

# Dyeing of Wool Yarn with Colourants Extracted From Biodegradable Sources

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**Abstract:** Wool is the only fiber possessing all the desirable characteristics of a textile fiber and considered as one of the most versatile fiber due to combination of its properties. It is plastic enough to be formed into any shape and resilient enough to retain the shape (Hess<sup>30</sup>, 1974). Turmeric rhizomes were used as source for pure vegetable dye for yellow colour. In conventional dyeing procedure, where dyeing is done post extraction from the source, the turmeric dye needs mordants to improve the hue and get fixed on substrate. The yellow colour of turmeric turns in to grayish yellow with the normal conventional method of dyeing. The study was aimed at achieving a pure natural yellow colour from turmeric rhizomes without use of any mordants with good fastness properties. Australian merino wool was used for the present study. A suitable procedure was optimized for wool through a series of experiments to achieve a variety of yellow hue. Colourimetric analysis of dye was performed at 380 nm. A number of shades were developed with good to excellent washing fastness, through variations in dyeing conditions only. The final range of shades was tested for colour fastness to light and washing as per ISO standards. These tests were done at the Department of Textile Technology, IIT Delhi. Dyed samples showed good to excellent washing fastness.

**Keywords:** *Turmeric Rhizomes, Curcumin, ecofriendly dyes, wool, Natural dyes, Protein fibers*

## I. INTRODUCTION

Wool is a natural protein fiber with a high molecular weight. Its an ideal fiber for use in the construction of clothing. In every civilization man used colourants in various creative ways and forms and thus a colourful and artistic history was created (Vashishtha<sup>29</sup>, 1995). The interest in natural dyes has been revived in recent years. Athaught of replacing synthetic dyes by natural biodegradable dyes is being considered all over the world. During the manufacturing process of synthetic dyes many carcinogenic chemicals are used which leads to formation of toxic bi-products. These bi-products are discharged in the rivers, ponds or left in open. Hence cause severe atmospheric pollution (Paul<sup>4</sup>, 1996; Vashishtha & Jahan<sup>5</sup>, 2017; Mohanty, Chandranouli & Nayak<sup>6</sup>, 1984). This has threatened the ecological balance and called attention of the environmentalists to develop eco-friendly technologies to produce dyes from natural sources (Pruthi, Chawla & Yadav<sup>7</sup>, 2007; Vashishtha<sup>8</sup>, 2017; Patel, Agarwal, Solanky & Panchal<sup>9</sup>, 2006; Gulrajani & Gupta<sup>10</sup>, 1993; Pradip, Pratibha, Kamal & Harish<sup>11</sup>, 2012; Clark<sup>12</sup>, 1973; Abrahart<sup>13</sup>, 1977). Today all over the world the environment restrictions are becoming stricter. The need to realize the importance and the technology of natural dye is more urgent (Ecolabel index, 2007<sup>1</sup>). This is then led to returning to traditional and more natural way of life (Comptons pictured encyclopedia<sup>2</sup>, 1955; Vashishtha<sup>3</sup>, 2017). Synthetic dyes are produced at very high temperature and pressure from chemicals isolated from petroleum derivatives (Cavendish<sup>28</sup>, 1978). The present study was aimed at

developing 100% natural yellow colour, for dyeing of wool without the use of any synthetic stuffs as mordants. No synthetic chemical was used at any stage. Various studies have been carried out by different scientists on turmeric dye. However optimizing the dyeing conditions for turmeric dye, using simultaneous dyeing and extraction technique for dyeing of wool, investigated in the present study have not been reported earlier.

## II. MATERIALS AND METHODS

### A. Collection of Raw materials and their preparation:

Plant material- Common name: Turmeric, Botanical name: Curcuma Longa, Family\_ginger family, **Zingiberaceae**. It is native to southern Asia, requiring temperatures between 20 and 30 °C (68 and 86 °F) and a considerable amount of annual rainfall to thrive (Watt<sup>14</sup>, 1972). Part used: Rhizomes, When not used fresh the rhizomes are boiled for about 30–45 minutes and then dried in hot ovens after which it is ready for sale in market for further use as a colouring agent for food and dyes. Dried Turmeric Rhizomes were collected from the local market complex in G. B. Pant University campus, Pantnagar, Udham Singh nagar, Uttarakhand. Rhizomes were dried in shade and pulverized in a powder form.

### Instruments:

- Wiley mill installed in the department of Post Harvest Technology, College of Technology, Pantnagar was used for pulverizing the Turmeric Rhizomes.
- Optical density of dye solutions was measured by Spectrophotometer- G5866C installed in the department of Chemistry, College of Basic sciences and Humanities, Pantnagar.
- Electronic balance was used for weighing the dye materials and wool.
- Water bath with thermostatic temperature control was used for dyeing the wool samples in glass beakers under controlled conditions (Image: VI).
- Mercury Bulb Tungston fluorescent lamp (MBTF) Light fastness Tester installed at the department of Textile Technology, IIT Delhi was used to test the colour fastness of dyed samples to light.
- Atlas Launder'ometer installed in the department of Textile Technology, IIT Delhi was used for testing washing fastness of dyed samples (Image: IV)
- Colour fastness rating was done with Grey Scale for evaluating changes in colour and staining as per ISO recommendations (ISO 105-A02: 1993 and ISO 105-A-03).

**Blue Wool standards:** Blue Wool standards were used (for fading along with samples) for testing colour fastness to light.

**Wool:** White Australian Merino wool was purchased from Shree Gandhi Ashram, Haldwani, Uttarakhand. **Processing of wool:** The scouring of wool was done according to procedure

mentioned by Hover 1976 (Shenai<sup>15</sup>, 1980). The skeins were squeezed and rinsed with tap water, till they were freed of the traces of detergent (care was taken not to scrub wring or mangle the skeins as it might cause hardening and matting of wool). Washed skeins of wool were allowed to dry and finally weighed for further experimentation.

#### Optimization of different variables:

A series of experiments were conducted in order to standardize the different variables; such as dyeing technique, concentration of the dye material, time for extraction of dye, time for dyeing, temperature for extraction and temperature for dyeing.

For dyeing of samples the MLR (material liquor ratio) selected was 1:10. The Optical density (OD) values of the dye solutions before and after dyeing were recorded. A sample of one ml was taken from each beaker and optical density was recorded by diluting it 20 times. The percent absorption was calculated by the following formula:

$$\% \text{ Absorption} = \frac{\text{OD before dyeing} - \text{OD after dyeing}}{\text{OD before dyeing}} \times 100$$

Dyed samples were judged by a panel of 15 judges visually on the criteria of luster, evenness of dye, depth of shade and overall appearance. From the total marks obtained the percentage ratings were calculated. Each optimized variable was used in further experiments where ever desired. The final range of shades was tested for colour fastness to light and washing at the Department of Textile Technology, IIT Delhi.

#### B. Dyeing Techniques:

##### Technique I - Dyeing after extraction:

Turmeric powder was tied in muslin bags (2 inch X 2 inch) with a thread to hold it. Pre soaked and weighed wool sample, and dye bag were put in a beaker containing 100 ml water. Beaker was placed in a boiling water bath (100°C). Simultaneous extraction and dyeing was carried out in glass beakers for one hour.

Samples were stirred and dye bags were pounded with a glass rod after every five minutes to ensure even dyeing and continuous extraction of dye from muslin bag. After one hour dye bags were taken out and dyed samples were allowed to cool in dye bath itself. Then the samples were rinsed under running water and dried in shade.

##### Technique II – Simultaneous extraction and Dyeing:

In this technique the conventional method of dyeing has been used. The dye was extracted for one hour in a boiling water bath (100°C). The solution was then cooled and filtered. Pre soaked wool sample of 10 g was added to this dye solution and dyeing was carried out at 80°C for one hour. The samples were stirred with glass rod after every 10 minutes in order to obtain an even dyeing on sample. After one hour beakers were taken out of water bath and samples were allowed to cool in dye bath itself. Dyed samples were then rinsed under tap water and dried in shade. According to results obtained Technique I was selected for Turmeric dye. For further experiments Technique I was used.

#### Measuring the Optical density while using Technique I

During further experiments while using technique I, each experiment was carried out as a pair of two. In each pair of two beakers, two dye bags with same contents and water were placed but presoaked weighed wool sample was added only to one beaker and both beakers were placed in dye bath for

extraction and dyeing. This was done in order to facilitate recording of the optical density before and after dyeing. Thus for each experiment the number of specimen dye solutions was doubled. For example, for optimization of concentration 3 different concentrations i.e. 1g, 2g, & 3g were tested. Two bags for each quantity- total 6 dye bags were placed in separate beakers to carry out the experiment.

#### Concentration of dye material:

For optimization of concentration of Turmeric dye, 3 different concentrations i.e. 1g, 2g, & 3g were tested, Simultaneous extraction and dyeing was carried out for one hour at 100°C. The remaining process was repeated as explained earlier. Optical density was measured for all six solutions and dyed samples were evaluated by judges.

#### Time for simultaneous extraction & dyeing:

Simultaneous extraction and dyeing was carried out with optimized concentration, for three different time durations i.e. 60, 120, 180 minutes respectively at 100°C. Rest of the procedure was done as mentioned earlier. Optical density was measured for all six solutions and dyed samples were evaluated by judges.

#### Temperature for simultaneous extraction & dyeing:

Simultaneous extraction and Dyeing was carried out with optimum concentration of dye for optimized time duration at four different temperatures i.e. 40°C, 60°C, 80°C, 100°C. Optical density was measured for all eight solutions and dyed samples were evaluated by judges.

#### Preparation of Final samples:

Final sample was prepared by dyeing the presoaked weighed wool sample with Turmeric using all optimized variables i.e. optimized dyeing technique, optimized concentration of dye material, optimized time for extraction and dyeing and optimized temperature for extraction and dyeing. This sample was prepared for final tests to colour fastness and washing (Table- A)

As per the results of evaluation by judges the shades out of 13 shades were selected. Finally 9 different shades of yellow were included in the final range of shades (table- A). Further Tests of the final range of shades, for colour fastness to light and washing were done at the Department of Textile Technology, IIT Delhi.

#### Colour fastness Tests of dyed samples:

**Colour fastness to Light:** The dyed wool yarns along with blue wool standards were mounted on a 6 cm wide card board frame in a sheet form (of parallel lengths). The samples half exposed were put inside Fadometer and faded as per ISO recommendations. The samples were compared with Blue wool standards and rated.

#### Colour fastness to washing:

Yarns of test samples were made into sheet form of parallel length measuring 10x4 cm and placed between two pieces of undyed fabrics of same size. The fabric on one side was wool and on other side was cotton. These three layers were sewn from all sides to form a composite specimen. Each composite sample was treated in the Launderometer for 45 minutes at a temperature of  $50 \pm 2^\circ\text{C}$ . The liquor: material ratio was 50:1. The samples were removed from Launderometer, rinsed thoroughly under running tap water, dried and ironed.

The samples were graded on the basis of change in colour of the samples and also the staining of the adjacent fabrics with the help of Grey scales (ISI handbook of Textile testing<sup>27</sup>,1982 ).

### III. RESULTS AND DISCUSSION

Turmeric is one of the oldest natural colouring agents used throughout the world from ancient times. The rhizomes of the perennial turmeric are the source of colour. Turmeric (*Curcuma Longa*) is cultivated throughout temperate parts of the world, and probably the native of India (Gulrajani & Gupta<sup>16</sup>, 1992; Vashishtha<sup>17</sup>, 2018). The rhizomes find use as spice and also colouring matter. Turmeric also has medicinal use for many diseases. The colouring matter is Curcumin, only deposited in the rhizomes with age. It imparts yellow to orange red shades (with alkali) on wool silk and cotton. The colour does not have good fastness properties especially to light (Watt<sup>14</sup>, 1972; Vashishtha<sup>18</sup>,2018; The Wealth of India<sup>26</sup>, 1948).

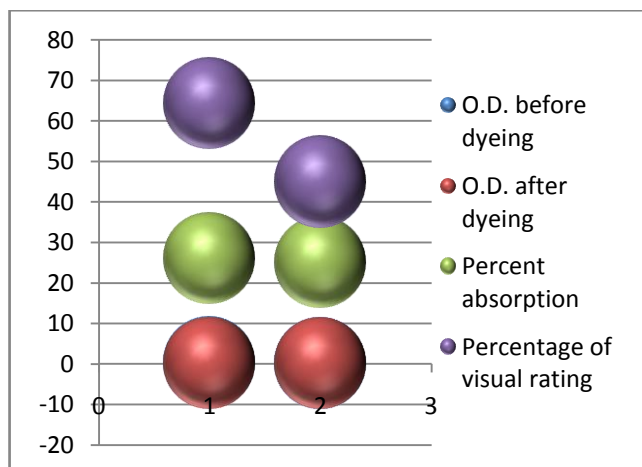
The proximate analysis of Turmeric shows:

13.1% - MOIST URE	6.3% - PROTE IN,	69.4% - CARBOHYD RATE	5.1 % - FA T	2.6 % - FIB ER	3.5%- MINER AL MATT ER
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Curcumin the yellow colouring matter of Turmeric has chemical formula either C<sub>10</sub>C<sub>10</sub>O<sub>3</sub> or C<sub>16</sub>H<sub>16</sub>O<sub>4</sub>.

It melts at 172°C, forms red-brown with alkalis, is converted by boric or sulphuric acid in to rococyanine, by reduction with zinc dust into oily body and by fusion with potash into protocatechuic acid. It is cultivated in almost all the parts of India Curcumin is the prime principal constituent of yellow dye, along with other constituents like monodesmethoxycurcumin and bidesmethoxycurcumin, which also contributes fewer amounts of pigment and flavour (Singh et. al<sup>19</sup>, 1983).

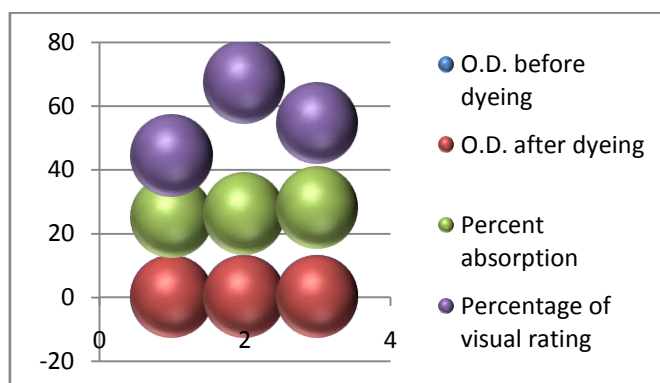
Under experimental trials, different methodologies were adopted for the extraction of colour and dyeing of wool. Figures and tables I to IV show the results of the experiments.



Dyeing Technique	O.D. before dyeing	O.D. after dyeing	Percent absorption	Percentage of visual rating
Dyeing technique I	0.27	0.2	25.92	64.33
Dyeing Technique II	0.2	0.15	25	44.83

Figure I & Table I: Percentage ratings of visual analysis and dye absorption percentage with different methods of dyeing for Turmeric (wave length- 380 nm)

Figure I & table I show the results of colourimetric analysis (at 380 nm) and visual analysis at various parameters explained earlier. As per the results technique one was selected as appropriate for turmeric dye as this produced much better hue as compared to dyeing technique II. Anshu<sup>20</sup> (1987) has recommended that 2 gm of dye is required per gm of wool to obtain optimum colour from natural dye Turmeric. This is a bit different from findings of the present study i.e. 2 gm of Turmeric dye is required to dye 10 gm of wool. This variation in findings may be due to natural variation in sources of dyes used and also wool used (Rama<sup>21</sup>, 1990). It's a well known fact that properties of natural products vary too much because of variation in environment and nourishment of the source used (Umbreen, Ali, Hussain & Nawaz<sup>22</sup>, 2008; Vashishtha<sup>23</sup>, 2018).



Conc. Of dye (g/100ml)	O.D. before dyeing	O.D. after dyeing	Percent absorption	Percentage of visual rating
1	0.24	0.18	25	44.33
2	0.26	0.19	26	67.33
3	0.25	0.18	28	54.5

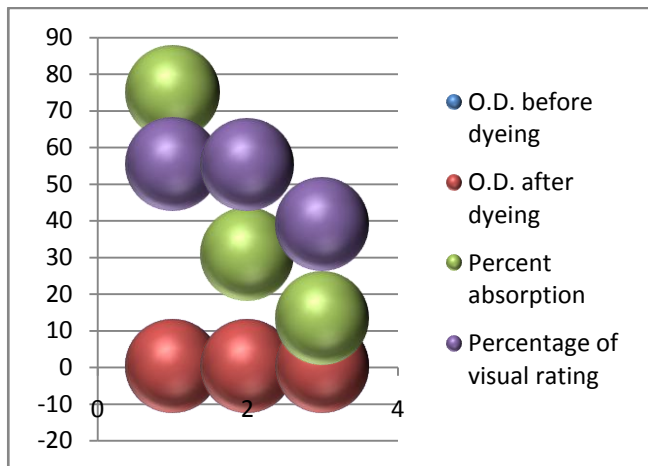
Figure II & Table II: Percentage ratings of visual analysis and dye absorption percentage (at 380 nm) with different concentrations of dye for Turmeric

Fig II & table II reveal the results of optimization of concentration of Turmeric dye. According to results 2gms of Turmeric per 100 ml of water, for dyeing 10 gm of wool was found as optimum concentration.



Table A: The Spectrum of yellow shades obtained

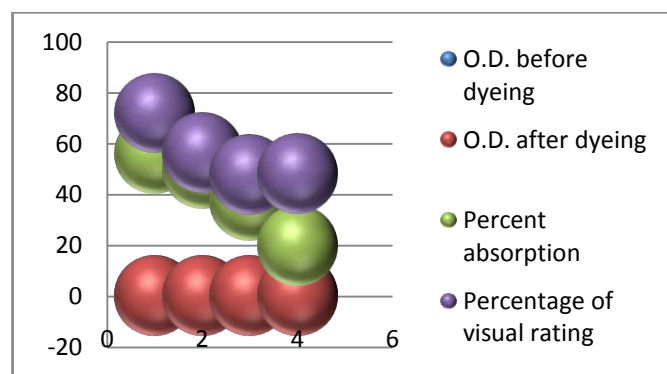
Sr No.	Sample Code	Sample
1	#111	
2	#222	
3	#333	
4	#444	
5	#555	
6	#666	
7	#777	
8	#888	
9	#999	



Time for simultaneous extraction and dyeing (min.)	O.D. before dyeing	O.D. after dyeing	Percent absorption	Percentage of visual rating
60 min.	0.16	0.04	75	55.5
120 min.	0.13	0.09	30.76	55.3
180 min.	0.15	0.13	13.33	38.83

Figure III & Table III: Percentage ratings of visual analysis and dye absorption percentage (at 380 nm) at different time durations for Turmeric dye

Results of colorimetric analysis and visual analysis (to optimize the time for dyeing) show that 60 min. dyeing time is appropriate for Turmeric. Maximum dye absorption was found at 60 min. duration (Table III and Figure III). Results show that dyeing beyond 60 min. makes the hue dull (Nagia & Mohammadi<sup>24</sup>, 2007; Sachan & Kapoor<sup>25</sup>, 2007). Fig IV and table IV show that 40°C temperature provides the best results for dyeing with Turmeric. Temperature beyond that made the hue more Greyish. The Dye absorption was also found maximum at 40°C. Although this is in contrast to the findings of Umbreen & et.al<sup>22</sup> (2008) who reported that maximum colour strength was obtained at 90°C. The results indicate that high temperature should be avoided for dyeing with turmeric if a bright hue is required. Table A shows the various shades achieved through different dyeing procedures during optimization process. The **sample #777** has been dyed with all optimized parameters (Dyeing technique I, conc. of dye 2%, temperature 40°C, Dyeing time- 60 min.)



Temperature for simultaneous extraction and dyeing	O.D. before dyeing	O.D. after dyeing	Percent absorption	Percentage of visual rating
40°C	0.25	0.11	56	72
60°C	0.2	0.1	50	56.33
80°C	0.16	0.1	37.5	47.66
100°C	0.15	0.12	20	48.16

Figure IV & Table IV: Percentage ratings of visual analysis and dye absorption percentage (at 380 nm) at different temperatures for Turmeric dye

Table B: Ratings for colour fastness to light and washing

SN	Sample	Ratings for Colour fastness to light	Colour fastness to washing		
			Ratings for colour change	Ratings for staining on cotton	Ratings for staining on wool
	#111	2- 3	4	3-4	4
	#222	3	5	4	4-5
	#333	2	4	3	4
	#444	3	4-5	3-4	4
	#555	3	3-4	5	3-4
	#666	2	3	2	4
	#777	2	4	3-4	4
	#888	3	5	4	4-5
	#999	2-3	4-5	4	4-5

Table B shows the results of color fastness tests to Light and Washing. Most of the shades exhibited poor to fair fastness to light. The samples were found to be affected by light rapidly. The shades processed at high temperature exhibited a bit better colour fastness to light. For shades done at high temperature a grade of 3 (fair) was observed. Over all the range of light fastness grades were between 2 to 3 (poor to fair).

Samples showed better grades for colour fastness to washing (3- 5). Best grades were observed for #222, #888 and #999 (4- 5) good to excellent for change in colour, good for staining on cotton (4) & good to excellent for staining on wool. Lowest ratings were observed for #666. Rest of the shades got a satisfactory rating of good to excellent (4-5) for colour change and staining as well.

## CONCLUSION

In every civilization colour has played a very important role. For present study turmeric rhizomes were used as a biodegradable source of yellow colour. The water soluble yellow dye from turmeric rhizomes (Curcumin) is very fugitive in nature. The hue becomes dull with exposure to time and temperature. The conventional aqueous extraction procedure involves a time gap between extraction of dye and actual dyeing of the substrate. This exposure to time gap makes the dye dull and unappealing. To achieve the best dyeing results, another way of dyeing was tried and tested, "Simultaneous extraction and dyeing" where dyeing of substrate is carried out in same water bath along with extraction of dye from the raw material. A series of experiments were conducted to compare and analyze the

results two different procedures of dyeing, by means of colorimetric analysis and visually by trained subject experts. Simultaneous extraction and dyeing method was found to yield best hues of yellow dye as decided by colorimetric tests and visual analysis.

Results of numerous experiments reveal that same dye produced different shades under different dyeing conditions. Standard recipes have been developed for each shade. Observations proved that less temperature and reduced time of dyeing give better results in terms of colour appeal. High temperature and prolonged dyeing time produced shades with more durable dyeing. Thus any of the dyeing technique can be used as per the requirement of end product. The various shades have exhibited good washing fastness and fair light fastness. The colours are quite stable even after a long time period gap. The dyeing procedure produced a more or less stable natural yellow dye for wool. Most of the shades exhibited poor to fair fastness to light. The samples were found to be affected by light rapidly. The shades processed at high temperature exhibited a bit better colour fastness to light. For shades done at high temperature a grade of 3 (fair) was observed. Over all the range of light fastness grades were poor to fair. Samples showed better grades for colour fastness to washing. Shades treated at higher temperature exhibited better grades as, good to excellent for change in colour, good for staining on cotton & good to excellent for staining on wool. Lower ratings were observed for shades done at low temperature. Rest of the shades got a satisfactory rating of good to excellent for colour change and staining as well. There is a lot of scope to use the Turmeric dye for obtaining a variety of yellow shades, using safe eco-friendly textile dyeing. Use of this dye will help in detoxifying the textile value chain.

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