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A Review on Natural Dyes: Raw Materials, Extraction Process, and their Properties

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Abstract

Synthetic dyes and all the processing used in general dyeing is harmful to our health. Dye from natural sources can reduce the risk of synthetic dyes. As indigo has been used for thousands of years for the coloration of textiles as a natural source. This review is aimed at a discussion of different raw materials used for the extraction of natural dyes, the extraction process for different natural dyes, and the properties of fabric dyed by those dyestuffs. Most of the natural dyes showed a very good fastness property in researches. The dyes can be extracted from trees, bark, leaves, flowers, and many more sources. Most of the natural dyes exhibit special properties like anti-microbial, less toxicity, less allergenic, UV protection.

Keywords: Natural dye; Eco-friendly; Extraction Process; Properties; Fastness

Introduction

The processing of textile is one of the most polluting events on earth. Making just a T-shirt needs around 250 gallons of water from raw to end position. The waste from dyeing carries thousands of pollutants and chemicals dumped onto rivers and canals nearby. That's why we should step forward for eco-friendly processing and use eco-friendly chemicals as well. Using natural dyes in the coloration process of textile can be a step towards a new era of less polluting processing of textile. All new trend for fashion and textile is green and eco-friendly textile. All fashion brands are now stepping forward for making the world less polluted and more livable. From the very first of the 21st century, the world is going towards safer, less hazardous, recyclable technology. Thus the use of environment friendly, biodegradable, non-toxic, less-polluting natural dye is increasing day by day in various sectors. The UV absorption property of most of the natural dyes makes it safer from harmful UV of sunlight. Foods, Drugs, Cosmetics, and Textiles are the main market for natural dyes. Some drawbacks of using natural dye may present as poor fastness property, research is going on to get rid of this.

Some of the most used natural components from which dyes are extracted are prickly pear fruit, Henna leaf, Turmeric, Turkish red pine, Saffron, Indigo, Pomegranate, Red Onion Peel, Eucalyptus Tree, and Marigold Flower.

Objectives of this Project:

- To bring more natural dye extraction process together on a single paper.
- To show the fastness properties of many types of natural dyes.
- To show different methods of extraction suitable for different sources.

Materials and Methods

Resources for natural dyes

Prickly pear fruit: Prickly pear is mostly known as a member of the cactus family scientific name is *Opuntia*. These are green trunk forming segmented cactus grows up to 5-7m with 2-2.5cm of spines and red-colored fruit. Grows mainly in regions of mild winter dry spell followed by hot summer [1].

Henna Leaf: Henna leaf from plant *Lawsonia inermis* has mainly used as Mehendi a dye for art on hand in Asian regions. The tree can also get in the Asian regions. The dye is now made to use as a textile coloration material. A henna tree grows up to 6-10ft [2].

Turmeric: Turmeric is a flowering tree root of which is used as a spice, coloring, and taste-enhancing material in cooking in Asian regions. The turmeric trees grow up to 1m tall. Produces yellow and red color on acidic and alkaline conditions respectively [3].

Saffron: Saffron is mainly a very costly spice collected from the flower of *Crocus sativus*. Used mainly for seasoning and coloring of food. Dye from saffron flower waste is very promising. This purple-colored flowering plant is seen in Asian regions, Europe and America also [4].

Pomegranate: Pomegranate *Punica granatum* is a tasty organic product grows all through the Middle East and Caucasus locale, north and tropical Africa, the Indian subcontinent, Central Asia, the drier lands of Southeast Asia, and parts of the Mediterranean Basin [5].

Indigo: Indigo often called true indigo is a species of plant from the bean family. Scientifically named *Indigofera tinctoria*. This can be found in different regions of the world but mostly cultivated in regions of Asia and Africa. The plant is a shrub which height might be 1-2 meters. The blue dye is derived from the leaves of the plant by fermenting the leaves in water [6].

Red onion peel: Onion is one of the largely used vegetables used

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in different dishes. India is one of the largest producer of Onion amongst the world. This has been used in almost all regions of the world. Asia is the largest producer of onions. The peel of onion is a huge waste in an amount which can be turned into dyes and increase the sustainable uses [7].

Marigold flower: The plants are local to the Americas, developing normally from the southwestern United States into South America, however, a few species groups have gotten naturalized the world over. One species, *T. minuta*, is viewed as a harmful obtrusive plant in certain territories. Species shift in size from 0.1 to 2.2 m tall. Most species have pinnate green leaves. Sprouts normally happen in brilliant, orange, yellow, and white tones, frequently with maroon features. Botanical heads are ordinarily (1-) to 4-6 cm in width, by and large with both ray florets and disc florets [8].

Eucalyptus tree: Plants in the family Eucalyptus have bark that is either smooth, sinewy, hard or wiry, leaves with oil glands, and sepals and petals that are melded to shape a "cap" or operculum over the stamens. The fruit is a woody container usually alluded to as a "gumnut". Most types of Eucalyptus are local to Australia, and each state and region has delegate species. Around 75% of Australian woodlands are eucalypt backwoods. Eucalypts trees can be found in various sizes. Trees normally have a solitary primary stem or trunk yet numerous eucalypts are mallees that are multi originated from ground level and once in a while taller than 10 meters (33 feet) [9].

Turkish red pine: Turkish pine generally grows in the eastern Mediterranean region. The bulk of its range is in Turkey, but it also extends to south easternmost Bulgaria, the East Aegean Islands of North-west Jordan, Iran, Crete, Georgia, and Northern Iraq, the Crimea western Syria, Israel, Aegean Sea, Azerbaijan, Lebanon, and Cyprus. It generally occurs at low altitudes, mostly from sea level to 600 meters (2,000ft), up to 1,200 meters (3,900ft) in the south of its range. Red pine is a medium-size tree, generally grows 20-35 meters tall and diameter of up to 1 meter. The bark is orange-red, thick and deeply fissured at the base of the trunk, and thin and flaky in the upper crown. Color of the leaves are bright green to slightly yellowish green [10].

Methods

Extraction methods for dyes:

Prickly pear fruit: This is a fruit of a cactus type plant. The Dye is extracted from the fruit.

First, the fruit is peeled off and the seeds also are removed. The fruit is then cut down to small pieces. The pieces of the fruit are homogenized with an equal amount of water. The liquid is heated at 80°C for about 5 minutes. Centrifuging of the extract is carried out after the temperature of the extract is cooled down to 8-10 °C [11].

Pigment can be extracted from the fruit and used as dye [12].

Henna leaf: There were many trials done and among them, the leaves in 0.30M aqueous solution of NaOH for 60 minute, at 100°C gives the best color strength and uniformity. Where the material to liquor ratio (M:L) is 1:10. [13].

In a different study maximum color strength obtained at pH 6.64 extraction for 78minutes at M:L of 1:44. [14].

Turmeric: From testing of 25 samples with turmeric for extraction of dye Umbreen S, Ali S, Hussain T and Nawaz R. (2008). Concluded to an observation [15].

Turmeric roots washed and dried before extraction and crushed into a coarse powder. The powder was then dissolved in distilled water with a liquor ratio of 50:1. Then the mixture was stirred for a different range of time and temperature. It has been observed that the strength of the extracted dye was best at boiling temperature and 100 minute of stirring [15].

Turkish red pine: Turkish red pine barks are used for extraction dyestuff. The dyestuff has been removed with the help of a natural dyestuff extraction machine. The pine barks were dried in a vacuum. The fluid from the vacuum was also retained for further use instead of freshwater [16].

In the extraction machine, the dyestuffs were milled to $1mm^3$ particles. Those the transported to the extraction zone where 2% (w/w) bark particles were processed with 78% ethanol and 20% of stocked water from the early process for 24hrs. The extract with solvents the go the solvent removing and extract is being separated from the solvents [16].

Saffron: The dye can get from saffron flower waste. The flower is collected after harvesting the stigma. The flowers then dried at normal temperature without direct sunlight. The dried flowers were ground in a grinding machine. 100gm of flower powder was soaked for 8-10 hrs in 2 Liters of water. Boiling of the extract was carried out for making it water-soluble and then filtered. A dark yellow colored dye was retained from the flower which yielded 12% of colorant by this aqueous extraction method [17].

Pomegranate: Soxhlet operation was used for extraction of dyestuff. [18] Powder from dried pomegranate peel is used in the soxhlet extractor. 7gm of powder with 168ml of Ethanol and 112ml of distilled water with material to Liquor ratio of 1:40. This is taken in a round flask. The solvent in that was heated for 100minute and at 95°C [18].

A condenser was fitted over the flask in which vapor is condensed. When the condensed solvent reaches the top level, the extracted material moves back into the flask. The pomegranate dye extracted was filtered through filter paper and purified further through a rotary evaporator [18].

Red onion peel: In an experiment Peels from raw onions are collected and a solution of Acetic Acid and Distilled water is taken with a ratio of 1:9 respectively and stirred continuously [8].

Extract from different time intervals was collected. A dark ink dye extract is then collected and processed for making a powder of dye. The yield of the dye was recorded at 23.3% [19].

Eucalyptus tree: According to an experiment dead bark of the eucalyptus tree was collected and ground to powder form after drying the barks. Then sieved through a 22-mesh strainer to get rid of unwanted and big particles. 25 of the sample experiment was carried out for finding the optimum condition of extraction. 5 samples of 20gm powder and 200ml water at a different stirring time with different temperatures were taken [20].

The color strength was better when stirring was done for 40 minutes with boiling temperature. The best result was obtained for the sample presoaked overnight and stirring for 80 minute in boiling temperature [20].

Marigold flower: 3.3gm of the dried marigold flower was taken in 100ml of distilled water at 95°C for 2hrs. Coloring materials were extracted from the flower and taken for dyeing of fabric. This method was used by N. Grover, V. Patni. 2011; 2: 403 [21].

Another method used by MD Luque De Castro, F Priego-Capote, J Chromatogr A, 2010, 1217, 2383. Aqueous Ethanol extraction method in which finely chopped dried flowers were taken in an ethanol solution of different concentrations and kept in a water bath incubation at 68°C for 4 hours. The extracted dyes were left overnight in a hot air oven for getting dye extracts [22].

Indigo: Dried crushed leaves of indigo were taken into a beaker containing 250ml of NaOH of Different concentrations for the experiment. So they can see the yield of dye at different concentrations. The mixture was stirred for five minutes and then filtered with a filter paper. The extracted residue was then taken to a beaker and dried at 110°C and ground to powder form. Olusola Adeyanju, Emmanuel SE and Akomolafe SF found that the optimum yield can get at 2.00m NaOH solution and for economical yield 2.00-2.50m concentration can be used [23].

Dyeing: Dyeing was carried out on different type of fabrics for different dyes. Authors used different dyeing techniques, mordants, and methods for dyeing. As we are focusing on properties we didn't discussed any of dyeing methods or techniques.

Tests for properties

Test of properties was carried out maintaining below mentioned standards. Different authors used different methods. All standards used by different authors are mentioned below:

Wash Fastness: ISO 105-C10; ISO 105-C03; ISO 105:C06

Rubbing Fastness: AATCC 8-1995; ISO 105-X12

Fastness to Perspiration: AATCC TM 15-2002; ISO 105-E04

Light Fastness: ISO 105-B02; AATCC 16-1987

Color Strength: UV-vis spectrophotometer

Results and Discussion

Properties of dyed fabric

Henna leaf: From the trial of MM Alam, ML Rahman and MZ Haque. It was seen that brilliant and uniform shades were delivered when degummed silk fiber was colored with 0.8% color. Above or beneath these rates of colors dull and uneven shades were acquired.

[0.8% color relating to 88% color depletion from the color bath]

Effect of time on strength of color

It is seen that the absorption of color by degummed silk fiber increases with the increase of coloring time also, it arrives at the most extreme when coloring time is 60 minutes. The ingestion of color from the color bath at harmony coloring time is 88 % separately. The ingestion remains the same on the additional expansion of coloring time [24].

Colorfastness

It is seen that with the progress of time of exposure to the daylight in the air, the shade of raw silk suddenly changes from brilliant yellow to dull yellow. However, in the instance of degummed silk, just a slight tone change happens from white to yellowish-white even following exposure for 250 hours [24].

Eucalyptus tree bark

Effect of dyeing condition on color strength: With the increase of temperature up-to 90°C the color strength increases. The best result was seen dyeing at 90°C. An increase in salt concentration and dyeing time also increases the color strength of the dye on fabric. The optimum condition 170g/l salt concentration and 120 minute of dyeing at 90°C [20].

Fastness property of fabric

A cotton sample dyed on the above-mentioned condition was checked for fastness and the below observations got

Washing Fastness (Staining on Cotton) - 4

Dry Rubbing - 4/5

Wet Rubbing - 3

Light Fastness - 5

The samples were washed in a standard soap solution at 60°C for 30min, keeping the liquor-to-material ratio as 1:50, according to ISO 105 CO3. The dry and wet rubbing fastness of the dyeing was tested according to ISO 105 X-12 method. Lightfastness was tested according to ISO 105 BO2 method [20].

Red onion peel

Color strength: It has been found by Astha and Junia Laldaihzovi that in 90°C and 60min of dyeing the color strength of onion dye is best which is 13.97 [25].

Fastness property: When dyed at 90°C for 60min the dyed cotton fabric shows:

Dry Rubbing - 3

Wet Rubbing - 3

Wash Fastness - 4

Whereas, dyeing at 30°C for 30min show little better wash fastness but pretty similar rubbing fastness

Dry Rubbing - (3-4)

Wet Rubbing - (2-3)

Wash Fastness - (4-5)

The rubbing fastness test was evaluated by ISO-105-X12 with greyscale ratings. (Ratings 1-5; where 1-poor, 2-fair, 3-good, 4-very good and 5-excellent).

For wash fastness, the solution containing 5gpl nonionic soap solution was used and samples were treated for 45mins at 50°C using a material to liquor ratio of 1:50. 4-very good and 5-excellent) [25].

Indigo

Jabar JM has done some experiments dyeing at 60°C for a different range of time. Fastness was tested after [26].

Good to very good fastness property seen on dyed fabric at all ranges of time except the fastness to alkaline perspiration. Dyeing for 15 and 30 minute shows average fastness on to alkaline perspiration.

The best result was seen at 75min of dyeing:

Washing - 5 (Both water and soap)

Rubbing - 5 (Both dry and wet)

Hot Ironing - 5

Perspiration - 5 (Both alkaline and acidic)

Light - 8

Wash fastness test was carried out keeping specimen at liquor ratio 1:50 and temperature 40°C for 30min with continuous stirring [26].

Indigo dyed cotton fabrics (specimens) were exposed to daylight under standard conditions (including protection from rain, heat from a fire, etc.) for 4hrs per day for 4 months [26,27].

Turkish red pine

Dyeing of fabric with different types of fiber content was dyed with the extracted dye powder 10% (OWF) at a 1:40 liquor ratio. Dyeing was carried out at 100°C for 60min [16].

Color Strength: The dyeing was carried out with both natural and synthetic mordant to Cotton, Wool, Silk, Flax, Polyamide-6,6, Acrylic, and Tencel. The best strength was shown by wool dyed with natural mordant and silk with synthetic mordant [16].

Wash fastness: Almost all fabrics showed average to excellent wash fastness dyed with synthetic mordant (Alum). Briefly, wool, silk, and polyamide 6,6 showed the best fastness among all those. Whereas, the fastness property of fabrics treated with natural mordant (Oak Ash) is lower than that, except Tencel [16].

Rubbing fastness: All fabrics treated with synthetic mordant shows very good to excellent dry rubbing fastness and average to good wet rubbing fastness. The fabrics treated with natural mordant showed better rubbing fastness both dry and wet [16].

Light fastness: Higher color yield leads to a color that is more resistant to fading caused by light exposure. When the results were investigated from this perspective, as expected, alum mordant samples exhibited higher light fastness performance due to their higher color yields, when compared to those of the oak ash mordant and dyed respective fiber types. Where alum mordant silk, acrylic showed better property from that of the natural cellulosic fibers. Among those of Oak ash mordant fibers to silk, flax showed better performance. Others were very poor [16].

Prickly pear fruit

Color strength: The best color strength was gained at pH level 5-6 and decreases thereafter [12].

Fastness property: P. Ganesan and T. Karthik found the fastness of the dyed silk fabric was average to the good that is 3-4 depending on mordanting [12].

Wash fastness: Being treated with natural mordant fabric showed wash fastness 3-4. Color staining on cotton and silk was 4 [12].

Perspiration: Fastness on perspiration was pointed 3-4 for both alkaline and acidic type of perspiration with both natural and synthetic mordant [12].

Light fastness: Lightfastness was marked for 6(pre-mordanting) with both natural and synthetic dye [12].

Rubbing fastness: Dry Rubbing fastness was recorded 3-4(pre-mordanting) for both natural and synthetic mordant.

Wet Fastness was recorded 3 and 3-4(Pre-mordanting) respectively for natural and synthetic mordant [12].

Anti-microbial activity: It was observed that the natural mordant samples have higher bioactive/bacterial resistance compounds that directly influence the antimicrobial activity of the dyed material in both qualitative and quantitatively [12].

Marigold flower

Color strength: It has been seen that the best color strength can be obtained by dyeing the cotton sample at 90°C for 45 minute. The value of color strength is 15.20 [25].

D. Jothi, 2008 found the best color strength with a mordant Copper Sulphate, K/S value 1.1736 [28].

Wash fastness: Astha and Junia Laldaihzovi found a very satisfying fastness property of Marigold flower dye. During the experiments, they have found excellent wash fastness that is 4-5 for dyeing at 30°C for 30min and very good that is 4 during all the tests [25].

Another research by D. Jothi, 2008 showed wash fastness of marigold dyed cotton fabric 4,5 for different types of mordant [28].

Rubbing fastness: For dyeing, at 30°C up to 60 minutes gives the same result for dry rubbing that is 3-4. Above this temperature, all tests show average dry rubbing fastness that is 3.

Wet rubbing fastness was not so good for marigold flower. Most of the experiments showed 2-3 Up to 60°C 45min and 3 after that up to 90°C and 60min. [25]. D. JOTHI showed very good rubbing fastness of dyed cotton fabric in her experiment using a different mordant [28].

Pomegranate

Color strength: Lyocel fabrics were dyed with pomegranate peel dye powder without any other chemical except mordant. The experiment was to see the dyeability of lyocel fabric and see the effect of dye without auxiliaries. The maximum K/S value was for Stannous Chloride that is 6.4 [18].

Wash fastness: The wash fastness property of lyocel fabric dyed with mordant $SnCl_2$ was found 5. The fabric with other mordants also performed very well as well scoring 4-5 [ISO 150-C10] [18].

Rubbing fastness: Dry rubbing fastness for all the mordants was all the same 4-5 except Potassium Dichromate which is 4. Wet rubbing was 4 for all the mordant fabrics [ISO 105-X12] [18].

Light fastness: The lightfastness for fabric treated with stannous chloride was the highest at 6 and others scored 4-5 [ISO 105-BO2] [18].

Fastness on perspiration: Fabric treated with a different type of mordants showed the same resistance to acidic perspiration that is 4-5. The fabric treated with Stannus Chloride scored 5 best amongst all other to alkaline perspiration [ISO 105-E04] [18].

Anti-microbial activity: Cotton Fabric without any treatment of mordant shows 9%, 69%, 28% reduction of *Staphylococcus* (*Aureus*), *Klebsiella* (Pneumoniae), *Candida albicans* (Fungus) respectively. Reduction efficacy increases to 100% for *Staphylococcus* (*Aureus*) if cotton fabric treated with $FeSO_4$ mordant but reduces for other ones [29].

Turmeric

Color strength: Turmeric dyed wool samples with a shade% of 9% show best K/S value of 5.770 [30]. In another experiment, the turmeric dyed cotton with synthetic mordant showed a K/S value of 3.46 [29].

Washing fastness: Cotton fabric treated with turmeric dye scored 4-5 staining on wool both with and without synthetic mordant [ISO 105-C01:1989] [29].

Light fastness: Cotton fabric dyed with turmeric and treated with mordant showed a lightfastness of 3-4. The best lightfastness property showed by wool fabric treated with mordant that is 5-6 [ISO 105-B02] [29].

Rubbing fastness: Cotton fabric treated with mordant scored 4-5 for dry rubbing and 4 for wet rubbing. Among all other fabric like silk, wool, nylon, Nylon only give better dry rubbing fastness others were very poor [ISO 105-X12] [29].

Anti-microbial activity: Cotton fabric without any treatment of mordant 87%, 15%, 55% reduction of *Staphylococcus* (*Aureus*), *Klebsiella* (Pneumoniae), *Candida albicans* (Fungus) respectively [29]. If treated with mordant it reduces to an extent [29].

Saffron flower

Color strength: Wool fabric dyed at both alkaline and acidic condition with saffron flower extract. Stannous Chloride (mordant) treated fabric showed the best K/S value at an acidic condition that is 1.986. But at the alkaline condition, the fabric treated with Ferrous Sulphate gives the best K/S value at 1.650 [17].

Wash fastness: Fabric treated with stannous chloride in acidic condition scored 4-5 and treated with ferrous sulphate in alkaline condition scored 3-4 [17].

Light fastness: For acidic medium, the fabric light fastness was 3-4 and alkaline condition 3 for all types of mordant used [17].

Discussions

There is a wrong perception regarding fastness properties of natural dyes that the dyes don't have required fastness property. That is one of the reasons industries doesn't use natural and consumers also don't want to use those dyes. On this paper in the result section we have seen that most of the dyes used for dyeing fabric had a good score on fastness test. It has been seen that using mordant also affect the color strength and fastness. Use of natural and synthetic mordant both have different effect on dyed fabrics.

So, to take natural dyes for industrial use we both industries and consumers have to step forward to make more and more use of natural dyes instead of synthetic ones.

Table 1: Fastness Properties of Dyes (Best Values Given).

Dye Name	Wash Fastness	Rubbing Fastness		Light Fastness
		Dry	Wet	
Eucalyptus tree bark	4	4/5	3	5
Red Onion Peel	4/5	3/4	2/3	*
Indigo	5	5	5	8
Red Turkish Pine	3/4	4/5	4/5	2
Prickly Peer Fruit	4	3/4	3/4	6
Marigold flower	4/5	3/4	3/4	*
Pomegranate	4/5	4/5	4	6
Turmeric	4/5	4/5	4	5/6
Saffron Flower	4/5	*		3/4
Henna Leaf	*	*		4/5

Note: *Not available on researches.

Extraction process

It has been seen that extraction of dye from natural sources mainly done by some methods. Extraction mainly carried out by drying, grinding and mixing with water or acetic acid or NaOH or ethanol solution for research purpose and little amount use.

Soxhlet extraction machine are used for extraction of dyes in bulk amount for industrial use.

Fastness comparison

All the dyes have well to very good fastness properties on average. Fastness varies mordant to mordant. In our review we have seen different mordant showed different behavior with different types of dyes. So mordant and type of mordant are also factor for fastness. But in many cases it has been seen that synthetic mordants works better to get more color strength and fastness property than the natural one (Table 1).

Conclusion

People are now more aware of sustainability and environment friendly products. As natural dye shows nontoxic, non-allergic effects and results in less pollution as well as fewer side effects, it becomes an important area in the field of textile dyeing research.

On this paper different type of extraction method of natural dye and properties of the dyed fabrics has been discussed. As it is difficult to get many types of dyes together in one place, this paper will help the new researcher to get an idea about extraction methods and properties of dyed fabric in one place.

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