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RESEARCH ARTICLE

# Sustainable application of *Cassia obovata*–based chrysophanic acid as potential source of yellow natural colorant for textile dyeing

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## Abstract

In the current pandemic scenario, sustainable green products particularly antiviral, antioxidant, and antibacterial in nature are gaining worldwide fame in almost every walk of life. *Cassia obovata* (*C. obovata*) has been valorized as a source of yellow natural dye for nylon dyeing. For the isolation of dye extracts and for surface tuning, nylon fabrics were treated with microwave rays up to 10 min. For getting new shades with good to excellent fastness characteristics, sustainable bio-mordants in comparison with chemical mordants have been used at 60 °C, 70 °C, and 80°C. It has been found that for getting effective colorant yield, acidic extract should be exposed to MW ray treatment up to 6 min, and for getting improved fastness rating, bio-mordants have given excellent color characteristics. Statistical optimization of dyeing variable shows that application of 40 mL of *C. obovata* acidic extract of RE of 6 *p*H containing 3 g/100 mL of salt when employed at 55 °C for 45 min has given excellent results onto irradiated nylon fabric (RNF). It is inferred that *Cassia obovata* has an excellent potential for coloration of surface-modified fabrics, where the application of low amount of bio-mordants under statistical optimized conditions has made process more ecological, economical, and sustainable.

**Keywords** *Cassia obovata* · Bio-mordants · Chrysophanic acid · Microwave · Nylon fabric · Sustainability

## Introduction

Today, color has occupied a special place around the globe; without color, life looks barren. Color in the life of a human is present since its advent either in the form of the painting of caves, body parts, or clothes (Arifeen et al. 2021; Chakraborty et al. 2020). Previously, colors from plants and animals were used for coloration, but with the formulation of mauveine by Henry Perkin, the mentality of community has changed (Mansour et al. 2020; Chakraborty et al. 2020). Now on a daily basis, thousands of dyes are formed and applied in every walk of life (Syafaatullah and Mahfud 2021; Özomay and Akalın 2020; Özomay et al. 2021). With the running of life-style, it has been found that the world is facing problems such as fatal diseases, global heat, destruction of land, and less greenery due to pollution and excessive chemical use (Ouazani et al. 2020). The world protection agencies have observed that one of the causes of these problems is textile effluents (Khan et al. 2021; Thakker 2020). The dyes formed need such intermediates and chemicals which during their processing shed carcinogenic effluent loads (Haule et al. 2020). This effluent load is nondegradable and when in

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contact with water bodies lowers their biological characteristics such as COD, BOD, and OD, disturbs eco-balance, and ruins the agri-land by changing its pH (Hossain et al. 2021; Tebeje et al. 2021). Now, these protection agencies have forced the industrial sector to move toward green or sustainable products such as natural dyes (Thakker 2020; Nathan and Rani 2021; Özomay and Özomay 2021). Eco-friendly awareness about the pros is on the way particularly on account of their excellent biological characteristics such as antiviral, antioxidant, anticancer, and antibacterial (Thakker 2020).

The current era is the era of sustainability, where pharmaceutical-based products which are eco-label and eco-friendly in nature are gaining fame (Islam et al. 2019; Tambi et al. 2021). These colors are separated without any chemical processing from natural sources without any disposal issues (Baaka et al. 2017). Their wastes not only act as fertilizers when added to the soil but also being soothing and attractive in nature (Ansari and Iqbal 2021). These colors from natural sources have a special place in the pharmaceutical world as a tonic to various diseases and are harmonized with nature (Shabbir et al. 2018). Almost every part of the plant yields such molecules which act as antiviral, antioxidants, antibacterial, antifungal, ant-UV, and anticancer agent, for the global community (Tambi et al. 2021; Verma et al. 2021; Rather et al. 2021). Also, the people who are keen to know about the art of cultural heritage in the form of plant dyeing are also urging to revive this art (Singh et al. 2020). Hence the move toward the utilization of herbal-based colorants is on the way around the globe (Mongkholrattanasit et al. 2021).

However natural dyes have some limitations such as low isolation yield and poor fastness ratings (Hosseinnezhad et al. 2021a). Researchers are now trying to overcome these limitations by finding and employing new ways (Haule et al. 2020). According to their approaches either conventional methods such as stirring, soaking, refluxing, and Soxhlet processes for isolation yield should be used or bio-polishing, mercerization, cationization, etc. should be used to improve the sustainability of fabrics. But these methods are less effective and time-, energy-, cost-, and labor-consuming (Adeel et al. 2021a). For fastness properties, salts of $Al^{3+}$, $Fe^{2+}$, $Co^{2+}$, $Cr^{3+}$, $Cu^{2+}$, etc. (Silva et al. 2020; Rani et al. 2020) are used, but owing to toxicity, these chemicals are also coming under strict observation (Alebeid et al. 2020**;** Atav et al. 2021). Now modern approaches have been adopted such as radiation tools for the isolation of colorant bio-mordants as a possible substitute of banned chemical anchors. Many radiation methods such as ultraviolet (Gözütok and Bahtiyari 2020), ultrasonic (Lei et al. 2021), microwave (Buyukakinci et al. 2021), gamma (Islam et al. 2021), and plasma (Krifa et al. 2021) have been employed, but microwave treatment (MW) has its unique role. These rays are considered as one of the clean, sustainable, and uniform heating sources and for the isolation of natural molecules (colorant) from plants (Karadag et al. 2020; Buyukakinci et al. 2021). These rays weaken the cell wall for rising the isolation yield of phytochemicals particularly potent isolate (natural dye) with solvent within a short time and consume less solvent money and labor (Suktham et al. 2021; Karadag et al. 2020).

To raise the fastness ratings, instead of chemicals, now plant isolates are being used. The isolates have such potential bioactive which are not only excellent in antiviral, antimicrobial, and antioxidant characters but also give soothing shades with improved fastness properties (Alebeid et al. 2020; Rani et al. 2020). The addition of such plants, which have excellent therapeutic nature, not only makes the process greener, valorized, and sustainable but also attracts the people on account of environmental health and global eco-balance (Baseri 2021; Verma et al. 2021).

Keeping in view the benefits of MW rays for isolation and bio-mordants for shade development process, the current study has been designed to explore *Cassia obovata* commonly known as *Senna italica* as a source of yellow natural dye for bio-mordanted nylon fabrics. *Cassia obovata* (*C. obovata*) being a member of Fabaceae family (Figure 1a) is found around the globe as a source of yellow natural coloring agent (Singh et al. 2013; Zin et al. 2020). Its extract is useful in the treatment of headache, fever, leprosy, skin diseases, ophthalmic, and ringworm infection and exhibits antioxidant, anticancer, antimicrobial, hypoglycemic, anti-inflammatory, anti-mutagenic, hyperglycemic, anti-hepatic, anti-asthmatic, and antiviral activities (Singh et al. 2013; Shehu et al. 2018). The extract of *Cassia* has several bioactive components such as flavonoids, glycosides, and anthraquinone, but chrysophanol (chrysophanic acid) is the main active colorant that imparts color onto fabric such as nylon, wool, silk, and cotton. Nylon is a synthetic polyamide fabric that has amido linkage for interaction with colorants (Figure 1c).

To the authors' knowledge, still no study has been made to employ this yellow source of colorant for nylon dyeing. For the first time, our group has explored the coloring behavior of *Cassia obovata*, its exploration, statistical optimization of dyeing parameters, and finding the suitable temperature and optimum amount of mordants to get colorfast shades with sustainable cost-, time-, and energy-effective processing.

The current study has been designed:

(i) To isolate colorant in a suitable medium through level of microwave rays, i.e., exposure time
(ii) To observe a physical and chemical change in polyamide fiber before and after microwave radiations
(iii) To optimize mordanting temperature for getting excellent shades with utilization of optimum concentration of mordants
(iv) To observe the colorfastness rating of dyed fabric before and after sustainable mordanting at optimum conditions

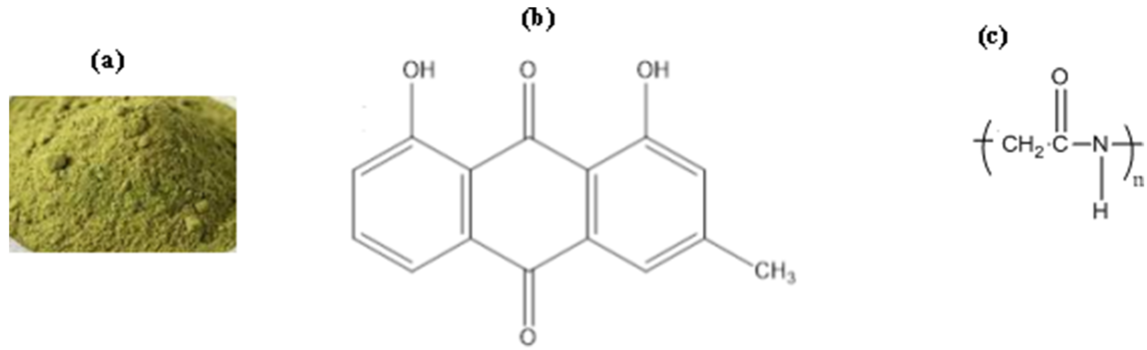



**Fig. 1** *C. obovata* powder (a), chrysophanic acid (b), functional unit of nylon fabric (c)

## Materials and methods

### Material collection

Leaves *of Cassia Obovata* (*C. obovata*) have been collected from herbal store of Faisalabad, Pakistan. These leaves were chopped into very small pieces and ground finely using a food blender. The ground stuff was passed through a sieve of 20 meshes to get uniform size granular powder. In the same way, bio-mordant sources such as turmeric rhizomes, pomegranate peels, acacia bark, and pine-nut hull were treated and sieved finally to get equal size granular powder. Both dye source and mordant sources were stored in airtight jars to be used for further isolation, coloration, and shade-making process (mordanting). Pre-treated nylon fabric was provided by the National Engineering Laboratory for Modern Silk, College of Textile and Clothing Engineering, Soochow University, Suzhou, China. All the chemicals used in this study were of commercial scale.

### Dye extraction and irradiation process

For the better extraction of natural colorant, powder of *C. obovata* was boiled in distilled water and acidic medium for 45 min, by keeping powder to solvent ratio of 1:25. After attaining extracts, both nylon fabric and extract of aqueous, acidic medium (pH= 6) were exposed to MW irradiations for 2, 4, 6, 8, and 10 min, using Orient Microwave irradiator (800 W, 2450MHz). To obtain the higher colorant yield onto nylon fabric, MW-irradiated (RE) and un-irradiated extracts were used to dye MW-irradiated (RW) and un-irradiated nylon (UNF) keeping extract to nylon fabric ratio (E:NF) of 1:25 at 80 °C for 45 min. The detailed scheme of extraction and dyeing has been narrated in Table 1. Dyeing variables have been optimized using response surface methodology as statistical tool by varying temperature, time, volume, pH, and salt amount (Table 2).

*NRE*, non-irradiated extract; *RE*, irradiated extract; *RNF*, irradiated nylon fabric; *NRNF*, non-irradiated nylon fabric; *MAD*, microwave-assisted dyeing

### Mordanting treatment

To enhance the fastness and color strength values as well as for making the process more sustainable and eco-friendly, various bio-mordants and sustainable metallic mordants were used. Bio-mordants such as pomegranate peels containing tannin, acacia bark containing quercetin, turmeric containing curcumin, and pine-nut hull containing catechin were used. For the extraction of bio-mordants, 1–5 g of crude powder of mordants was boiled in 100 mL of aqueous medium for 45 min keeping bio-mordant source to water ratio (BS:W) of 1:25. Three metallic mordants (1–5g/100 mL) such as salts of $Al^{3+}$, $Al_2(SO_4)_3$, $Fe^{2+}(FeSO_4)$, and tannic acid (TA) were used for the mordanting nylon fabric before dyeing using *Cassia obovata* extract at 60 °C, 70 °C, and 80 °C. The whole process of application of chemical and bio-mordants before and after dyeing of nylon at given conditions was carried out by following already documented methods of Adeel et al. 2021a, b, c) and Arifeen et al. (2021).

### Optimization of dyed and undyed fabrics

Nylon fabrics before and after irradiation for 6 min were subjected to FTIR (Perkin Elmer, USA) analysis for viewing any change in characteristics peak of amido linkage. Similarly, SEM analysis was performed for viewing any change in surface morphology of nylon fabric through SEM (SEM-model Tescan; 5 kV). Datacolor SF 600 was used to assess all dyed and mordanted fabrics through Kubelka-Munk equation ($K/S = (1–R)^2/2R$). To observe the role of biomolecules (bio-mordants) in comparison with chemical mordants, ISO standards for light, washing, and rubbing fastness were used, and the results were assessed at gray scale to get final rating.

## Results and discussion

Microwave treatment day by day has taken the place as the state-of-the-art tool in the field of green chemistry (Buyukakinci et al. 2021; Pal and Jadeja 2020; Popescu

**Table 1** Microwave-assisted extraction and dyeing conditions for coloration of nylon fabric from *C. obovata*

| Fabric used | Sample code | Microwave irradiation time | Dyeing conditions |
|---|---|---|---|
| Nylon | NRE/NRNF | 2–10 min | 40 mL volume of pH=6 for 45 min |
| | RE/NRNF | 2–10 min | at 55°C |
| | RE/RNF | 2–10 min | |
| | NRE/RNF | 2–10 min | using 3g/100mL of table salt |
| | MAD | 2–10 min | |

et al. 2019). This is because it is a green, leveled, and sustainable isolation tool that takes less energy, solvent, time, and labor to abstract the novel potent biomolecules (colorant) from plant molecules (Majumder et al. 2020; Karadag et al. 2020). These rays by penetrating into the layers of the matrix rupture its cell wall and by forming excellent solid (plant)-liquid (solvent) interaction, give high color yield (Sanchez-Camargo et al. 2021; Taqi et al. 2020). This mass transfer kinetic process occurs by collapsing the heat from solvent to the plant cell wall after exposure to MW rays, which in turn, ruptures this cell wall to evolve the biomolecules (Adeel et al. 2021b; Elshemy and Haggag 2019). The same is the situation that has been observed in our studies while exploring the efficacy of *C. obovata* for nylon dyeing. The results given in Figure 2a using an aqueous extract show that the extract (RE) irradiated for 4 min has given high color yield (K/S up to 5.1138) onto irradiated nylon fabric (RNF 4min) as compared to unirradiated extract (NRE) used to color unirradiated nylon fabric (NRF). Upon changing the medium, it has been observed that acidic extract (RE) was irradiated for 6 min onto irradiated nylon fabric (Figure 2b). Here again, it can be seen that irradiation of fabric is of equal importance.

It is concluded that for getting effective yield, extract should be irradiated for 6 min and fabric surface should be tuned for 6 min to get its improved substantivity (Figure 3a and b). The other factor is surface scaling of nylon where the changes at the fiber surface cause the improvement in its dyeing behavior. The scales formed at the surface sorb dye significantly which, in turn, show high color strength (K/S). Upon assessment before and after microwave irradiation up to 6 min, the SEM images given in Fig. 3a and b reveal that surface of nylon after MW treatment for 6 min has been upgraded by producing the scales at the surface, which in turn the tuned surface of nylon has held dye molecules promisingly. Previous studies also revealed that microwave rays cannot change the chemistry of the functional site of nylon (Peets

**Table 2** Optimization of dyeing parameters for nylon using irradiated acidic extract of *C. obovata through* response surface methodology (RSM)

| Expno. | A (pH) | B (mL) | C (min) | D (°C) | E (g/100mL) | Exp no. | A (pH) | B (mL) | C (min) | D (°C) | E (g/100mL) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 4 | 30 | 45 | 45 | 4 | 17 | 2 | 40 | 55 | 55 | 3 |
| 2 | 8 | 30 | 45 | 45 | 2 | 18 | 10 | 40 | 55 | 55 | 3 |
| 3 | 4 | 50 | 45 | 45 | 2 | 19 | 6 | 20 | 55 | 55 | 3 |
| 4 | 8 | 50 | 45 | 45 | 4 | 20 | 6 | 60 | 55 | 55 | 3 |
| 5 | 4 | 30 | 65 | 45 | 2 | 21 | 6 | 40 | 35 | 55 | 3 |
| 6 | 8 | 30 | 65 | 45 | 4 | 22 | 6 | 40 | 75 | 55 | 3 |
| 7 | 4 | 50 | 65 | 45 | 4 | 23 | 6 | 40 | 55 | 35 | 3 |
| 8 | 8 | 50 | 65 | 45 | 2 | 24 | 6 | 40 | 55 | 75 | 3 |
| 9 | 4 | 30 | 45 | 65 | 2 | 25 | 6 | 40 | 55 | 55 | 1 |
| 10 | 8 | 30 | 45 | 65 | 4 | 26 | 6 | 40 | 55 | 55 | 5 |
| 11 | 4 | 50 | 45 | 65 | 4 | 27 | 6 | 40 | 55 | 55 | 3 |
| 12 | 8 | 50 | 45 | 65 | 2 | 28 | 6 | 40 | 55 | 55 | 3 |
| 13 | 4 | 30 | 65 | 65 | 4 | 29 | 6 | 40 | 55 | 55 | 3 |
| 14 | 8 | 30 | 65 | 65 | 2 | 30 | 6 | 40 | 55 | 55 | 3 |
| 15 | 4 | 50 | 65 | 65 | 2 | 31 | 6 | 40 | 55 | 55 | 3 |
| 16 | 8 | 50 | 65 | 65 | 4 | 32 | 6 | 40 | 55 | 55 | 3 |

*A*, extract *p*H; *B*, extract volume; *C*, contact time; *D*, contact temperature; *E*, salt concentration

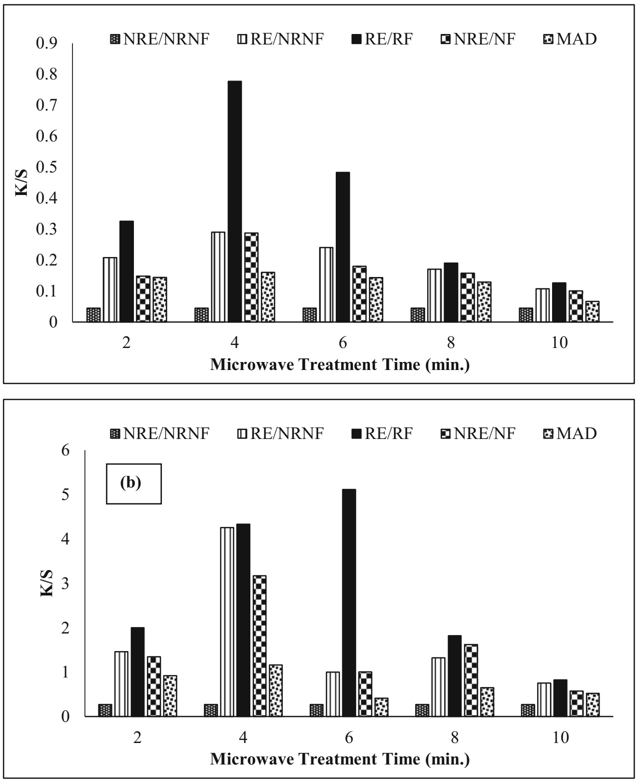



**Fig. 2** Application of *C. obovata* aqueous (**a**) and acidic extract (**b**) for dyeing of nylon fabric before and after microwave treatment

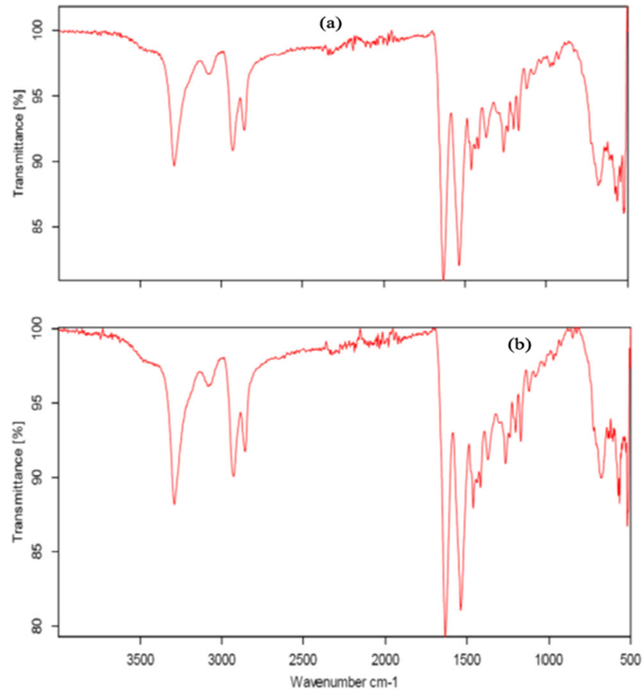



**Fig. 4** FTIR spectra of control (**a**) and microwave-treated nylon fabric (**b**)

et al. 2019). However, it modified its surface to enhance its substantive nature toward colorant to give high color depth (Elnagar et al. 2014; Gashti et al. 2013). The result given in Figure 4a and b reveals that after MW treatment up to 6 min, there is no change in the characteristic peak of amido linkage of nylon. ATR-FTIR spectral images of fabric before and after MW rays obtained reveal that the –NH stretching peak (3500–3000 cm$^{-1}$), C=O stretching peaks (1630 cm$^{-1}$), and C–N–H bending peak (1520 cm$^{-1}$) have not been changed after microwave treatment up to 6 min. The presence of characteristic peak of amido linkage as functional units of polyamide fabric, i.e., nylon, shows that these rays have not changed its chemical nature which is a very useful thing in the field of textile processing. Hence microwave rays do not cause any chemical nature in the characteristics of nylon.

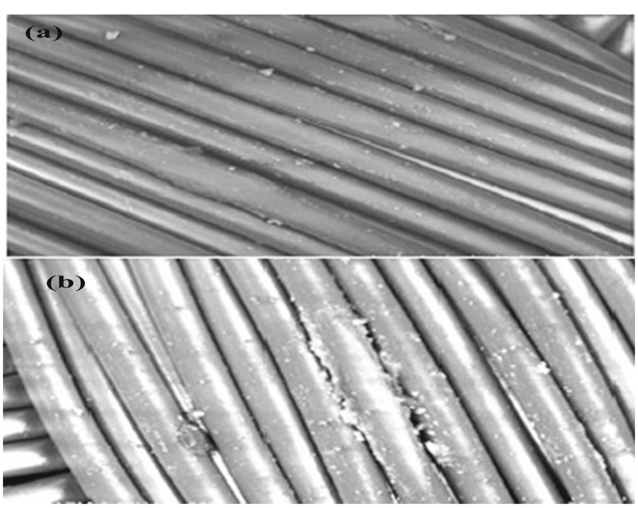



**Fig. 3** SEM images of control (a) and microwave-treated nylon fabric (b)

Previously, it can be seen that contact levels always promote dyeing, but after microwave treatment, their levels become reduced. This is because after MW treatment, cluster of colorant molecules are broken into small size molecules which are accelerated at 55 °C for 55 min to get excellent results. Another factor is the nature of extract (*p*H), where nylon being polyamide in nature is prone to acidic dyeing. However, MW treatment has made it possible at mild acidic condition (*p*H 6). Salt is another factor which adds value in coloration by giving maximum exhaustion of reduced size molecules through utilization of low levels of table salt (3g/100mL). Hence, practically the utilization of 40 mL of *C. obovata* acidic extract of RE of 6 *p*H containing 3g/100mL of salt when employed at 55 °C for 45 min has given excellent results onto irradiated nylon fabric (RNF). Hence, microwave treatment has reduced the level which shows that this treatment is a cost-, time, and energy-effective tool. The experimental results have been statistically analyzed in Tables 3–4. We employed a central composite design (CCD) as shown in Tables 2. Design includes 32 trial; each variable pH, volume, time, temperature, and salt was applied at five levels; and response was observed. The behavior of response (K/S) is graphically assessed through 3D response graphs in Figure 5a–c. Graphs are constructed between two factors and the response, while the remaining factors are held at middle levels. Table 3 shows ANOVA

**Table 3** ANOVA: K/S versus *p*H, vol, time, temperature, and salt second-order polynomial regression

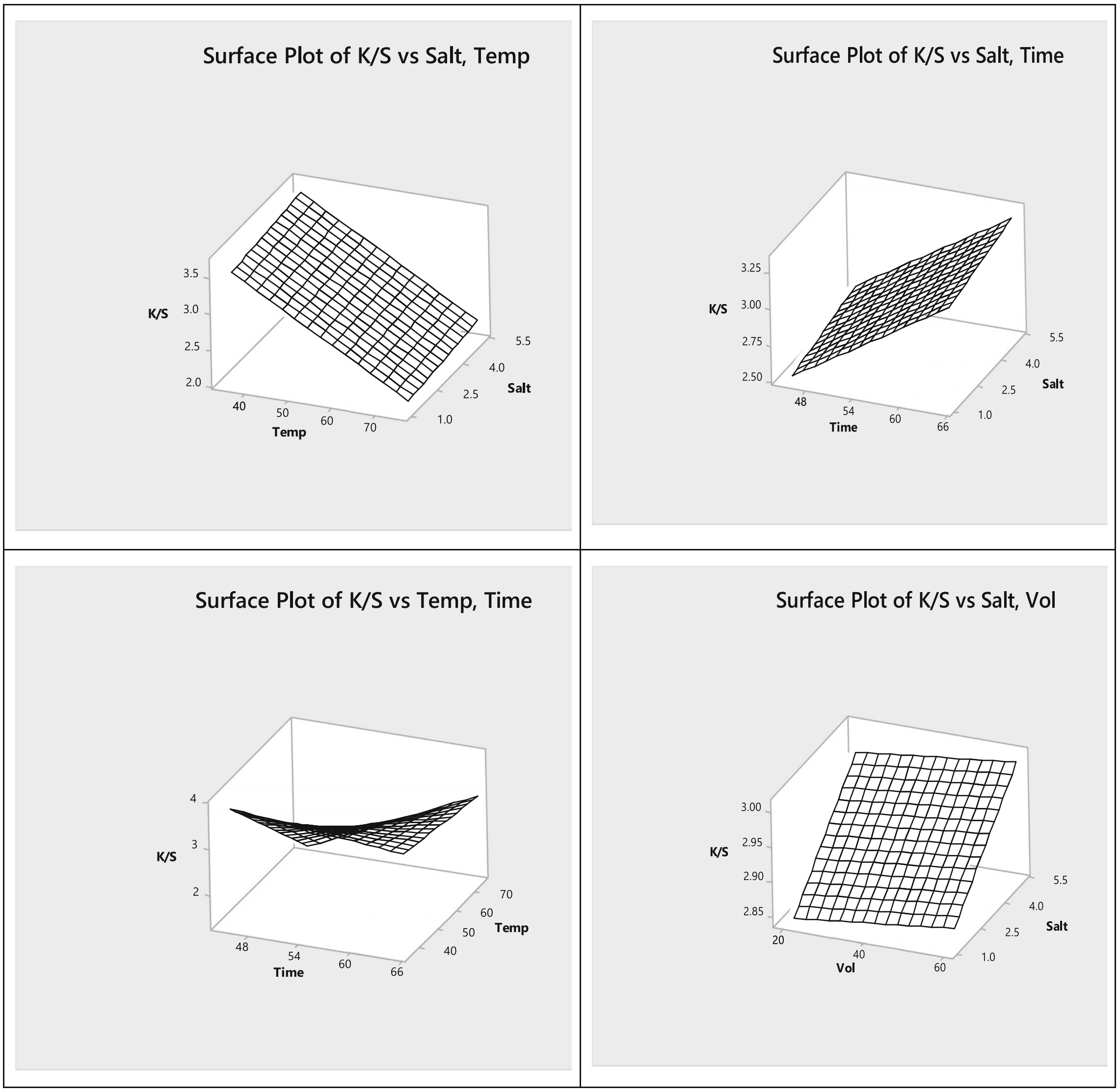
| Source | DF | Adj SS | Adj MS | *F*-value | *p*-value |
|---|---|---|---|---|---|
| Model | 11 | 9.30798 | 0.84618 | 27.55 | 0.000 |
| Linear | 5 | 3.73498 | 0.74700 | 24.32 | 0.000 |
| *p*H | 1 | 0.21690 | 0.21690 | 7.06 | 0.019 |
| Volume | 1 | 0.00082 | 0.00082 | 0.03 | 0.873 |
| Time | 1 | 1.46688 | 1.46688 | 47.76 | 0.000 |
| Temperature | 1 | 2.58136 | 2.58136 | 84.05 | 0.000 |
| Salt | 1 | 0.02628 | 0.02628 | 0.86 | 0.371 |
| Square | 1 | 4.75113 | 4.75113 | 154.69 | 0.000 |
| *p*H**p*H | 1 | 4.75113 | 4.75113 | 154.69 | 0.000 |
| 2-way interaction | 5 | 2.57194 | 0.51439 | 16.75 | 0.000 |
| *p*H*volume | 1 | 0.46765 | 0.46765 | 15.23 | 0.002 |
| *p*H*time | 1 | 0.61491 | 0.61491 | 20.02 | 0.001 |
| *p*H*temperature | 1 | 0.82688 | 0.82688 | 26.92 | 0.000 |
| Volume*temperature | 1 | 0.16415 | 0.16415 | 5.34 | 0.037 |
| Time*temperature | 1 | 0.90906 | 0.90906 | 29.60 | 0.000 |
| Error | 14 | 0.42999 | 0.03071 | | |
| Lack-of-fit | 10 | 0.38275 | 0.03828 | 3.24 | 0.134 |
| Pure error | 4 | 0.04724 | 0.01181 | --- | ----- |
| Total | 25 | 9.73797 | | | |

narrated results under second-order model which includes five main effects, five quadratic effects, and ten 2-way interactions. We gradually improved the model by eliminating insignificant terms of the model except the main effects which were included altogether. We can see from the *p*-value ($p<0.05$) that main effect of salt and volume is not significant for the K/S value. The significant quadratic effect and the interaction effects can be seen from the table. The contribution of different terms of the model and their significance under *T*-test is presented in Table 4. Variance inflation factor (VIF) greater than 1 justifies the results. Statistically, the optimization of dyeing variables has been done by employing various levels using optimum irradiation and extraction conditions given in Figure 5a–c. The result reported in Tables 1 and 2 for response surface recognization of color strength versus extract pH, extract volume, time, and temperature of dye bath as well as salt for getting excellent exhaustion shows that all these parameters have good effects but *p*-values show that effects of contact points (time and temperature) and dye bath pH are highly significant ($p$=0.000).

Mordanting in natural coloration is the need of the hour because this process can only overcome the issue of their poor fastness characteristics. In this study, the sustainable salt of $Al^{3+}$, $Fe^{2+}$, and tannic acid (TA) as chemical and in comparison the herbal-based plant anchors such as extracts of acacia, pomegranate, pine-nut hull, and turmeric have been used at 60–80 °C. The results given in Figure 6a revealed that at 60 °C, 2% of $Al^{3+}$, $Fe^{2+}$, and TA as pre-chemical mordanting whereas 3% of pomegranate and 2% of acacia, turmeric, and pine-nut hull as a pre-bio-mordanting have given excellent shade strength as well as good fastness properties. Similarly, the results given in Figure 7a have shown that 1% of $Al^{3+}$ and $Fe^{2+}$ and 3% of TA as post chemical mordanting and 2% of acacia, 1% of pomegranate, 2% of turmeric, and 1% of pine-nut hull as a post-bio-mordanting at 60 °C have given excellent color strength. At 70 °C, it has been observed that 3% of $Al^{3+}$, 2% of $Fe^{2+}$, and 3% of TA as chemical mordant whereas 3% of acacia, turmeric, and pine-nut hull and 2% of pomegranate extract as bio-mordant have given promising shades (Figure 6b). In the same way, acidic extract of *Cassia obovata* is used before the application of chemical mordanting such as 2% of $Al^{3+}$, $Fe^{2+}$, and TA, and 1% of acacia and 2% of pomegranate, turmeric, and pine-nut hull were used as a post-bio-

**Table 4** Coded coefficients for optimization of dyeing parameters using two-way ANOVA analysis

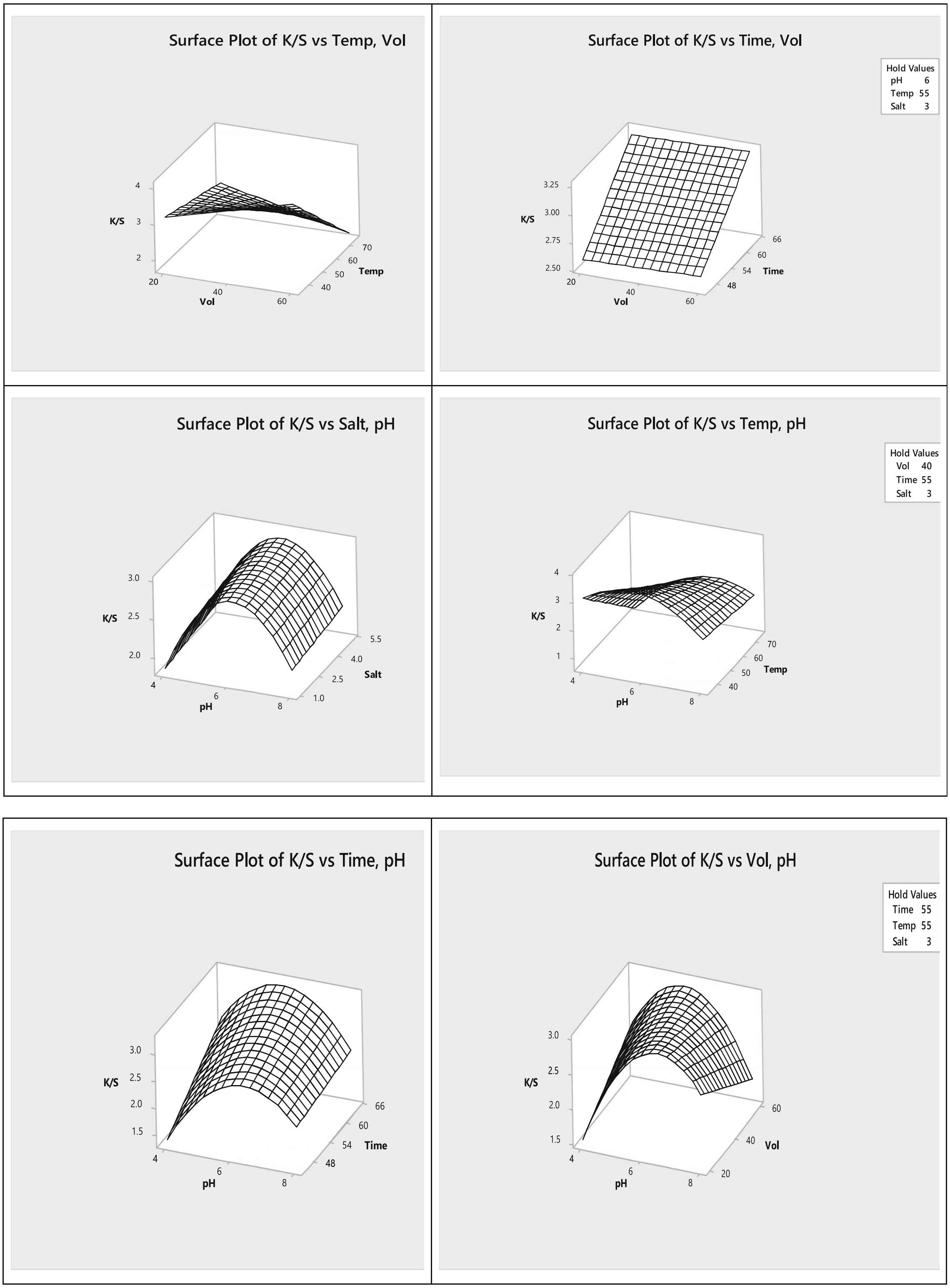
| Term | Effect | Coef | SE Coef | *T*-value | *p*-value | VIF |
|---|---|---|---|---|---|---|
| Constant | | 0.9271 | 0.0528 | 55.40 | 0.000 | 1.09 |
| *p*H | 0.5030 | 0.2515 | 0.0946 | 2.66 | 0.019 | 1.06 |
| Volume | 0.0246 | 0.0123 | 0.0754 | 0.16 | 0.873 | 1.09 |
| Time | 1.3082 | 0.6541 | 0.0946 | 6.91 | 0.000 | 1.06 |
| Temperature | −1.3828 | −0.6914 | 0.0754 | −9.17 | 0.000 | 1.06 |
| Salt | 0.1395 | 0.0698 | 0.0754 | 0.93 | 0.371 | 1.04 |
| *p*H**p*H | −7.058 | −3.529 | 0.284 | −12.44 | 0.000 | 1.09 |
| *p*H*Volume | −1.477 | −0.739 | 0.189 | −3.90 | 0.002 | 1.09 |
| *p*H*time | −1.694 | −0.847 | 0.189 | −4.47 | 0.001 | 1.09 |
| *p*H*temperature | 1.964 | 0.982 | 0.189 | 5.19 | 0.000 | 1.09 |
| Volume*temperature | −0.875 | −0.438 | 0.189 | −2.31 | 0.037 | 1.09 |
| Time*temperature | 2.060 | 1.030 | 0.189 | 5.44 | 0.000 | 1.09 |




**a**

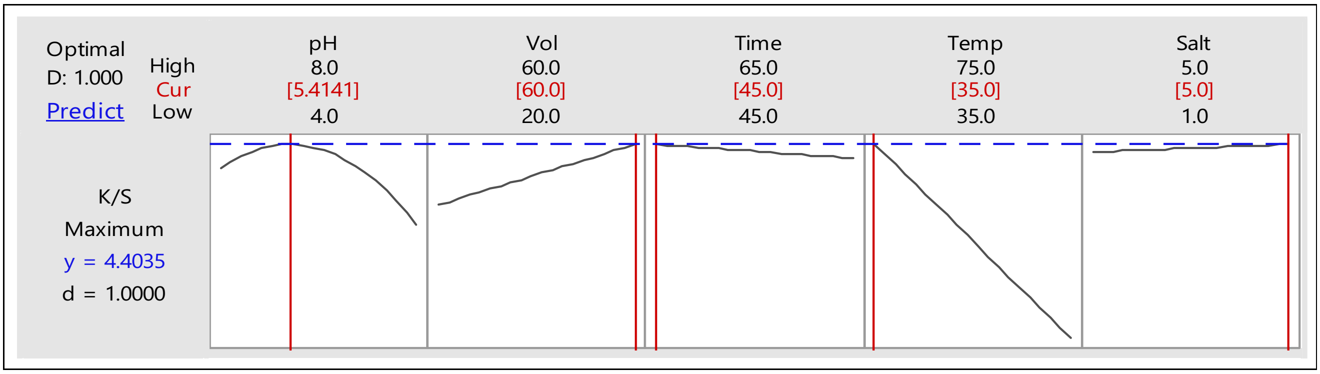
**Fig. 5** **a, b** 3D response graphs of *C. obovata* K/S versus two factors while holding remaining factors at center level. **c** Graphical representation of two-way ANOVA analysis of *C. obovata* K/S versus time *p*H, volume, temperature, and salt using response surface regression

mordanting and acceptable color strength given in Figure 7b. Using microwave ray treatment, during the dyeing of nylon fabric with the acidic extract obtained after irradiation of extract upon pre-mordanting at 80 °C shown in Figure 6c, 2% of $Al^{3+}$ and 1% of $Fe^{2+}$ and TA, as chemical, and 2% of acacia, pomegranate, and pine-nut hull and 1% of turmeric extracts as shade developing anchors have given good color depth (K/S). Similarly, using 80 °C the application of 2% of $Al^{3+}$ and TA and 3% $Fe^{2+}$ as post chemical and 2% of acacia, turmeric, and pine-nut hull and 3% of pomegranate extracts as post-bio-mordant has given desired results (Figure 7c). Overall, it has been observed in Figure 4a–c that the optimum color strength is obtained at 60 °C mordanting temperature using 2% of $Al^{3+}$, $Fe^{2+}$, TA, acacia, pine-nut hull, and turmeric and 3% of pomegranate before mordanting. Likewise, at the same mordanting temperature, fabrics were dyed after (post) mordanting using 1% of $Al^{3+}$, Fe, pomegranate, and pine-nut hull, 2% of acacia and turmeric, and 3% of TA showing brilliant color strength (K/S). The color coordinates given in Table 5 reveal using optimum mordanting temperature (60 °C), mostly dyed fabric produced brighter shades with reddish and more yellow hue using chemical mordants, but a fabric dyed after (pre) the application of 2% of Al salt has given brighter ($L^*$=69.77) shade with redder ($a^*$=6.52) and yellower ($b^*$=25.40) hue. Among bio-mordants used, all the fabric dyed using optimal acidic extract shown much brighter shade, less reddish-greener tint, and yellower hue given in Table 6. But fabric dyed after the application of 2% of turmeric has shown brighter shade ($L^*$=75.85) with less redder hue ($a^*$=1.01) and yellower tone ($b^*$=37.18). Similarly, the color




**b**

**Fig. 5** (continued)




**c**

**Fig. 5** (continued)

coordinates given in Table 5 displayed that after the application of chemical mordants, mostly fabric dyed at (60 °C) exhibit much brighter shades with reddish-bluer tone and more yellow hue, but using 1% of Fe salt used to dye the fabric displayed brighter shades ($L$*=72.08) with redder ($a$*= 7.79) and more yellowish tone ($b$*=35.62). Moreover, 1% of pomegranate is applied before dyeing at 60 °C, and then dyed fabric obtained exhibits brighter redder and yellower tint ($L$*=76.07; $a$*=0.01; $b$*=31.60) given in Table 6. The good color strength is due to the formation of metal dye complex onto surface-modified nylon fabric after MW treatment up to 6 min at optimum mordanting temperature. The proposed interaction of the metal ($M^{n+}$) with dye and fabric has been displayed in Figure 8a. During bio-mordants, the plant functional molecules, i.e., quercetin in acacia, the tannin in pomegranate, curcumin in turmeric, and catechin in pine-nut hull, have

**Table 5** Color characteristics of chemical-mordanted nylon fabrics at various temperatures dyed with *C. obovata* extract

| Temperature | Mordant conc.% | $L$* | $a$* | $b$* | LF | WF | | RF | | DCF |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | CC | CS | DRF | WRF | |
| | Ctrl | 66.54 | 11.30 | 37.45 | 3/4 | 3/4 | 3/4 | 3/4 | 3 | 3/4 |
| 60 °C | 2% Al (pre) | 69.77 | 6.52 | 25.40 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| | 1% Al (post) | 82.51 | −1.02 | 23.00 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| 70 °C | 3% Al (pre) | 76.12 | 3.60 | 22.34 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| | 2% Al (post) | 83.05 | −1.94 | 22.89 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| 80°C | 2% Al (pre) | 75.52 | 2.60 | 21.38 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| | 2% Al. (post) | 82.73 | −0.70 | 20.21 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| 60 °C | 2% Fe (pre) | 65.67 | 8.43 | 29.22 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| | 1% Fe (post) | 72.08 | 7.79 | 35.62 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| 70 °C | 2% Fe (pre) | 76.35 | 2.83 | 27.98 | 5 | 4/5 | 4/5 | 5 | 5 | 5 |
| | 2% Fe (post) | 73.15 | 7.91 | 35.31 | 5 | 4/5 | 4/5 | 5 | 5 | 4/5 |
| 80°C | 1% Fe (pre) | 74.55 | 3.93 | 29.93 | 5 | 4/5 | 4/5 | 5 | 5 | 4/5 |
| | 3% Fe. (post) | 75.08 | 4.35 | 31.28 | 5 | 4/5 | 4/5 | 5 | 5 | 5 |
| 60 °C | 2% TA (pre) | 71.66 | 7.09 | 24.86 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| | 3% TA (post) | 78.02 | 2.09 | 20.05 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| 70 °C | 3% TA (pre) | 72.15 | 6.77 | 23.67 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| | 2% TA (post) | 80.53 | 0.51 | 20.78 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| 80°C | 1% TA (pre) | 75.04 | 4.90 | 21.31 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| | 2% TA (post) | 80.04 | 0.98 | 21.31 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |

*LF* light fastness, *WF* wash fastness, *CC* color change, *CS* color stain, *DRF* dry rubbing fastness, *WRF* wet rubbing fastness, *DCF* dry clean fastness

**Table 6** Color characteristics of bio-mordanted nylon fabrics at various temperatures dyed with *C. obovata* extract

| Temperature | Mordant conc.% | *L** | *a** | *b** | LF | WF | | RF | | DCF |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | CC | CS | DRF | WRF | |
| | Ctrl | 66.54 | 11.30 | 37.45 | ¾ | 3/4 | 3/4 | 3/4 | 3 | 3/4 |
| 60 °C | Acacia (2% pre) | 74.86 | 1.89 | 29.36 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 5 |
| | Acacia (2% post) | 75.17 | 2.83 | 26.31 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 5 |
| 70 °C | Acacia (3% pre) | 78.43 | −1.01 | 29.96 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 5 |
| | Acacia (1% post) | 75.65 | 2.87 | 26.20 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| 80°C | Acacia (2% pre) | 81.72 | −2.00 | 21.22 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| | Acacia (2% post) | 74.81 | 3.03 | 25.57 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| 60 °C | Pomegranate (3% pre) | 67.38 | 4.54 | 35.33 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| | Pomegranate (1% post) | 76.07 | 0.01 | 31.60 | 5 | 4/5 | 4/5 | 5 | 4/5 | 4/5 |
| 70 °C | Pomegranate (2% pre) | 65.66 | 4.98 | 31.31 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| | Pomegranate (2% post) | 77.33 | −0.51 | 31.69 | 5 | 4/5 | 4/5 | 5 | 4/5 | 5 |
| 80°C | Pomegranate (2% pre) | 67.35 | 5.17 | 33.84 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| | Pomegranate (3% post) | 77.09 | −0.37 | 32.10 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| 60 °C | Turmeric (2% pre) | 75.85 | 1.01 | 37.18 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| | Turmeric (2% post) | 73.10 | 2.09 | 33.27 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| 70 °C | Turmeric (3% pre) | 79.31 | −1.22 | 28.76 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| | Turmeric (2% post) | 79.36 | −1.04 | 36.06 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| 80°C | Turmeric (1% pre) | 81.07 | −3.23 | 30.31 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| | Turmeric (2% post) | 74.42 | 1.31 | 30.79 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| 60 °C | Pine-nut hull (2% pre) | 75.57 | 1.58 | 31.61 | 5 | 4/5 | 4/5 | 4/5 | 4 | 4/5 |
| | Pine-nut hull (1% post) | 80.49 | −0.12 | 22.19 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| 70 °C | Pine-nut hull (3% pre) | 77.54 | 0.42 | 28.09 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| | Pine-nut hull (2% post) | 81.82 | −0.78 | 19.67 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 5 |
| 80°C | Pine-nut hull (2% pre) | 77.19 | 0.20 | 27.40 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| | Pine-nut hull (2% post) | 82.52 | −1.23 | 18.57 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |

*LF* light fastness, *WF* wash fastness, *CC* color change, *CS* color stain, *DRF* dry rubbing fastness, *WRF* wet rubbing fastness, *DCF* dry clean fastness

various –OH sites which interact with –OH of colorant and amido linkage (–CO, –NH) of nylon to give new tints with improved color depth (Rani et al. 2020; Thakker 2020). The proposed interaction of bio-mordanted, with nylon fabric and colorant, has been displayed in Figure 8b. Overall in comparison, bio-mordants have not only given soothing tints but also high color strength. Hence the addition of plant-based anchors as an herbal substitute of metal salt can be used to make the process more green, sustainable, and eco-friendly (Yaqub et al. 2020; Hosseinnezhad et al. 2021b).

The color fastness rating given in Tables 5 and 6 reveals that the mordants employed at given conditions have improved the fastness ratings. This is due to the nature of mordants used, the active functional sites of fabric surface modified and colorant (Jabar et al. 2020; Sadeghi-Kiakhani et al. 2020). Mostly, the tannin present in pomegranate extract after application has given excellent fastness ratings. Although chemical anchors due to the formation of firm metal dye complex onto fabric have given good results, bio-mordants have given promising ratings due to the additional benzene ring –OH group and conjugation (Silva et al. 2021; Adeel et al. 2021b, c). Hence, it is recommended that plant-based anchors should be used because of having biological and herbal characteristics as well as to get the desired results. Their application is very useful for fabric coloration, from the global point of view, but also the recycling of their waste to get low shade with good fastness can also be achieved.

## Conclusion

Natural dyes are bio-compatible, less hazardous, and non-allergic due to which these natural colorants are making their place in the world of textiles. It has been found that

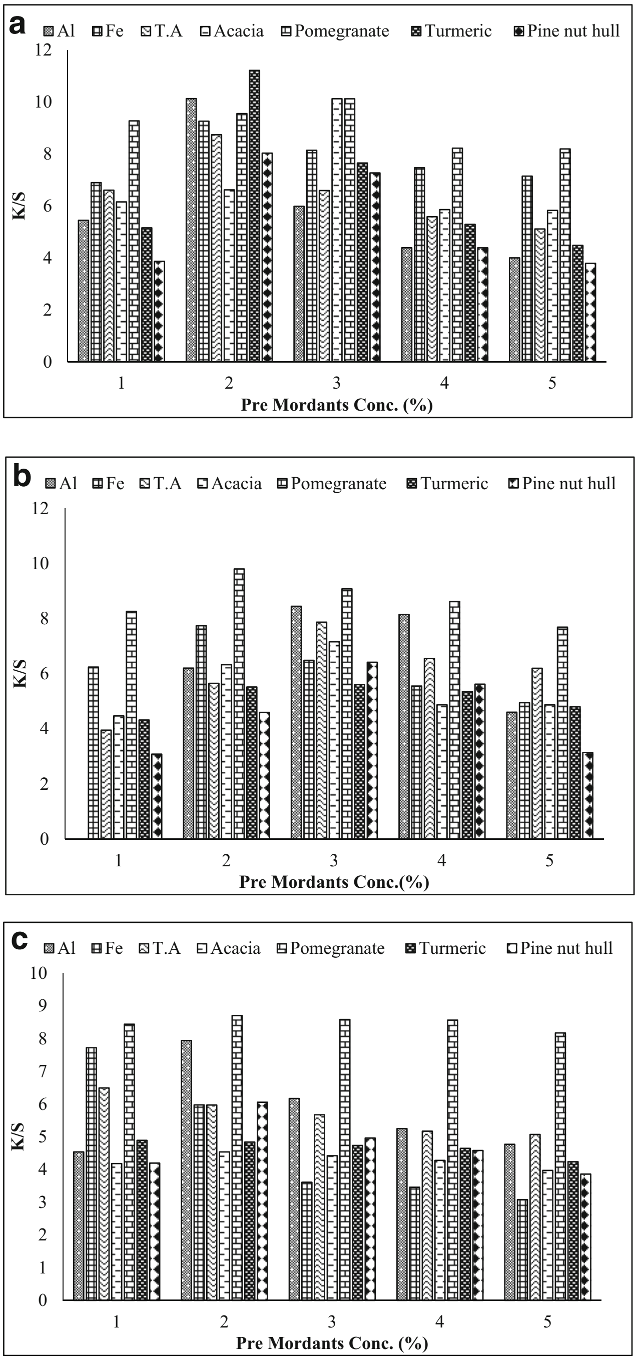



**Fig. 6** **a** Effect of pre-chemical and bio-mordanting at 60 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract. **b** Effect of chemical pre- and bio-pre-mordanting at 70 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract. **c** Effect of chemical post- and bio-post-mordanting at 80 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract

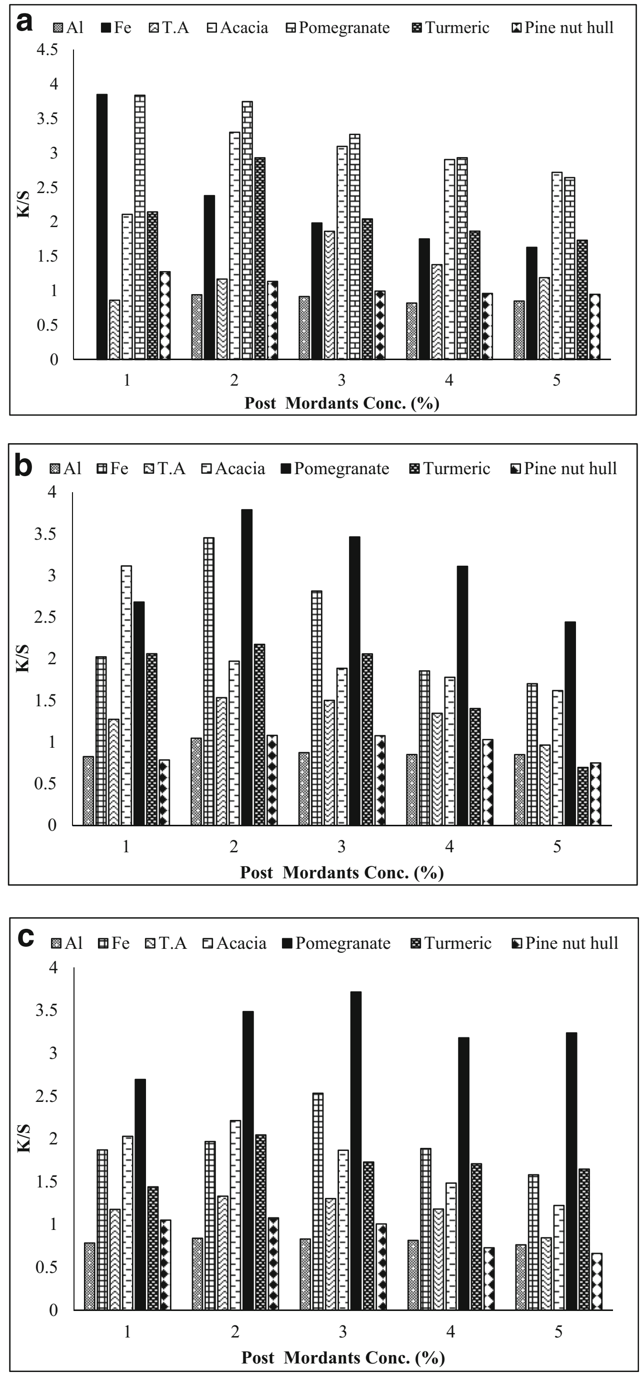



**Fig. 7** **a** Effect of post chemical and bio-mordanting at 60 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract. **b** Effect of chemical post and bio-post-mordanting at 70 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract. **c** Effect of chemical pre and bio-pre-mordanting at 80 °C on dyeing of microwave irradiated nylon fabric using optimum *C. obovata* extract

an innovative method of extractions, i.e., microwave extraction, which has a good potential to yield the natural product though less consumption of solvent, energy, and time should be used. To enhance the shade strength of the dye, bio-mordanting as the newly state-of-the-art tool should be used. In this study, *Cassia obovata* as the new dye yielding plant has been explored under MW

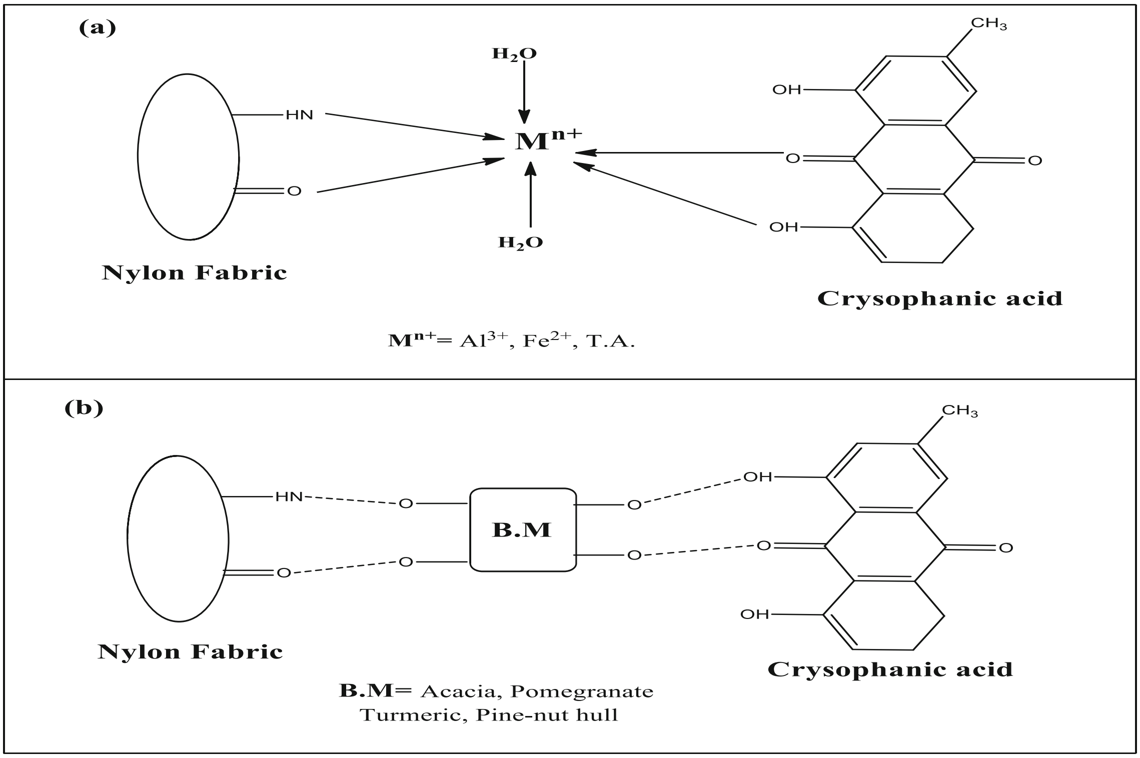



**Fig. 8** Proposed interaction between chemical mordant (**a**) and bio-mordant (**b**) with nylon fabric and colorant of *C. obovata* (chrysophanic acid)

treatment. It is observed that chrysophanic acid dye had good color fastness as compared to methods in which mordants were absent. It has been concluded the presence of bio-mordants and environmentally friendly chemical mordants deepen the color strength into the fabric by forming an extra binding with fabric and dye giving a new tint with improved fasting.

**Author contribution** Dr. Shahid Adeel is the supervisor, where Prof. Dr. Ren Cheng Tang has scientifically guided the work. Mr. Mahmood ul Hasan along with Dr. Fatima Batool and Ms. Nimra Amin have jointly conducted the experiments. Mr. Shahid Rehman Khan has analyzed the effect of radiation on fabrics via physiochemical techniques and Dr. Tanvir Ahmad has statistically analyzed the data.

**Data availability** As this is part of M.Phil studies, so whole data is present in M.Phil thesis.

## Declarations

**Ethics approval** We approve that this manuscript is part of M.Phil studies.

**Competing interests** The authors declare no competing interests.

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