Extraction of Natural Dye from Red Ochre and their Applications on Cotton Fabrics

Latha Muthusamy[1], Ragavi Subaramaniyam[2], Sumithra Murugesan[3]

[1]Assistant Professor of Textiles and Apparel Design, Periyar University, Salem, Tamil Nadu, India-636011.
[2]Department of Textiles and Apparel Design, Periyar University, Salem, Tamil Nadu, India- 636011
[3]Research Scholar of Textiles and Apparel Design, Periyar University, Salem, Tamil Nadu, India-636011

Abstract
This Research explores the use of natural dyes extracted from the natural mineral of ochre for dyeing cotton fabric. While plants and insects undoubtedly dominate the world of natural dyes, minerals hold their own in terms of color possibilities. By employing proper preparation methods, utilizing effective techniques, and understanding the effects on different fibers, mineral-based dyes can bring a touch of natural beauty to textiles. Using mineral dyes offers a sustainable and environmentally friendly alternative to synthetic dyes in the textile processing industry. Natural minerals are widely used in numerous fields of pollution remediation because of their unique structure, characteristics, and perfect environmental harmony. In Ancient Egypt, Red Ochre was used in celebrations as a symbol of life and victory and was used as an early form of cosmetics by women. It is made by grinding red earth or clay, which contains Hematite (red iron oxide), into a fine powder. Mineral dye is a sustainable and ethical solution for fast fashion. Mineral dye has another advantage; it is resistant over time to washing water, perspiration, friction, and light. Disposal of mineral dyes into landfills will not cause any pollution to the environment as it is a land source. The characteristics of red ochre were characterized by Color fastness and hydro analysis.

Keywords: Natural dyeing, Mineral Ochre's, Pigment

1. Introduction

Nature is superbly colourful and is an abundant source of colourants. Both living and non-living members of nature store an unending amount of colouring substances, coloration is an essential step of the textile value chain where textile goods are dyed or printed to increase their fashion value. A dye is a coloured substance that chemically bonds to the substrate to which it is being applied. The dyes are classified as natural and synthetic dyes. Synthetic dyes are produced from various chemicals. Natural colours are created or extracted from edible sources like fruits, vegetables, seeds, and minerals.

Natural dyes and their use in dyeing is probably the most ancient art of all times. People started using natural dye as their first intellectual tool to portray their surroundings and themselves by art. Though natural dyes have been used to colour textiles from ancient times, but in the 19th century, synthetic dyes replaced natural dyes after discovery of mauve colorant. Natural dye is defined as any dye, pigment, or any other substance derived from natural sources such as plants, animals and minerals are renewable and sustainable bioresource products with minimum environmental impact[1] .They have been known since antiquity for their use in colouring of textiles, food substrate, natural protein fibre like wool, silk and cotton, and leather as well as food ingredients and cosmetics.

Minerals have long been admired for their vibrant colours and captivating beauty. While plants and insects are often associated with natural dyes, minerals also possess the ability to create stunning hues that can transform fabrics and fibres. Mineral-based dyes are generally considered more eco-friendly than synthetic dyes, as they are derived from natural minerals and do not contain harmful chemicals. Mineral-based dyes are generally safe for sensitive skin, as they do not contain harsh chemicals commonly found in synthetic dyes. However, individual sensitivities may vary, so it's always advisable to perform a patch test before wearing dyed fabrics directly on the skin. The colourfastness of mineral-based dyes varies depending on the specific mineral and the dyeing techniques used with proper care, mineral-based dyes can retain their vibrancy for extended periods.

Ochre is a family of natural mineral pigments, which includes yellow ochre, red ochre, purple ochre, sienna, and umber [2].Ochres generally refers to iron oxide and hydroxide[3] .Natural Fe-bearing ochres ranging in colour from yellow to deep red and brown abound on earth's crust. Quite often, ochres can be used for pigmenting purposes upon little or no processing, and, hence the pertinent materials have been widely employed for decorative and artistic purposes since the dawn of prehistory; ochres have also found medicinal applications.

Deposits of red ochre are found chiefly from India in Bharatpur, Bhilwara, Bikaner, Chittorgarh and Rewa districts in Madhya Pradesh; Anantapur, Kadapa, Visakhapatnam districts in Andhra Pradesh; Bhavnagar, Kachchh and Patan districts in Gujarat; Ballari and Bidar districts in Karnataka and Chandrapur district in Maharashtra[4].

Cotton fabric is one of the most commonly used types of fabrics in the world. This textile is chemically organic, which means that it does not contain any synthetic compounds. Cotton fabric is derived from the fibres surrounding the seeds of cotton plants, which emerge in a round, fluffy formation once the seeds are mature. Approximately 75 percent of the world's clothing products contain at least some amount of cotton. In sheer numbers, cotton is the most widely used textile fibre in the world, and manufacturers can spin this fabric into a myriad of different types of products. For instance, most T-shirts contain at least some amount of cotton, and blue jeans are 100 percent cotton. Since cotton is highly breathable and absorbent, it is commonly used to make warm-weather clothing. Its softness makes it a good option for formal and business wear, and its notable draping abilities make it an ideal fabric for dresses. This fabric is used to make bathrobes, bathmats, and towels, and it is also used to make bedsheets, blankets, and duvets. Manufacturers may even use cotton to make curtains, wall-hangings, and other types of home decorations. Manufacturers use cotton to make medical supplies, and this fabric is also used to make industrial thread and tarps. In summation, cotton can be used to make practically any type of textile for consumer or industrial use.

This research study focuses on the natural dye from selected mineral ochres. While plants and insects undoubtedly dominate the world of natural dyes, minerals hold their own in terms of colour possibilities. Iron, copper, and tin-based dyes offer a wide range of vibrant hues, each with its own unique charm. By employing proper preparation methods, utilizing effective techniques, and understanding the effects on different fibre, mineral-based dyes can bring a touch of natural beauty to textiles. Additionally, their historical and cultural significance adds depth and richness to their allure. Embrace the fascinating realm of mineral-based dyes and unlock a world of captivating colours.

2. Materials and Methods

2.1 Selection of fabric

Cotton cloth is the most important natural fiber. Cotton is very breathable and absorbent. This cloth is chemically organic, meaning it contains no synthetic ingredients. It is sourced from Salem. The de-sizing procedure was used to pre-treat 100% cotton cloth with a simple plain weave. To eliminate impurities, the cotton fabric was boiled in boiling water for up to fifteen minutes prior to dying. After such treatment, the fabric was free of sizing ingredients and suitable for dyeing.

2.2 Mineral sources for dyeing

Occurrences of ochre are widely distributed in India. Deposits of red ochre are chiefly found in Gujarat, Karnataka, Madhya Pradesh, and Rajasthan. The researcher obtained the mineral sources from Vinarghya Pharmaceuticals, Akola, Maharashtra.

Preparing ochre dye powder involves extracting from ochre-containing minerals and then processing it into a fine powder.

1. **Cleaning the rocks:** Remove any dirt, debris, or other impurities from the ochre-containing rocks. This can be done by gently cleaning them soft brush or cloth.
2. **Crushing and grinding:** Break the cleaned minerals into smaller pieces using a hammer or similar tool. Then, grind the pieces into a fine powder using a mortar and pestle or a mechanical grinder.
3. **Sifting:** Pass the powdered ochre through a fine mesh sieve to remove any remaining coarse particles. This step helps in obtaining a smoother powder.

Figure 1: Red ochre- preparation of dye powder

Red ochre - Stone form | Crushed Stone | Powdered form | Red ochre - Powder

2.3 Selection of Mordant

Myrobalan is a natural mordant that is widely used in textile dyeing and printing. It is made from the dried fruit of numerous plants of the Terminalia genus, including Terminalia chebula and Terminalia bellirica. In the field of natural dyeing, myrobalan mordant is renowned for its versatility, color-enhancing properties, and environmental friendliness.

Simultaneous method

The simultaneous method, also known as the one-bath method or the combined dyeing and mordanting method, is a natural dyeing technique in which both the mordanting and dyeing processes take place in the same dye bath.

2.4 Dyeing Process

The dyeing experiment was carried out using the boiling procedure in a vessel with a material to liquid ratio of 1:30 for 30 minutes. In dyeing, two types of water were used: distilled water and river water. During the simultaneous procedure with the dyeing processes, the mordants Myrobalan and chemical mordants ferrous sulphate and copper sulphate were introduced individually. Next, the fabric was stained using red ochre powder. The dyed fabric was washed with cold water and dried.

2.4.1. Red ochre with distilled water

In this dyeing process, simultaneous method 20% of red ochre powder was added into distilled water and 10% of myrobalan sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes. In this dyeing process, simultaneous method 20% of red ochre powder was added into distilled water and 10% of ferrous sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes.

In this dyeing process, simultaneous method 20% of red ochre powder were added into distilled water and 10% of copper sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes.

Figure 2: Dyeing process with distilled water

Mordant of myrobalan (RDM001) | Mordant of ferrous sulphate (RDF001) | mordant of copper sulphate (RDM001)

2.4.2. Red ochre with River water

In this dyeing process, simultaneous method 20% of red ochre powders were added into river water and 10% of myrobalan was used. Then mixture stirred, heated at 120°C for 30 minutes. In this dyeing process, simultaneous method 20% of red ochre powder was added into river water and 10% of ferrous sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes. In this dyeing process, simultaneous method 20% of red ochre powders were added into river water and 10% of copper sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes.

In this dyeing process, simultaneous method 20% of red ochre powders were added into river water and 10% of copper sulphate was used. Then mixture stirred, heated at 120°C for 30 minutes.

Figure 3: Dyeing Process with River water

Mordant of myrobalan (RDM001) | Mordant of ferrous sulphate (RDF001) | Mordant of copper sulphate (RDC001)

Table 1: Dyeing Process details

DYEING PROCESS	RED OCHRE WITH DISTILLED WATER			RED OCHRE WITH RIVER WATER		
Source	Powdered Red ochre	Powdered Red ochre	Powdered Red ochre	Powdered Red ochre	Powdered Red ochre	Powdered Red ochre
Dyeing water	Distilled water	Distilled water	Distilled water	River water	River water	River water
Dyeing Method	Boiling	Boiling	Boiling	Boiling	Boiling	Boiling
Dyeing time	30mins	30mins	30mins	30mins	30mins	30mins
Dyeing temperature	120℃	120℃	120℃	120℃	120℃	120℃
Dye stuff	20%	20%	20%	20%	20%	20%
Mordant	Myrobalan-10%	Ferrous sulphate - 10%	Copper sulphate-10%	Myrobalan-10%	Ferrous sulphate-10%	Copper sulphate-10%
M:L:R	1:30	1:30	1:30	1:30	1:30	1:30

3. Results and Discussion

3.1 Colour fastness to washing

The sample is taken and treated with soap and soda solution containing 5gms of soap, and & 2gms of hydrous sodium carbonate with material liquor as 1:50 along with this solution 6om solutions is heated for 45 min by forward and backward movement in laundrometer.

- Standard: AATCC
- Method: AATCC 61

Table 2: Colourfastness to laundrometer properties

Name of the Test	Standard	SPECIMEN SIZE	MLR (ml)	TEMPERATURE	DETERGENT LIQUID (ml)	TIME
Laundro meter	AATCC	6×2	200 ml	40℃	1.1 ml	45 mins

After the specified time the sample is taken and washed with distilled water for 10 minutes and squeezed. The stitches along the 3 sides are removed and dried in shades below 60℃. The colour changing and staining are evaluated using the respected grey scales.

Table 3: Color fastness to Laundrometer

S.No	RED ochre Dyed sample	Sample Name	Colour fastness to washing-Laundrometer	
			Change in colour	Staining in colour
1	RED OCHRE DYED SAMPLE (DISTILLED WATER)	Red ochre with mordant of Myrobalan (RDM001)	3/4	5
2		Red ochre with mordant of Ferrous Sulphate (RDF001)	4/5	3/4
3		Red ochre with mordant of Copper Sulphate (RDC001)	4/5	5
4	RED OCHRE DYED SAMPLE (RIVER WATER)	Red ochre with mordant of Myrobalan (RRM001)	3/4	5
5		Red ochre with mordant of Ferrous Sulphate) RRF001	3/4	5
6		Red ochre with mordant of Copper sulphate (RRC001)	3	4/5

Table 3 and figure 4 demonstrates the colour fastness to washing (laundrometer) the dyed samples of red ochre (distilled water) with mordant of myrobalan, ferrous sulphate and copper sulphate. The mordant of ferrous sulphate, copper sulphate has very good colourfastness and mordant of myrobalan have moderate to good colourfastness properties of change in colour. The mordant of myrobalan and copper sulphate has excellent colourfastness and mordant ferrous sulphate has moderate to good colourfastness of staining in colour.

Table 3 demonstrate the colour fastness to washing (laundrometer) of the dyed samples of red ochre (river water) with mordants of myrobalan, ferrous sulphate, and copper sulphate. The mordant of myrobalan, ferrous sulphate has moderate to good colourfastness and the mordant of copper sulphate has good colourfastness of change in colour. The mordant of myrobalan, ferrous sulphate has excellent colourfastness and mordant of copper sulphate has very good colour fastness of staining in colour.

3.2 Calorimetric test

Beer-Ambert's Law, which states that a liquid sample's absorption and concentration are directly proportionate, forms the basis of a colorimeter's operation. The colorimeter shines light through a liquid sample to compare the colour to an established standard. The tristimulus absorption filter and lens of the colorimeter convert the light beam into a distinct wavelength. The device's digital display shows the findings of the photocell's evaluation of the wavelength that was absorbed.

Figure 4: Calorimeter tester

Table 4: Calorimetric tests- colour analysis

SPECIMEN SIZE	STANDARD	TESTING PARAMETERS		
		COLOUR ANALYSIS		
5×5	CIELAB (CIE-International commission on illumination)	L*	a*	b*

The testing parameters of colour analysis are listed in Table 4 and the test is conducted by the Department of Food Science and Nutrition Laboratory, Periyar University, Salem.
This reflected light intensity was then converted into a b* value using established equations or algorithms.

Table 5: Parameters analyze

Parameters	Standard	Differences	Values
ΔL*	L* sample minus L* standard	Lightness and darkness	(+ = lighter, – = darker)
Δ a*	a* sample minus a* standard	Red and green	green (+ = redder, – = greener)

Δ b*	b* sample minus b* standard	Yellow and blue	blue (+ = yellower, – = bluer)

To begin, the sample colour and the standard colour should be measured, and the values for each measurement saved[5]. The colour differences between the sample and standard are calculated using the resulting colorimetric values. (Eqs.1)

$$\Delta E^*_{ab} = \sqrt{(L_2^*-L_1^*)^2 + (a_2^*-a_1^*)^2 + (b_2^*-b_1^*)^2} \quad \text{(Eqs.1)}$$

To ensure accuracy, multiple measurements may have been taken, and the average value recorded and the results are analyzed.

3.3 Hydro analysis

Table 5, shown hydro analysis (river water and distilled water) insightful results regarding various parameters crucial for evaluating water quality. The pH level, registering at 7.85 river water higher than distilled water 6.56, falls comfortably within the optimal range of 6.5 to 8.5, indicating a neutral to slightly alkaline nature conducive to aquatic ecosystems and human consumption. Total Dissolved Solids (TDS) are measured river water at 413 mg/L, distilled water at 2.0 mg/L and, slightly below the acceptable limit of 500 mg/L, suggesting relatively low concentrations of dissolved substances in the water. Electrical conductivity, recorded river water at 635 µS/cm and distilled water at 3.08 µS/cm, remains well below the recommended threshold, indicating minimal presence of ions and pollutants, thus affirming water's purity to a significant extent. Total Hardness, quantified river water at 198.2 mg/L and distilled water at 1.0ppm (Below detectable limit) as CaCO3, reflects a level within acceptable bounds, ensuring water's suitability for various applications. Calcium content is measured river water at 41.7 mg/L and distilled water at 0.2ppm (Below detectable limit) indicating a presence conducive to maintaining water hardness within acceptable limits. Chlorides, notably river water at 82.7 mg/L, distilled water at 1.0ppm (Below detectable limit) are significantly lower than the permissible threshold of 250 mg/L, suggesting minimal salinity or industrial contamination. Sulphate levels, measured river at 73.9 mg/L, also fall within acceptable limits, indicating a moderate concentration with no immediate concerns for water quality. Distilled water measured at 1.0ppm (Below detectable limit).

Table 5: Testing parameters – Hydro analysis river water

S. No	Parameters	Units	Test method	Acceptable limit	Results	
					River water	Distilled water
1	Ph Value at 25°C	-	IS 3025 P.11: 1983 RA 2017	6.5 – 8.5 Agreeable	7.85	6.56

2	Total Dissolved Solids	mg/L	IS 3025 P.16: 1984 RA 2017	500 mg/L	413 mg/L	2.0 mg/L
3	Electrical Conductivity	µS/cm	IS 3025 P.14: 1984 RA 2019	3000µS/cm (Not recommended for drinking water)	635 µS/cm	3.08 µS/cm
4	Total Hardness (as CaCO3)	mg/L	IS 3025 P.21: 2009 RA 2019	200 mg/L	198.2 mg/L	BDL(DL=1.0ppm)
5	Calcium (as Ca)	mg/L	IS 3025 P.40: 1991 RA 2019	75 mg/L	41.7 mg/L	BDL(DL=0.2ppm)
6	Chlorides (as Cl-)	mg/L	IS 3025 P.32: 1988 RA 2019	250 mg/L	82.7 mg/L	BDL(DL=1.0ppm)
7	Sulphate (as SO4)	mg/L	IS 3025 P.24/sec-1 :2022	200 mg/L	73.9 mg/L	BDL(DL=1.0ppm)

4. Conclusion

Mineral dye is a sustainable and ethical solution for fast fashion. Minerals can also produce bright colours same as other sources like plant and animal. Mineral dye has another advantage; it is resistant over time to washing water, perspiration, friction and light. Compared to chemical dyeing techniques, mineral dyes consuming less water. Disposal of mineral dyes into landfills will not cause any pollution to the environment as it is a land source. Using mineral dyes offers a sustainable and environmentally friendly alternative to synthetic dyes in the textile processing industry.

Data Availability

The datasets generated during the current study can be requested from the corresponding authors upon reasonable request.

References

1. " Mazharul Islam kiron, Natural dyes: Properties, Classification, Production, Advantages and Disadvantages, october22, 2022 https://textilelearner.net/natural-dyes-propertis-types-production/ - Yahoo India Search Results." Accessed: Sep. 07, 2024. [Online].
2. S. S. Shanmathi and S. G. M. Soundri, "SCOPE OF NATURAL DYES IN PRESENT SCENARIO".
3. "Minerals | Free Full-Text | Mineral Pigments in Archaeology: Their Analysis and the Range of Available Materials." Accessed: Sep. 07, 2024. [Online]. Available: https://www.mdpi.com/2075-

163X/8/5/201
4. “Heritage | Free Full-Text | A Review on Historical Earth Pigments Used in India’s Wall Paintings.” Accessed: Sep. 07, 2024. [Online]. Available: https://www.mdpi.com/2571-9408/4/3/112
5. “Colorimetric Measurement and Functional Analysis of Selective Natural Colorants Applicable for Food and Textile Products | IntechOpen.” Accessed: Sep. 07, 2024. [Online]. Available: https://www.intechopen.com/chapters/80556