



# Exploring natural colorant behavior of husk of durum (*Triticum durum* Desf.) and bread (*Triticum aestivum* L.) wheat species for sustainable cotton fabric dyeing

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## Abstract

Revival of natural colorants in textile dyeing is one of the important strategies to reduce synthetic chemical-based environmental pollution. The study has been conducted to explore the coloring potential of durum (*Triticum durum* Desf.) and bread (*Triticum aestivum* L.) wheat husk for fabric dyeing. The results showed that both wheat species husk could be an excellent source of natural dye, if extracted in alkaline medium. It has been observed that durum wheat husk based dye worked best at 70°C with a pH 11.0 and salt concentration of 8.0 g/100 ml of solution. Similarly, alkaline extract of bread wheat husk worked better at 80°C with dyeing solution pH 9.0 and salt concentration of 8.0 g/100 ml. Bio-mordanting experiments results revealed pomegranate rind (7%) as most effective bio-mordant to obtain high color strength of wheat husk treated fabric. In chemical-mordanting, tannic acid (5%) as pre-mordant and chrome (5%) as post-mordant have improved the color strength more than all other quantities of employed mordants. FTIR analysis indicated the presence of flavonoids as major colorant compounds in wheat husk-based natural dye. Suggested ISO standards for colorfastness illustrated good color strength ratings of husk-based dyed fabric when treated with bio-mordants as compared to chemical counterparts. Hence, husk of both bread and durum wheat species has great potential to be used as source of eco-friendly natural colorant for cotton dyeing.

**Keywords** Bio-mordanting · Eco-friendly dyeing · Natural colorant · Wheat husk

## Introduction

Synthetic dyes are being repeatedly used in many areas like textile, food, cosmetics, and pharmaceutical industries (Adeel et al. 2019; Haddar et al. 2014). Though these dyes impart strong colors to different materials which attract the people's attention, the effluents are not only hazardous to environment but for human life as well (Khatri et al. 2016; Yusuf et al. 2017; Batoool et al. 2013). Synthetic dyes take a long time to break down and are ecologically unfavorable or environmentally hazardous (Khan et al. 2013; İşmal 2017; Hussaan et al.

2017). Hence, fresh water ecosystems are polluted and becoming unfit for living organisms existence (Hossain et al. 2018) due to continuous addition of the coloring waste (Hassan and Carr 2018). The textile sector is charged as one of the biggest environmental polluters and consumes high amounts of fuels and chemicals (Bhatia 2017). Most of the synthetic dyes, being used in textile processing, show stability against breakdown by microorganisms and hence are not biodegrade under aerobic environment (Mia et al. 2019). The toxicity associated with waste and the harmful materials, being released during the preparation and utilization of chemicals, have emphasized the search for ecofriendly alternatives of synthetic dyes.

Bio-colors, extracted from plants, fungi, insects, and animals (Baaka et al. 2017; Adeel et al. 2018a, b), have gained considerable importance to be utilized in industry owing to awareness regarding environmental pollution associated with the use of synthetic dyes (Rather et al. 2016; Zuber et al. 2020). Contrary to synthetic dyes, natural colorants are safe for humans and harmonized with nature (Kundal et al. 2016). The bio-based colorants have good compatibility with human

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skin with wonderful functional properties such as antimicrobial activity as well as insect repellent, UV protection, and deodorizing nature (Baliarsingh et al. 2012; Bukhari et al. 2017). The fascinating and attracting colors obtained from natural flora and fauna are another reason of attention of human being towards the revival of natural colorant-based textile. Reduced colorants yield and the poor fastness properties of plant-based dyes are the main problems being faced in traditional dyeing. Hence, extraction of maximum colorants from varying plant parts is an important step for the dyeing of textile substrate (Adeel et al. 2020). Studies have indicated that these problems could be minimized using eco-friendly solvents and bio-mordants (Haddar et al. 2018).

A few plant species have been explored with hidden benefits of natural dye yielding characteristics. Overuse of this limited number of dye yielding plants is continuously disturbing bio-diversity and consequently the ecosystems. Utilization of plant leftovers as potential alternatives of significant plant parts, like flowers, seeds, or whole plants in fabric dyeing, is gaining considerable importance as sustainable strategy (Batool et al. 2019). The plant leftovers are easily available, cost effective, and do not have deleterious effects on bio-diversity and eco-balance of nature. Moreover, the crop leftover material may be an additional income-generating source for growers, if utilized in dye yielding process.

Wheat is among the most broadly grown cereal crops, traditionally cultivated for multiple benefits. Durum (*Triticum durum* Desf.) and bread (*Triticum aestivum* L.) wheat species are of significant importance as food source and commercial products (Serpen et al. 2008). Wheat husk comprising lignocellulosic material is used as cattle food and fuel. Most of the times, wheat husk is treated as agricultural raw material and usually burn by farmers. However, beneficial utilization of wheat straw may have value addition characteristic for growers (Kuta and Golab 2014). Considering the utilization of natural dyes in recent industrial processes and bio-diversity losses due to overuse of dye yielding plants, the current study has been conducted to evaluate dyeing parameters for durum and bread wheat species. The objective of the study is to use wheat husk in value added form to increase economic importance of wheat and to provide a variety of shades of colorants extracted from selected species of wheat to textile industry by utilizing bio- and chemical-mordants.

## Material and methods

### Experimental material and sample preparation

The experimental work was conducted at Economic Botany Lab, Govt. College University Faisalabad, Pakistan. Husk of two wheat species, *Triticum durum* Desf. (durum wheat) and *Triticum aestivum* L. (bread wheat), was collected from Wheat Research Institute, AARI, Faisalabad, Pakistan. The husk was

washed with tap water, dried under shade, and grounded into fine powder followed by meshing (20 meshes) to get uniform particle size. Plain, mill mercirized cotton was obtained from Noor Fatima fabrics Faisalabad Pakistan.

### Extraction of colorant

Extraction of colorant from husk of both wheat species was carried out using grinded powder. Following media used to extract the colorant from the wheat husk powder.

1. Aqueous media: tape water
2. Three alkaline media, potassium hydroxide (KOH), sodium hydroxide (NaOH), sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), were used in different concentrations like 1, 3, 5, 7, and 9%.
3. Organic solvents, methanol, ethanol, acetone, and N-hexane were used in different concentration such as, 20, 40, 60, and 80%
4. Acidic media, 1, 2, and 3% HCl, were used for acidic extraction
5. Methanol + KOH and ethanol + KOH media were also prepared using 100% pure ethanol or methanol as solvent and KOH (1, 3, 5, 7, and 9%)

After extraction, the respective filtrate were cooled and used to dye cotton fabric. The fabric was dyed at 60°C for 60 min keeping M:L ratio 1:25.

### Optimization of dyeing conditions

The optimum extracts of both wheat species were used to evaluate the dyeing parameters or dyeing conditions. A range of pH values (1, 3, 5, 7, 9, and 11) of extract was tested to evaluate optimum pH of dyeing extract. The dyeing temperature was optimized using different temperatures (20, 30, 40, 50, 60, 70, and 80°C). The optimum salt concentration was evaluated using different concentrations of salt (2, 4, 6, 8, and 10 g/100ml). The dyeing time was also optimized by dyeing the cotton fabric for different time intervals like 20, 30, 40, 50, 60, and 70 min.

### Color development process

For the improvement of colorfastness and color strength properties, pre- and post-mordanting was performed. The mordanting was done by using the bio-mordants like Acacia bark, pomegranate peel, and mango bark. Metallic mordants, like Fe (iron sulfate) and Al (aluminum sulfate) and tannic acid, were used. The cotton fabrics were treated separately with various concentrations (1, 3, 5, 7, or 9%) of each of abovementioned bio-mordants and metallic mordants (Tables 1 and 2).

**Table 1** Fastness ratings of pre- and post-chemical-mordanted of cotton fabrics dyed using extracts of colorant

<i>Triticum durum</i> Desf.					<i>Triticum aestivum</i> L.				
Mordant Conc.	LF	WF	DRF	WRF	Mordant Conc.	LF	WF	DRF	WRF
Al 9% pre	4	4	4	4	Al 1% pre	4	4	3	¾
Al 9% post	4	4	4	4	Al 3% post	4	4	4	4
T.A 9% pre	4	5	5	4	T.A 9% pre	4	4	4	4
T.A 7% post	4	4	4	5	T.A 7% post	4	4	4/5	4
Fe 5% pre	4	4	4	4	Fe 9% pre	5	5	4	4
Fe 5% post	4	4	4	4	Fe 9% post	5	5	4	4

LF light fastness, WF wash fastness, DRF dry rub fastness, WRF wet rub fastness

## Evaluation of the quality characteristics of dyed fabrics and FTIR analysis

The color strength values of dyed fabrics and mordanted fabrics were investigated by CIE lab system using the Spectraflash (SF 600). Color fastness properties of all mordanted and dyed fabrics were evaluated by following ISO standard methods. The protocol, ISO-105 CO3, was followed for washing test, ISO 105 X 12 for rubbing and ISO 105 BO2 light fastness test of dyed fabric. Fourier transform infrared spectroscopy (FTIR) of the dye powder was performed

## Results

The results (Fig. 1a and b) from natural colorant extraction experiments showed that different concentrations of all the extraction media used in the study produced varying color depth of dyed cotton fabric. The good color strength from husk-based dye could be obtained using mild alkaline extraction medium (8% Na<sub>2</sub>CO<sub>3</sub>). In general, the natural colorants of darker shades were obtained from durum wheat husk using Na<sub>2</sub>CO<sub>3</sub>, ethanol, acetone, and HCl extraction media compared with bread wheat. The result given in Fig. 1a showed that extract obtained in 40% acetone medium

using durum wheat husk and 80% acetone for bread wheat husk resulted darker shades. Other organic solvents such as methanol and N hexane did not prove better to extract colorants from husk of both wheat species. The data presented in Fig. 1 revealed that extraction of dye in the acidic and organic + alkaline solvents (methanol + KOH and ethanol + KOH) did not produce shades of good color strength. Overall, alkaline medium (8% Na<sub>2</sub>CO<sub>3</sub>) produced good color strength (K/S) on fabric treated with husk of both wheat species.

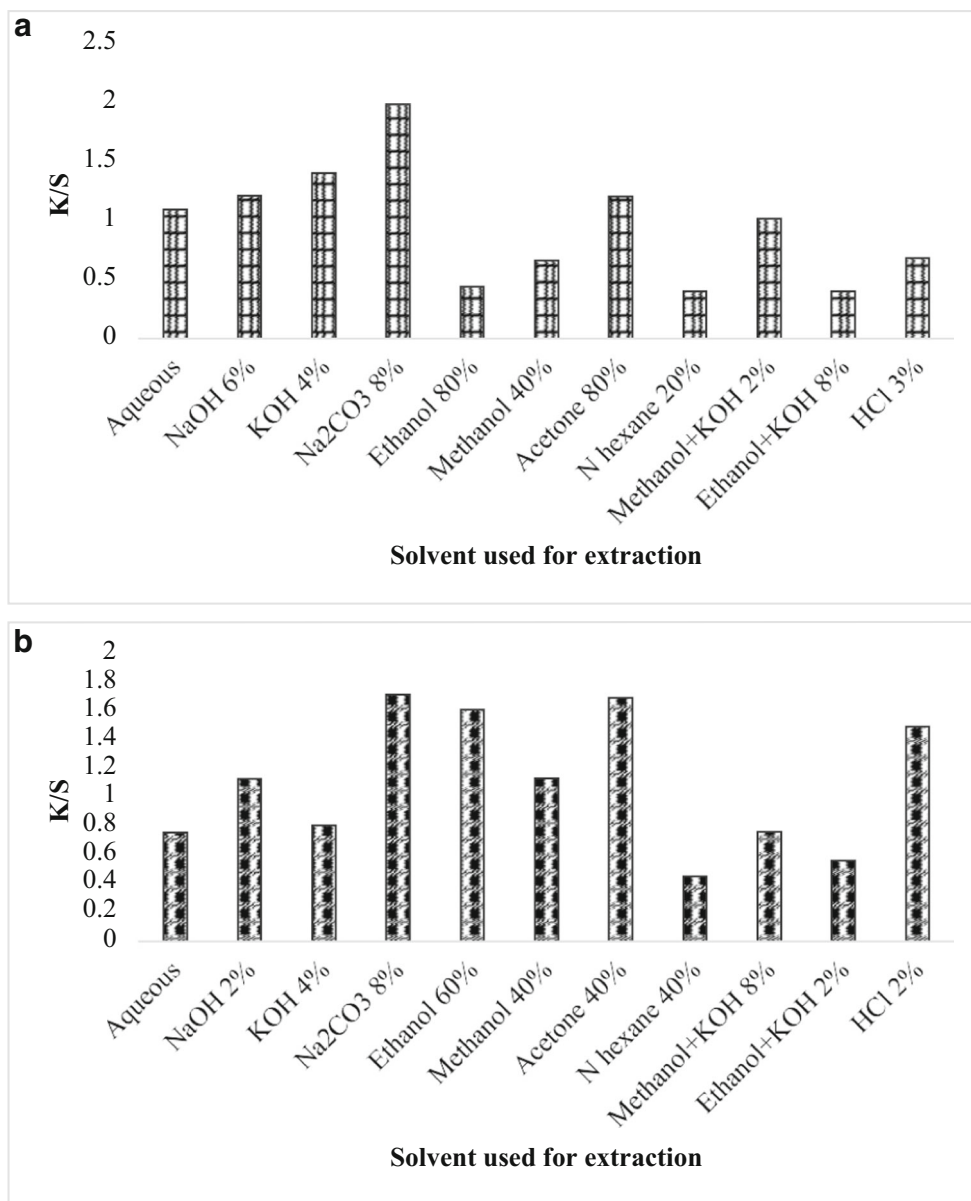
Heating of wheat plant-based natural colorants using dye bath resulted in promising effect on dyeing of cellulosic fabrics (Fig. 2). The results showed in Fig. 2 also revealed that 70°C was the optimum heating level for getting darker shades from durum wheat-based colorants. Similarly, 80°C was the best dyeing temperature for maximum colorant extraction from bread wheat residues. The results from exhausting agent optimization experiments (Fig. 3) revealed that 8.0 g of sodium chloride salt was the best level to enhance the color strength of fabric dyed separately with colorants obtained from both wheat species compared with all other levels of salt used in the study. Among neutral to alkaline (Fig. 4) and acidic dye bath medium, employed during current study, alkaline extract of durum wheat produced darker shades on cellulosic fabric in basic medium of pH 9.0. Similarly, dyeing bath medium having pH 11.0 gave darker shades of fabric

**Table 2** Fastness ratings of pre- and post-bio-mordanted of cotton fabrics dyed using extracts of colorant

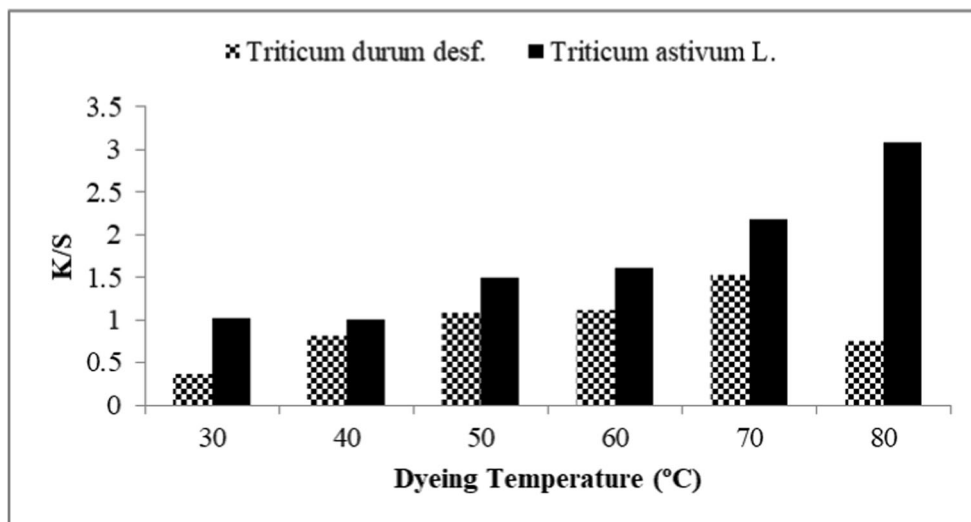
<i>Triticum durum</i> Desf.					<i>Triticum aestivum</i> L.				
Mordant Conc.	LF	WF	DRF	WRF	Mordant Conc.	LF	WF	DRF	WRF
Pomegranate 5% pre	4	5	5	4	Pomegranate 3% pre	5	5	4	4
Pomegranate 7% post	4	4	4	5	Pomegranate 3% post	¾	¾	4	4
Acacia 1% pre	4	4	¾	4	Acacia 9% pre	¾	3	4	3
Acacia 7% post	5	4	4	4	Acacia 5% post	5	4	4/5	4/5
Mango 3% pre	4	¾	4	4	Mango 1% pre	3	4	3	¾
Mango 1% post	4	4	4	4	Mango 9% post	4	4	4	4

LF light fastness, WF wash fastness, DRF dry rub fastness, WRF wet rub fastness

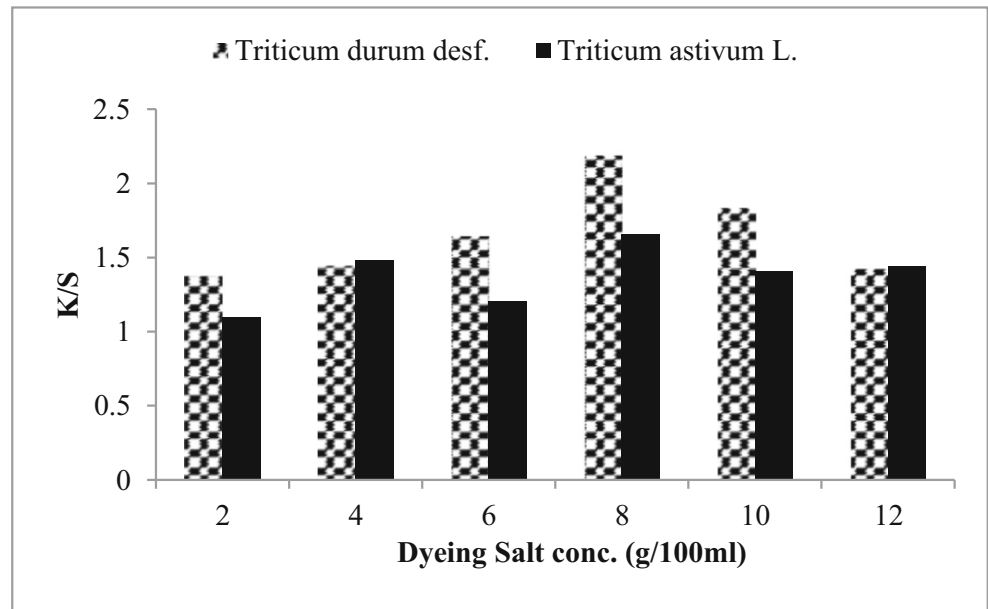
**Fig. 1 a** Extraction of colorant using different solvents from bread wheat (*Triticum astivum* L.) husk. **b** Extraction of colorants using different solvents from durum wheat (*Triticum durum* Desf.) husk



**Fig. 2** Effect of temperature on dyeing of cotton fabric using husk extracts of two wheat species, *Triticum durum* Desf. and *Triticum astivum* L.



**Fig. 3** Effect of exhausting agent on dyeing of cotton fabric using husk extracts of wheat species, *Triticum durum* Desf. and *Triticum astivum* L.

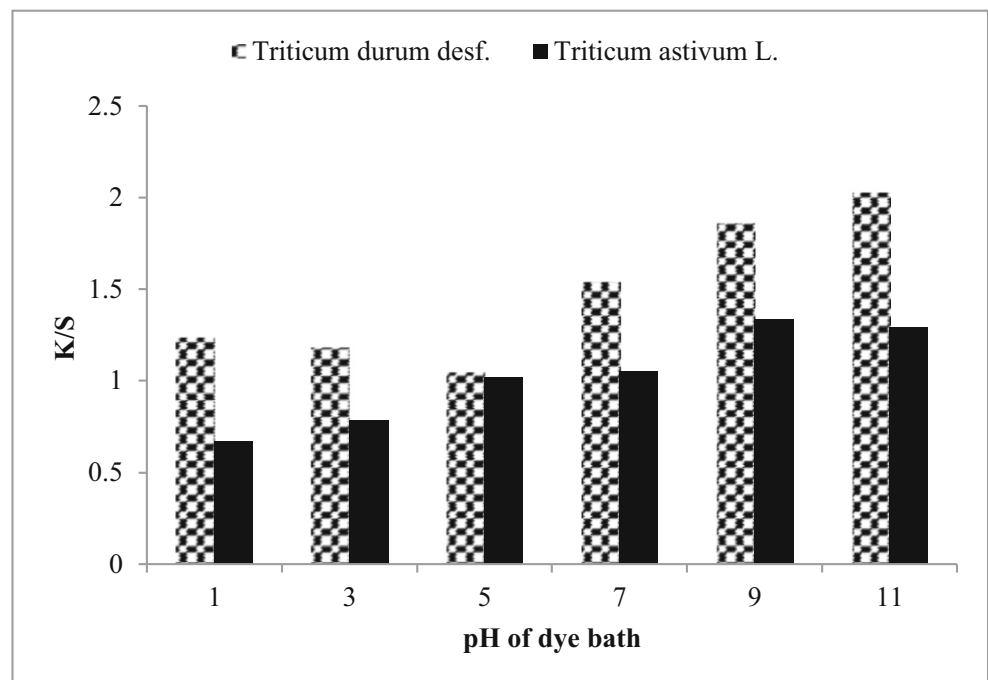


treated with bread wheat husk than all other pH levels used in the study. The results presented in Fig. 5 showed that sorption of colorant into cotton fabric was more favored after treatment of fabric with dye extract of both wheat species for 60 min.

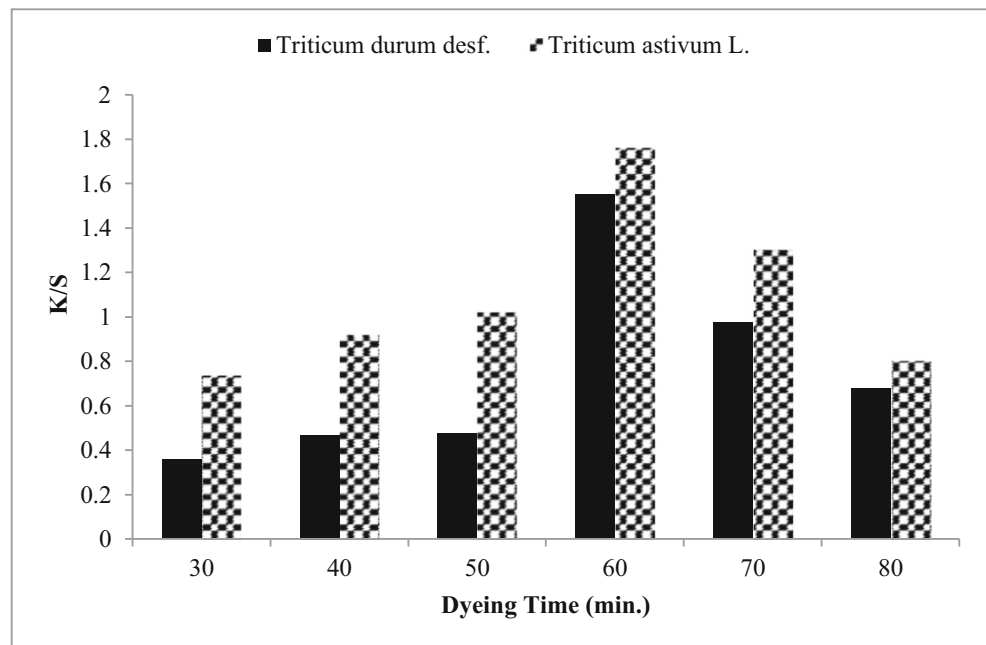
Metallic mordanting results revealed that post-mordanting with iron sulfate and tannic acid produced relatively darker shades and excellent colorfastness characteristics of fabric treated separately with extract of both wheat species (Fig. 6a). Post-mordanting with iron sulfate level of 7% showed best results for cotton fabric dyed with bread wheat husk, while durum wheat-treated fabric showed high color strength

with 5% iron sulfate post-mordanting. Other metallic mordants aluminum sulfate and tannic acid also have produced variety of shades but with relatively low  $k/s$  values and fastness properties as compared with iron sulfate. Mordanting of cotton fabric before dyeing with metallic mordants revealed that 7% iron sulfate produced darker shades with acceptable colorfastness characteristics (Fig. 6b). Iron sulfate as pre-mordant also produced relatively good color depth on wheat extract-treated fabric compared with other metallic mordants used in the study except tannic acid. In bread wheat-treated fabric, 7% iron sulfate as post-mordant gave

**Fig. 4** Effect of dye bath pH on dyeing of cotton fabric using husk extract of wheat species, *Triticum durum* Desf. and *Triticum astivum* L.



**Fig. 5** Effect of time duration on dyeing of cotton fabric using husk extracts of wheat species, *Triticum durum* Desf. and *Triticum aestivum* L.



high color yield followed by 5% tannic acid. The post-mordanting was more effective in both the plant species than the pre-mordanting.

Bio-mordants application also added more value in shade variability and produced high color strength of wheat husk extract-treated fabric. The results narrated in Fig. 7a revealed that use of pomegranate rind and Acacia bark was more effective in producing high color depth on cellulosic fabric dyed with alkaline extract of durum wheat husk. Results also revealed that pre-mordanting with the pomegranate rind extract showed best color strength at 7% extract in mild alkaline medium of both durum and bread wheat husk. The post-mordanting with bio-mordants also imparted good color strength of wheat husk-treated fabric. The results given in Fig. 7b showed that bread wheat husk powder produced excellent *k/s* values with 3% pomegranate rind extract. Other bio-mordants like Acacia and mango tree bark also imparted good color characteristics and fastness properties but less than the pomegranate rind extract. Durum wheat husk extract produced good color fastness with 7% pomegranate rind extract, followed by 1% mango tree bark powder and 7% Acacia bark. Overall, 3% pomegranate rind showed best result in bread wheat husk and 7% pomegranate rind extract showed best result in durum wheat (Fig. 8).

## Discussion

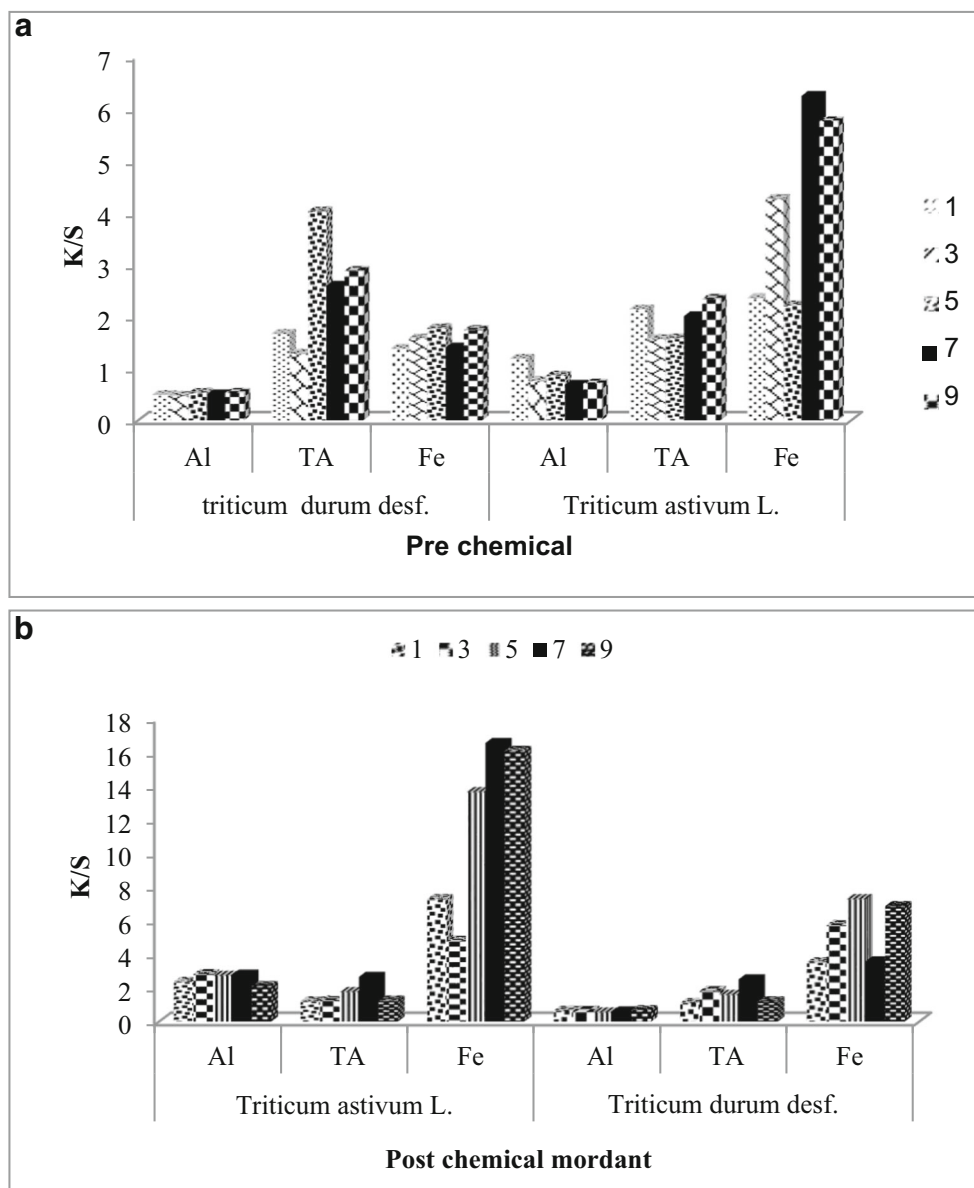
As evident from the results of current study that phytochemicals did not involve with colorant by using aqueous, acid, and highly alkaline media. Similarly, data presented in Fig. 1a and b revealed that alkaline and organic medium imparted high

color strength indicating high extractable power of this solvent towards colorant isolation from the cell wall resulting good color yield (Khan et al. 2014). The results from dye bath temperature optimization experiments indicated that heating at relatively low temperature did not stimulate the colorant to move towards fabric for significant sorption. Similarly, heating at much higher temperature resulted in degradation of colorant and involvement of other phytochemical for sorption onto the fabric. Hence, heating at 70–80°C was the optimum temperature for color extraction from husk of both wheat species.

The better color strength of 8.0 g NaCl-treated fabric indicated that this concentration of exhausting agent efficiently minimized the repulsion between negative charge of the fabric and colorant molecules thus produced darker shades (Zuber et al. 2020). The low amount of salt (less than 8.0 g/100 ml) was not sufficient enough to make significant interactions between fabric and colorant and hence produced relatively less color strength of wheat husk-treated fabric, whereas, high amount of salt (higher than 8.0g/100 ml) might have caused over exhaustion, resulting in clusters of molecules at cellulosic surface and upon washing and investigation in spectrafash, showed low color depth. The optimum concentration of 8.0 g/100 ml of table salt as an exhausting agent as proved during current investigation had also been reported for other photosynthetic organisms like algae (Azeem et al. 2019).

The results of pH optimization experiments during current investigation indicated that acidic conditions did not favor cellulosic –OH group to counteract with functional moiety of flavonoids of durum wheat straw and hence produced low color strength. Whereas, too much alkaline

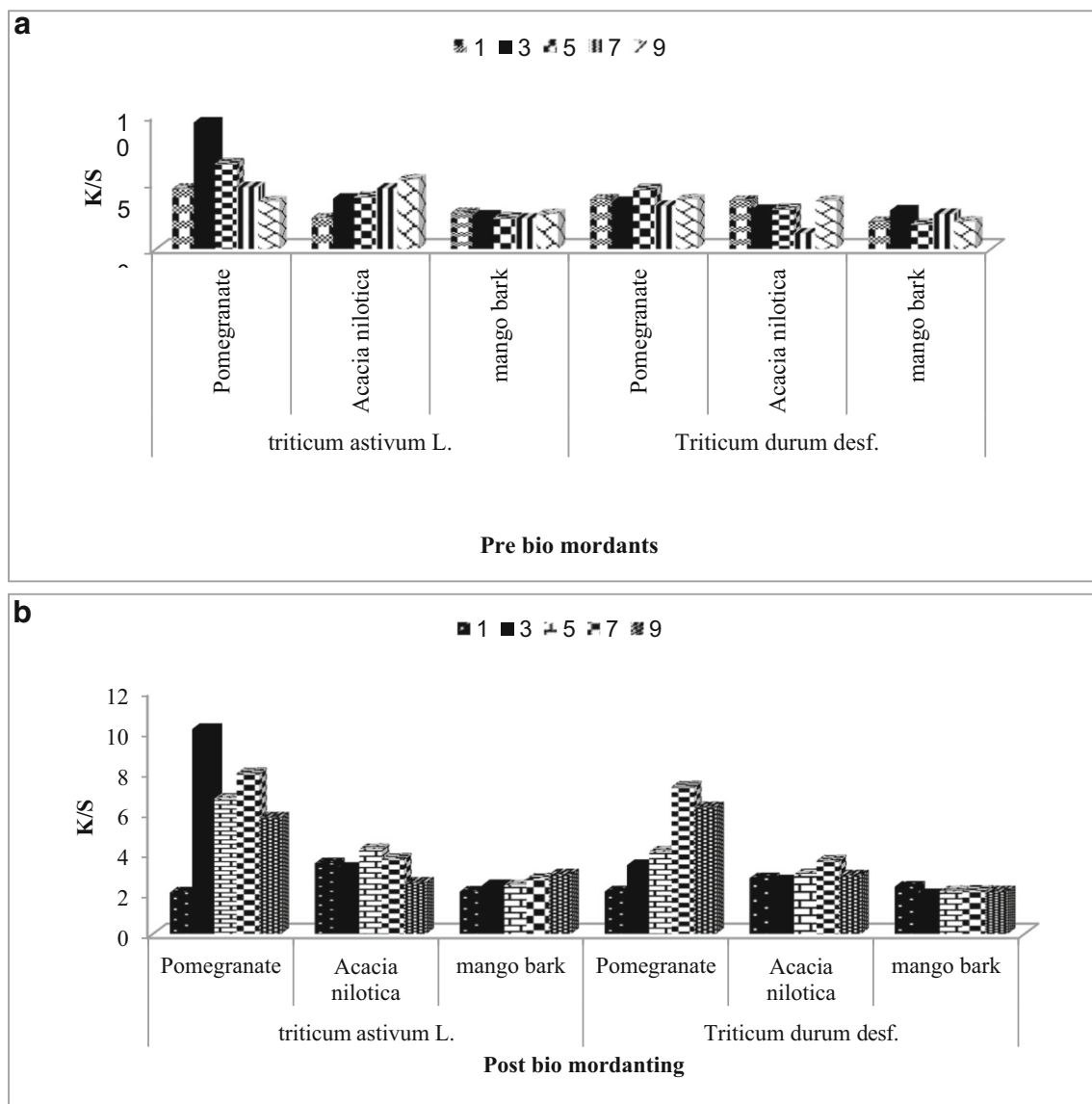
**Fig. 6** **a** Application of pre-metallic mordanting on cotton dyeing using husk extracts of wheat species, *Triticum durum* Desf. and *Triticum astivum* L. **b** Application of post-metallic mordanting on cotton dyeing using husk extracts of wheat species, *Triticum durum* Desf. and *Triticum astivum* L.



medium caused swelling and weakening of cellulosic fabric resulting relatively low color strength of fabric as that of optimum pH. The imbibitions of cellulosic fabrics in highly alkaline medium resulted into weak interaction of colorant which in turn gave low shades (Bukhari et al. 2017). Hence, dyeing of cellulosic fabric was darker in alkaline medium using alkaline solubilized extract of wheat husk to get good color coordinate. Dyeing for optimal time not only helped to make a viable bond between fabric and colorant but also added value in coloration and uptake ability. This was because the colorant imparted darker yellowish shade while contacting with fabric for 60 min due to better colorant interaction. Hence, to get darker shades with maximum  $k/s$  value, 60-min time was

the optimal extract duration for dyeing of cotton using wheat husk powder during current investigation and as described by Batool et al. (2019) for vegetable residue-based colorant.

The effectiveness of 7% of iron sulfate as post-mordanting agent to produce better color strength as well as fastness characteristics as recorded during present study pointed towards good metal dye complex formation through good diffusion of this concentration (Adeel et al. 2017). Tannic acid also imparted good color depth that might be due to the high reduction power and metal dye complex formation (Yusuf et al. 2017; Adeel et al. 2020) as well as intermolecular covalent bonding of OH of tannic acid with  $-OH$  of colorant and cellulosic functional moiety (Amin et al. 2020). Variety of color shades produced



**Fig. 7** **a** Application of pre-bio mordanting on cotton dyeing using extracts of wheat husks *Triticum durum* Desf. and *Triticum astivum* L. **b** Application of post-bio-mordanting on cotton dyeing using extracts of wheat husks *Triticum durum* Desf. and *Triticum astivum* L.

with mordanting agents might also be due to intermolecular H-bonding with colorant –OH and active site of cellulosic fabric.

The characteristic peak obtained during FTIR analysis showed that optima extract of both the species of wheat contained flavonoids as major colorant component. The peak at  $3370\text{cm}^{-1}$  corresponds to the presence of OH group whereas at  $1570\text{cm}^{-1}$  and at  $1406\text{cm}^{-1}$  show the presence of aromatic ring (MaciejHenczkowski et al. 2001).

### Conclusion

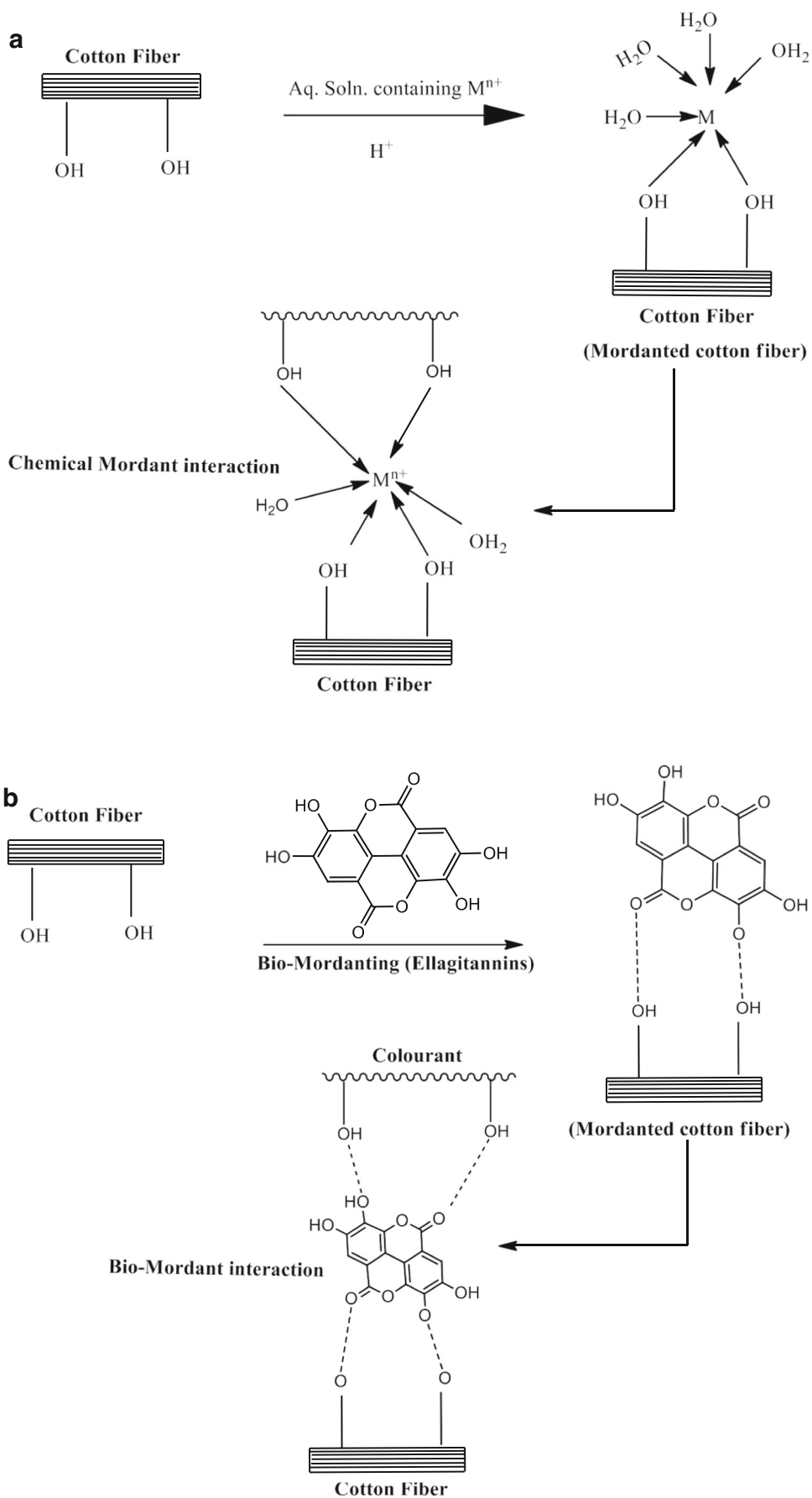
In conclusion, both wheat species husk could be used as eco-friendly source of natural dye. The optimal conditions for extraction of colorant from durum wheat husk were  $70^\circ\text{C}$  temperature

with dyeing bath pH 11.0 and salt concentration of  $8.0\text{ g}/100\text{ ml}$  of solution. The bread wheat husk-based colorant worked better at  $80^\circ\text{C}$  with dyeing solution pH 9.0 and salt concentration of  $8.0\text{ g}/100\text{ ml}$  as exhausting agent. Pomegranate rind (7%) was most effective bio-mordant to obtain high color strength with both wheat species husk extract. Tannic acid (5%) as pre-mordant and chrome (5%) as post-mordant produced good color strength of wheat husk-treated fabric.

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**Fig. 8** **a** Proposed mechanism for interaction between chemical-mordant, cotton, and colorant. **b** Proposed mechanism for interaction between bio-mordant, cotton, and colorant



**Author contribution** The current article is an outcome of PhD research work of Mr Ali Ahmad Khan. Ali A Khan, Naeem Iqbal, and M Azeem contributed in conceptualization, formal analysis, data curation, project administration, and writing. Shahid Adeel contributed in formal analysis, data curation, project administration, and writing.

**Availability of data and materials** All data generated or analyzed during this study are included in this published article (and its supplementary information files).

## Declarations

**Ethical approval** Not applicable

**Consent to participate** Not applicable

**Consent to publish** Not applicable

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

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