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# Dyeing Of Cotton Cloth with Natural Dye Extracted From Pomegranate Peel and its Fastness

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## Abstract

A vast array of colorants obtained from natural sources such as plants, insects/animals and microbes have been scrutinized in recent past for their use in different kinds of applications. Research into new natural dyes sources along with eco-friendly, robust and cost-effective technologies for their processing and application have greatly aided in widening the scope of natural dyes in various traditional and advanced application disciplines. This paper contains application of natural dye extracted from pomegranate peel using ethanol water mixture as a solvent in soxhlet apparatus on cotton cloth. Different tests were conducted to observe the strength of dyeing and presented the results.

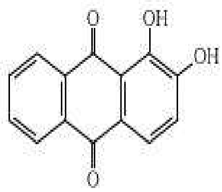
**Keywords**: Mordants, colorfastness, Punicagranatumis, light fastness, alizarin, indigo, granatonine, chromatophore

## Introduction

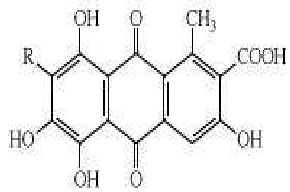
Dyeing is an ancient art which predates written records. It was practiced since Bronze Age. The widely and commercially used synthetic dyes impart strong colors but causes carcinogenicity and inhibition of benthic photosynthesis. Germany was the first to take initiative to put ban on numerous specific azo-dyes for their manufacturing and applications. Netherlands, India and some other countries also followed the ban. Certain problems with the use of natural dyes in textile dyeing are color yield, complexibility of dyeing process, reproducibility results, limited shades, blending problems and inadequate fastness properties. But these problems can be overcome by using chemicals called as mordants. Mordants are metal salts which produce an affinity between the fabric and the dye. Metal ions of mordants act as electron acceptors for electron donors to form co-ordination bonds with the dye molecule, making them insoluble in water. Alum, chrome, stannous chloride, copper sulphate, ferrous sulphate etc. are the commonly used mordants. Cotton textile dyeing was done since the medieval period using cheap natural dyes. Nature has gifted us more than 500 dye-yielding plant species. Coloring agents of these plants are derived from roots, leaves, barks, trunks or fruits. All colors of rainbow are obtained from plants (Cage). Natural dyes have better bio degradability and generally have higher compatibility with the environment. They are non toxic, non-allergic to skin, non-carcinogenic, easily available and renewable. Color fastness is the resistance of a material to change any of its color characteristics or extent of transfer of its colorants to adjacent white materials in touch. Generally light fastness, wash fastness and rub fastness are considered for textile fibers. Punicagranatumis from the family Punicacea. It grows in all warm countries of the world and was originally a native of Persia. The rind of pomegranate contains a considerable amount of tannin, about 19% with pelletierin. The main coloring agent in the pomegranate peel is granatonine which is present in the alkaloid form N-methylgranatonine .This compound gives color to the dye. Its study will enable us to understand the structural chemistry of the coloring compound.

## History of Dyes

Until the 1850s virtually all dyes were obtained from natural sources, most commonly from vegetables, such as plants, trees, and lichens, with a few from insects. Solid evidence that dyeing methods are more than 4,000 years old has been provided by dyed fabrics found in Egyptian tombs. Ancient hieroglyphs describe extraction and application of natural dyes. Countless attempts have been made to extract dyes from brightly colored plants and flowers; yet only a dozen or so natural dyes found widespread use. Undoubtedly most attempts failed because most natural dyes are not highly stable and occur as components of complex mixtures, the successful separation of which would be unlikely by the crude methods employed in ancient times. Nevertheless, studies of these dyes in the 1800s provided a base for development of synthetic dyes, which dominated the market by 1900. Two natural dyes, alizarin and indigo, have major significance. Alizarin is a red dye extracted from the roots of the madder plant. Two other red dyes were obtained from scale insects. These include kermes, obtained from Coccus ilicis (or Kermes ilicis), which infects the Kermes oak, and cochineal, obtained from Dactylopius coccus, which lives on prickly pear cactus in Mexico. One kilogram (2.2 pounds) of cochineal dye can be obtained from an estimated 200,000 insects. The principal colored components in these dyes are kermesic and carminic acids, respectively, whose similarity was established by 1920. In their natural state many colorants are rendered water-soluble through the presence of sugar residues. These sugars, however, are often lost during dye isolation procedures.

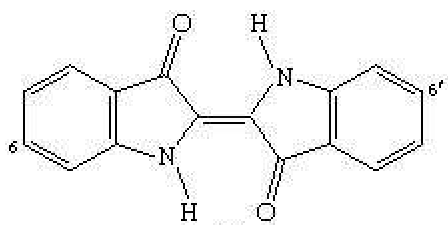



alizarin



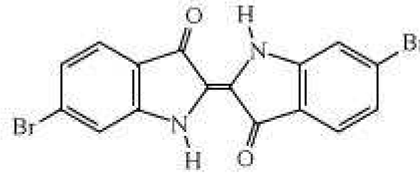

kermesic acid (R = H)
carminic acid (R = glucosyl)

Probably the oldest known dye is the blue dye indigo, obtained in Europe from the leaves of the dyerswoad herb, and in Asia from the indigo plant. Even by modern standards, both alizarin and indigo have very good dyeing properties, and indigo remains a favored dye for denim, although synthetic indigo has replaced the natural material.



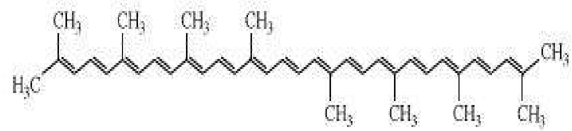

indigo

With a process developed by the Phoenicians, a derivative of indigo, Tyrian purple, was extracted in very small amounts from the glands of a snail, indigenous to the Mediterranean Sea. Experiments in 1909 yielded 1.4 grams (0.05 ounce) from 12,000 snails. Historically, this dye was also called royal purple because kings, emperors, and high priests had the exclusive right to wear garments dyed with it, as is well documented in the Hebrew Bible and illustrated for Roman emperors on mosaics in Ravenna, Italy. By the 1450s, with the decline of the Eastern Roman Empire, the Mediterranean purple industry died out.

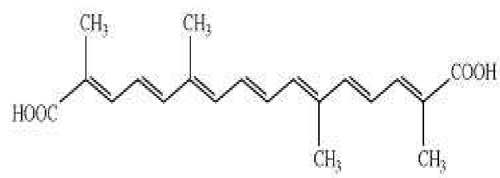



Tyrian Purple

Natural yellow dyes include louting, from the leaves of weld, and quercetin, from the bark of the North American oak tree. These are in the flavonoid family, a group of compounds occurring almost exclusively in higher plants and producing the colors of many flowers. In fact, these compounds can produce all the colors of the rainbow except green. Luteolin, a yellow crystalline pigment, was used with indigo to produce Lincoln green, the color associated with Robin Hood and his merry men. Another group of compounds, the carotenoids, present in all green plants, produce yellow to red shades. Lycopene, from which all carotenoids are derived, produces the red color of tomatoes. An ancient natural yellow dye, crocetin, was obtained from the stigmas of Crocus sativus; this dye is undoubtedly derived from lycopene in the plant. Few of the flavonoid and carotenoid colorants would have survived ancient extraction processes.

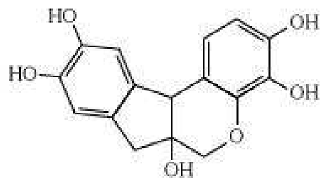



lycopene

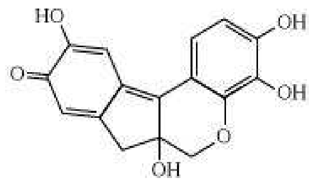



crocetin

Logwood is the only natural dye used today. Heartwood extracts of the logwood tree, Haematoxylon campechianum, yield hematoxylin, which oxidizes to hematein during isolation. The latter is red but in combination with chromium gives shades of charcoal, gray, and black; it is used mainly to dye silk and leather.



hematoxylin



hematein

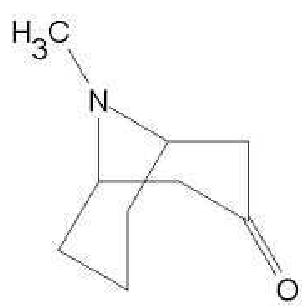
**Wave length and absorption in organic dyes**:

| Wavelength Absorbed(nm) | Colour Absorbed | Colour Observed |
|---|---|---|
| 400–435 | Violet | Yellow-Green |
| 435–480 | Blue | Yellow |
| 480–490 | Green-Blue | Orange |
| 490–500 | Blue-Green | Red |
| 500–560 | Green | Purple |
| 560–580 | Yellow-Green | Violet |
| 580–595 | Yellow | Blue |
| 595–605 | Orange | Green-Blue |
| 605–700 | Red | Blue-Green |

## Punicagranatum

Use of natural dyes has increased several folds in the past few years due to the eco-friendly approach of the people. This paper concerns with the purification of natural dyestuff extracted from an abundantly occurring plant ‘Punicagranatum’. The main coloring agent in the pomegranate peel is granatonine which is present in the alkaloid form N-methyl granatonine. Solvent extraction method was used for the extraction of the dye. The pomegranate peel dye was used for dyeing of scoured cotton cloth using two mordants-copper sulphate and ferrous sulphate in the ratios 1:1, 1:3, 3:1. Dyeing along with mordanting techniques which included pre mordanting, simultaneous mordanting and post mordanting was carried out. Study about fastness tests of dyed clothes was undertaken. Large range of shades was obtained because of varying mordant ratios and combinations.

**Structure of Granatonine:**



In these days of sophisticated technology, we made many synthetic dyes to impart color to our cloths of which some are carcinogenic, some are allergic to skin and other body organs. These dyes are mostly made from nonbiodegradable materials and thus are so. Therefore the dyes prepared and applied to fabric are accumulating on the earth causing a huge disposal problem. Therefore man is in search of eco-friendly materials and methods to develop them. Hence preferred natural dyes to synthetic dyes due to their faster biodegradability, no or less pollution in the process preparation and wide ranges of applications with slight modifications. In search of sources to extract natural dye stuff of plant and animal origin, the ruddy and attractive punicagranatum (pomegranate), caught attention.

**Preparation of Raw Material:**

The pomegranate peels are collected and washed thoroughly with water to remove any impurities. They are dried at room temperature; the samples were ground into powder with the help of grinder. They are shown in the figures below.


**Extraction of Crude Dyestuff*:***

The crude dyestuff is manufactured by taking 100 g of powder in a round bottom flask and 500ml of solvent (ethanol water) in the ratio 40:60 was added to it. The flask was heated in a water bath at 60°C for 60minutes.The solution was then filtered to obtain crude dyestuff. We used different concentrations of solvent (ethanol) while extracting the crude dye stuff. When laboratory grade 95% Ethanol was used the yield of the crude dyestuff is 18.52%. When laboratory grade 75% Ethanol was used the yield of the crude dyestuff is 13.21%

**Purification of Crude Dyestuff*:***

The crude dyestuff is distilled to get 1/3rd of the solution using the Soxhlet apparatus at 70°C for 3hrs. In this process ethanol is recovered and the concentrated dye is obtained. The solution is kept overnight at room temperature for precipitation. The precipitation in ethanol water is obtained by decanting the solution. The obtained particles are dried in the oven overnight at 60°C. Water was added in the Soxhlet apparatus. By addition of water, the boiling points of the compounds

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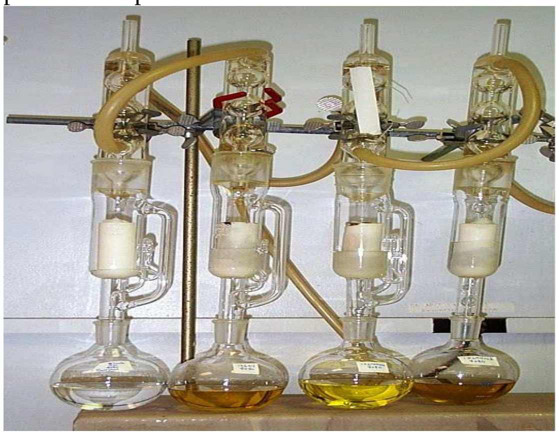
are lowered, allowing them to evaporate at lower temperatures.
Whatever the concentration of solvent may be, the purification process remains the same.



**Application on substrate:**
**Scouring of Cotton Cloth*:***

Scouring of cotton cloth was done by washing it in a solution containing 0.5g/lit Sodium carbonate and 2g/lit nonionic detergent (Tween 80) at 50°C for 25 minutes, keeping the material to liquor ratio at 1:40.The scoured cotton was thoroughly washed with tap water and dried at room temperature. The scoured material was soaked in clean water for 30 minutes prior to dyeing and mordanting.

**Dyeing and Mordanting*:***

Accurately weighed cotton cloth was treated with different metal salts (mordants used-cupric sulphate and ferrous sulphate).The processes of mordanting used were Pre mordanting: It (Pre-mordanting) is a technique that involves applying the mordant first and then introducing the dye to the material .It is also called on-chrome treatment. Simultaneous mordanting: Simultaneous mordanting employs the approach of combining the dye and then mordant agent first, then applying the mixture to the material that is to be dyed. It is also known as meta-chrome or meta-mordanting. Post mordanting: Post-mordanting involves dyeing the fabric first, then exposing the material to mordants as a final treatment. It is also known as after-chrome treatment. After dyeing, the dyed material was washed with cold water and dried at room temperature.

The mordanted cotton cloth was immediately used for dyeing because some mordants are light sensitive. The chromatophore of the dye makes it resistant to photochemical attack, but the auxochrome may alter the fastness. This is due to the formation of complex with the metal which protects the chromatophore from photolytic degradation. Wash fastness of the dye is influenced by the rate of diffusion of the dye and state of the dye inside the fiber.

## Conditions for Dyeing and Mordanting

| Dye | Mordant | Material:Liquor | Temp. | Time |
|---|---|---|---|---|
| 4% | 2% | 1:40 | 80$^0$C | 60 minutes |

## Results and Discussion of Fastness Tests

Dry rubbing: With the dry rubbing cloth flat in place over the end of the finger of the crock meter, same should be rubbed 10 times to and fro in a straight line along a track 10 cm long on the dry specimen in 10 seconds and with a downward force of 9 N.

Wet rubbing: The above described procedure should be repeated with a fresh dry specimen and with wet rubbing cloth. The wet rubbing cloth should be prepared by soaking a fresh rubbing cloth in distilled water and squeezing the same to contain its own weight of water.

**Before testing**

By using $CuSO_4$ as mordant

By using $FeSO_4$ as mordant

**After testing**

By using $FeSO_4$ as mordant

By using $CuSO_4$ as mordant

| S.No | type | Dark shade | Medium shade | Light shade |
|---|---|---|---|---|
| 1 | Dry | 3-4 | 4 | 4-5 |
| 2 | Wet | 2-2.5 | 3 | 3.5-4 |

From the pictures above, we can infer that the dye from pomegranate showed good rub fastness using the dye extracted from the pomegranate peel.

**Wash fastness test:-**

Washing Fastness Tester is used widely for determining color fastness of textile materials to washings**.** The color fastness of textile material is determined by way of mechanical hesitation of a specimen of textile with the piece of specified adjacent fabrics in Standard Soap Solution followed by rinsing and drying. Thereafter, the change in color of specimen and stains of the adjacent fabrics are assessed with standard grey scale.

**Before testing:**

By using $FeSO_4$ as mordant

By using $CuSO_4$ as mordant

**After testing:**

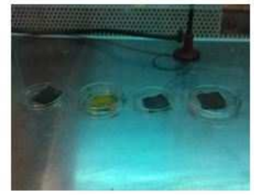
By using $FeSO_4$ as mordant

By using $CuSO_4$ as mordant

From the pictures above, we can infer that the dye from pomegranate showed moderate wash fastness using the dye extracted from the pomegranate peel.

**Light fastness test:**

Color fastness is the resistance of a material to change any of its color characteristics or extent of transfer of it's colorants to adjacent white materials in touch. It is a term used in the dyeing of textiles materials, meaning resistance of the material's color to fading or running. In general, cloths should be tested for color fastness before using bleach or other cleaning products. Fastness tests establish the fitness for purpose of the fabric and help identify the appropriate care label instructions. Color fastness is a measure of two different properties of a textile, the degree to which it changes color when subjected to a particular treatment or environment, and the tendency for the textile to cause staining of other fabrics with which it comes into contact. The color fastness properties of a textile will be related to the type of fibers present, the construction of the material, and especially to the type of dyestuff used and the method by which it has been applied. Fastness properties depend on the strength of the attractive forces between the dyes and the fiber. Light fastness, wash fastness and rub fastnesses that are standardized. The light fastness of textile dye is categorized from one to eight. The higher the number the better fastness is obtained.



Similarly certain types of dyestuff offer better fastness, but at an increased cost to the customer. Occasionally, the dyeing process can result in a reduction in other physical properties and it is up to the dyer to obtain a balance in all these respects. Generally, dyes based on metal complex tend to have better light fastness. The color fastness to perspiration (acid and alkaline) shall be at least level 3-4 (color change and staining). This criterion does not apply to white products, to products that are neither dyed nor printed, to furniture fabrics, curtains or similar textiles intended for interior decoration. A level of 3 is nevertheless allowed when fabrics are both light colored and made of silk or of blends with more than 20% silk. This kind of test is specially applied for the sports wear and heavy dresses which is used specially. A normal cloth is also tested by

**Before testing**

**After testing**

From the pictures above, we can infer that the dye from pomegranate showed very good light fastness using the dye extracted from the pomegranate peel.

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