

## **Nanoparticles Assisted Natural Dyeing of Silk Fabric**

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### **ABSTRACT**

The current study was carried out to assess the effect of different nanoparticles on dyeability of silk fabric using different natural dyes. Four different naturally extracted dyes *i.e.* barberry bark, henna leaves, marigold flower and pomegranate rinds along with four different inorganic nanoparticles *i.e.* Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Copper oxide ( $\text{CuO}$ ), Titanium dioxide ( $\text{TiO}_2$ ) and Zinc oxide ( $\text{ZnO}$ ) as mordant were used to dye the silk fabric. The cross combination of all four natural dyes and four inorganic nanoparticles were executed for the study. The phytochemical analysis of all the four natural dye extracts was done to ensure the presence of active compounds. The colour properties in terms of per cent dye absorption, colour strength and wash fastness (CC and CS) grades were examined. Significant increase was observed in dyeability, colour strength and wash fastness of silk fabric dyeing with barberry bark, henna leaves, marigold flower and pomegranate rinds dyes. Hence, the inorganic nanoparticles can be successfully used for natural dyeing of silk fabric.

**Keywords:** Nanoparticle, Natural Dyes, Silk, Colour fastness grades, Dyeing

### **INTRODUCTION**

For production of natural dyes, the vast biodiversity of India is harbouring enough potential raw materials. Natural dyes are derived either from plant or animal source. It is believed that out of 47000 plant species occurring in India more than 500 plant species can be used as raw material for production of natural colouring pigments. The most commonly used natural colourants are generally obtained from insects or vegetable matters such as leaves, stems, twigs, prunings, flowers, barks, roots, hulls, fungi, etc. They produce very unique, soothing and soft shades as compared to synthetic dyes without chemical processing. The natural dyes are believed to be eco-friendly, can be cultivated in an environmentally sustainable manner and are inherently carbon neutral. The discovery of man-made synthetic dyes in the mid-19th century triggered a long decline in the large-scale market for natural dyes. Natural dyes are always superior to synthetic dyes in terms of eco-friendliness but natural dyes were re-placed by synthetic dyes because the

synthetic dyes could be mass produced with consistent shades of colour. Also there are some disadvantages related to the application of natural dyes which reduced its applications *i.e.* poor colour fastness properties, poor reproducibility of shades, no standard colour recipes and methods available, use of metallic mordants for better dyeing, some of which are not eco-friendly (Singh and Bharti, 2014). In the early 21st century, the world is heading towards safer, less hazardous, recyclable technologies and the use of natural dyes for textile colouration can be a step towards minimal polluting textile processes and the market for natural dyes in the fashion industry which revive the use of natural dyes in textile dyeing (Affat, 2021).

Mordant is a substance which helps a chemical reaction to take place between a fibre and dye molecules. The mordants which are usually metallic salts form metal complex with the fibre during mordanting, and during the dyeing process it attracts the dye and creates a coordinating bond and attaches the dye firmly to the fabric. Use of mordants which are usually metallic salts

form metal complex result in the generation of highly contaminated wastewater containing non-biodegradable and persistent substances, posing a threat to the environment and human health. As a result, researchers are now shifting their focus towards eco-friendly methodologies that enhance the colour characteristics of natural dyed textiles without causing environmental pollution (Affat, 2021). Nanoparticles of metal oxide are progressively gaining global attention because it is widely perceived as offering vast potential in a broad array of end uses. The unique characteristics of nanomaterials attracted attention of scientists and research personnel to utilize nanoparticles for improving colour strength during dyeing various textile materials (Jabasingh, 2019; Cheng *et al.*, 2020).

Hence, the current study was aimed to impart nanoparticle treatment to silk fabric for dyeing with natural dyes and assess the effect on colour properties of nano treated dyed silk fabric.

## METHODOLOGY

### Materials:

#### *Fabric:*

The silk fabric was acquired from the local market of Hisar, Haryana.

#### *Natural dyes:*

On the basis of easy availability and colour yielding properties of natural dyes, four natural dyes obtained from different parts of plants namely barberry bark, henna leaves, marigold flowers and pomegranate rind were used for the study.

#### *Nanoparticles:*

Four different inorganic nanoparticles (25-100 nm) *i.e.* Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Copper oxide (CuO), Titanium dioxide ( $\text{TiO}_2$ ) and Zinc oxide (ZnO) were used as mordants for dyeing of silk fabric with natural dyes. All the nanoparticles were procured from Central Drug

House (P) Ltd., New Delhi, India.

#### *Enzyme:*

The papain enzyme was used for enzymatic degumming of silk fabric and procured from the Aum Enzymes, Ahmedabad, Gujarat, India.

### Methods:

#### *Preparation of silk fabric:*

To make the fabric free from the gum, make process eco-friendly and increase the absorbency of silk fabric, enzymatic degumming was carried out using papain enzyme as per protocol of Feng *et al.* (2020).

#### *Phytochemical analysis of natural dyes:*

For identification of active chemical compounds present in the selected natural dye, phytochemical screening of the dye extracts was done following the standard procedures given by Pandey *et al.* (2018); Samant *et al.* (2020).

#### *Dyeing of silk fabric:*

For dyeing of silk fabric with natural dyes, the nanoparticles treatment and simultaneous dyeing of silk fabric with different natural dyes was carried out in treatment bath containing 1.00 per cent concentration of all four nanoparticles using optimized dyeing conditions as mentioned in Table 1.

#### *Dye absorption:*

To determine the per cent dye absorption in silk fabric, values of dye bath were measured by taking the absorbance of the dye liquor before and after dyeing on UV visible spectrophotometer by recording optical density (OD).

#### *Colour measurement:*

The dyed samples were assessed for colour measurements in terms of colour strength using Computer Colour Matching system (Premier Colour Scan

**Table 1 : Optimized dyeing conditions of natural dyes used for dyeing of silk fabric**

| Natural Dyes | Optimized dyeing conditions |           |                         |                      |           | References                  |
|--------------|-----------------------------|-----------|-------------------------|----------------------|-----------|-----------------------------|
|              | Dye concentration (%)       | Dyeing pH | Dyeing temperature (C°) | Dyeing time (minute) | M:L ratio |                             |
| Barberry     | 8                           | 4         | 70                      | 45                   | 1:80      | Pruthi <i>et al.</i> (2008) |
| Henna        | 10                          | 7         | 75                      | 45                   | 1:40      | Hasan <i>et al.</i> (2015)  |
| Marigold     | 4                           | 4         | 80                      | 30                   | 1:40      | Jothi (2008)                |
| Pomegranate  | 4                           | 6         | 70                      | 40                   | 1:40      | Ajmal <i>et al.</i> (2014)  |

Spectrophotometer SS5100A).

#### **Wash fastness properties:**

Washing fastness test was carried out as per IS: 3361-1979 Standard test method (BIS, 1979). The change in colour and colour stain of dyed samples were assessed with grey scale number 1 and 2, respectively as per the recommendations of the ISO: 105 method.

## **RESULTS AND DISCUSSION**

#### **Phytochemical analysis of natural dyes:**

The attractive colours and fragrance produced by the plant based dyes is due to the presence of specific phytochemicals present in them. The phytochemical namely tannins, flavonoids, glycosides, saponins, steroids and alkanoids are helpful in imparting various functional properties to textile materials. Therefore, phytochemical analysis is an important step to identify the presence of different active chemical compounds in the plant extract. The data related to phytochemical analysis of barberry bark, henna leaves, marigold flowers and pomegranate rinds dye were presented in Table 2. It is obvious from the table that in barberry bark extract phenol, tannins and

terpenoids were present whereas in henna leaves dye extract, flavonoids, glycosides, phenol and tannins are present. The marigold flowers dye extract confirmed the presence of glycosides, phenol and terpenoids while in pomegranate rinds dye extract, phenol and terpenoids were present. The results are in line with findings of Bhandari *et al.* (2021); Supian and Osman (2023); Mokhber-Dezfuli *et al.* (2014); Kar and Patra (2022) and Sweidan *et al.* (2023).

#### **Colour properties of natural dyed silk fabric:**

The colour properties of natural dyed silk fabric samples *i.e.* per cent dye absorption, colour strength and wash fastness in terms of change in colour and colour staining were examined. The data in Tables 3-6 illustrate the effect of four inorganic nanoparticles on the colour properties of the silk fabric with dye extracts of barberry bark, henna leaves, marigold flowers and pomegranate rinds dyes.






The data in Table 3 represent the colour properties of nanoparticles treated silk fabric dyed with barberry bark dye. It is clear from the table that an improvement in colour properties of silk fabric was observed with use of nano particles as mordant. The maximum per cent dye

**Table 2 : Phyto-chemical analysis of natural dye extracts**






| Phyto-chemicals | Barberry bark | Henna leaves | Marigold flowers | Pomegranate rinds |
|-----------------|---------------|--------------|------------------|-------------------|
| Flavonoids      | -             | +            | -                | -                 |
| Glycosides      | -             | +            | +                | -                 |
| Phenol          | +             | +            | +                | +                 |
| Saponins        | -             | -            | -                | -                 |
| Tannins         | +             | +            | -                | -                 |
| Terpenoids      | +             | -            | +                | +                 |

+ Presence; - Absence

**Table 3 : Colour properties of silk fabric dyed with barberry bark dye**

| Nano particles        | Dye absorption (%) | Colour strength (K/S) | Wash fastness grades |      | Shade obtained  |
|-----------------------|--------------------|-----------------------|----------------------|------|---|
|                       |                    |                       | (CC)                 | (CS) |   |
| Untreated (Control)   | 21.56              | 13.85                 | 3/4                  | 3/4  |  |
| Nano aluminium oxide  | 23.85              | 15.56                 | 4                    | 4    |  |
| Nano copper oxide     | 23.23              | 16.23                 | 4/5                  | 4    |  |
| Nano titanium dioxide | 24.23              | 15.56                 | 4/5                  | 4    |  |
| Nano zinc oxide       | 22.86              | 14.23                 | 4/5                  | 4    |  |

**Table 4 : Colour properties of silk fabric dyed with henna leaves dye**

| Nano particles        | Dye absorption (%) | Colour strength (K/S) | Wash fastness grades |      | Shade obtained  |
|-----------------------|--------------------|-----------------------|----------------------|------|---|
|                       |                    |                       | (CC)                 | (CS) |   |
| Untreated (Control)   | 19.23              | 14.84                 | 4                    | 3/4  |  |
| Nano aluminium oxide  | 25.45              | 16.52                 | 5                    | 4    |  |
| Nano copper oxide     | 27.15              | 17.52                 | 5                    | 4    |  |
| Nano titanium dioxide | 26.15              | 18.02                 | 4                    | 4/5  |  |
| Nano zinc oxide       | 22.86              | 15.95                 | 4                    | 4/5  |  |






absorption was noticed when dyeing of silk fabric was done along with nano titanium dioxide *i.e.* 24.23 per cent followed by nano aluminium oxide (23.85%), nano copper oxide (23.23%) and nano Zinc oxide (22.86%). The colour strength was noticed with treatment of nano copper oxide is 16.23, with treatment of nano aluminium oxide and nano titanium dioxide is 15.56 and colour strength was noticed with treatment of nano zinc oxide is 14.23 whereas good (4) to very good (4/5) wash fastness grades in terms of change in colour and colour staining were found for all nanotreated silk fabric sample dyed with barberry bark. The shades and tints of gold colour were obtained on silk fabric dyed with barberry bark dye.

It is obvious from the data in Table 4 that for dyeing of silk fabric with henna leaves dye, the use of nano particles of zinc oxide exhibited highest dye absorption (27.15%) whereas 17.52 colour strength was observed. The highest colour strength (18.02) was noticed with


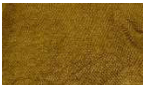



treatment of nano titanium dioxide whereas 26.15 per cent dye absorption was exhibited. The treatment of nano aluminium oxide and nano zinc oxide exhibited 16.52 and 15.95 colour strength and 25.45 per cent and 22.86 per cent dye absorption respectively. The wash fastness grades were good (4) to excellent (5) in terms of CC and CS were found for all nanotreated silk fabric sample dyed with henna leaves. The data in table further narrate that use of different nanoparticles in dyeing of silk fabric with henna leaves dye improved the colour properties in comparison to untreated dyed fabric and cherry wood colour shades and tints obtained on fabric.

The data presented in Table 5 describe that use of different nanoparticles as mordant in dyeing of silk fabric with marigold flowers dye. The shades and tints of old gold colour was noticed on dyed fabric. The improved the colour properties in comparison to untreated dyed fabric were noticed on all nano treated fabric samples. It

**Table 5 : Colour properties of silk fabric dyed with marigold flowers dye**

| Nano particles        | Dye absorption (%) | Colour strength (K/S) | Wash fastness grades |      | Shade obtained  |
|-----------------------|--------------------|-----------------------|----------------------|------|---|
|                       |                    |                       | (CC)                 | (CS) |   |
| Untreated (Control)   | 21.85              | 15.07                 | 4                    | 3/4  |  |
| Nano aluminium oxide  | 24.73              | 17.89                 | 5*                   | 4    |  |
| Nano copper oxide     | 23.46              | 16.23                 | 4/5                  | 4    |  |
| Nano titanium dioxide | 24.23              | 17.89                 | 5                    | 4    |  |
| Nano zinc oxide       | 23.03              | 15.74                 | 4                    | 4    |  |

**Table 6 : Colour properties of silk fabric dyed with pomegranate rind dye**

| Nano particles        | Dye absorption (%) | Colour strength (K/S) | Wash fastness grades |      | Shade obtained  |
|-----------------------|--------------------|-----------------------|----------------------|------|---|
|                       |                    |                       | (CC)                 | (CS) |   |
| Untreated (Control)   | 23.51              | 17.23                 | 4                    | 3/4  |  |
| Nano aluminium oxide  | 27.61              | 18.47                 | 4*                   | 4    |  |
| Nano copper oxide     | 26.86              | 19.58                 | 4/5                  | 4    |  |
| Nano titanium dioxide | 26.06              | 18.97                 | 4/5*                 | 4    |  |
| Nano zinc oxide       | 24.34              | 17.86                 | 5                    | 4    |  |

can be inferred from the data that that maximum per cent dye absorption (24.73%) was exhibited when dyeing of silk fabric along with nano aluminium oxide was done followed by nano titanium dioxide (24.23%), nano nano copper oxide (23.46%) and nano zinc oxide (23.03%). The highest colour strength (17.89) was noticed with both nano titanium dioxide and nano aluminium oxide followed by nano copper oxide (16.23) and nano zinc oxide (15.74). The wash fastness grades, good (4) to excellent (5) in terms of CC and CS were noticed for all nano treated silk fabric sample dyed with marigold flowers.

It is inferred from the data incorporated in Table 6 regarding colour properties of silk fabric treated with different metallic nano particles and dyed with pomegranate rind dye and ochre colour shades and tints were observed. When silk fabric was treated with nano aluminium oxide, exhibited the maximum dye absorption (27.61%) followed by nano copper oxide (26.86%), nano nano titanium dioxide (26.06%) and nano zinc oxide (24.34%). The highest colour strength (19.58) was noticed with treatment of nano copper oxide, 18.97 colour strength exhibited with treatment of nano titanium dioxide followed by 18.47 colour strength with treatment of nano aluminium oxide and 17.86 with nano zinc oxide whereas good (4) to excellent (4/5) wash fastness grades in term of CC and good (4) wash fastness grades in term of CS were found for all nano treated silk fabric sample dyed with pomegranate rind.

The use of natural dyes for textile colouration can be a step towards minimal polluting textile processes and the unique characteristics of nanomaterials attracted

attention of scientists and research personnel to utilize nanoparticles for improving colour strength during dyeing various textile materials (Gola *et al.*, 2021). Hence, to enhance the dye uptake silk fabric was dyed with four natural dyes namely barberry bark, henna leaves, marigold flowers and pomegranate rind in the presence of four metallic nanoparticles *viz.*, aluminum oxide ( $\text{Al}_2\text{O}_3$ ), copper oxide ( $\text{CuO}$ ), titanium dioxide ( $\text{TiO}_2$ ) and zinc oxide ( $\text{ZnO}$ ). The results showed enhancement in colour strength, per cent dye absorption and wash fastness in terms of change in colour and colour staining in nanotreated dyed silk fabric samples comparing to untreated natural dyed silk fabric samples. The findings also line up with Cheng *et al.* (2020); Tangkawanit and Keawsri (2023); Shahin *et al.* (2020); Khatib *et al.* (2022) and Zou *et al.* (2018).

### Conclusion:

The use of nanoparticles of inorganic salts of Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Copper oxide ( $\text{CuO}$ ), Titanium dioxide ( $\text{TiO}_2$ ) and Zinc oxide ( $\text{ZnO}$ ) enhanced the colour properties of silk fabric dyed with barberry bark, henna leaves, marigold flowers and pomegranate rind natural dyes. Hence, nanoparticles for treatment during the dyeing of silk fabric can be used for enhancing colour properties and wash fastness grades of the silk fabric.

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