EVALUATION OF FASTNESS PROPERTIES FOR COTTON FABRICS DYED WITH EXTRACT OF SAFFRON PETALS

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Abstract

Nowadays, there is a growing demand in natural colorants from plants because of negative impact of the synthetic ones. The natural dyes are non-toxic and eco-friendly. In this study extracted colorant from saffron petals is used. The dyeing process is conducted both with and without the use of mordants, employing three distinct mordanting methods: pre-mordanting, simultaneous-mordanting, and post-mordanting. The experimental results showed that the simultaneous-mordanting method produces good grades of fastness compared to pre-mordanting and post-mordanting methods. The fastness properties were improved by mordanting process in all cases. The fabrics gained different hues with different mordants resulting in different hue profiles.

Key words: *cotton, natural colorant, fastness, mordant, saffron petals.*

Përmbledhje

Në ditët e sotme ka një kërkesë në rritje për ngjyruesit natyrorë nga bimët për shkak të ndikimit negativ të ngjyruesve sintetikë. Ngjyruesit natyrorë janë jo-toksikë dhe eko-miqësorë. Në këtë studim është përdorur ngjyruesi i ekstraktuar nga petalet e shafranit. Procesi i ngjyrosjes kryhet me dhe pa përdorimin e mordantëve, duke përdorur tre metoda mordimi: para-mordim, bashkë-mordim dhe pas-mordim. Rezultatet eksperimentale treguan se metoda e bashkë-mordimit jep nota më të mira të qëndrueshmërisë në krahasim me metodat e para-mordimit dhe pas-mordimit. Qëndrueshmëria u përmirësua nga procesi i mordimit në të gjitha rastet. Fibrat morën nuanca

të ndryshme me mordentë të ndryshëm duke rezultuar në profile të ndryshme nuancash.

Fjalë kyçe: *pambuk, ngjyrues natyror, qëndrueshmëri, mordent, petale shafrani.*

Introduction

Textile dyeing using natural pigments is a sustainable alternative, as natural substances are biodegradable and compatible with the environment (*Alemayehu et.al., 2014; Mortazavi et.al., 2012; Rahman et.al., 2023; Rehman et.al., 2022; Lachguer et.al., 2023; Janani et.al., 2015*). They are obtained from renewable resources, compared to synthetic dyes, which are derived from non-renewable petroleum resources. The major environmental pollution today is from industries, with textile industries being the largest contributors of liquid effluent due to the large amount of water used in textile processing. It is estimated that globally, 280 000 tons of textile dyes are released annually as textile industrial effluent. Effluents from industries containing synthetic dyes limit light penetration into water bodies, and hence affect the activities of life. They also reduce the amount of dissolved oxygen by forming a thin layer on the water's surface, affecting aquatic fauna (*Jelagat & Cherutoi, 2022; Rehman et.al., 2022; Lachguer et.al., 2023*). Therefore, it is important to implement sustainable dyeing processes that have minimal or benign environmental impacts (*Janani et.al., 2015; Rahman et.al., 2023*).

Most of the natural dyes have no substantivity for the fiber, and mordants must be used. Mordants are usually derived from metallic salts (*Khin et.al., 2017; Alemayehu et.al., 2014; Mortazavi et.al., 2012; Wangatia et.al., 2015; Yusuf et.al., 2017*), or other acidic or basic chemical agents which facilitate the fixing of natural dyes on fabric through chemical reactions (*Mortazavi et.al., 2012; Rehman et.al., 2022*). The commonly used metal salts include alum, chrome, stannous chloride, copper sulphate and ferrous sulphate (*Mortazavi et.al., 2012; Wangatia et.al., 2015; Ahmed et.al., 2019; Rehman et.al., 2022; Yusuf et.al., 2017*). Another advantage of mordanting is that when metallic or mineral salts are added to a natural dye bath, the dye is either intensified or the hue is altered (*Borah et.al., 2023; Rahman et.al., 2023; Zubairu et.al., 2015*). Mordants form a link between the fiber and the dye, which allows certain dyes with no or little affinity for the fiber to be fixed (*Zubairu et.al., 2015; Janani et.al., 2015; Yusuf et.al., 2017*).

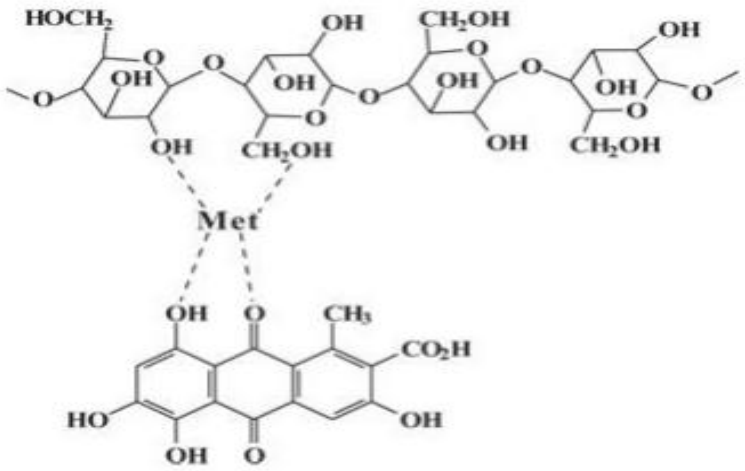

Figure 1. Coloring mechanism of cotton fabric with mordant and natural colorant

Cotton is a natural and eco-friendly fiber. It is used as an essential textile material because of its affordability and abundance, as well as several other qualities including absorbency, strength and durability (*Borah et.al., 2023*).It is a pure natural cellulosic fiber (*Rahman et.al., 2023*) containing the groups $–CH_2OH$ and –CHOH as main chemical functions. The functional hydroxyl groups are the points at which dye fixation and bonding take place (*Lachguer et.al., 2023*).

The dyeing of cotton fabric with natural dyes is reported from different authors (*Kumaresan 2013; Khin et.al., 2017; Borah et.al., 2023; Ahmed et al., 2019; Wangatia et.al., 2015; Rahman et.al., 2023; Rehman et.al., 2022; Lachguer et.al., 2023*). They have concluded that cotton can be dyed using simultaneous mordanting with natural dyes. The color fastness of cotton fabrics is improved by mordants usage.

In this study, saffron petals were selected as a source of natural dye due to their ease of extraction, availability and low cost. They are produced locally in the Dumrea area, Elbasan, Albania. After harvesting stigma the remaining petals are just a waste. They are reused as a natural color resource, thus increasing their economic profit. The extract obtained from saffron petals has no side effects on skin and no harmful effect on the environment as well. The main objective of this research work was to analyze the colour fastness

properties of dyed cellulosic fabric with a natural extract from saffron petals under different mordants and mordanting methods.

Figure 2. Saffron flower cultivated in Dumrea, Elbasan, Albania

Materials and methods

Materials for dye extraction

Saffron petals were supplied from Victus Fed company. Saffron was cultivated in Dumrea area, Elbasan, Albania. The colouring matter used in this work was extracted from saffron petals after stigma harvesting process.

Fabrics

The cotton fabric was supplied from the Department of Textile and Fashion, Polytechnic University of Tirana. The samples used for dyeing process were prepared with dimensions 40x100mm.

Mordants

The mordants used in this study were aluminium sulphate, iron sulphate (III), copper sulphate, zinc sulphate, nickel sulphate, kalium bichromate and sodium sulphate. All the chemicals were of analytical grade.

Extraction of coloring matter

The coloring matter was extracted from saffron petals via the traditional boiling method for one hour. Then the extracted solution left cooled down and filtered off to obtain the natural coloring matter. The natural dye was kept in dark place to avoid photo degradation.

Characterization of the petals as source of coloring components

The mid-infrared vibrational spectra of the dried saffron petals was recorded using a Fourier transform infrared (ATR-FTIR) spectrometer in transmission mode using ATR as sampling technique. The absorbance spectrum from 4000 to 600cm^{-1} was obtained with a number of 64 scans at a resolution of 4cm^{-1} using the software provided by the manufacturer.

Dyeing process

The cotton fabrics were dyed using the traditional method at a liquor ratio of 1:75, pH value of 4.57, time of dyeing 60 min, dyeing solution 2% at boiling temperature and with 10% by fibre weight of mordant concentration.

Mordanting of cotton fabrics

Three mordanting methods were used to dye cotton fabrics. In the pre-mordanting method the fabric is treated initially with the mordant and then is dyed with coloring extract of the saffron petals. In the simultaneous-mordanting, the mordant and the dye are applied at the same time to the cotton fabric, while in the post-mordanting method the fabrics are dyed with the coloring extract and then are treated with the mordant.

Colour measurements

The colour obtained from the cotton samples was determined by measuring the absorbance of the remaining solution on the dyebath after the colouring process is finished. The lowest value of absorbance means the higher amount of the coloring solution obtained by the fiber. UV 1200 Prixma spectrophotometer was used to perform all the measurements of absorbance.

Fastness properties

Colour fastness is used to determine the degree to which dye holds fast to the fiber. A good or high fastness means that they do not bleed in washings and perspiration processes. The colour fastness to washing test and acid and alkaline perspiration test were performed and evaluated according to ISO

standards respectively ISO 105-C10:2006, ISO 105-E04, ISO 105-A02, ISO 105-A03.

Color fastness to washing: A specimen of the textile in contact with two specified adjacent fabrics is mechanically agitated under specified conditions of time and temperature in a soap solution, then rinsed and dried. The change in colour of the dyed cotton specimen and the staining of the adjacent fabrics are assessed with reference to the original fabric with grey scale, ISO 105-C10:2006.

Color fastness to perspiration: Specimens of the textile in the contact with adjacent fabrics are treated in two different solutions, acidic and alkaline solution containing histidine, drained and placed between two plates under a specified pressure in a test device. The change in colour of each specimen and the staining of the adjacent fabrics are assessed by comparison with grey scale ISO 105-E04.

Results and discussion

Characterization of the dried petals

The dried petals were characterized with ATR-FTIR method. The obtained FTIR spectrum is showed below:

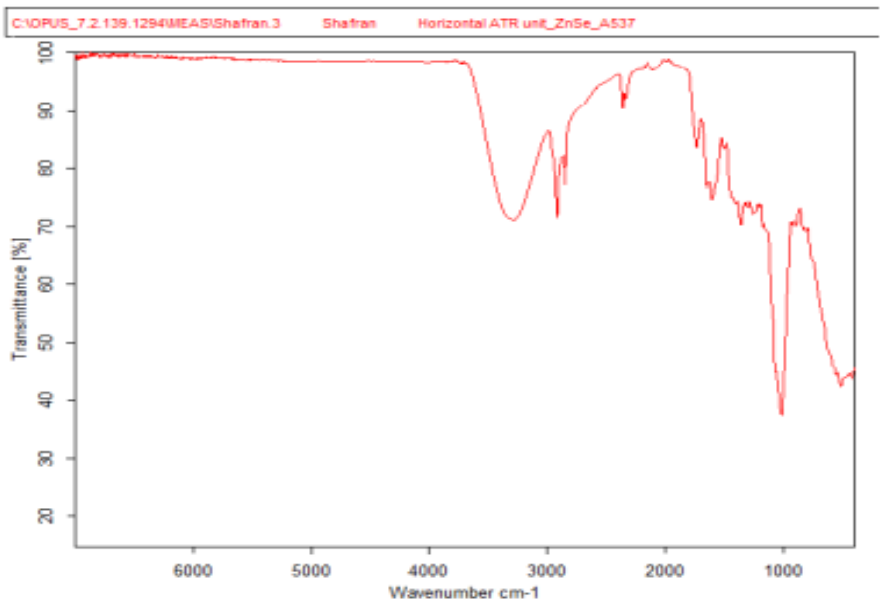

Figure 3. FTIR spectra of dried saffron petals

FTIR analysis was performed to identify the chromophore and auxochrome groups, which are responsible for the substance of the colorant. The spectrum of saffron petals presents visible points in the area 3300 cm^{-1} which corresponds to the vibrational motions of the phenolic OH hydroxyl groups (Ar-OH). These intense points at 3300 cm^{-1} verify the presence of polyphenolic groups which are mostly present in saffron petals. These polyphenolic groups help in the combination of most chromophore groups to be captured on the fabric increasing in this way the color fastness.

The sharp group of peaks at 2900 cm^{-1} and 2800 cm^{-1} are attributed to asymmetric and symmetric aliphatic -C-H bonding. Small dots displayed at 2350 cm^{-1} and 2300 cm^{-1} correspond to aromatic -C-H groups. The peak at 1700 cm^{-1} is characteristic of the carbonyl and ester group. The bands at 1650 cm^{-1}, 1600 cm^{-1} and 1500 cm^{-1} correspond to the C=O group conjugated with two aromatic rings. Peak at 1400 cm^{-1} corresponds to the C=C group of rings of naphthalene, while the peak at 1350 cm^{-1} corresponds to the $-CH_2$ group. Peaks at 1250 cm^{-1} and 1050 cm^{-1} are related to the presence of the C-O group. All these characteristics match the molecular structure of phenolic and flavonoid compounds and confirm their presence in saffron petals (*Naim et.al., 2022; Elhamdaoui et.al., 2022; Jaff et.al., 2014; Mortazavi et.al., 2012*). This spectrum is characteristic of saffron petals and indicates the presence of all the functional groups responsible for the color in the saffron petal extract. These groups form complexes with metals and cellulosic fibers, fix the dye and increase the fastness of the color in the dyed fiber.

Characterization of the aqueous extract of saffron petals

The aqueous extract of saffron petals was analyzed using a UV-VIS spectrometer to identify its λ_{max}, determining the wavelength at which measurements for the dyeing experiments will be conducted. The obtained UV-VIS spectrum showed significant peaks only in UV region. The UV range spectrum is showed below:

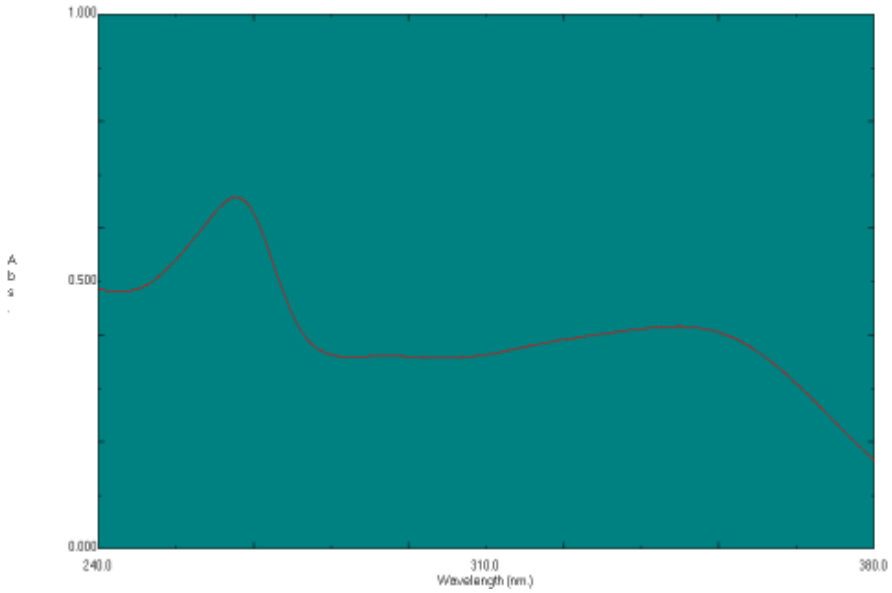

Figure 4. UV spectra of aqueous saffron extract

It is observed that the extract of saffron petals presents two peaks with different intensities at two different wavelengths. A peak with maximum absorbance intensity is observed at a wavelength of 265 nm. This peak appears as a result of the presence of aromatic rings and is detected in the spectrum of all compounds with phenolic structure (anthocyanins). The second peak, which has a lower intensity, but is wider, is positioned at the 340 nm wavelength. It also belongs to the UV zone. This peak at this wavelength together with the first peak at 265nm, confirms the results previously obtained from the FTIR analysis for the presence of flavonoid compounds (*Vihakas 2014*). The peak at 265nm will be used in further measurements of dyeing efficiency.

Washing fastness of dyed cotton fabrics

In the table below are shown the results obtained for colour fastness to washing test for cotton fabrics dyed with saffron petals extract.

Table 1. Rating of colour fastness to washing of mordanted dyed cotton fabrics on grey scale

Mordant	Mordanting method	Value of colour fastness to washing
		Color Change
Without mordant		1
Aluminium sulphate	Pre-mordanting	1
	Simultaneous mordanting	2-3
	Post-mordanting	1
Iron sulphate (III)	Pre-mordanting	1-2
	Simultaneous mordanting	3
	Post-mordanting	2
Copper sulphate	Pre-mordanting	2
	Simultaneous mordanting	3
	Post-mordanting	3
Nickel sulphate	Pre-mordanting	2
	Simultaneous mordanting	2-3
	Post-mordanting	1
Sodium sulphate	Pre-mordanting	1
	Simultaneous mordanting	1-2
	Post-mordanting	1
Kalium dichromate	Pre-mordanting	1-2
	Simultaneous mordanting	3-4

	Post-mordanting	3
Zinc sulphate	Pre-mordanting	1-2
	Simultaneous mordanting	1-2
	Post-mordanting	1-2

From the results presented in table 1, all the dyed samples have moderate values of fastness. It is observed that the mordanting process increases the color fastness. Since the washing fastness of a colored textile is affected and depends on the rate of diffusion of the colorant molecules and the state of the dye inside the fiber, it can be said that the dye molecules tend to aggregate inside the fibers. This brings about an increasing in the molecule size which, on the other hand, leads to good washing fastness properties. In the case of mordanted samples, complexation with the mordant has the effect of reducing the solubility of the dyes, thus increasing the stability of their color.

The color fastness to washing in the case of sample dyes without mordant is related to the affinity of the coloring components with the fiber through hydrogen bonds and Van der Waals bonds. However, this stability is very low. For almost all mordants, the best color fastness to washing is noted in the simultaneous-mordanting process, where grades 2, 2-3, 3 and 3-4 are obtained, while for two other mordanting methods, the results were lower, thus showing a decrease in values of color fastness in washing.

These values indicate moderate to good color fastness to washing. However, we can say that the use of saffron petals as a coloring material for cellulosic samples can be seen as an alternative in the future. Other methods of mordanting, other mordants and pre-treatment of fibers must be further studied in order to produce more wash-fast colors. The dye extracted from saffron petals produced different colors with mordants. The formation of these multiple colors with different mordants tells us that the saffron petal dye is classified as a polygenetic dye.

Perspiration fastness of dyed cotton fabrics

The cotton fabrics dyed with extract of saffron petals were tested for perspiration fastness according to the standard ISO 105-E04. In the following table are presented the obtained results.

Table 2. Acidic and alkaline perspiration fastness properties of mordanted, dyed cotton fabrics

Mordant	Mordanting method	Acidic perspiration colour fastness	Alkaline perspiration colour fastness
		Colour Change	Colour change
Without mordant		1	1
Aluminium sulphate	Pre-mordanting	2	1
	Simultaneous mordanting	2-3	2
	Post-mordanting	1	2
Iron sulphate (III)	Pre-mordanting	2	1-2
	Simultaneous mordanting	2-3	2-3
	Post-mordanting	2	2
Copper sulphate	Pre-mordanting	1-2	1
	Simultaneous mordanting	3	1-2
	Post-mordanting	1-2	1
Nickel sulphate	Pre-mordanting	1	1
	Simultaneous mordanting	1-2	1-2
	Post-mordanting	1	1
Sodium sulphate	Pre-mordanting	1	1
	Simultaneous mordanting	1-2	1-2
	Post-mordanting	1	1
Kalium dichromate	Pre-mordanting	1	1
	Simultaneous	2-3	2-3

	mordanting		
	Post-mordanting	2-3	2-3
Zinc sulphate	Pre-mordanting	1	1
	Simultaneous mordanting	1-2	1
	Post-mordanting	1	1

From the obtained results, the cotton samples have higher color fastness properties in the simultaneous-mordanting process for both acidic and alkaline perspiration tests. The color fastness is higher in acidic perspiration compared to alkaline perspiration. These results confirm again that the cotton fabrics adsorb the colorant better in this mordanting method. The color staining is lower.

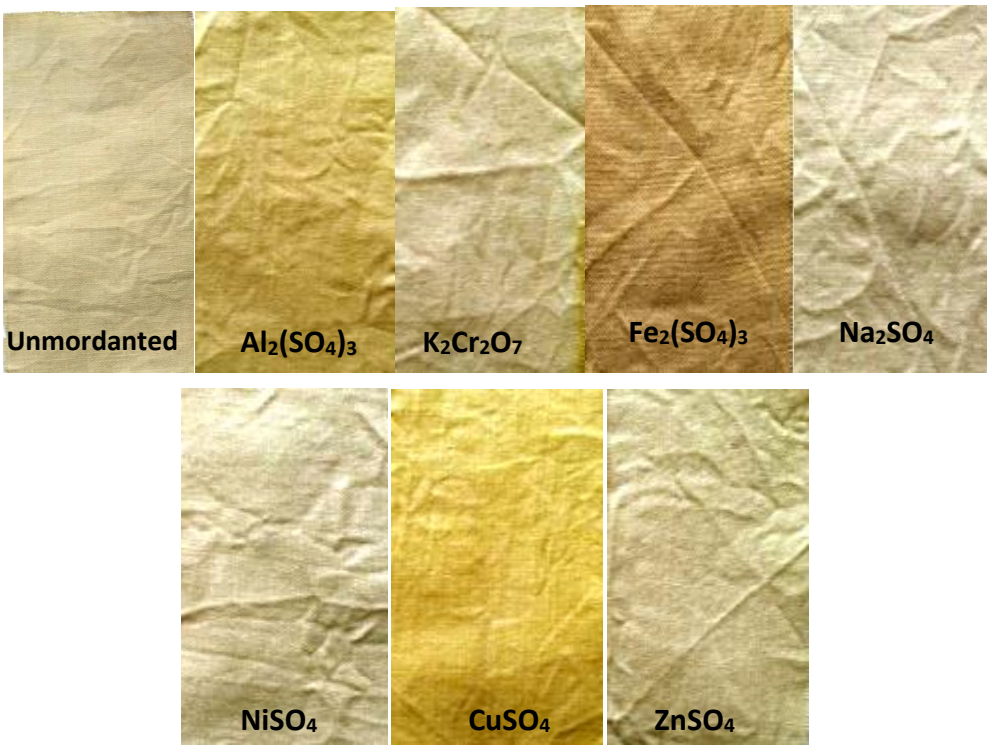

Figure 5. Saffron dyed cotton fabrics with different mordants

Figure 5 shows coloured cotton fabrics obtained from the simultaneous-mordanting method with different mordants. As it can be seen, the colored cotton fabrics have different hues for different mordants.

Conclusions

The results obtained from this experimental work concluded that the extract of saffron petals can be successfully used for dyeing of cotton yarns; a wide range of soft and light colours can be derived by using different mordants and mordanting techniques. Simultaneous mordanting increased affinity of cotton fabrics for saffron extract dye molecules, with development of attractive shades on dyed fabrics for each mordant. The mordants also enhanced the colour quality of the fabrics due to the increased efficiency in absorption and fixation and better colour fastness grades in terms of washing and perspiration, respectively. The grades of washing and perspiration fastness were higher in simultaneous-mordanting process compared to two other mordanting methods. The good fastness properties of mordanted cotton fabrics demonstrated that petals of saffron can be a potential eco-friendly alternative to synthetic dyes in the textile industry.

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References

Alemayehu T. and Teklemariam Z., (2014): Application of natural dyes on textile: a review. International Journal of research-Granthaalayah, 2(2)

Ahmed M., Islam T., Karim M.R., Kaiser S. H., Barua P., (2019): Assessment of fastness properties of knitted cotton fabric dyed with natural dyes: a sustainable approach of textile coloration. Journal of Textile Engineering & Fashion Technology, 5(3), 177-182

Borah P., Boruah R.R., Konwar M., Baruah S., Dutta S. H., (2023): Evaluation of colour fastness properties of cotton fabric dyed with pomegranate rind extract dye. The Pharma Innovation Journal, 12(8), 2127-2131

Elhamdaoui O., El Orche A., Cheikh A., Laarej K., Karrouchi K., El Karbane M., Bouatia M., (2022): Tracing the geographical origin of moroccan saffron by mid-infrared spectroscopy and multivariate analysis. Brazilian Journal of Analytical Chemistry, 9(37), pp. 115-128, https://doi.org/1030744/brjac.2179-3425.AR-23-2022

ISO 105-C10:2006, Textiles-Tests for colour fastness – Part C06: Colour fastness to washing with soap or soap and soda

ISO 105-E04, Textiles-Tests for colour fastness – Part E04: Colour fastness to perspiration

ISO 105-A02, Textiles-Tests for colour fastness – Part A02: Grey scale for assessing change in colour

ISO 105-A03, Textiles-Tests for colour fastness – Part A03: Grey scale for assessing staining

Janani L., Hillary L., Phillips K., (2015): Mordanting methods for dyeing cotton fabrics with dye from albizia coriaria plant species. International Journal of Scientific and Research Publications, 4(10), ISSN: 2250-3135

Jaff P.M., Rasheed B.O., Omer T.A., (2014): Discrimination of saffron available in local markets using spectroscopic methods. IOSR Journal of Applied Physics, 6(6), pp. 56-61, e-ISSN: 2278-4861

Jelagat L. and Cherutoi J., (2022): Dyeing of cotton fabric with natural dye from Flavoparmelia caperata. Advances in Phytochemistry, Textile and Renewable Energy Research for Industrial Growth, ISBN: 978-1-032-11871-0, 125-131

Khin O.O., Yee O.S., (2017): A study on the fastness properties of cotton fabric dyed with turmeric dyestuff. International Journal for Innovative Research in Multidisciplinary Field, 3(8), ISSN – 2455-0620

Kumaresan M., (2013): Comparison of fastness properties and colour strength of dyed cotton fabrics with eco-friendly natural dyes. International Journal of Science and Technology, 8(3), 483-489

Lachguer K., Boudadi I., Fayzi L., El Merzougui S., El Bouchiti M., Cherkaoui O., Serghini M. A., (2023): Natural extraction of dyes from saffron (Crocus sativus L.) flower waste, cotton dyeing and antioxidant effectiveness. Pollution, ISSN: 2823-451X,

https://doi.org/10.22059/poll.2023.351631.1700

Mortazavi S.M., Moghaddam M.K., Safi S., Salehi R., (2012): Saffron petals, a by-product for dyeing of wool fibers. Progress in Color, Colorants and Coatings, 5(2), pp. 75-84, https://doi.org/10.30509/PCCC.2012.75800

Naim N., Fauconnier M.L., Ennahli N., Tahiri A., Baala M., Madani I., Ennahli S., Lahlali R., (2022): Chemical composition profiling and antifungal activity of saffron petal extract. Molecules, 27(24), https://doi.org/10.3390/molecules27248742

Rahman M.M., Kim M., Youm K., Kumar S., Koh J., Hong K.H., (2023): Sustainable one-bath natural dyeing of cotton fabric using turmeric root extract and chitosan biomordant. Journal of Cleaner Production, 382, https://doi.org/10.1016/j.jclepro.2022.135303

Rehman A., Irfan M., Hameed A., Saif M.J., Qayyum M.A., Farooq T., (2022): Chemical-free dyeing of cotton with natural dye: A pollution-free and cleaner production approach. Frontiers in Environmental Sciences, 10, https://doi.org/10.3389/fenvs.2022.848245

Vihakas M., (2014): Flavonoids and other phenolic compounds: Characterization and interactions with lepidopteran and sawfly larvae; doctoral thesis

Wangatia L.M., Tadesse K., Moyo S., (2015): Mango bark mordant for dyeing cotton with natural dye: Fully eco-friendly natural dyeing. International Journal of Textile, 4(2), 36-41, doi:10.5923/j.textile.20150402.02

Yusuf M., Mohammad F., Shabbir M., (2017): Eco-friendly and effective dyeing of wool with anthaquinone colorants extracted from Rubia cordifolia roots: Optimization, colorimetric and fastness assay. Journal of King Saudi University – Science, 29, pp. 137-144, https://dx.doi.org/10.1016/j.jksus.2016.06.005

Zubairu A., Mshelia Y.M., (2015): Effects of selected mordants on the application of natural dye from onion skin (Allium cepa). Science and Technology, 5(2): 26-32,

https://doi.org/10.5923/j.scit.20150502.02