in a frame and then introduced into the steaming chamber.

#### Dryer

An Electrolux TS 560 forced-air-flow convection drying chamber was used to dry the samples. The samples were dried at either  $40^{\circ}$ C or  $65^{\circ}$ C in a tension-free state. Uniform airflow minimized shade differences between the face and back of the fabric.

### Chemicals

All chemicals used were of analytical grade using doubly distilled water (18.5 M $\Omega$ .cm-1). NaOH was analytical grade (Koch-Light Co.), Hydrogen peroxide (30% LR grade) from Aldrich. Sodium carbonate (LR grade), sodium silicate (136° Tw, 27% SiO2), Sodium alginate, and potassium sulfate supplied by Merck. Propane and hexane was HPLC grade.

#### Methods:

#### Drying of the orange peel residue

The orange peel was dried under vacuum with a vacuum pump RV12 from Edwards of 200V before extracted. For drying desiccators silica with silica gel was used. The drying was done to remove the water from the residue to make sure that only the residue without any water was used for the further extraction. The samples were dried in a dark place to prevent degradation of the  $\beta$ -carotene due to light exposure. The drying was continued until the dry matter weight was not changing anymore to be sure all moisture was removed from the samples. To achieve a higher surface area the dried orange peel was ground in a porcelain mortar after the drying.

#### Extraction of natural dye from orange peel

Carotenoids were extracted from fresh orange peel with propanone according to the method described by Aravantinos-Zafiris, et al <sup>19</sup>. Two successive extractions with propanone after an initial washing with propanone were adequate to remove 89% of the total carotenoids. The extracts were concentrated, then carotenoids transferred to hexane and a crude pigment hexane concentrate was obtained by evaporation. Water washings prior to propanone extraction eliminated the solventsolvent transfer to hexane. The extraction residue was used for pectin recovery. Carotenoid removal from the peel did not affect the yield and quality of the pectin.

#### Padding technique

The extracted solution of Orange peel was padded individually on cotton fabric. The squeeze roll pressure was set to achieve 80% wet pick-up. In order to obtain a nominal 1% depth of shade, the dye liquor for padding was prepared by using the extracted dye 15g/l, sodium alignate 2g/l, and sodium sulphate Freshly prepared liquor was allowed to stand for 2–3 hours for the sodium alginate to swell and become completely effective.

#### Steaming and washing off:

Steaming was conducted at 100°C and 100% RH for 10 minutes, and followed by drying at 65°C. The dried samples after padding were washed off (soaped) with 1 g/l anionic detergent solution at the boil for 20 minutes to simulate extreme washing conditions. This was followed by consecutive rinses in hot and cold water respectively.

#### Measurements:

Dyed samples were conditioned for at least 24 hours under standard conditions of 65% RH and  $20 \pm 2 \circ C$ . color measurements were carried out using the double beam spectrophotometer (Perkin Elmer Company -USA, of model Lambda 35) using the CIE system to evaluate the shades obtained on scoured and scoured and bleached dyeing cotton. The color strength (K/S value), intensity or saturation of color (Chroma C), Hue angle (H) is derived from the two coordinates a\* and b, L\*, and color difference  $\Delta E$  was measured.

The tensile strength (kg/force) and elongation (%) were measured according to ASTM D412-98a using Zwick testing machine of model Z010 and equipped with 10Kn load cell and the testing was conducted at speed of 100mm/min. The results obtained were based on an average of ten tests in the wrap direction of each sample.

Wash fastness was evaluated according to the AS 2001.4.15-2006 test method using an Atlas launder-o-meter. The color of the wash solution, staining of adjacent multi fiber fabric and dyed fabric color change were evaluated against AATCC grey scales for color change and staining. Light fastness was tested according to the AS 2001.4.21-2006 test

method for a period of 48 hours. Wet and dry rubbing fastness of samples was evaluated according to the AS 2001.4.3-1995 test method using a crock meter. Dyed samples were rubbed with a standard white cotton fabric, in a dry or wet state, under specified test conditions. The staining of the cotton fabric was evaluated using standard AATCC grey scales for staining.

#### **Results and Recommendations**

B-carotene is a natural yellow-red colored pigment with the chemical structure  $C_{40}H_{56}$  as shown in Figure 1.



Figure 1. B-carotene structure **Evaluation of color components** 

The results for the colorimetric measurements of cotton weave fabric dyed with  $\beta$ -carotene dyestuff of orange peel residue are given in table 1. The measurements were as well done by the CIE color system. The shade of orange peel extract dyed on cotton fabric was greenish yellow. The cotton fabric was evenly dyed. The color uptake ( $\Delta E$ ) was for the scoured samples slightly higher than for the scoured and bleached samples. The presence of some non-cellulosic material in the scoured samples increased the absorption of the dyestuff molecules on the cotton fabric. The color strength K/S is higher for the scoured samples. The scoured samples were compared with the scoured and bleached samples slightly lighter due to their higher L\* value. The C\* values for color saturation were for the post scoured and bleached samples little lower, which means the color saturation decreases with bleaching. Considering both values L\* and C\* the shades of both scoured and scoured and bleached samples cotton fabric dyed with orange peel extract are light and weak in shade. The slight negative values of a\* and the more positive b\* values indicate shades of greenish yellow and slightly more greenish and less yellowish for the scoured and bleached samples.



 Table 1: Color coordination of the dyed cotton fabrics with orange peel extract

# Evaluation of the tensile strength and elongation

The results obtained from Figure 2 revealed that fabric tensile strength and elongation% for the  $\beta$ -carotene dyestuff of orange peel

residue for the scoured Giza 93, (raw material) had higher tensile strength among the scoured and bleached samples. This is due to the damage effect of the scouring and the bleaching materials on the cellulosic chain of the fabrics. More concentration of the orange peel extracts increased the tensile strength for the two fabrics due to the formation of cross linking with the free hydroxyl ions of cotton causing orientation of the cellulose chain. On the other hand, the scoured and bleached samples had higher elongation% than that of the scoured sample. This is due to the damage effect of the scoured or the bleached agents on the elongation% of the fabrics.

#### **Evaluation of washing fastness**

The results for the color change of scoured and scoured and bleached samples dyed with β-carotene dyestuff of orange peel residue were of grade 1-2 as shown in table 2. Most of the color was washed away by the washing test. The samples were partially colored after the test in much lighter and weaker color saturation. Almost no color was left on the cotton samples after washing at 60°C for 30 min. whereas the color change gave poor results, the staining results were with the grades 4-5 for all samples very good. The poor results for color change of the washing test is interpreted by the lack of functional groups and the relative small molecule sizes of the  $\beta$ -carotene dye and their consequential low affinity towards the textile fibres<sup>16</sup>. A possibility to improve the wash fastness properties could be the increase of dye concentration<sup>20</sup>.

Table	2.	Washing	fastness	of	Giza	93	dyed
with o	ran	ige peel ex	tract				

	Wash 60°C	
	Gray	Stain
S- Samples	2	5
S+B- Samples	1	4

As shown in Table 1

# Evaluation of light fastness

The light fastness of the scoured samples was better than that of scoured and bleached samples as shown in table 3. The results for  $\beta$ -carotene dyestuff of orange peel residue were reasonable, with the grade 4 for scoured and 3 for scoured and bleached samples. Scoured samples of  $\beta$ -carotene dyestuffs were compared with their scoured and bleached counterparts each with 1 grade

better. The poorer results of the light fastness for  $\beta$ -carotene dyestuff of orange peel residue could be interpreted due to the high sensitivity of  $\beta$ -carotene in light exposure and its higher and purer amount in this dye<sup>19</sup>. In its natural form  $\beta$ -carotene is higher concentrated and it is surrounded by proteins that can keep it more stable<sup>19</sup>. Another reason is the small molecule size and the resulting lower affinity towards the cotton fibers <sup>21</sup>. As shown in Table 1

Table 3. Light fastness of Giza	93 dyed with				
orange peel extract					

U1	Light fastness
S- Samples	4
S+B- Samples	3

## CONCLUSION

The use of solution from orange peel as natural waste source to dye Giza 93 Egyptian cotton fabric has been achieved. The treated and untreated fabrics samples were tested for their mechanical properties expressed as tensile strength (kg h) and elongation %. Dyeing performance in terms of color parameter (K/S, L, a, b and  $\Delta E$ ), and fastness properties (wash, and light) were studied. The samples show a high tensile strength, high color strength, and poor fastness properties. We believe that the results obtained here are interesting for industrial application due to the production of a natural dye as an inexpensive source from orange peel as a by- product. Another objective is to contribute in solving or decrease the problem of environmental pollution.

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