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REGULAR ARTICLE

Sustainable Coloration and Bioactive Functionalization of Wool Fabric via Plant-based Naphthoquinone Compounds

Mohd Shabbir[1,2] · **Faqeer Mohammad**[2]

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Abstract

Phytochemicals, obtained from natural sources, have high importance for their practical implications owing to their inherent biological characteristics. Naphthoquinone compounds (alkannin and shikonin) from *Alkanna tinctoria* are well known for antioxidant and coloring properties. Here, naphthoquinone-rich dyeing solution was obtained by aqueous extraction of alkanet roots and applied on protein-based fabrics, i.e., wool. Bioactive colored fabrics were characterized for UV protection, antioxidant activity, and color characteristics. Ultraviolet (UV) protection ability was measured in terms of UV transmittance and UPF (UV protection factor) values, and antioxidant activity in percentage. Some non-hazardous metal salts in permissible concentrations were used as mordants, and their effects on coloration and bioactivities were studied. These salts significantly improved color characteristics and broadened the shade range, while it had little effect on bioactivities. Control dyeing with alkanet extract showed great potential for UV protection and antioxidant activity owing to the conjugated systems in naphthoquinone compounds. Polar functional groups of naphthoquinones were responsible for adsorption on fabric surfaces via chemical interactions.

Keywords *Alkanna tinctoria* · Naphthoquinone · Mordants · Coloration · UV protection · Antioxidant activity

1 Introduction

Pollution, due to explosive industrialization, and public's enhanced awareness increased the health concerns of human beings and their desires to use compatible products including textiles [1, 2]. Overproduction of gaseous pollutants depleted the ozone layer in stratosphere, due to which the ultraviolet (UV) radiations reach onto the surface of earth. Among the three categories of UV radiations (UV-A (320–400 nm), UV-B (280–320 nm), and UV-C (< 280 nm)), depending on their wavelength range, UV-B (280–320 nm) radiations have more detrimental effects on lipids, nucleic acids, and proteins in our body. Skin sunburn, premature aging, and even skin cancers are the end health effects of long-term exposure to these radiations [3, 4]. Exposure to UV radiations can also trigger the overproduction of reactive oxygen species (ROS), which are also produced due to some other factors such as defense of pathogens, nutrient deficiency, and pollutants toxicity. In normal cells, these ROS are produced in low levels and are not problematic in nature, but overproduction leads to associated health effects, particularly on skin as it is more prone to radiations' exposure. Various sunscreens are usually applied directly on skin and UV protective clothing also used. Both these methods cannot avoid use of synthetic materials which in other ways may be harmful [3].

Textiles are used primarily to protect human body from environmental effects and exposure to pathogens to some extent. Functional textiles also act as barrier for UV radiations depending on the weaving and knitting of fabrics, and functionalization of fabrics with the help of organic compounds (organic dyes) and nanomaterials (ZnO, TiO_2, etc.). Organic synthetic dyes and functional nanomaterials leached out are associated with environmental and health concerns and are not easy to be degraded [5–7]. Since humans nowadays are eager to use materials of natural origin owing to the associated characteristics such as biocompatibility, biodegradability, and eco-friendliness. Sometimes waste of agricultural or industrial crops can be source for dyeing and textile industry. Organic compounds such as tannins,

✉ Mohd Shabbir
shabbirmeo@gmail.com

1 Department of Chemistry, GL Bajaj Institute of Technology and Management, Greater Noida, UP 201306, India

2 Department of Chemistry, Jamia Millia Islamia, New Delhi 110025, India

anthraquinones, naphthoquinones, and other polyphenols obtained from plants are well used for textile coloration from ancient times [8–10]. But, since a few years ago, natural dyes have also been explored for other functional properties including antimicrobial, antioxidant, and UV protection. Extended conjugation and polyphenolic functional groups of such compounds might also be responsible to utilize reactive oxygen species and can act as antioxidant [11–13].

Despite the superiority of natural dyes in terms of eco-friendly, biocompatibility, and waste management areas, natural dyes are not easy to bind with the textile surfaces such as cotton and wool. Various methods or surface treatment of textiles have been evolved by the time to enhance the affinity of natural dyes toward textile fibers. Use of mordants has been a common practice in the past decades to enhance affinity via coordination complex formation in case of metal mordants. Keeping in mind the environmental safety, the use of natural mordants or bio-mordants has also been evolved, and compounds from bio-mordants help in acting as a bridge between the dye molecules and textile fibers resulting in higher affinity [14–16]. Pretreatment of textile surfaces via chemical and physical means (plasma treatment, microwave-assisted dyeing, sericin pre-treatment) has also been a technique to enhance affinity of natural dyes toward textiles [17–19]. Here in our study, we chose metal mordants in ecological permissible limits.

Alkanna tinctoria or commonly known as alkanet is an herb from *boraginaceae* family, and its roots' extract contains naphthoquinone compounds well known for coloration and various pharmaceutical importance. Alkannin and shikonin are the primary compounds which are also used as food colorants. These compounds bear both the quinone (naphthoquinone) and the phenolic moiety expected for the functional activities [20, 21]. In this study, naphthoquinone compounds from alkanet roots' extract were used for wool fabric coloration and functionalization for UV protection and free radical scavenging activity. This research may lead to easy and sustainable replacement for synthetic dyes and functional agents.

2 Experimental

2.1 Materials

Powdered alkanet dye was purchased from Sam Vegetable Colours Pvt. Ltd., Moradabad, UP, India. 100% knitted wool fabric of 0.54 mm thickness, 316 g/m^2 weight, 34 × 22 fabric count per inch was used in this study. ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt) was procured from Shanghai D & B Chemicals Technology Co. Ltd., China. $Al_2K_2(SO_4)_4 \cdot 24H_2O$ (Alum), $FeSO_4 \cdot 7H_2O$ (ferrous sulfate), and $SnCl_2 \cdot 2H_2O$ (stannous chloride) were used of laboratory grade.

2.2 Extraction of Naphthoquinone Colorants

Dye was extracted in aqueous medium at 1:20 M:L (material to liquor) ratio and 90 ℃ temperature for 45 min with constant stirring of the solution. Extraction was repeated 3 times to get maximum yield of colorant. Obtained extract was cooled, filtered, and used in the dyeing process.

2.3 Mordanting with Metal Salts

Pre-mordanting method was opted for mordanting of textile substrate with three eco-friendly (up to permissible limits) metal mordants; alum (10% o.w.f.), iron sulfate (5% o.w.f.), and tin chloride (1% o.w.f.). Firstly, square-shaped wool fabric samples of area 100 cm^2 (approx.) were soaked in water to make them dirtless and then transferred into mordant solution at temperature nearly 30 ℃. Mordant bath was heated constantly up to 90 ˚C temperature at constant rate and kept for 60 min with constant agitation. Finally, mordanted textile samples were rinsed with tap water to remove unused mordants.

2.4 Dyeing

Un-mordanted and mordanted wool samples were immersed into dye baths with material to liquor (M:L) ratio of 1:40, pH 7, and dyeing was carried out for 60 min at 90 ℃. Constant stirring was carried out during the dyeing process to achieve uniform dyeing. Dyed wool fabric samples were given mild wash with Safewash (non-ionic detergent), Wipro of concentration 5 ml/*l*, and were dried in shade.

2.5 Color Characteristics

Colorimetric characteristics of dyed wool in terms of color strength (*K/S*) & CIE $L^*a^*b^*$ values were recorded on a HunterLab UltraScan PRO reflectance spectrophotometer (10° standard observer; illuminant D65).

Chroma (c^*) and hue angles (h^o) were obtained with the help of following equations:

$$\text{Chroma}(c^*) = \sqrt{(a^*)^2 + (b^*)^2} \quad (1)$$

$$\text{Hueangle}(h^o) = \tan^{-1}(b^*/a^*) \quad (2)$$

2.6 Color Fastness Properties

Standard testing methods for measuring color fastness were adopted using Digi light Nx™ based on test method ISO

105-B02:1994 for light fastness, Digi wash SS™ (Launderometer) based on test method ISO 105-C06:1994 for wash fastness, and Digi crock™ (Crockmeter) based on test method ISO 105-X12:2001 for both dry and wet rub fastness [22–24]. Samples were also evaluated for color staining on white cotton and wool.

2.7 UV Protection

Ultraviolet protection ability in terms of UV transmittance and ultraviolet protection factor (UPF) of naphthoquinone-dyed wool fabric was assessed on a Labsphere UV-1000F ultraviolet transmittance analyzer (Labsphere Inc., USA) (Fig. 1).

2.8 Colored Textile's Antioxidant Activity

The antioxidant activity of untreated and naphthoquinone-dyed wool fabric was assessed using ABTS radical decolorization assay, an already developed method [3, 25, 26]. ABTS stock solution (conc. 7 mM) was reacted with potassium persulfate (conc 2.45 mM) for production of ABTS radical cation ($ABTS^{\cdot+}$) and kept still in dark for 14 h at 25 ℃. With the help of phosphate buffer (0.1 M and 7.4 pH), the $ABTS^{\cdot+}$ solution was diluted up to absorbance of 0.700 ± 0.025 at 734 nm before measurements; further, 10 mg sample was added to 10 ml $ABTS^{\cdot+}$ solution. Scavenging ability of $ABTS^{\cdot+}$ was calculated after 30 min at 734 nm by Eq. (3):

$$\text{Antioxidantactivity}\% = \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \times 100 \quad (3)$$

where A_{control} is initial absorbance of $ABTS^{\cdot+}$ and A_{sample} is absorbance of remaining $ABTS^{\cdot+}$

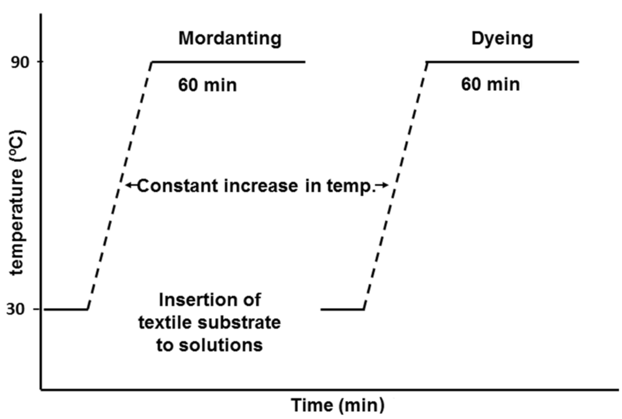

Fig. 1 Mordanting and dyeing process

3 Results and Discussion

3.1 Colorimetric Properties

Colored fabric samples were characterized for colorimetric properties on a HunterLab UltraScan PRO reflectance spectrophotometer in terms of CIE $L^*a^*b^*$ (Table 1) and color strength (*K/S*) values (Figs. 2 and 3).

Color strength of the colored fabric samples in the range of wavelength 400–700 nm is graphically represented. In the first study, the dye concentration (Fig. 2) was variable, and in the second one, metallic mordants are variant (Fig. 3). Increased diffusion rates with the high concentration of dye solution enhanced the color strength values, and gradual increase in color strength can be observed from the graph with the concentration of dye. Functional groups of naphthoquinones are arranged in such a fashion that coordination complexation occurs with the introduction of metallic ions in between the textile and dye molecules. From the results, this fact is well established. Metallic salts significantly increased the color strength values, and iron highly affected the results among them. This might be explained by strong coordination complexation ability of Fe with naphthoquinone compounds and fabric surface [27, 28]. Al does not affect the color strength much, and it is usually known for lightening the shades on fabrics, while Fe and Sn generally produce dark shades owing to the higher adsorption of dye molecules via coordination complexation.

Color coordinates are represented in color space diagram (Fig. 4). Actual color tone variation was very little with the

Table 1 Fastness properties and color characteristics of dyed wool fabric samples (AT = Alkanet)

S. no.	Wool sample	Light fastness	Wash fastness	Rub fastness		*L*	*C*	*h*
				Dry	Wet			
1	5% AT	4–5	4–5	5	4–5	50.39	3.45	76.95
2	10% AT	4–5	4	5	4–5	47.78	4.46	78.79
3	15% AT	4–5	4–5	5	4	45.26	3.72	74.66
4	20% AT	4–5	4	5	3–4	42.42	2.83	80.03
5	Al + 20% AT	4–5	4–5	4	3–4	46.86	7.64	90.27
6	Fe + 20% AT	5	5	4–5	3–4	39.69	9.77	61.28
7	Sn + 20% AT	4	4	4–5	3–4	40.84	9.63	66.17

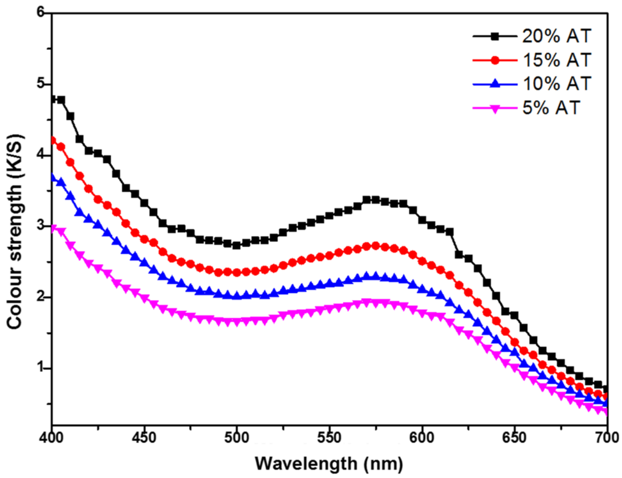

Fig. 2 Color strength of dyed fabrics with the variation of alkanet extract

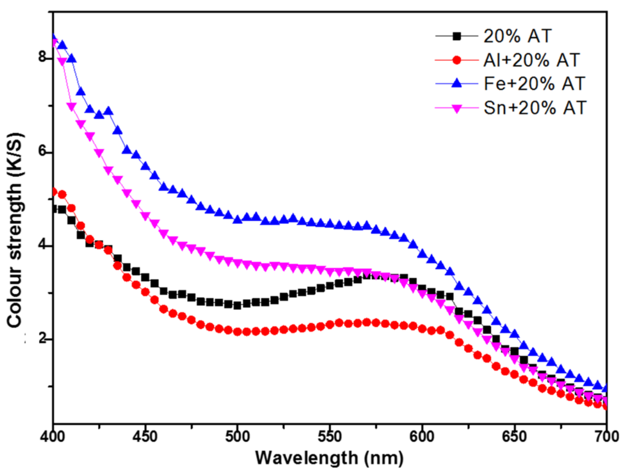

Fig. 3 Color strength of mordant-dyed fabrics with the effect of alum, iron, and tin salts

effect of dye concentration. All the color coordinates lie in yellow–red quadrant, and the samples of unmordanted are closely placed since there was not much variation with the effect of concentration. Fe and Sn salts shifted the color coordinates toward red axis, and shades of red tone were produced (Fig. 5), while Al shifted the color coordinate toward yellow axis and literally placed on the axis. This effect of shade lightening is well known for alum salt and here also produced shade of yellow tone (Fig. 5).

3.2 Fastness Properties

Colored fabric samples were tested for light, wash, and rub fastness properties (Table 2) and evaluated on gray-scale. These results range from average to excellent grades.

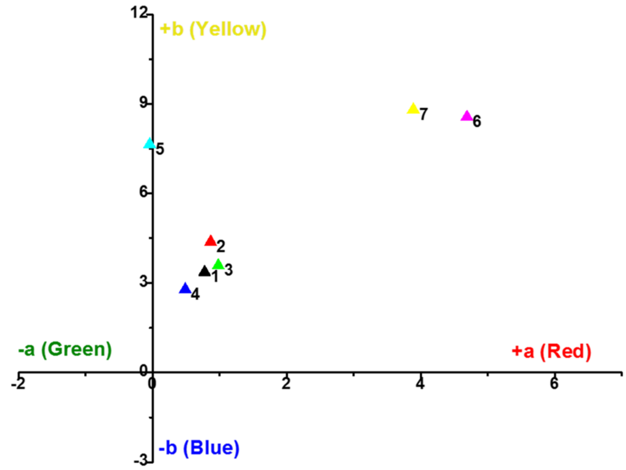

Fig. 4 Color space diagram of alkanet-dyed wool fabrics

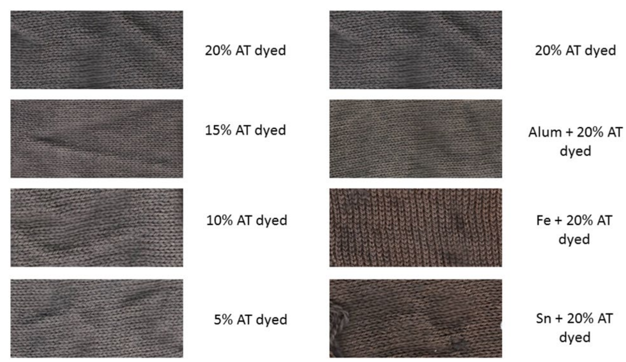

Fig. 5 Shades obtained from naphthoquinones-based dye

Table 2 Dye conc. and mordants effect on UV protection parameters (AT = Alkanet)

Wool sample	%*T* (UV-A)	%*T* (UV-B)	UPF	Rating
Undyed	22.39	13.58	< 15	Bad
5% AT	2.20%	1.13%	50+	Excellent
10% AT	1.84%	1.07%	50+	Excellent
15% AT	0.13%	0.55%	50+	Excellent
20% AT	1.51%	0.91%	50+	Excellent
Al + 20% AT	2.52%	1.77%	50+	Excellent
Fe + 20% AT	3.05%	2.37%	50+	Excellent
Sn + 20% AT	3.17%	2.47%	25–39	Very good

Naphthoquinones from *Alkanna tinctoria* either in control dyeing or with metal mordants made strong bonds with protein fabric. Color shades were highly resistive to light effects, and results in every combination were of excellent grades. Higher concentration of dye sometimes left some extra molecules attached on the surface of fabric (double-layer adsorption) and results in lowering of fastness in wet conditions, and it can be observed from the results of samples 4, 5, 6, and 7. In control dyed wool samples, dye molecules and fabric are usually bound via electrostatic interactions or H-bonding and these interactions in some cases show good fastness results owing to the strength of electrostatic interactions. In case of mordanted dyeing, however, interactions between dye molecules and fabric are completely covalent-coordinate bonds (Fig. 6), usually resulting in much better fastness. Al and Sn give lighter shades generally and make weak complexes, resulting in relatively low fastness. Fe among the metals made shades more resistive to light, washing, and rubbing owing to their strong complexation abilities [29, 30].

3.3 UV Protection Activity

Exposure to UV light is harmful in any way and have detrimental effects on human health. Long-term exposure may lead to skin burning and even epidermal carcinoma [31]. There are many ways to avoid these radiations' exposure, and protective clothing is one of them. UV protection ability of clothing varies with many factors such as nature of textile material, weaving structure and density, coating, and color properties. UV protective clothing is developed by various means, and molecules from both natural and synthetic origin are utilized in recent times. Here, naphthoquinones from *Alkanna tinctoria* are used to functionalize wool fabrics for UV protection abilities. UV protection capacity of functional fabrics was observed in terms of UV transmittance (UV-A and UV-B %) and UPF values (Table 2).

UV transmittance of colored and bare fabric was recorded in the wavelength range of 250–450 nm, which covers all the three regions of UV radiations (Figs. 7 and 8). UV transmittance of bare fabric shows increasing pattern from UV-C region to UV-A, and it goes from around 12 to 30%. This level of UV transmittance falls in the bad category of UV protection rating and so requires some functionalization. Functionalization with naphthoquinone compounds significantly improved these results, and UV transmittance consistently lies below 5%, which is considered as excellent UV protection.

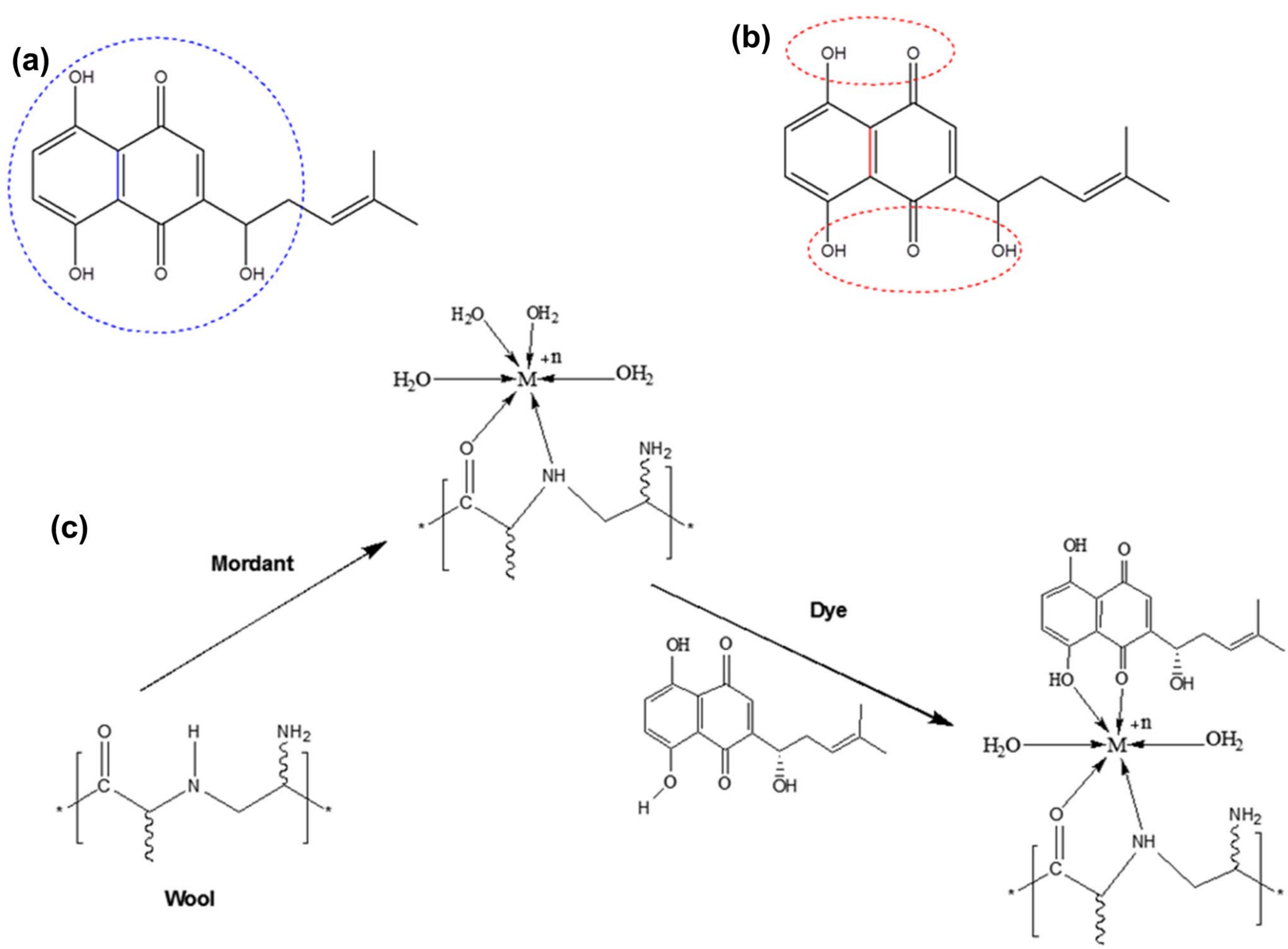

Fig. 6 Chemical structure of naphthoquinone compound depicting functional moiety and proposed complex of dye–mordant–fiber

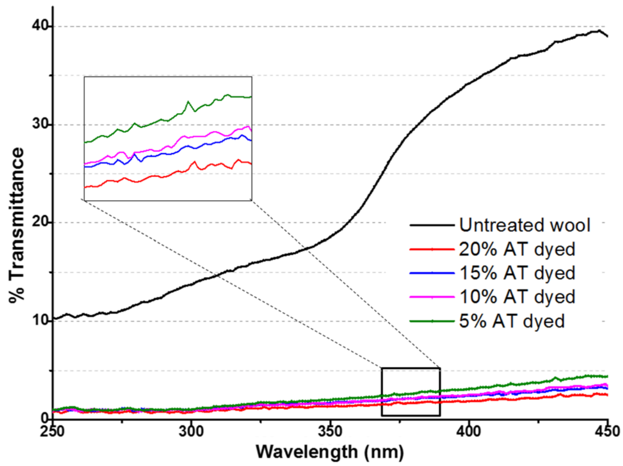

Fig. 7 UV transmittance results with the effect of dye concentration

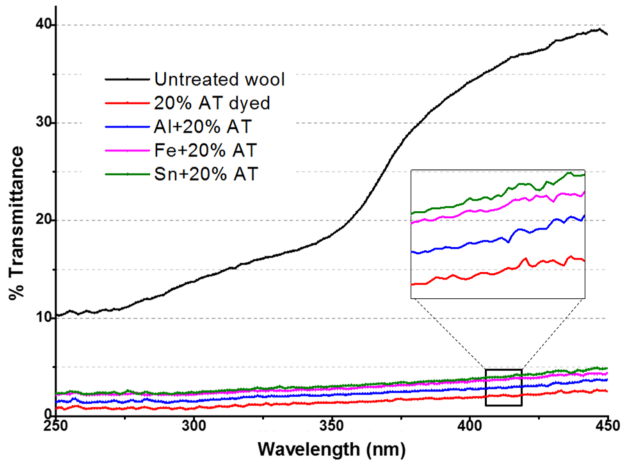

Fig. 8 UV transmittance results with the effect of metal salts

Arrangement of alternative double bonds along with carbonyl and phenolic groups in naphthoquinones' chemical structure can easily dissipate the absorbed radiations' energy (Fig. 6a). Generally, UV radiations cause change or destruction of chemical bonds and sometimes processes like tautomerism [32]. These types of chemical changes or reactions help to absorb these harmful radiations and protects human skin. Metal salts affected these results adversely, still in the good range of less than 5% (Fig. 8). This might be due to accumulation of some functional groups in formation of coordination complexes between fabric surface and naphthoquinone compounds (Fig. 6b), and consequently less absorption of UV radiations. This effect of metal salts can also be seen in UPF values of mordant-dyed fabrics (Fig. 9).

UPF results are represented graphically in Fig. 9. Continuous increment in UPF values observed with the dye concentration from 5 to 20%, even 5% dye concentration gives UV protection of excellent grades with UPF value of 50+. It is the arrangement of functional groups and double bonds in dye molecules responsible for UV light absorption and dissipation through the moiety. Introduction of metal mordants in dyeing significantly decreased the UPF values which are also in the line of the explanation of energy dissipation by the moiety arrangement. Metal salts consume functional groups and disturb the chemical structure or arrangement of the naphthoquinone coloring compounds. Still mordant-dyed samples have UPF values in the acceptable range.

3.4 Antioxidant Activity

Human skin is a barrier between internal organ system and outside environment or atmosphere. Atmospheric environment consists of sun radiations including UV radiations and pollutants, which produce reactive oxygen species (ROS). These ROS may lead to serious skin disorders or sometimes skin cancer. Areas around skin wounds are also susceptible to reactive oxygen species and may enhance severity of the wounds. Clothing and bandages can be functionalized to neutralize ROS or antioxidant activity via natural compounds from plant extracts.

Organic compounds having phenolic groups and extended conjugation (Fig. 6) in their chemical structure are well known for antioxidant or free radical scavenging activities. Naphthoquinone compounds from alkanet roots were tested for antioxidant activity on textiles which can further be extended to biomedical applications such as wound dressings [21].

Antioxidant activity in percentage of naphthoquinone compounds from alkanet roots on fabrics was evaluated and compared with raw wool fabric (Fig. 10). Antioxidant activity of raw wool fabric was less than 30%, which was exponentially increased with the application of naphthoquinone

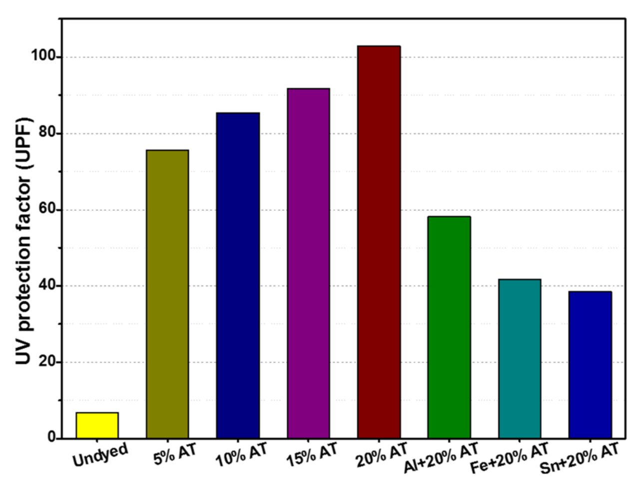

Fig. 9 UPF results of colored and functional fabrics

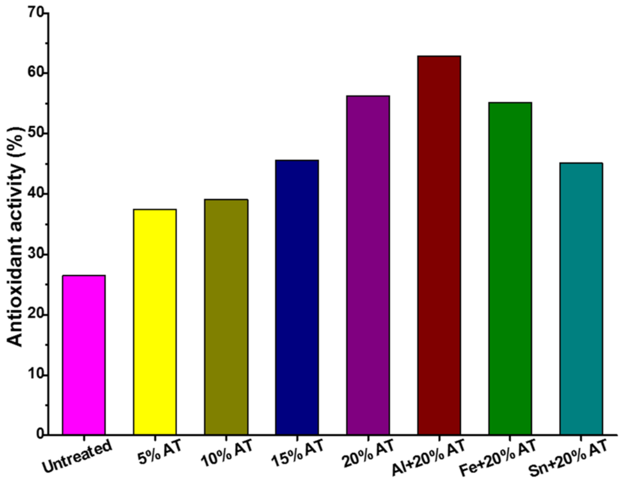

Fig. 10 Antioxidant activity results of colored and functional fabrics

compounds on fabric. Activity reached up to about 60% for 20% o.w.f of naphthoquinones' extract. This enhancement of the activity is attributed to the phenolic, quinone groups, and extended conjugation in compounds' chemical structure. Metal salts are generally known for suppressing activity due to utilization of functional groups of coloring compounds' structure [33]. But in case of naphthoquinones, alum enhanced the activity which might be due to dissipation of free radical electrons in vacant d orbitals of Al, while it involved in coordination complexation between fabric surface and coloring compounds. Iron and tin salts did not affect much the antioxidant activity. Fe might have suppressed a little but that might be overcome due to accumulation of more compounds to the fabric via strong complexation properties. Tin salts suppressed antioxidant activity to some extent might be due to involvement of functional groups in complexation.

4 Conclusion

Industrialization and enhanced awareness of human beings motivated researchers to find solutions in natural origin compounds for every aspect of life. In this study, naphthoquinone compounds from alkanet roots were utilized for coloration, UV protective, and antioxidative functionalization of wool fabrics.

In terms of coloration, brown to gray shades were obtained on wool fabrics. Excellent colorimetric and fastness properties toward washing, light, and rubbing were observed suggestive of industrial application of naphthoquinone colorants on fabrics. Use of metal salts broadened the shade range and enhanced the color and fastness properties significantly which were confirmed by colorimetric properties and color space diagram. These compounds were found highly effective for UV protection capability of dyed fabrics. UV protection results in terms of UPF values were enhanced from bad rating for raw wool to excellent rating for dyed and mordant-dyed wool fabrics. Naphthoquinones increased the UPF values in 50 + range even for lowest concentration of 5% o.w.f. Metal salts adversely affected the UV protection results owing to the fact, accumulation of functional groups in complexation. But that adverse effect did not lead to downgrade UV protection rating below good. Antioxidant activity results were also better and improved from less than 30% for raw fabric to about 60% for colored fabric with or without metal salts. Ultimately, results of naphthoquinone colorants on textiles could be of great importance in textile coloration and functionalization for UV protection and antioxidant activity.

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Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors have no relevant financial or non-financial conflicts of interests.

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