Textile & Leather Review

ISSN 2623-6281 | www.tlr-journal.com | 10.31881/TLR

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How to cite: Nasreen H, Adeel S, Yameen M, Amin N, Ozomay M, Qayyum MA. Green Application of Ultrasonic Waves for Extraction of Yellow Colorant from *Haar Singhar* and its Colouring Behaviour in Cotton Dyeing. Textile & Leather Review. 2023; 6:18-36. https://doi.org/10.31881/TLR.2022.67

How to link: https://doi.org/10.31881/TLR.2022.67

Published: 16 January 2023

Green Application of Ultrasonic Waves for Extraction of Yellow Colorant from *Haar Singhar* and its Colouring Behaviour in Cotton Dyeing

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Article
https://doi.org/10.31881/TLR.2022.67
Received 30 August 2022; Accepted 4 January 2023; Published 16 January 2023

ABSTRACT

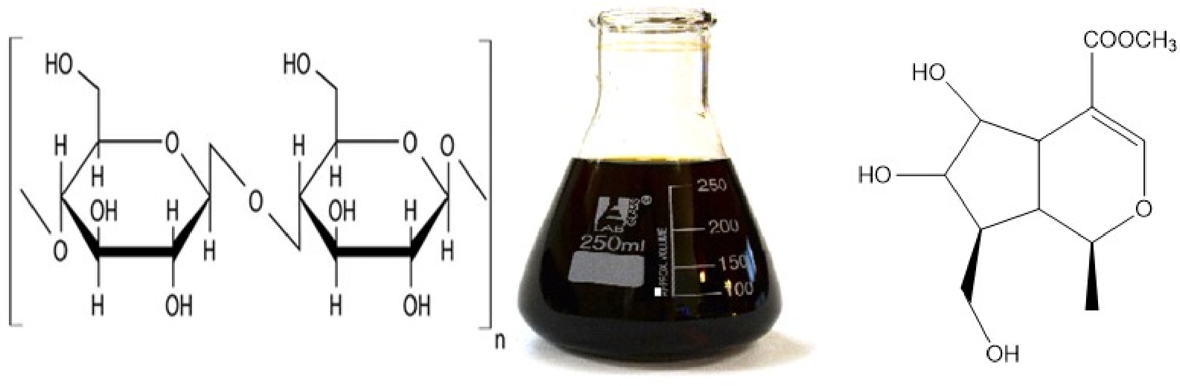
Natural dyes have grown in popularity due to their eco-friendliness. They can be used as a substitute for synthetic dyes to minimize environmental pollution. This research investigates the natural colouring behaviour of haar singhar flower (HSF) extract in cotton dyeing. Colorant was extracted under various conditions utilizing various extraction mediums such as aqueous, basic, and acidic. On cotton, it was discovered that applying 55 mL of aqueous extract containing 1.5 g/100 mL sodium chloride for 40 minutes at 70 °C yielded the best colour output. A new hue with good colour fastness was developed utilizing chemical and bio-mordants. The existence of nyctanthin as a colouring agent in haar singhar flowers was discovered through FTIR analysis of the extract. The CIE Lab system revealed that using 2 g/100 mL of pistachio shell as a bio-mordant resulted in good quality reddish yellow hues. It was discovered that ultrasonic radiations have a high potential for isolating colourants and dyeing cotton fabric under decreased conditions of temperature, time and volume. The application of biomordants has made the procedure greener, more efficient, and safer.

KEYWORDS

bio mordants, chemical mordants, cotton, haar singhar, nyctanthin, ultrasound

INTRODUCTION

Synthetic dyes are widely utilized in textile, food, cosmetic, and pharmaceutical industries because of their high colouring power, reproducibility and low price [1,2,3]. These dyes offer a wide spectrum of colours as well as excellent fastness [4]. The widespread use of synthetic dyes is one of the major components of environmental pollution [5]. Around 8500 mt of dyestuff is discharged each year worldwide, with 55% of their effluent being spilt into water bodies without being purified. This high concentration of dyes stuff obstructs light from entering deep into water bodies, disrupting the aquatic environment. They affect the aquatic photosynthetic activity and degrade water quality, posing a threat to agricultural land and marine life [6]. These pollutants contain non-biodegradable carcinogenic elements and need a significant amount of time, money, and work to treat [7,8]. In all sectors, including textiles, food, pharmaceuticals, and cosmetics the manufacturers are compelling to switch such products with green, and sustainable products [9]. Antioxidant, anti-allergic, and antibacterial characteristics are found in most natural dyes [10,11]. Because of their significant advantages, these dyes can be labelled green, eco-friendly with high UV protection, and therapeutic qualities [12,13]. Some natural dyes yield brighter, better, faster, luminous, attractive, softer, and calmer colour shades in the textile industry, as well as insect-repellent, and sanitizing qualities [14]. These dyes have some disadvantages, such as a low exhaustion rate, slowness and unsatisfactory light and washing fastness. In natural dyeing, mordants are used to increase colouration qualities and fastness [15]. Mordants are significant in natural dyeing processes because they interact and make fabric-mordant complexes. Biomordants substitute chemical mordants in fabric dyeing for environmentally friendly textile dyeing [16,17,18]. Traditional extraction processes are eco-friendly and greener for isolating colourants, but they are laborious time-consuming, and inefficient [19,20]. Modern methods of extraction and dyeing such as ultrasonic radiations, ultraviolet radiations, microwave radiations, and gamma rays have aided the green dyeing process by increasing colour strength and colourfastness [21,22]. Ultrasonic radiation is the most effective and energy-efficient advanced technique of radiation. The cavitation mechanism in the ultrasound extraction method results in two strategies of cavitation and heating, as cavitation molecules knock at the surface of a solid substrate and create strong vibrations that quickly mix the altered layer by fully dispersing the molecules at the surface, cavitation removes air. It improves the fabric's surface physically, which boosts dye adsorption [23,24,25]. The ultrasonic approach is more effective, saving money, energy, and labour since it bursts the cell wall without physically harming the physiologic property [26,27]. This research focuses on the use of a natural colourant (nyctanthin) extracted from the haar singhar flower for the dyeing of cotton fabric. Flavanols, glycosides, D-mannitol, and nicotiflorin are among the phytochemicals found in this plant [28]. The haar singhar flowers have antibacterial, anti-inflammatory, and antimicrobial activities, and the whole plant has medicinal activities. Haar singhar treats rheumatoid arthritis, pyrexia, malaria, cancer, parasitic infections, skin ailments, sciatica etc. [29]. Cotton is an excellent natural fabric having a hydroxyl bond allowing it to interact with colourant molecules and mordant through terminal hydroxyl groups [30].



a) b) c)

Figure 1. a) Chemical structure of (cellulose) cotton b) Haar singhar flower extract c) Colorant (Nyctanthin)

To the best knowledge of the researchers, no such detailed investigation on the dyeing of haar singhar plant for textiles has been conducted. Colouring has been examined in *Nyctanthes arbortritis* as a natural yellow colourant to dye cotton fabric under the effect of US radiations for the first time, with the addition of a long-lasting and pleasing colour with excellent fastness attributes.

The goals of this research are:

a) Isolation of colourant in a selective medium using the US radiations.
b) Evaluation of the physical and chemical makeup of materials before and after US irradiation via FTIR.
c) Production of the best shade with the least amount of mordants, optimizing the mordanting temperature.
d) Assessment of the colourfastness of coloured cotton fabric by pre and post-mordanting under optimal conditions.

METHODOLOGY

Sample collection

Haar singhar (*Nyctanthes arbortritis*) flowers were bought from the herbal market in Faisalabad. Flowers were washed with water, dried in the air, and then ground into a fine powder. Cotton fabric was obtained from a fabric store in Faisalabad, Punjab, Pakistan for dyeing. All the chemicals used were commercial-grade, including sodium hydroxide, hydrochloric acid, and iron sulphate. Pine nutshell (*Pinus gerardiana*), walnut shell (*Juglans regia*), and Pistachio shell (*Pistacia vera*) were among the natural bio-mordants procured from the herbal market Faisalabad.

Extraction of colorant

Aqueous, alkaline (NaOH), and acidic media (Conc. HCl) was used to extract colourants from the powder. Extraction of colourant was carried out by boiling 4.0 g of haar singhar flower powder (HSFP) in the selected media (HCl 1 mL/100 mL, Na_2CO_3 2 g/10 mL) for 45 minutes at 60 °C, with the material to liquid ratio of 1:25 and then filtered.

Optimization of dyeing parameters

Using an optimum extract of haar singhar flowers, different dyeing parameters were examined to achieve higher colour strength, along with the following:

- Dye powder (g/100 mL)	4
- Time (min)	20, 30, 40, 50, and 60
- Salt concentration (%)	0.5, 1, 1.5, 2, and 2.5
- Temperature (°C)	50, 60, 70, 80, and 90
- pH	6, 7, 8, 9, and 10
- Volume (mL)	25,35,45,55, and 65

Mordanting

Before and after the dyeing of cotton fabric bio and chemical mordants were utilized at defined conditions to produce a range of colours as well as increase the fastness characteristics of dyed fabrics. Bio-mordants included 0.5, 1, 1.5, 2, and 2.5% extracts of Pine nutshell (*Pinus gerardiana*), walnut shell (*Juglans regia*), and Pistachio shell (*Pistacia vera*), as well as chemical mordants such as salts of iron and aluminium along with sodium potassium tartrate (0.5, 1, 1.5, 2, and 2.5%) were employed at selected conditions.

Analysis of the properties of coloured cotton fabric

Spectra Flash SF 600 was used to investigate the colour strength of all mordanted and dyed fabrics. Mordanted dyed fabrics were examined for colourfastness using ISO standard rubbing, washing, and light fastness methods. FTIR was used to determine the colourant molecule isolated from haar singhar flowers. To evaluate the research findings, ANOVA was utilized as a statistical analysis.

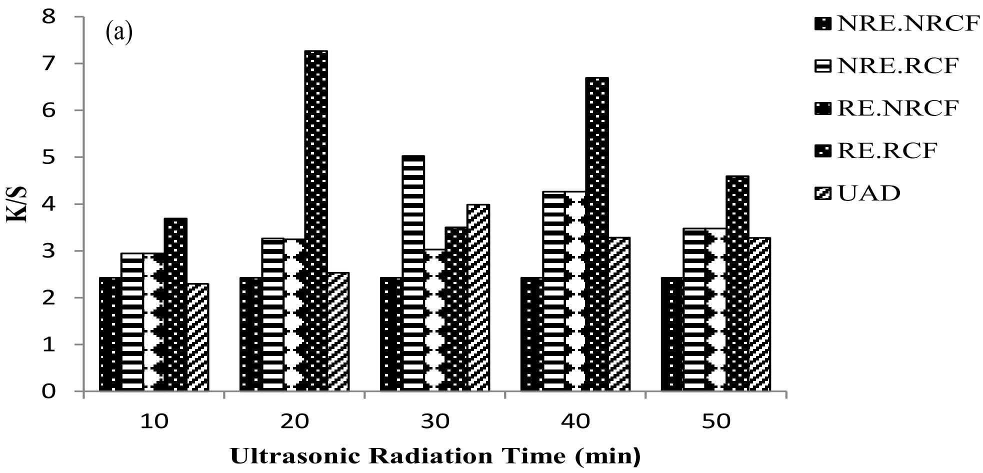
RESULTS AND DISCUSSION

Ultrasonic radiation has been proven to have potential applications in every sector of life. This is because these waves cause acoustic cavitation, which transfers energy into the matrix by scratching the outer layer of the matrix. The plant cell walls rupture when these rays collide with the matrix, allowing more mass to be transferred into the solvent by permeating deeper, and this kinetics results in a beneficial powder-solvent interaction for isolating functional biomolecules [31]. Plant-based dyes, particularly from the haar singhar flower, have produced a variety of hues when separated in various mediums.

Table 1. Shade strength and tonal impact of cotton fabric dyed with floral extracts under ultrasonic radiation

Extracts used	Time (min)	Sample code	K/S	L*	a*	b*	c*	h
Aqueous	0	NRF/NRE	2.4235	73.08	6.24	40.35	22.52	30.34
	20	RF/RE	7.2657	73.12	8.09	46.66	53.78	31.53
Basic	0	NRF/NRE	1.1311	79.50	5.63	55.97	18.62	25.81
	40	RF/RE	6.8059	77.51	7.36	61.29	35.64	34.92
Acidic	0	NRF/NRE	1.5926	65.74	5.02	28.58	26.91	27.68
	30	RF/RE	4.9999	76.37	7.41	30.35	47.41	27.64

Ultrasonic irradiated cotton fabric in a wet state increases its dye ability toward natural dyes. In the aqueous medium, both the extract and the cotton were irradiated for 20 minutes, providing excellent results, as shown in Figure 2a. In Figure 2b, it was revealed that when the medium was switched to acidic conditions, both the extract and the cotton fabric irradiated for 30 minutes generated excellent results. While a short irradiation duration did not encourage cell wall breaking, a prolonged irradiation time may have helped in the separation of other components that influenced shade firmness during dyeing. It was reported that irradiating cotton fabric for 30 minutes gave excellent results when using irradiated alkaline extract (Figure 2c).



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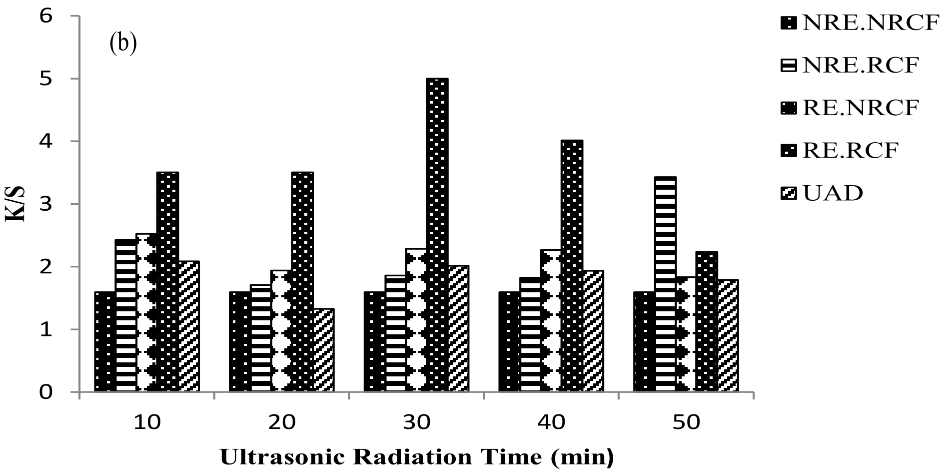

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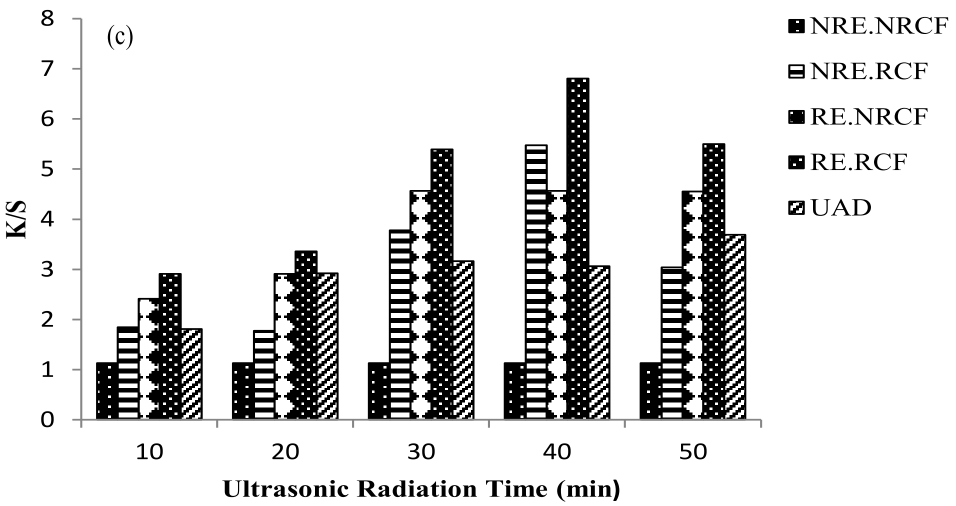
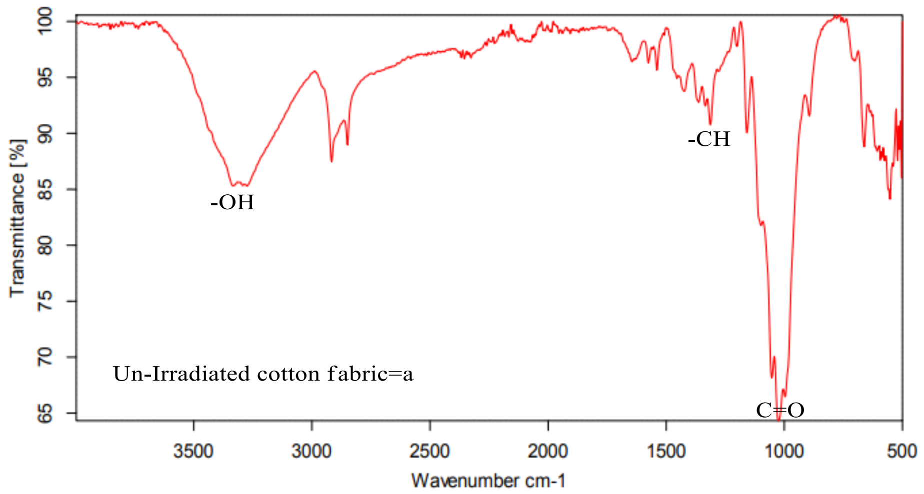


c)

Figure 2. Extraction of colourant from haar singhar flower (HSF) in media such as a) aqueous, b) acidic, and c) basic for colouration of cotton fabric before and after ultrasonic irradiation

The results reveal that employment of ultrasonic irradiations for up to 20 minutes, there is no alteration in the functional peak of H-bonding in the cellulosic fabric (Figure 3a) and 3b)). According to FTIR analysis taken from untreated and ultrasonic-treated cotton fabric, the -OH stretching peak (3276 cm^{-1}) and CH and C=O stretching peaks 1250 cm^{-1} 1010 cm^{-1} did not alter following ultrasonic irradiations for up to 20 minutes. Hydroxyl linkage characteristics peak as functional units of cellulosic fabric, such as cotton, demonstrates that the chemical nature of cotton fabric was not altered by these radiations, which is very beneficial to the textile sector. The FTIR spectra dyed cotton fabric with haar singhar flower extract revealed strong IR bands indicating hydroxyl (3300–3450 cm^{-1}), amino stretching peaks at 2890 cm^{-1}, C=C of benzene (1200 cm^{-1}), and 1015 cm^{-1} (C=o of benzene). FTIR spectrum shows the existence of nyctanthin, representing that it is used as a dye in cotton.



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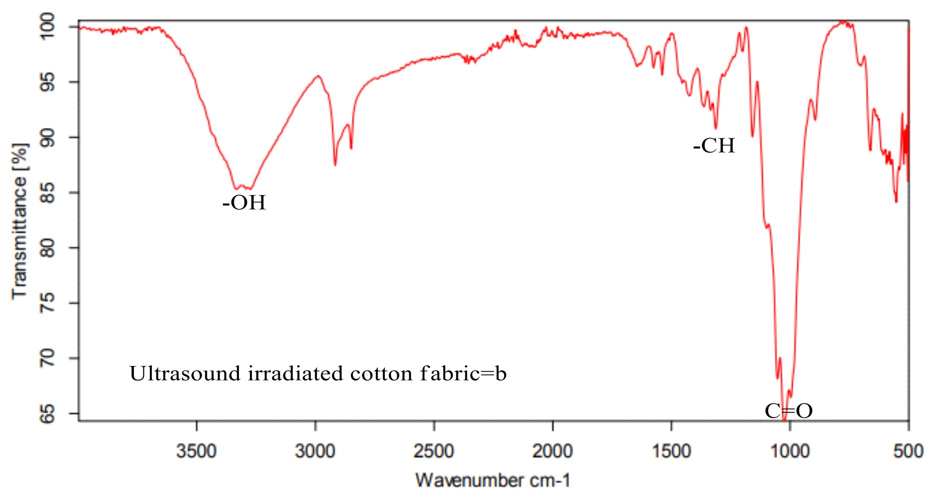


b)

Figure 3. a) FTIR spectral analysis of un-radiated and b) irradiated cotton fabric

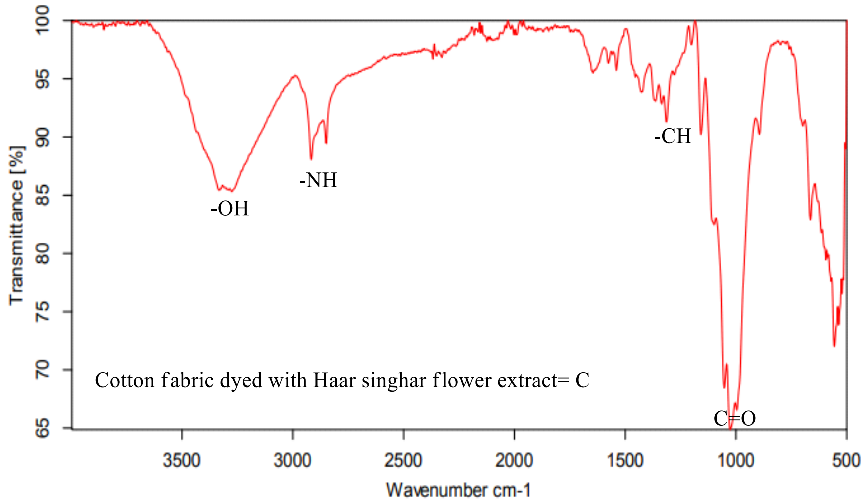


Figure 4. FTIR analysis of dyed cotton fabric with haar singhar flower extract

Only the optimal amount of powder for extracting colourant from haar singhar flower extract in an aqueous media can produce significant colour on fabric. Other functional components are isolated above the optimal concentrations, affecting the adsorption of the active colourant (nyctanthin) and reducing the colour strength after 40 minutes of ultrasonic treatment. It was observed that 4 g of the crude powder, followed by 40 minutes of irradiation, yielded excellent shades in an aqueous media, with satisfactory results on irradiated cotton fabric.

Application of salt in textile dyeing aids to obtain optimum colourant depletion because it transfers the colourant molecules from extract to cotton fabric within a short range of attractive forces. Results show that 1.5 g/100 mL of sodium chloride gave maximum exhaustion, and increase the colourant output.

The pH of the dye bath is critical for cotton fabric colouring, for the reason that the active sites of cotton fabric (OH) are susceptible to binding in an aqueous extract with active sites of nyctanthin. Table 2 show that following US treatment, an aqueous extract with a pH of 7 gave excellent results on irradiated cotton fabric. As a result, an aqueous extract of 55 mL at 7 pH produced from 4 g of dye powder gave good results on cotton fabric after being exposed to US radiation for 40 minutes.

Table 2. Selection of dyeing conditions using central composite design for cotton dyeing using flower extract

Experiment no.	pH	Volume (ml)	Time (min)	Temperature (^{0}C)	Salt concentration (g/100ml)	Colour strength (K/s)
1	7	55	30	80	1	2.3318
2	9	55	30	60	1	1.8689
3	7	35	30	60	1	2.1736

Experiment no.	pH	Volume (ml)	Time (min)	Temperature (^{0}C)	Salt concentration (g/100ml)	Colour strength (K/s)
4	9	35	30	80	1	2.6943
5	7	55	30	60	2	2.5707
6	9	55	30	80	2	2.4320
7	7	35	30	80	2	1.7221
8	9	35	30	60	2	2.3320
9	7	55	50	60	1	2.4575
10	9	55	50	80	1	1.8923
11	7	35	50	80	1	1.7692
12	9	35	50	60	1	2.0571
13	7	55	50	80	2	2.7580
14	9	55	50	60	2	1.7467
15	7	35	50	80	2	1.996
16	9	35	50	70	2	2.0326
17	7	55	40	70	1.5	3.5438
18	9	55	40	70	1.5	1.8084
19	6	45	40	70	1.5	2.8694
20	10	45	40	70	1.5	1.8408
21	8	25	40	70	0.5	1.9100
22	8	65	20	70	2.5	1.9498
23	8	45	60	70	1.5	1.7868
24	8	45	40	70	1.5	1.7674
25	8	45	40	50	1.5	3.1204
26	8	45	40	90	1.5	1.8548
27	8	45	40	70	1.5	1.9344
28	8	45	40	70	1.5	1.7123
29	8	45	40	70	1.5	2.0634
30	8	45	40	70	1.5	1.5984
31	8	45	40	70	1.5	1.5593
32	8	45	40	70	1.5	1.6640

Because the equilibrium of the dye bath can only be obtained at low heating, and too much heating promotes dissociation rather than adsorption, lowering the colour, the level of dyeing of cotton fabric with plant-based colourant is constantly reliant on the time and temperature. Dyeing ultrasonic-treated cotton fabric at 70 °C for 40 minutes using an aqueous extract with 7 pH made from 4 g powder including 1.5% sodium chloride yielded the highest colour strength as compared with other samples (Table 2)

ANOVA statistics for a 2nd order model with 5 key variables, 5 quadratic impacts, and 2 way interactions have been seen in Table 4. We progressively changed the model by removing non-significant components, excluding the key variables, which were included intact. Table 4 shows the p-value is supported by a variance inflation factor (VIF) greater than 1. By experimenting with several permutations of extraction and irradiation conditions, the colouring parameters were statistically optimized. Most of these variables have positive effects on colour strength versus extract amount, time, temperature, pH of the dye bath, and concentration of salt for attaining high exhaustion, but the effects of salt, pH of dye bath and time are extremely significant (p=0.000).

Table 3. Statistical evaluation of results obtained after dyeing of cotton with flower extracts using various trials

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		5.6008	0.0957	65.48	0.000	
pH	-1.544	-0.622	0.120	-7.32	0.000	2.00
Volume	-0.129	-0.125	0.135	-1.00	0.044	1.30
Time	-0.614	-0.367	0.126	-3.25	0.009	1.12
Temperature	-3.927	1.978	0.132	-16.16	0.000	1.52
Salt	-0.450	0.365	0.135	-2.33	0.045	1.30
pH* pH	4.561	2.431	0.199	11.49	0.000	1.06
Volume* volume	3.251	1.631	0.300	5.45	0.000	1.90
Temperature* temperature	-2.999	-1.484	0.250	-6.12	0.000	1.35
pH* volume	6.690	3.360	0.255	14.17	0.000	1.29
pH* time	-4.544	-2.297	0.255	-9.96	0.000	1.29
pH* temperature	6.5.01	3.200	0.279	12.61	0.000	1.34
Volume* time	-4.140	-2.070	0.240	-9.79	0.000	1.010
Volume* temperature	2.295	1.150	0.270	5.27	0.003	1.34
Volume* salt	-2.810	-1.405	0.215	-8.55	0.000	1.79
Time* salt	5.860	2.930	0.239	13.44	0.000	1.10
Temperature* salt	-5.200	-2.605	0.269	-8.69	0.000	1.30

Table 4. ANOVA: Colour strength versus dyeing parameters (temperature, volume, pH, time, and salt concentration)

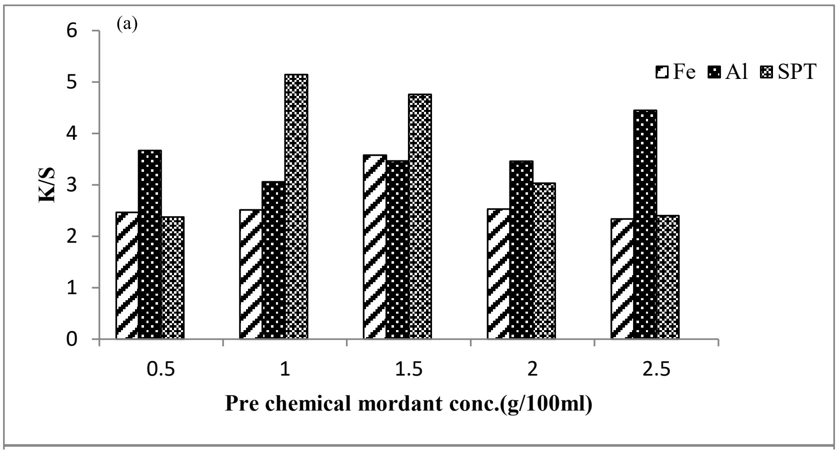
Source	DF	ADJ SS	ADJ MS	F-Value	P-Value
Model	164	41.1195	2.5574	50.19	0.000
pH	1	15.4198	2.9038	59.57	0.000
Volume	1	3.6450	2.6950	55.08	0.034
Time	1	0.0507	0.0494	0.100	0.000
Temperature	1	0.5835	0.6036	12.35	0.008
Salt	1	0.2675	0.3005	5.31	0.045
Square	3	12.7145	13.7355	230.81	0.000
pH* pH	1	8.9383	8.8483	159.92	0.000
Volume* volume	1	1.5059	1.5059	30.34	0.000

Source	DF	ADJ SS	ADJ MS	F-Value	P-Value
Temperature* temperature	1	1.9080	1.9380	40.06	0.000
2-Way Interaction	9	21.2860	2.9845	55.61	0.000
pH* volume	1	8.7877	8.8777	169.56	0.000
pH* time	1	4.1086	4.0986	79.20	0.000
pH* temperature	1	5.7703	6.8903	140.77	0.000
Volume* time	1	3.9481	4.9281	80.25	0.000
Volume* temperature	1	0.8906	0.8006	18.25	0.001
Volume* salt	1	3.1859	3.1867	45.90	0.000
Time* salt	1	6.9031	8.8831	144.83	0.000
Temperature* salt	1	5.9015	4.8055	92.87	0.000
Error	11	0.4998	0.0600		0.000
Lack-of-fit	6	0.2031	0.0406	0.60	0.000
Pure error	6	0.4167	0.0553		0.000
Total	25	43.6383			0.000

Mordanting is regarded as essential in natural colouring since it is the only approach to address the problem of low fastness [32,33]. In comparison to chemical salts of Al^{3+}, Fe^{2+}, and sodium potassium tartrate, green plant anchors i.e. extracts of pistachio, walnut, and pine nut shell were used at 20–90 °C. At 60 °C, pre-chemical mordanting with 2.5% Al^{3+}, 1.5 g/100 ml Fe^{2+}, and sodium potassium tartrate, as well as pre-bio-mordanting with 0.5 g/100 ml walnut and pine nut shells and 2.5% pistachio shell gave good shade strength and fastness features. Chemical mordants have given vibrant colours as well as superior colour strength when compared to bio-mordants [34-36]. As a result, plant-based anchors can be used to substitute metal salt in the process, making it more sustainable.

Table 5. Shade variables of optimum dyed and mordanted cotton fabrics using haar singhar flower extract

Mordanted samples conc.(g/100ml)	K/S	L*	a*	b*	c*	h*
Fe (1.5g/100mL) (Chemical pre)	3.5775	72.64	7.73	34.74	40.52	39.44
Fe(1g/100mL) (Chemical post)	1.6239	70.36	4.22	59.27	44.86	41.76
Al (2.5g/100mL) (Chemical pre)	4.4485	72.86	4.17	44.74	34.46	33.70
Al (2g/100mL) (Chemical post)	2.8908	61.89	8.16	43.61	48.22	48.12
SPT(1g/100mL) (Chemical pre)	5.1453	76.60	1.22	48.18	47.63	46.82
SPT (2g/100mL) (Chemical post)	1.6671	87.79	0.95	12.99	35.92	34.89
Walnut (0.5g/100mL) (Bio pre)	3.1399	69.92	0.99	40.78	30.53	29.35
Walnut (1g/100mL) (Bio post)	1.3091	82.71	3.33	31.73	19.19	16.66
Pistachio (2.5g/100mL) (Bio pre)	0.8632	75.65	3.88	59.78	36.62	36.12
Pistachio (2g/100mL) (Bio post)	7.9806	82.59	3.66	23.59	35.64	32.92
Pine nut (0.5g/100mL) (Bio pre)	1.5584	73.21	4.55	39.24	24.61	24.84
Pine nut (2.5g/100mL) (Bio post)	1.6967	71.27	4.09	45.41	27.64	26.91



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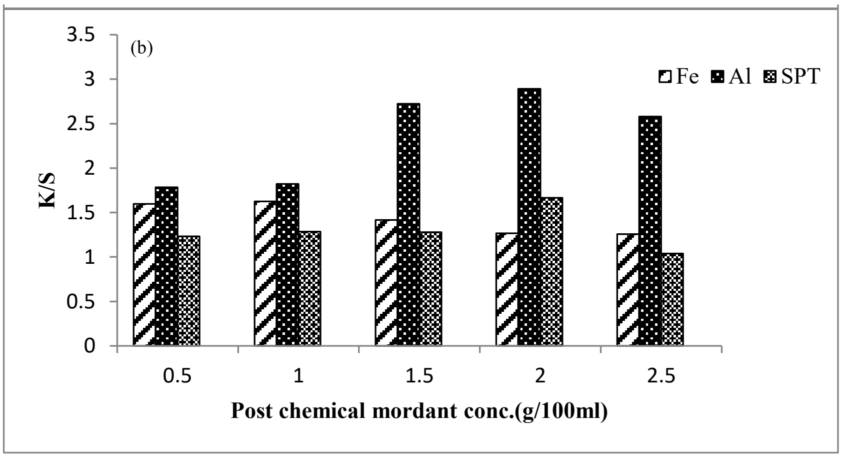
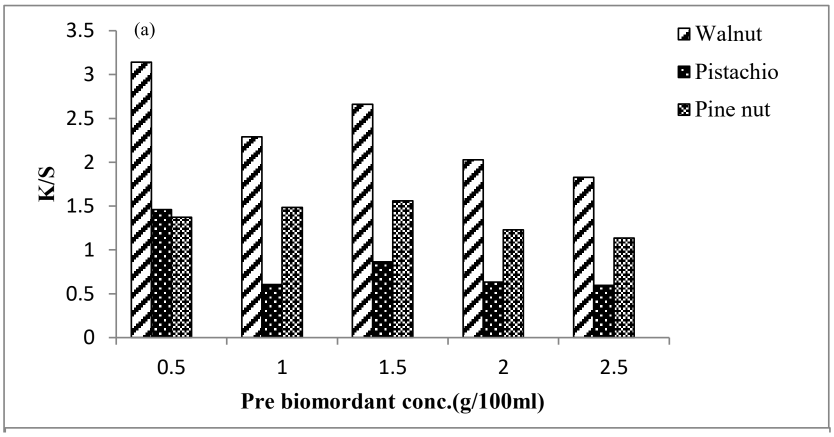


b)

Figure 5. Effect of pre a), and post chemical mordants b) on haar singhar dyed on cotton fabric colour strength



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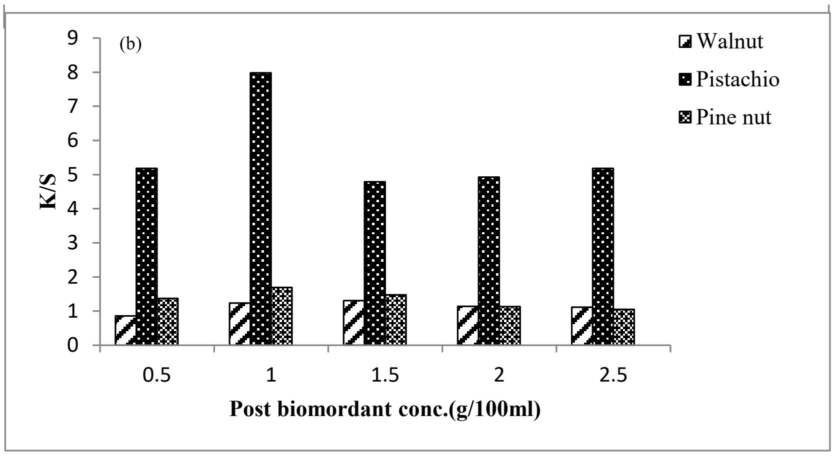

b)

Figure 6. Effect of pre-bio-mordants a), and post-bio-mordants b) on haar singhar dyed on cotton fabric colour strength.

Fastness characteristics following the application of chemical and bio mordants, it was discovered that functional molecules formed such a strong bond with the functional component of the colourant and the cotton fabric (–OH) that the shade formed hindered receding when exposed to light, washing, and dry and wet rubbing to a significant extent [37, 38]. The colourant molecule obtained from the haar singhar flower extract is light, rubbing, and washing resistant. This is because biomordants employ their –OH groups and ligation system to create new colours that resist separation from fabric. In chemical mordanting, the salts of Al, SPT, and Fe contribute to the establishment of a stable metal and dye complex on cotton fabric resulting in less colour separation and a good fastness rating. Thus, utilizing environmentally friendly mordants before and after dyeing has improved not only the natural dyeing of cotton using irradiated haar singhar flower extract but has also made the process more sustainable by introducing soothing hues, wash fastness, light fastness, dry and wet rubbing fastness are all processes to enhance colourfastness.

Table 6. Fastness ratings of pre and post mordanted dyed fabric

Mordanted samples concentration (g/100 ml)	LF	WF	DRF	WRF	Dyed mordanted fabrics
Fe (1.5 g/100 mL) (Chemical pre mordanted)	4	¾	4	3/4	
Fe(1 g/100 mL) (Chemical post mordanted)	5	4	5	4/5	

Mordanted samples concentration (g/100 ml)	LF	WF	DRF	WRF	Dyed mordanted fabrics
Al (2.5 g/100 mL) (Chemical pre mordanted)	5	4/5	5	4/5	
Al (2 g/100 mL) (Chemical post)	5	4/5	5	4/5	
SPT(1 g/100 mL) (Chemical pre mordanted)	5	4/5	5	4/5	
SPT (2 g/100 mL) (Chemical post mordanted)	5	4/5	5	4/5	
Walnut (0.5 g/100 mL) (Pre biomordanted)	5	4/5	5	4/5	
Walnut (1 g/100 mL) (Post bio mordanted)	5	4/5	5	4/5	
Pistachio (2.5 g/100 mL) (Pre bio mordanted)	5	4/5	5	4/5	
Pistachio (2 g/100 mL) (Post bio mordanted)	5	4/5	5	4/5	
Pine nut (0.5 g/100 mL) (Pre bio mordanted)	5	4/5	5	4/5	
Pine nut (2.5 g/100 mL) (Post bio mordanted)	5	4/5	5	4/5	

LF (light fastness), WF (wash fastness), DRF (dry rubbing fastness), and WRF (wet rubbing fastness).

CONCLUSION

Natural dyes are less toxic, and non-allergic, which is why they are becoming more popular in the textile industry. It has been discovered that a novel technique of extraction, namely ultrasonic extraction, has the potential for yielding the natural colourant while using less time, energy, and solvent. Mordanting is utilized to improve the colour strength of the dyed fabric. Under ultrasonic irradiation, the haar singhar flower extract was investigated as a new dye-producing plant. The

https://doi.org/10.31881/TLR.2022.67

introduction of bio-based mordants and ecologically friendly chemical mordants have been found to enhance the colour strength of cotton fabrics by making an additional binding with the dye and fabric, resulting in a new shade with outstanding colour fastness.

Author Contributions

Conceptualization: Adeel S; Methodology: Nasreen H and Amin N; Supervision: Yameen M; Investigation: Ozomay M; Writing draft: Nasreen H; Writing-Review and editing: Qayyum MA; Formal Anlaysis: Ozomay M.

Conflicts of Interest

The authors declare no conflict of interest.

Funding

This research received no external funding

Acknowledgements

We are highly thankful to Department of Biochemistry for providing necessary chemicals and assorted glassware and to Department of Applied Chemistry for providing facilities to conduct experiments.

This article was published under the competence of Guest Editor Shahid Adeel.

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