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# Natural Extraction of Dyes from Saffron '*Crocus sativus* L' Flower Waste, Cotton Dyeing, and Antioxidant Effectiveness

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**ABSTRACT**

The production of saffron, the spice obtained from the dried stigmas of *Crocus sativus* L. (Iridaceae family) flowers, generates, after pruning, considerable quantities of waste containing natural dyes. Saffron flower waste could be a source of extraction of natural dyes with antioxidant activity. In this study, we investigate the possibility of using saffron flower waste for dyeing cotton and evaluating the antioxidant effect of this dye by the DPPH free radical, reducing power and β-carotene bleaching assay. The dye has been evaluated for the composition of the color by the UV-visible spectrum and tested for the dyeing of cotton. The results indicate the presence of polyphenols and flavonoids. The dyeing conditions have been optimized at 6% dye concentration, dye bath pH of 3, dyeing temperature at 98 °C and dyeing time of 60 min. 2% dye concentration with 5 to 10% mordant concentration remains sufficient for dyeing with pre-mordanting. The exhaustion of the bath after dyeing has been improved by a rate of 20% in the case of addition of mordants which have produced a shade of green color. The dye contributes to the significant antioxidant activities with more DPPH scavenging capacity, FRAP reducing power, and β-carotene bleaching inhibition. Cotton fabrics dyed with bio-dyes obtained from saffron flower waste show good color fastness properties and could be a potential source of natural antioxidant agent. It presents an important eco-friendly alternative to synthetic dyes for large-scale application in textile and food industries.

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## INTRODUCTION

The use of natural dyes in the coloring of textiles has been known since prehistoric times; however, the advent of synthetic dyes with better fastness properties and low cost in the 19th century, has contributed considerably to the decrease of natural dyes (Slama et al., 2021).

The textile processing industry is one of the main polluters of the environment due to a heavy load of chemicals released, including the dyes used. It dispose of toxic chemicals in water bodies and the environment and produce toxic, allergic, carcinogenic and harmful effects on the skin seriously affecting the entire food chain (Hynes et al., 2020). Growing concerns about climate change, greenhouse gas emissions, energy instability, human health, and environmental pollution have led to serious interest in waste recovery practices in line with the UN Sustainable Development Goals (SDG) 2030. These global goals of around 17

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goals have challenged interested stakeholders to contribute to updating these overriding and complex issues in a world that is undoubtedly changing and seeking new approaches to address sustainable development (Rago et al., 2018). Therefore, industrials are turning more and more to natural substances because the list of authorized organic synthetic dyes tends to decrease under pressure from regulations and consumers (Thakker, 2020).

The rich biodiversity provides many raw materials for producing natural dyes; they cover all the dyes derived from the natural sources like plants, animal and minerals. They are eco-safe, renewable and biodegradable (Adeel et al., 2022). The revival of cultural heritage by being natural dyes to dye textiles is trending due to their soothing nature and luminous shades (Adeel et al., 2018). Most natural dyes have antioxidant, antiallergic, antibacterial, fluorescent, anti-UV and other medicinal properties. They also produce colors pleasing to the human eye and completely in line with today's fashion (Yusuf, 2016; Verma et al., 2021) The main problems with natural dyes are their high costs; one kilogram of plant material is required for each kilogram of fiber (Karaboyaci, 2014).

On the other hand, there is growing interest in the use of natural antioxidants to preserve foods from oxidation and manage some pathophysiological diseases, some of which are caused by free radicals. The implication of oxidative stress in the etiology as a causative agent of certain diseases has demonstrated that antioxidants can have health benefits as preventive agents (Tain and Hsu, 2022). Several studies reveal that antioxidants play a preventive and curative role in human health, due to their power to reduce oxidative stress. The measurement of the antioxidant activity or capacity of natural products is therefore essential not only to guarantee their quality, but also to study the effectiveness of their antioxidants in the prevention and treatment of diseases linked to oxidative stress (Munteanu and Apetrei, 2021). Some natural dyes have notable antioxidant and antimicrobial activities that can be used in the food, medical system and antimicrobial fnishing of textiles (Sadeghi-kiakhani et al., 2021).

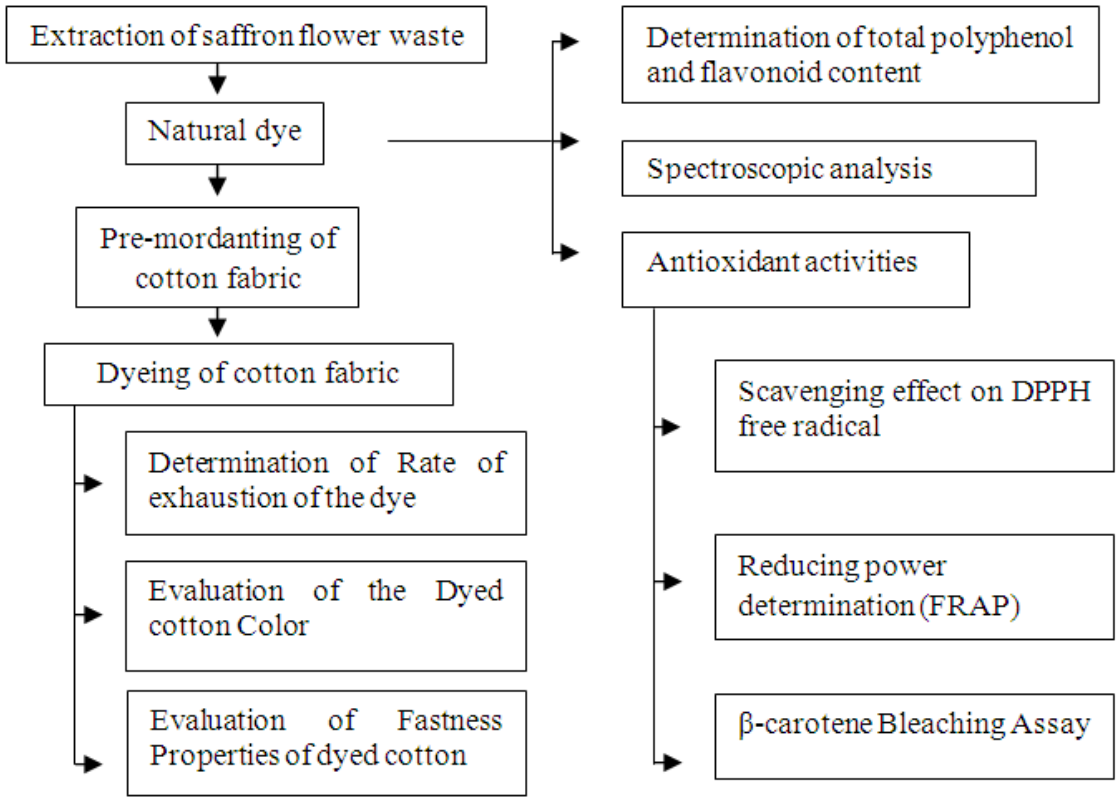
In Taliouine-Taznakht, a remote region of south-west of Morocco, the production of saffron *Crocus sativus* L. is of significant economic importance; it constitutes the main and sometimes the only source of income for around 1 400 families (Aboudrare, 2009).

Saffron is the most expensive spice in the world. Its high cost is mainly due to its very strong involvement of the workforce. The pruning of the flowers to extract the stigmas which, once dried constitute the spice, generates floral waste rich in dyes. Their development in terms of bio-dye constitutes added value for producers and an alternative able to reduce the polluting effect of artificial chemical dyes used in the textile industry.

The chemistry of saffron flowers has caught the attention of professionals in the field for a long time, but the valorization of such a practice is still unsatisfactory. It has been found that saffron petals which are the most abandoned part in the flowers of saffron contain the anthocyanidins (mainly pelargonidine) which are responsible for the purple color of the flowers; this color oxidizes to flavonol (Kaempferol) which gives a yellow color for aqueous extracts (Hadizadeh et al., 2003; Zeka et al., 2015).

Cotton is the basis of much of the textile industry, and it is a pure natural cellulosic fiber (92 to 97% cellulose) containing the groups $–CH_2OH$ and –CHOH as main chemical functions. These hydroxyl functional groups on the glucose units are the points at which dye fixation and bonding take place. It can also be dyed by coordination bonding of cellulose molecules to dyes with metal ions (Baaka et al., 2019).

This work focuses on the use of saffron flower waste as a source of natural dye and its application to the coloring of cotton in order to better enhance this culture and provide an alternative to reduce the polluting effect of chemical dyes. In addition to this coloring power, its antioxidant effect has been studied using different antioxidant tests such as DPPH free radical, reducing power and β-carotene bleaching assay.




## MATERIAL and METHODS

*Flowchart of methodology*

*Materials and chemicals*

Cotton fabrics, which are supplied by ITEX Casablanca, Morocco, with a weight of 125g/ $m^2$, have been used for dyeing. The saffron waste used is made of flower parts (petals and stamens), they come mainly from Taznakht region. Acetic acid, copper sulfate ($CuSO_4$. $5H_2O$) and aluminum and potassium sulfate ($KAl(SO_4)_2.12H_2O$) were obtained from Panreac Quimica SA, Spain. Sodium hydroxide and ferrous sulfate ($FeSO_4.7H_2O$) are purchased from Loba Chemie, India. Tin chloride ($SnCl_2.2H_2O$) is supplied by Sigma Aldrich, USA.

*Extraction of saffron flower*

According to the optimized extraction conditions for the natural dye from the saffron flower waste (Lachguer et al., 2021), flowers (petals and stamens) mean the waste thrown away after collecting the stigmas of saffron have been dried, crushed and dissolved in distilled water at a liquid/solid ratio of 30:1 before boiling at 100 °C for one hour. The solution has been filtered by folded filters (Ø = 150 mm, Porosity = 7-10 µm, PRAT DUMAS, France) and allowed to cool for later use. The colorant has been concentrated under reduced pressure using a rotary evaporator (Buchi R-215) until an extract is obtained. The yield of this extract is 77 ± 0.1% based on the weight of the dried flower and has been used without further purification.

*Determination of total polyphenol and flavonoid content*

The total polyphenol content (TPC) of saffron flower extract is determined according to the Folin-Ciocalteu method modified by Singleton and Rossi (Singleton and Rossi, 1965). Test

tubes containing 0.25 ml of extract, 1.25 ml of Folin-Ciocalteau reagent (10% v/v) and 1 ml of sodium carbonate solution (7%) are prepared and placed in a water bath at 50°C for 5 min, then the absorbance is measured at 760 nm using a visible UV spectrophotometer (Thermo Fisher Scientific, Evolution ™ 300). Quantification has been performed using gallic acid as the standard and the TPC of samples is expressed in milligrams of gallic acid equivalent per gram of sample dry matter (mg GAE/g DM).

The total flavonoid content (TFC) is determined by using aluminum chloride colorimetric method with some modifications (Nan et al., 2009). An aliquant of $AlCl_3$ solution (1 mL, 2 %, w/v) is added to 1 mL of the test solution (standard or sample). The mixture has been vigorously shaken and then after one hour of incubation at room temperature, the absorbance is measured at 430 nm. Quercetin is chosen as the reference compound as it is widely found in plants and the TFC of samples is expressed in milligrams of Quercetin equivalent per gram of sample dry matter (mg QE/g DM).

The total phenolic and flavonoid contents are tested 3 times, and the mean value ± standard error is calculated.

*Spectroscopic analysis*

Absorption spectrum of dye solution is recorded from 300 to 800 nm with an interval of 1nm with Evolution™ 300 UV-Vis Spectrophotometer (Thermo Fisher Scientific, USA) using a quartz cell with a path length of 1 cm.

*Pre-mordanting*

According to our previous research (Lachguer et al., 2021), the mordanting and dyeing protocol is as follows. Before the mordanting and/or dyeing, the cotton fabrics have been treated using 5g/l of sodium carbonate and 1g/l of non-ionic detergent (Marseille soap, Flota) at 50˚C for 30 min to remove the fat in the fibers while maintaining at a liquor ratio of 1:40 (m/v). The fabrics are washed carefully with water and dried at room temperature.

Mordants are used to facilitate the absorption and fixing of the dye on the fibers; in this study a pre-mordanting method is adopted using aluminum and potassium sulfate, copper sulfate, sulfate ferrous and tin chloride. The cotton fabrics have been treated with different concentrations of mordant from 2%, 5%, 10% and 15% on weight of fibers (owf) at a liquor ratio of 1:20. The temperature is slowly rised over thirty minutes to 90°C, and then held at this temperature for one hour. The mordanted fabrics are then cooled and well rinsed.

*Dyeing process*

Cotton fabric samples have been dyed with dye extract at a liquor ratio of 1:20. The samples are immersed in the dye bath in an oscillation dye machine (GESTER, GT-D18, China) at 70°C. The dye parameters are optimized by comparing different factors; concentration of dye (2, 4 and 6 g/100 ml of water), dye bath pH (3, 4, 6 and 8), bath temperature (70, 80, 90 and 98°C) and dyeing time (30, 60, 90 min). The dyed samples are thoroughly rinsed in cold water and washed using non-ionic detergent at 50°C for one hour, then dried at room temperature. For pre-mordanted samples, a dye concentration of 2% is used.

*Rate of exhaustion of the dye*

The remains of the solution dye baths are preserved and designated by residual dye baths. They have been used to determine the exhaustion rate and are analyzed using a UV-Vis spectrophotometer at an appropriate scanning wavelength. The exhaustion rate of the dye is calculated by the equation (1) (Hou et al., 2012):

$$Rex = [(C_0-C_t)/C_0] \times 100 \quad (1)$$

Where, $C_0$ and $C_t$ are concentrations of the initial dye bath and the residual dye bath respectively.

*Evaluation of the Dyed cotton Color*

The spectral reflectance of all cotton fabrics samples is measured using a Datacolor 400 bench-top spectrophotometer with an illuminant D65 and 10° standard observer. The dyed cotton fabric samples are folded into four layers and three different positions are measured. Color strength values (*K/S*) of the dyed samples are calculated using the Kubelka-Munk equation (2) (Hou et al., 2013).

$$K/S = (1-R)^2/2R \quad (2)$$

Where, *R* is the decimal fraction of the reflectance observed at the maximum absorption wavelength, *K* is the absorption coefficient and *S* is the light scattering coefficient.

Since the samples of cotton fabrics dyed with different mordants or methods give different colors and shades, the color values of *L**, *a**, *b**, *c** and *h*° have been evaluated to characterize the colored dyed fabrics. They represent, respectively, lightness-darkness, redness - greenness, yellowness -blueness, saturation and hue.

*Evaluation of Fastness Properties*

Washing fastness of the dyed samples has been tested according to ISO standard 105-C06: 2010. The dyed cotton samples are washed with non-dyed multifibers in a soap solution of 4 g/L at 40 ° C for 30 min, and then the change in color and staining on the adjacent tissues have been evaluated. Color fatness the dyed samples to dry and wet rubbing is determined using a Crockmeter (GESTER-D05) according to ISO105-X12: 2016.

Color fastness to perspiration has been evaluated according to ISO 105-E04: 2013. Cotton samples with non-dyed multifibers are immersed in the acidic and alkaline solution for 30 minutes, then pressed between two plates of the transpirometer and are kept in the oven at 37 °C for 4 hours and classified. These three tests have been evaluated using a gray scale.

Light fastness of dyed cotton is tested in accordance with ISO105-B02: 2014. The light fastness has been determined based on the color change of the fabrics tested and the blue wool reference materials after their exposition for 24 h in a XE-3 xenon test chamber (Q -sun, USA).

*Scavenging effect on DPPH free radical*

In order to study the anti-free radical activity of the dye, we used the method based on DPPH (2,2-diphenyl-1-picrylhydrazyl) as a relatively stable radical, according to the protocol described by Sanchez-Moreno (Sánchez-Moreno, 2002). 50 µl of the solutions of the extract or the standard were added to 1950 µl of DPPH (0.025 mg / ml of the methanol), the mixtures were incubated in the dark for 20 min and the discoloration compared to the negative control containing 1950 µl of solution of DPPH and 50 µl of ethanol was measured at 517 nanometers using a UV / visible spectrophotometer. The DPPH radical scavenging effect was calculated using the following equation (3):

$$\text{DPPH scavenging effect (\%)} = ((A_0 - A_1)/A_0) \times 100 \quad (3)$$

$A_0$ was the absorbance of the control and $A_1$ was absorbance of the extract sample and standard.

*Reducing power determination (FRAP)*

The FRAP method is based on the reduction of ferric ion ($Fe^{3+}$) to ferrous ion ($Fe^{2+}$). The reducing power was determined according to the method of (Oyaizu, 1986). In a test tube containing 0.5 ml of sample solution, were added 1.25 ml of phosphate buffer (0.2M: pH 6.6) and 1.25 ml of potassium hexacyanoferrate 1%. The whole was incubated to 50 ° C in a water bath for 30 minutes. 1.25 ml of trichloroacetic acid (10%) was added and the mixture is centrifuged at 2000 rpm for 10 minutes. Three aliquots of 0.625 ml were made into three eppendorf tubes to which were added 0.625 ml of distilled water and 0.125 ml of $FeCl_3$ 1% freshly prepared in distilled water. A blank without sample is prepared under the same conditions. The absorbance was measured at 700 nm. Ascorbic acid was used as positive control.

*β-Carotene Bleaching Assay*

The antioxidant activity of saffron flower waste extract was evaluated according to the protocol described by Taga, Miller and Pratt, 1984. The β-carotene / linoleic acid emulsion was prepared by dissolving 0.5 mg of β-carotene in 1 ml of chloroform. Then, 25 µl of linoleic acid and 200 mg of tween 40 were added. The chloroform was completely evaporated in the Rotavapor at 40 °C, then 100 ml of distilled water was added with vigorous shaking. 140 µl of different concentrations of solution of extract was added to 1 ml of the previous emulsion. Absorbance was determined at 470 nm immediately against the blank solution before and after heat treatment with regular time intervals of 20 min for 2 hours. Bleaching inhibition percentage was calculated using the following equation (4):

Bleaching inhibition (%) = (β-carotene content after 2 h of assay/initial β-carotene content)/100 (4)

## RESULTS AND DISCUSSION

*Composition of saffron flower extract*

The total phenolic content is 104.82 ± 0.43 mg of gallic acid equivalents per gram of extract. The total flavonoid content is 134.43 ± 0.94 mg of Quercetin equivalents per gram of extract.

Fourier-transform infrared (FTIR) and UV-Vis spectroscopies were used to characterize the chemical nature and the functional group in saffron flower waste responsible for the solubilization and mordanting power of the dye. Fig. 1 shows the UV−Vis absorbance curve of dye solution. UV-Vis spectrophotometry is successfully applied for the determination of major compounds in a solution. As shown in this Figure, dye solution has the highest absorbance in the ultraviolet range (300−400 nm) with the maximum absorbance peak at 344 nm; this may be due to the presence of aromatic phenols perceiving in this wavelength range. This shape remains stable even if the temperature is changed and with pH varying. Therefore, it presents good thermal and pH stability under acidic conditions (Lachguer et al., 2021).

Obviously, The FTIR spectral analysis carried out in our previous research (Figure 2) showed distinct peaks at 3269, 2924, 1763, 1603, 1361, 1021, and 627 $cm^{-1}$. The intense absorption peaks were around 3269 $cm^{-1}$ and 1021 $cm^{-1}$. The first peak corresponded to the stretching vibration O-H of phenols and the second corresponded to the stretching vibration of C-O (Liang et al., 2010). These two intense hydroxyl peaks indicated that the compounds in saffron flower waste dye had hydroxyl functional groups.

*Dyeing properties*

For direct dyeing, the effect of dye concentration, pH, dye bath temperature and dyeing time on the color strength expressed as K/S has been tested and is shown in Fig. 3. The optimized direct dyeing parameters are as follows: 6% dye concentration, dye bath pH of 3, dyeing temperature at 98 °C and dyeing time of 60 min for better dyeing conditions.

Color strength has been shown to increase with increasing saffron flower waste dye concentration. This could be attributed to the population effect of dye molecules present in

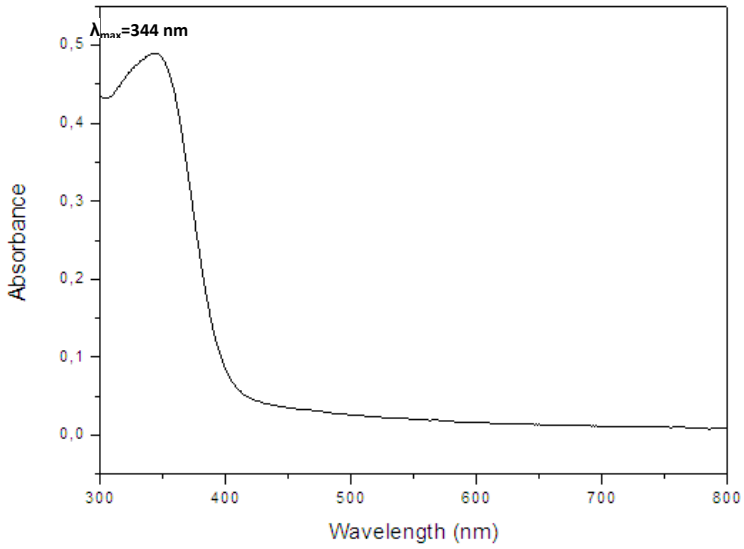



**Fig. 1.** UV–visible absorbance curve of saffron flower dye

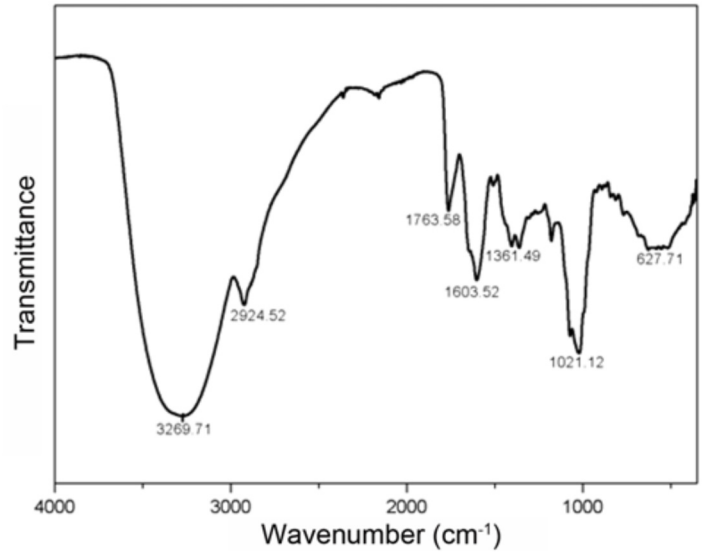



**Fig. 2.** FTIR spectrum of saffron flower dye (Lachguer et al., 2021)

the dye bath at a higher concentration. Moreover, it was found that the K/S exhibits a gradual decrease with the increase of the pH value of the dye bath from pH 3 to pH 8. The maximum color depth was observed under acidic conditions. This could be attributed to the structural characteristics of the dye, which possibly functions as a cationic dye under acidic conditions. However, dye uptake on cotton decreased with increasing pH. But the color remains always weak, so the use of mordants remains a solution to improve the fixation of the dye.

Also, it can be seen that the color intensity increases with increasing dyeing temperature. Because the temperature increases, the reaction speed of the dye increases, the swelling degree of the fiber increases, the diffusion speed of the dye increases, which is beneficial to the chemical reaction between the dye and the cotton. It is also observed that the color strength increases with increasing duration of dyeing time. This effect can be attributed to the ability of the dye to be absorbed by the cotton fibers for up to one hour. Beyond that, the absorption remains stable or can be reduced by the fact that the dye can move from the fabric to the dye bath for a longer

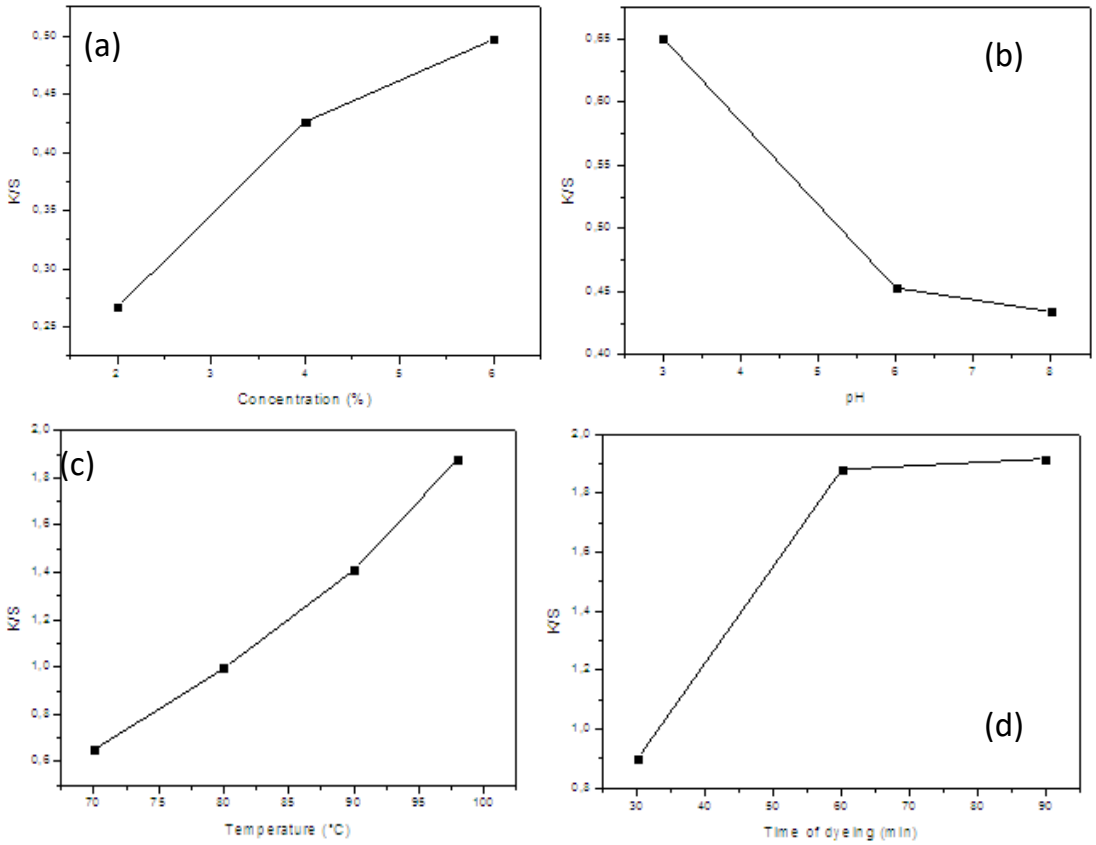



**Fig. 3.** Effect of saffron flower dye concentration, pH, temperature and time on color strength (K/S) of dyed cotton fabrics without pre-mordanting (a) dyeing condition: 70 °C, 60 min, and pH 4, (b) dyeing condition: dye concentration of 6 %, 70 °C, and 60 min, (c) dyeing conditions: dye concentration of 6 %, pH 3, and 60 min, and (d) dyeing conditions: dye concentration of 6 %, 95 °C, and pH 3.

dyeing time.

These results are in agreement with those found by Kamel *et al.* (Kamel et al., 2009) on the increase in K/S by increasing the concentration, temperature and duration of dyeing. However, these authors have found that the optimum pH for dyeing is 8 contrary to what we have found. Also, a study carried out on the dyeing of cotton fabric with *Cuminum cyminum* L. shows that the value of color intensity on cotton increases with increasing pH of the dye bath, with optimum pH for dyeing of 9 and duration of 60 minutes considered as the duration for which the exhaustion of the dye reaches its equilibrium (Tayade and Adivarekar, 2013). Contrary to our results and what is in the literature, these authors found that the color intensity decreases with increasing temperature and that the maximum dye absorption is at 40°C. Our results agree with other studies such as (Deo and Desai, 1999) which have shown that good dyeing of cotton by tea requires optimization of the dyeing conditions, essentially the pH which is an essential element. Maximum color depth was observed under stronger acidic conditions (pH=3) and cotton fabrics can be dyed at medium depths (K/S=2.0).

For dyeing with mordants, cotton is treated using different concentrations of mordants: 2%, 5%, 10% and 15% (owf) with only 2% of the dye.

The color strength (K/S) and the calorimetric values of dyed cotton with or without mordant are presented in Table 1. The results show that all mordanted cotton fabrics have a high color strength compared to the unmordanted fabric attributed to the ability to form a complex between the mordant and the dye.

**Table 1.** Color characteristic values of the cotton fabrics dyed with saffron flower and or without mordant

| Mordant type | | Concentration (%) | K/S | L* | a* | b* | C* | h* |
|---|---|---|---|---|---|---|---|---|
| Without mordant |  | - | 0,9 | 85,85 | 0,03 | 15,3 | 15,3 | 89,9 |
| Alum |  | 2 | 1,46 | 80,34 | -1,55 | 18,99 | 19,05 | 94,68 |
| | | 5 | 1,60 | 80,4 | -1,51 | 18,23 | 18,29 | 94,72 |
| | | 10 | 1,23 | 80,7 | -2,14 | 20,36 | 20,49 | 96 |
| | | 15 | 1,28 | 79,87 | -2 | 20,63 | 20,72 | 95,53 |
| Copper sulfate |  | 2 | 2,08 | 75,07 | -1,06 | 18,32 | 18,35 | 93,32 |
| | | 5 | 2,18 | 76,33 | -2,14 | 23,88 | 23,97 | 95,13 |
| | | 10 | 1,85 | 76,57 | -2,3 | 23,73 | 23,84 | 95,54 |
| | | 15 | 1,96 | 76,02 | -1,87 | 25,07 | 25,14 | 94,26 |
| Iron sulfate |  | 2 | 1,18 | 78,32 | -0,73 | 14,55 | 14,57 | 92,89 |
| | | 5 | 1,43 | 77,24 | 0,47 | 12,98 | 12,99 | 87,95 |
| | | 10 | 1,88 | 83,85 | 0,39 | 16,84 | 16,84 | 88,67 |
| | | 15 | 1,98 | 73,67 | 0,99 | 18,15 | 18,18 | 86,87 |
| Tin chloride |  | 2 | 2,58 | 68,41 | -0,63 | 12,49 | 12,5 | 92,88 |
| | | 5 | 3,72 | 60,83 | -1,9 | 12,98 | 13,12 | 98,35 |
| | | 10 | 4,35 | 58,74 | -2,61 | 13,36 | 13,61 | 101,03 |
| | | 15 | 3,58 | 61,81 | -3,1 | 16,14 | 16,44 | 100,87 |

Cotton treatment with tin chloride gives the highest color strength while the alum shows the lowest one.

This may be due to the aluminum salt which forms weak coordination complexes with the dye, which results in the formation of quite strong bonds with the dye but little with the fiber. Thus, they block the dye and reduce its interaction with the fiber. It is known, that alum does not bond as readily with cotton as it does with other fibers like wool (Ding and Freeman, 2017). Treatment with copper and iron sulfate gives good color strength. The highest K/S values are obtained with 10% (owf) of tin chloride, 5% (owf) of copper sulfate, 15% (owf) of iron sulfate and 5% (owf) of sulfate aluminum and potassium. These results show firstly that with lower concentrations of mordants we can have high color strengths, a phenomena that can be explained by the aggregation of the dye with excess metal salts which decreases its solubility and its fixation on the fibers (Ali and El-Mohamedy, 2011); and secondly that pre-mordanting is the technique which gives the best color strength of cotton fabrics dyed with natural coloring matter extracted from saffron (Kamel et al., 2009); They observed that this strength follows the order: copper sulfate> aluminum and potassium sulfate> stannous chloride> ferrous sulfate.

The lightness value L * is high in the alum corresponding to good luminosity, while this value is lower for stannous, ferrous and copper mordants which show a darker shade.

The color depth of all samples is more yellowish and less greenish as indicated by the values of a* and b*. Negative values of a* for cotton fabrics treated with tin chloride, copper sulfate and alum correspond to shades of green, while yellowing of all fabrics is indicated by average values of b*. Saturation of the colors which tends to be green is indicated by the values C* and h°. The direct dyeing method leads to dull yellow shade of dyed cotton fabrics. The use of

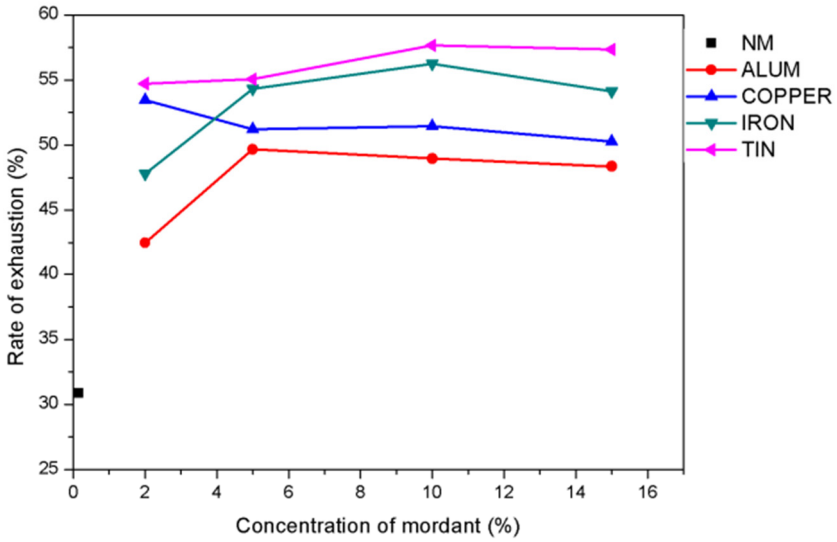



**Fig. 4.** Plot of the rate of dye exhaustion vs. the concentration of mordants.
NM: bath exhausted after cotton dyeing without mordanting. ALUM: bath exhausted after dyeing cotton mordanted with aluminum and potassium sulphate. COPPER: bath exhausted after dyeing cotton mordanted with copper sulphate. IRON: bath exhausted after dyeing cotton mordanted with ferrous sulphate. TIN: bath exhausted after dyeing cotton mordanted with tin chloride.

mordants has produced shades of green; alum and copper sulfate produce a light green shade, while stannous chloride produces a dark green color and iron sulfate produces a brownish green color. The different shades of colors have been found in other studies carried out on the dyeing of cotton by the petals of saffron. (Kamel et al., 2009) revealed that mordanting with copper sulfate gives a light reddish-brown shade, while ferrous sulfate gives a darker and duller shade, and with stannous chloride, the color is lighter in shade. The deeper shades were found through the use of tannic acid and potassium aluminum sulfate. Furthermore, varied hues (ranges from light yellow-dark green to light brown) were obtained from mordanted wool fiber with metal salts when dyed by aqueous extract of saffron petals (Raja et al., 2012; Mortazavi et al., 2012).

Fig. 4 shows the exhaustion rate of the bath after dyeing cotton directly or after a pre-mordanting. We find that the exhaustion rate of the bath after dyeing has been improved by about 20% more by using mordants.

The rate of exhaustion increases from a direct dye (30.87%) to higher values related to presence of mordant. It is raised 57.67% using tin chloride, then 56.27% with iron followed by 53.47% with copper and lastly 49.67% with alum. This is due to the fixing of the dye with the molecules of the mordant used and the cotton fiber. The same results were found in previous works. By comparing the rate of bath exhaustion after dyeing wool with saffron petals without mordant (40.33%) to that treated with mordants, (Lachguer et al., 2021) show that there is an increase in the exhaustion rate by about 20% more by using mordants. It is very high for the tin chloride followed by copper and iron and then alum. (Swamy, 2017) also showed that pre-mordanting of silk fabric dyed with *Casuarina equisetifolia* L. Leaf extract increased the maximum exhaustion rate of the dye bath from 30% to 60%. Maximum exhaustion is observed by alum followed by copper sulphate and ferrous sulphate. (Freeman, 2017) also compared the rate of bath exhaustion after dyeing cotton with natural dyes and different mordants. Decline levels increased from 27% to 56% for CI Mordant Blue 13 and from 38% to 55% for CI Mordant Orange 6. The bath exhaustion rate was also calculated by varying parameters such as pH, temperature, concentration of NaCl added. For example, absorption of Sodium Copper

**Table 2.** Colorfastness rating of the cotton Fabrics Dyed

| | Rubbing | | Washing | | | | | | Ch | Acidic perspiration | | | | | | Ch | Alkaline perspiration | | | | | | Ch | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | D | W | a | b | c | d | e | f | | a | b | c | d | e | f | | a | b | c | d | e | f | | 24 h |
| N. M | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 4-5 | 3 | 5 | 5 | 5 | 4-5 | 4-5 | 4 -5 | 4 | 4 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4 | 3 |
| Al | 5 | 5 | 5 | 5 | 5 | 3 | 5 | 4-5 | 3 | 5 | 5 | 5 | 5 | 5 | 5 | 4-5 | 4-5 | 5 | 5 | 4-5 | 4-5 | 5 | 4 | 3 |
| Cu | 5 | 4-5 | 5 | 5 | 5 | 4 | 5 | 5 | 3 | 4 | 5 | 5 | 4-5 | 3-4 | 4-5 | 4 | 4 | 4-5 | 5 | 4 | 4 | 5 | 3 | 3 |
| Fe | 5 | 4-5 | 5 | 5 | 5 | 5 | 5 | 5 | 3-4 | 4-5 | 5 | 5 | 5 | 4-5 | 5 | 5 | 4-5 | 4-5 | 5 | 4-5 | 4-5 | 4-5 | 4 | 3 |
| Tin | 4-5 | 4 | 5 | 5 | 5 | 5 | 5 | 5 | 3 | 5 | 4-5 | 5 | 4-5 | 4-5 | 5 | 3 | 3-4 | 5 | 5 | 3-4 | 4 | 4-5 | 3 | 1 |

**Legend** N.M: without mordant, Al: Alum, Cu: Copper sulfate, Fe: Ferrous sulfate, Tin: Tin chloride, D: dry, W: wet, a: wool, b: Acrylic, c: polyster, d: Nylon, e: cotton, f: Acetate, L: light, Ch: color change.

Chlorophyllin dye from dyed silk fabrics increased with increasing NaCl concentration from 30% to 70% (Hou et al., 2012). The residual dye bath could even be reused; attempts have been made to take advantage of this and have produced a full range of color intensities. It seems that, from an economic and environmental point of view, the optimization and reuse of the dye bath is of great importance (Kamel et al., 2011).

*Colorfastness properties*

The ability of the fabric to retain the primary color is one of the most important properties of textiles. It is a property of a dye that allows it to maintain its various characteristics despite degrading conditions such as exposure to washing, wet and dry rubbing, light and acid and alkaline perspiration. Colorfastness is then the resistance of the fabric to change in any of its color characteristics. On the other hand, it can prevent water ecosystem pollution, reduce waste and protect human skin. Therefore, color fastness grading is a crucial step in the quality control of fabrics.

The fastness test results of dyed cotton samples with the extract of saffron flowers are given in Table 2. It is clearly evident that the fabrics are found to be moderate to good color fastness to washing, wet and dry rubbing and acid and alkaline perspiration. In case of fastness to rubbing, all samples show high ratings of 4–5, as can be seen in table 2. The acid and alkaline perspiration is moderate to good for all tissues. In addition, the color has moderately changed after washing even with the use of mordants which do not improve the color fastness for all the tests while comparing them with the non-mordanted samples. All tissues have rating higher than 3 corresponding to a moderate resistance to light except tin chloride which shows very low sensitivity.

*DPPH radical scavenging activity*

Antioxidant properties, especially radical scavenging activities, are very important due to the serious role of free radicals in food and in biological systems. The presence of free radicals accelerates the oxidation of lipids in food and decreases the quality of food and therefore consumer acceptance (Min and Boff, 2002). Antioxidant effect on DPPH radical scavenging may be due to their hydrogen donating power, it reduces the purple DPPH free radical to yellow non-radical DPPH-H (Xie et al., 2015).

Fig. 5 shows that the capacity for scavenging DPPH radicals is high in the dye arriving at 80% inhibition for the highest concentration. The extract indicates a dose–response relationship between concentration and DPPH radical scavenging activity corresponding to the decrease in absorbance at 517 nm.

In general, the percent inhibition of DPPH should increase with increasing concentration of plant extract if the extract contains antioxidants. The high concentration of the dye from saffron flower waste corresponds to a multitude of phenolic and flavonoid compounds present in this dye and which are known for their antioxidant power. The antioxidant effect of the dye on

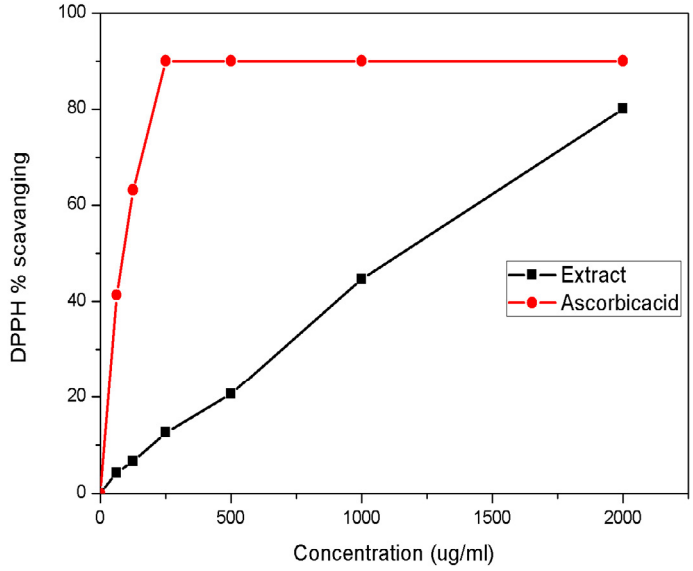



**Fig. 5.** Free radical scavenging activity of extract and standard

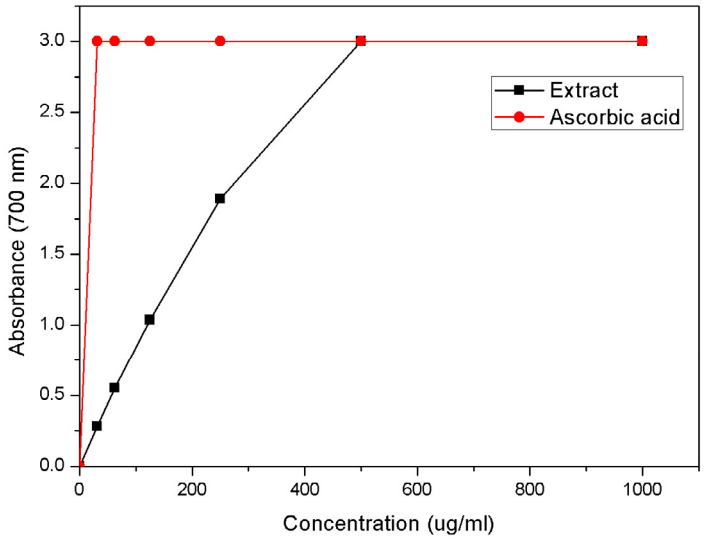



**Fig. 6.** Total reductive potential of different concentrations of extract and ascorbic acid

DPPH radical scavenging may be due to its hydrogen donating ability, which reduces the purple DPPH free radical to yellow non-radical form DPPH-H. The $IC_{50}$ value is 1.22mg/ml. Ascorbic acid presented the highest scavenging ability.

*Reducing Power Determination (FRAP)*

In this analysis, the antioxidant activity is determined by measuring the reduction of the ferric ion ($Fe^{3+}$) to ferrous ion ($Fe^{2+}$) by electron transfer from an antioxidant.

Figure 6 depicts the high absorbance in 700 nm indicated the reducing power of the extract and ascorbic acid standard which are increased with increasing concentration. The reducing power assay is used to assess the ability of an antioxidant to donate an electron which is an essential mechanism of phenolic antioxidant action. In reducing power tests, the increase in absorbance at 700 nm indicates an increase in reducing power from $Fe^{3+}$ into $Fe^{2+}$, thus

transforming the solution into different shades from green to blue, depending on the reducing power of the compounds. However, strong reducing agents formed a blue color were absorbed at 700 nm. Consequently, reactions between the transferred electrons and the free radicals are created making the products more stable. The dye induces a reduction of ferric ions to ferrous ions less effective than ascorbic acid. The $IC_{50}$ value was 64.71 µg/ml.

*β-Carotene Bleaching Assay*

The antioxidant capacity of the dye was also determined using β-carotene bleaching assay. The antioxidant activity, which reflects the ability of the dye to inhibit β-carotene bleaching, was measured and compared to the standard which did not contain any antioxidant component.

The β-carotene bleaching kinetics of the extract is shown in Fig 7 and its inhibition is shown in Fig 8. The β-carotene bleaching kinetics corresponds to the decrease in β-carotene absorbance

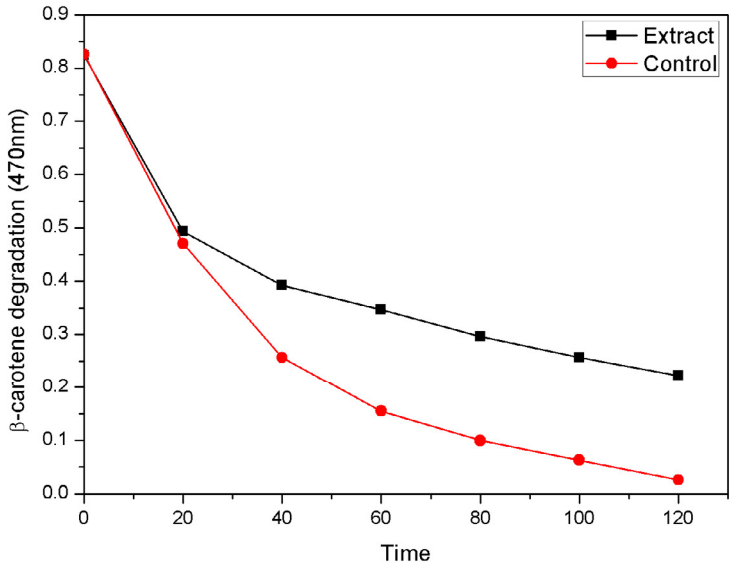



**Fig. 7.** Bleaching kinetics of β-carotene at 470 nanometers

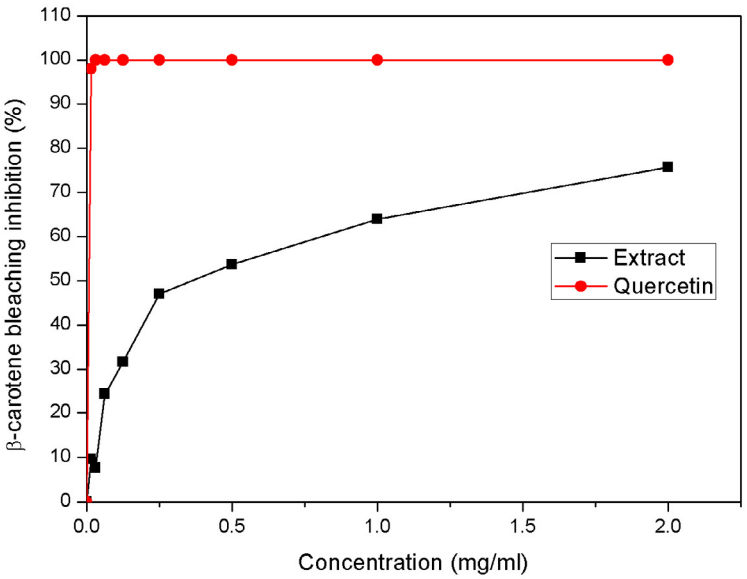



**Fig. 8.** β -carotene bleaching inhibition

at 470 nm due to the antioxidant power of the dye. The β-carotene bleaching test showed the dose-dependent response curve for the extract at concentrations ranging from 0.01 to 2 mg/ml. The control oxidized more quickly, this bleaching is reflected by the discoloration of the β-carotene and the disappearance of its yellow color. However, the presence of an antioxidant prevents this oxidation by neutralizing the free radicals formed from linoleic acid. $IC_{50}$ values were 398.84 ug /ml.

The difference in results obtained for DPPH scavenging ability, FRAP reducing power and β-carotene bleaching inhibition is due to the specificity and sensitivity of each method (Kulisic et al., 2004). These results confirm that the dye has a remarkable ability to react with free radicals to convert them to more stable non-reactive species and to terminate the radical chain reaction. The antioxidant capacity of the dye could be explained by the presence of phenolic and flavonoid compounds. Many reports have revealed that there is a direct correlation between the antioxidant activities and DPPH scavenging, reducing power and β-carotene bleaching of Saffron flower waste. (Lachguer et al., 2022) revealed that the six extracts of saffron flower waste; Methanol, Water, Diethyl ether, Ethyl acetate, n-Butanol, and Aqueous fraction extracts showed a solid scavenging effect, an excellent reducing power, and prevention of the bleaching of β-carotene &, thus, a high antioxidant activity.

Other studies showed a moderate antioxidant capacity of saffron flowers by comparing with its leaves (Lahmass et al., 2017; Jadouali et al., 2018). A study showed that juices obtained from saffron (*Crocus sativus* L.) floral by-products obtained 48 h post-harvest contained higher levels of total polar phenols and then the highest antioxidant activity using FRAP and DPPH methods (Tuberoso et al., 2016). Azghandi et al. showed the antioxidant power of methanolic extract in various parts of saffron (Azghandi et al., 2021). Also, Ouahhoud et al. suggest that C. sativus by-products contain natural antioxidant, metal chelating and DNA protective compounds, which are capable of reducing the risk of many diseases (Ouahhoud et al., 2022). Other authors reported that aqueous and methanolic extracts of saffron petals are a source of nutraceutical substances and have antioxidant activity determined by DPPH, FRAP, and ABTS methods (Caser et al., 2020; Stelluti et al., 2021).

## CONCLUSION

The left out waste of saffron, an agricultural by-product, could be a promising resource for natural dye of cotton and antioxidant with high capacity. Dyeing conditions have been optimized. Thus, the cotton fabrics have a moderate to good color fastness properties with varying hues ranging from dull yellow to brownish green. The natural dye is rich of phenols and flavonoids compounds and revealed a high scavenging effect, an excellent reducing power, and prevention of the bleaching of β-carotene &, therefore, a high antioxidant activity. The availability of the raw material as a valuable product of saffron which is a water-saving crop in arid regions with all its environmental properties and easy extraction techniques will provide natural dyes which have become a new trend in textile industries. The valorization of saffron floral waste in bio-dyes with antioxidant properties which once introduced into the textile industry and in food system will constitute an additional gain for the saffron sector.

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## CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

## LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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