




Article

Microwave-Assisted Exploration of Yellow Natural Dyes for Nylon Fabric

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Abstract: Today, the global community is appreciating green technologies in the application of green products in textiles. The aim of the current study is to use a sustainable heating technique for the isolation of colorant from plant sources and to use eco-friendly anchors to improve the fastness of dyed fabrics with new shades. The current study used microwave radiation to isolate natural colorants from saffron (*Crocus sativus*) and safflower (*Carthamus tinctorius* L.) petals for polyamide (nylon) fabric dyeing. For this purpose, acidic extract and fabric were exposed to MW treatment for up to 6 min and employed at various conditions. To make the dyeing process sustainable, bio-mordants have been employed and compared with synthetic mordants. It has been found that 6 min is the optimal radiation time for the isolation of colorant to get good results onto irradiated polyamide (nylon) fabric when employed at 65 °C for 45 min containing 1 g/100 mL of table salt for saffron and 3 g/100 mL of table salt for safflower dyeing. For improving color strength and giving an acceptable rating of fastness, 7% of turmeric as a pre-bio mordant and 7% pomegranate as a post-mordant has given high results using saffron extract. Similarly, with safflower extract, 5% of turmeric as a pre-mordant and 5% of turmeric extracts as a post-mordant have given high results as compared to chemical mordants used. It is concluded that microwave treatment has a high potential for investigating the coloring efficacy of crocin-containing saffron petals and safflower petals as carthamin as a yellow natural dye for bio-mordanted polyamide fabrics. It is recommended that such tools for the isolation of colorant from new dye-producing plants should be used, whereas green mordants should be used to develop new colorfast shades to make process more green and sustainable.

Keywords: bio-mordant; saffron; microwave; polyamide fabric; sustainability



Citation: Rehman, F.U.; Adeel, S.; Haddar, W.; Bibi, R.; Azeem, M.; Mia, R.; Ahmed, B. Microwave-Assisted Exploration of Yellow Natural Dyes for Nylon Fabric. *Sustainability* **2022**, *14*, 5599. <https://doi.org/10.3390/su14095599>

Academic Editor: Yu Yang

Received: 4 March 2022

Accepted: 31 March 2022

Published: 6 May 2022

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1. Introduction

The fashion industry has become a major polluter and one of the largest emitters of greenhouse gases in the world. The use of environmentally harmful chemicals and textile waste accumulation causes the textile industry to remain the second worst polluting industry worldwide [1]. The production of colored garments is ensured through the use of synthetic chemicals, which entails the use of non-renewable and non-green raw materials, which are strongly linked to environmental and health problems [2,3]. Nowadays, the chemicals used to manufacture dyes are frequently extremely toxic, carcinogenic, or even explosive [4]. The chemical aniline serves as the basis for a popular class of dyes known as Azo dyes. These dyes are considered lethal poisons and are extremely dangerous to work with [5]. Additionally, dioxin (a known carcinogen and possible hormone disruptor)

is used in the conventional dyeing process today, as are toxic heavy metals such as chrome, copper, and zinc, as well as formaldehyde (a suspected carcinogen) [6,7]. All of these toxic chemicals wreak havoc on the environment by contaminating the air, water, and soil. Water is used in large quantities to flush conventional synthetic dyes from garments, and then the wastewater must be treated to remove heavy metals and other dangerous substances before it can be handed back to water systems, sewer lines, and rivers [8,9]. The natural biosorbent from *Codium decortatum* alga (CDA) waste has emerged as a promising strategy for removing toxic dyes from wastewaters [10].

The interest in green products continues to grow daily, and it is becoming increasingly important to provide environmentally friendly dyed fabrics and garments [11]. Due to increased awareness of these health and environmental risks, a renewed international interest in natural dyes has emerged in recent years, with the dyes being proposed as a permanent solution to the environmental problems caused by synthetic colorants [12,13]. This will reduce the pollution burden and associated remediation costs associated with these harmful components. Apart from providing attractive natural colors, natural dyes can impart additional properties to the fabric, such as ultraviolet protection and antibacterial and antifungal properties [14,15]. To enhance cellulose's dyeability, it was extracted from sugarcane bagasse (SCB) and treated with xylanase to remove residual non-cellulosic polymers (hemicellulose and lignin). After that, the fibers were dyed with natural dyes extracted from *Ceasalpinia sappan* Linn. and *Artocarpus heterophyllus* Lam. heart wood [16]. Numerous natural dye sources have been identified, including catechin, gardenia, coffee sludge, and pomegranate [17].

Crocus Sativus Linn locally known as Saffron is the perennial plant that belongs to *Iridaceae* [18,19]. The spice made from the dried red stigma of the flowers of this plant is considered the world's most expensive spice. It has culinary and medicinal properties. Safflower (*Carthamus tinctorius* L.) is a source of famous natural pigment called carthamin that is widely used in textiles, food and flavors, herbal medicine, and cosmetics [20]. It has been shown to have excellent nutritional value and biological activity [21]. Safflower petals contain red and yellow pigments found as carthamin which give a pink to yellowish color. As early as 4500 BC, these pigments were used in cloth dyeing and natural cosmetics [22,23]. Currently, the yellow pigment (water-soluble) is used as a natural yellow food colorant in rice, bread, candy, jelly, and beverages, while the red pigment (water-insoluble) is primarily used as a fabric dye [22].

The purpose of this study was to extract dye from saffron and safflower petals for application on nylon fibers. Nylon is a generic term for a group of widely used synthetic polymers, more precisely aliphatic or semi-aromatic polyamides in which at least 85% of the amide linkages (-CO-NH-) are directly attached to aliphatic or cycloaliphatic units. The primary fiber-forming substance is any long-chain synthetic polyamide that contains recurring amide groups in its backbone. Indeed, polyamide fibers contain protonated terminal amino groups, which enables them to be dyed with the same wool dyes that operate at an almost acidic pH [24]. These fibers are extremely tough and resistant to abrasion, and they are easy to wash and dye in a variety of colors. The filament yarns create a smooth, soft, and light fabric with a high degree of resilience. The nylon fabrics have been dyed using acid dyes [25].

Several techniques are used to enhance the uptake of a natural colorant such as the ultrasonic method [26], gamma radiation [27], plasma treatment [28], supercritical fluid extractions [29], microwave radiations [30], and UV-radiation technique [31]. All these modern approaches try to overcome the limitations of traditional methods which are not capable of saving energy and money and have no effective yield of obtained shades and fastness. However, microwave radiation has been found to be more effective as a sustainable heating tool, resulting in decreased dyeing time, energy consumption, and solvent consumption. Indeed, this technique enables the generation of promising solvent-powder interactions in the presence of a uniform heating source, resulting in excellent mass transfer kinetics and the potential for high colorant yields in textile fabrics [32].

The purpose of the study is to utilize the selected MW ray level to extract the colorant in a suitable medium, to investigate the physicochemical properties of fabrics before and after dyeing, and to select the dyeing variable for mordanting for colorfast shade with better fastness ratings.

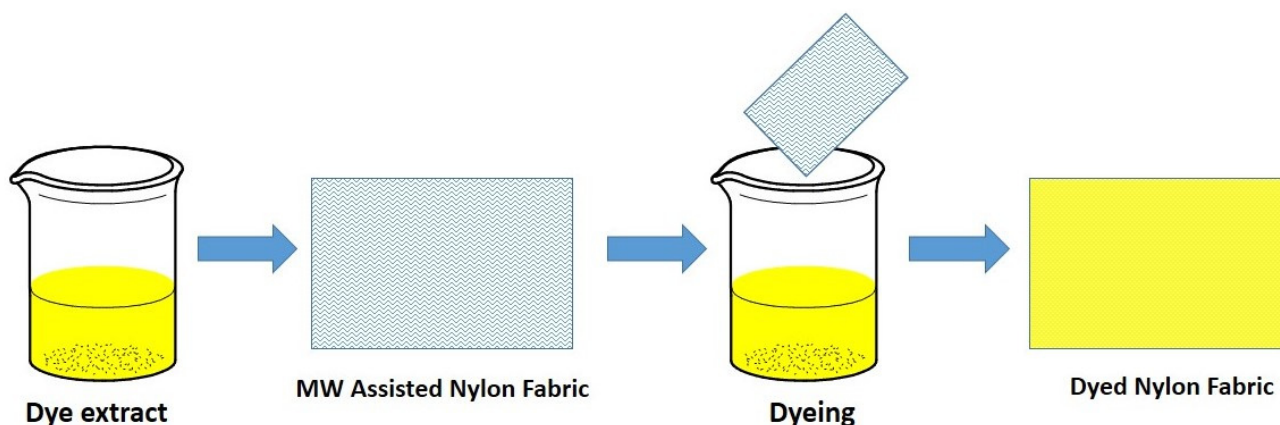
2. Materials and Methods

2.1. Materials

The raw powder of saffron and safflower dry petals has been collected from supermarket and sieved (20 meshes). Plant powders used as biological mordants have been ground, sieved, and stored. Nylon woven fabric (110×80 , weight = 20 g/m^2) was also stored for the coloration process after being pretreated with neutral soap. The remaining chemicals used for isolation, dyeing, mordanting, and fastness were purchased commercially (Pakistan-made).

2.2. Dye Extraction and Irradiation Process

Aqueous (water solubilized) and acidic (acid solubilized) extracts were made by combining 4 g of powder with 100 mL of boiling media. After the isolation process, the rough extract was filtered through muslin cloth and the extracts were stored and residues were discarded. Pre-treated nylon and respective extracts were exposed to MW rays from 1.0–6.0 min. After irradiation, the application of un-irradiated (NRE) and irradiated extracts (RE) were made to dye irradiated (RF) and un-irradiated nylon fabrics (NRF) at 80°C for 45 min. The set of experiments was designed to optimize extract salt amount for exhaustion (1.0–5.0 g/100 mL) and dyeing time (15–60 min.) with 15 min intervals using optimal irradiation and extraction conditions. For the application of green chemical and bio-mordants employed before and after dyeing, the process was followed given by Hayat et al. [33]. A schematic representation of the nylon fabric dyeing process is shown in Scheme 1.



Scheme 1. A Schematic representation of the nylon fabric dyeing process.

2.3. Fabric Assessment

The undyed nylon fabric was subjected to physicochemical analysis before and after a 6 min microwave treatment [34]. For the examination of dyed fabrics, a data color, SF 600 equipped with a light source (D 65, 10° observer) was used. Methods of ISO standards for washing and rubbing (ISO 105-CO3, ISO 105-BO2, and ISO 105-X12) were as described in our previous studies to test shade fastness [23], and the results were compared on a greyscale [35].

3. Results and Discussion

On account of the energy, time, and economy-saving concept, the microwave is an excellent heating source. In the chemistry of natural products, its role has been widely utilized because it is clean, uniform, and leveled in its action [20,36]. These rays collapse

with the plant matrix and tear up its boundary to give access of biomolecules toward solvent. These rays also cause mass transfer into the solvent and provide an effective matrix-solvent intention to take a high yield [37]. The same is the action of MW rays that have been found in our studies. The results shown in (Figure 1a,b) representing that using saffron, its aqueous extract irradiated for 3 min has given high yield ($K/S = 1.6245$) onto irradiated polyamides fabric. Similarly using safflowers, its aqueous extract irradiated for 3 min has given high yield ($K/S = 7.898$) onto un-irradiated polyamide fabric. Using the acidic extract of saffron, irradiation of extract for 6 min has developed good strength ($K/S = 7.2558$) onto un-irradiated polyamide fabric (Figure 2a). Similarly using acidic extract of safflower irradiation of extract and fabric for 6 min. has developed good results ($K/S = 12.444$) (Figure 2b). The color coordinates shows that fabric dyed with acidic saffron extract is more-brighter with redder and more-yellow in tone ($L^* = 80.42$; $a^* = 2.85$; $b^* = 45.11$). The color coordination shows that fabric dyed with acidic safflower extract is darker with redder and yellow in tone ($L^* = 52.96$; $a^* = 14.36$; $b^* = 46.65$), as shown in Table 1.

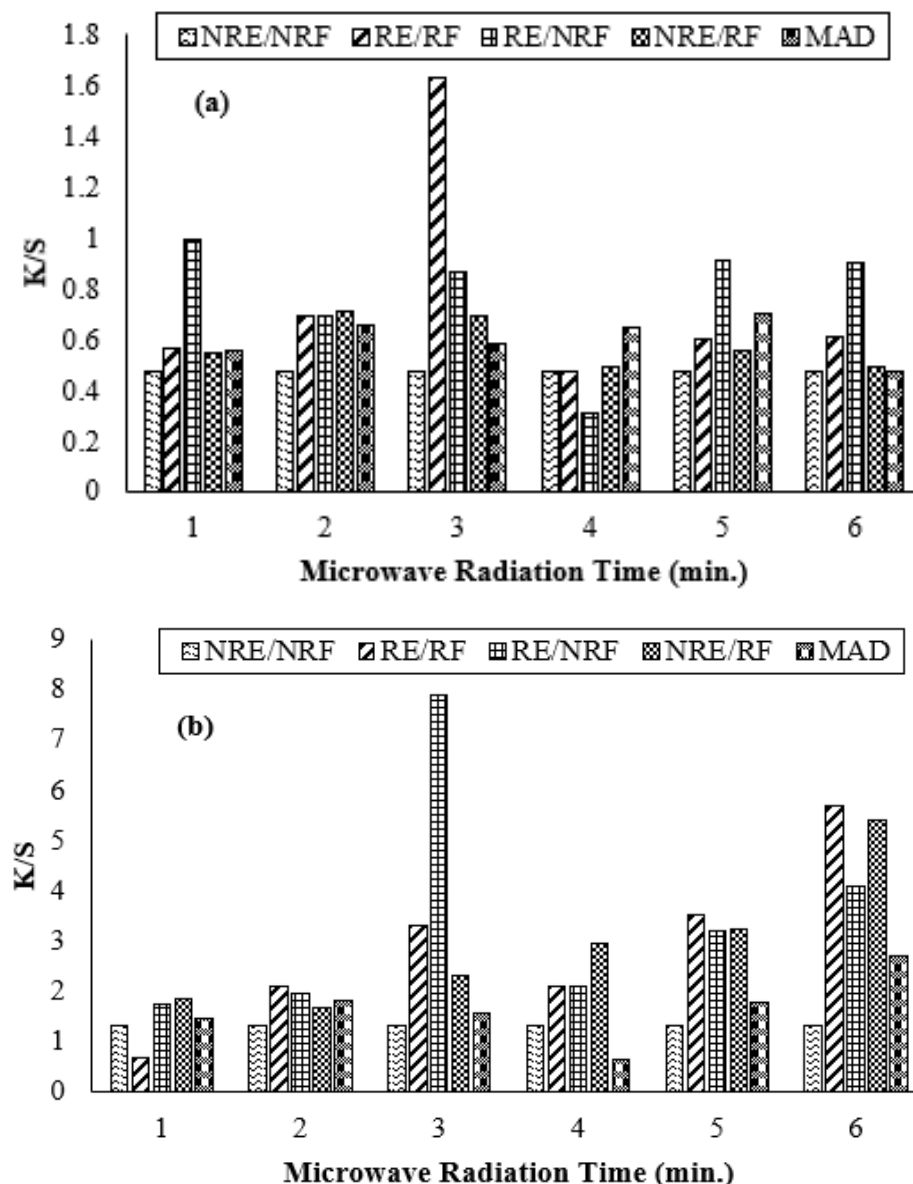


Figure 1. Dyeing of polyamide fabric with aqueous saffron (a) and safflower (b) extract before and after microwave radiations.

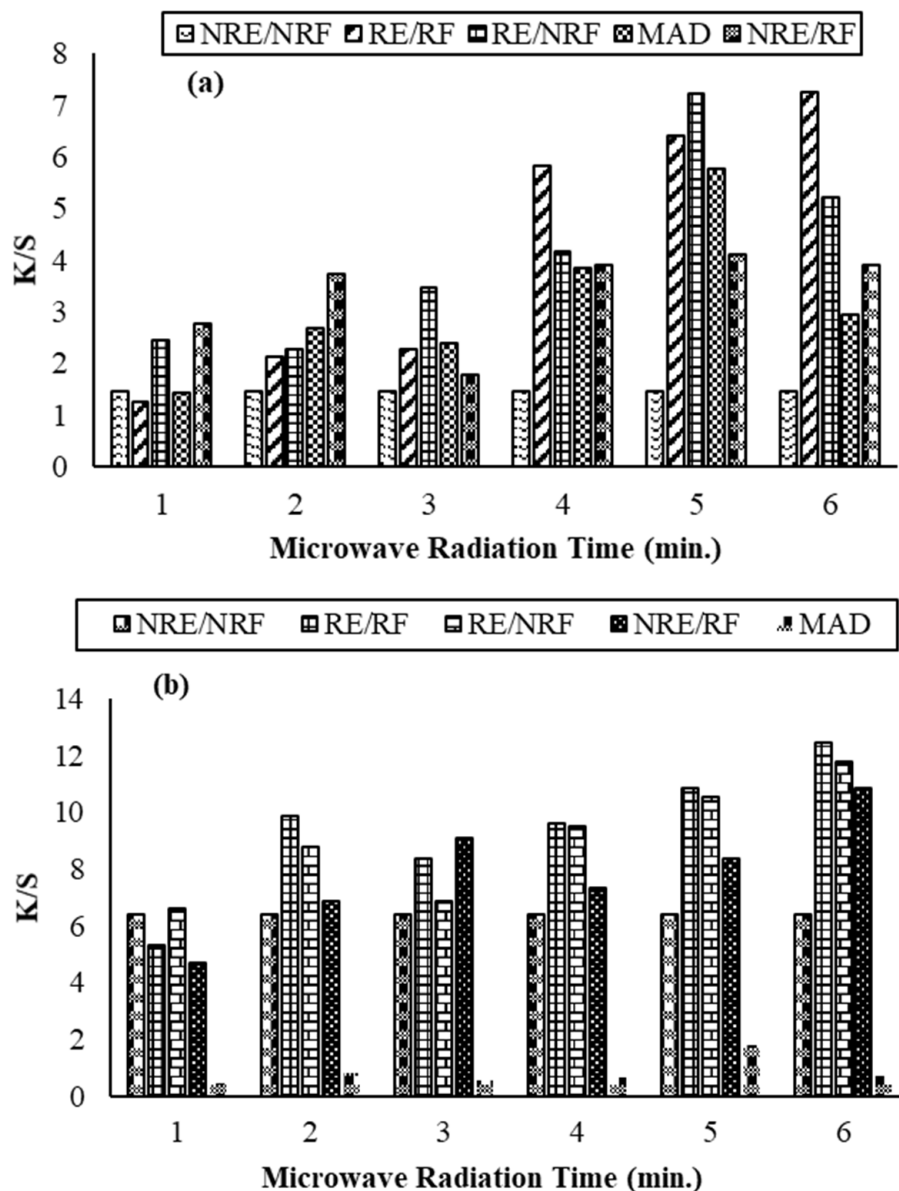


Figure 2. Dyeing of polyamide fabric with acidified saffron (a) and safflower (b) extract before and after microwave radiations.

Table 1. Color co-ordinates of nylon fabric bio-dyed at selected conditions.

	Saffron			Safflower			
Extraction Conditions	L^*	a^*	b^*	Extraction Conditions	L^*	a^*	b^*
Acidic				Acidic			
6 min RE/RC	80.42	2.85	45.11	6 min RE/RC	52.96	14.36	46.65
	Parameters						
Dyeing Conditions	L^*	a^*	b^*	Dyeing Conditions	L^*	a^*	b^*
Time = 45 min				Time = 45 min			
Salt = 1 g/100 mL	70.14	5.43	34.72	Salt = 3 g/100 mL	63.90	8.57	29.90
	91.70	−2.46	22.70		49.96	7.81	31.71

L^* = lighter/darker shade, a^* = redder/greenr tone and b^* = yelower/greenish huse.

In the dyeing of synthetic fabric, the contact of stuff with the colorant particularly in natural dyeing is very important using saffron and safflower acidic extract, the contact with irradiated polyamide fabric for 45 min has given a maximum yield, where the application of irradiated acidic safflower extracts into the un-irradiated fabric for 45 min has given

an excellent yield (Figure 3a,b). Previously, it has been found that for a low contact-time, the rate of dyeing is low, whereas a long contact-time in the dye bath results in over desorption, due to which a low yield has been observed. However, microwave has reduced the contact time which show that this tool is time effective [38]. The amount of salt is always there to overcome the limitation of the low yield of natural dye. Salt when added always tries to make an effective environment between fabric dye and solvent. A low amount cannot result in exhaustion, whereas a high amount causes over-exhaustion, due to which colorants gather onto fabric in the form of aggregates [30]. After microwave ray treatment using acidic saffron extract 1 g/100 mL of salt and using acidic safflower extract 3 g/100 mL of salt has given excellent exhaustion which has been observed in terms of color strength (Figure 4a,b). Again, it can be seen that by reducing the salt amount to get a high yield in natural dyeing polyamide fabric, the MW treatment again has been observed as a cost-effective tool.

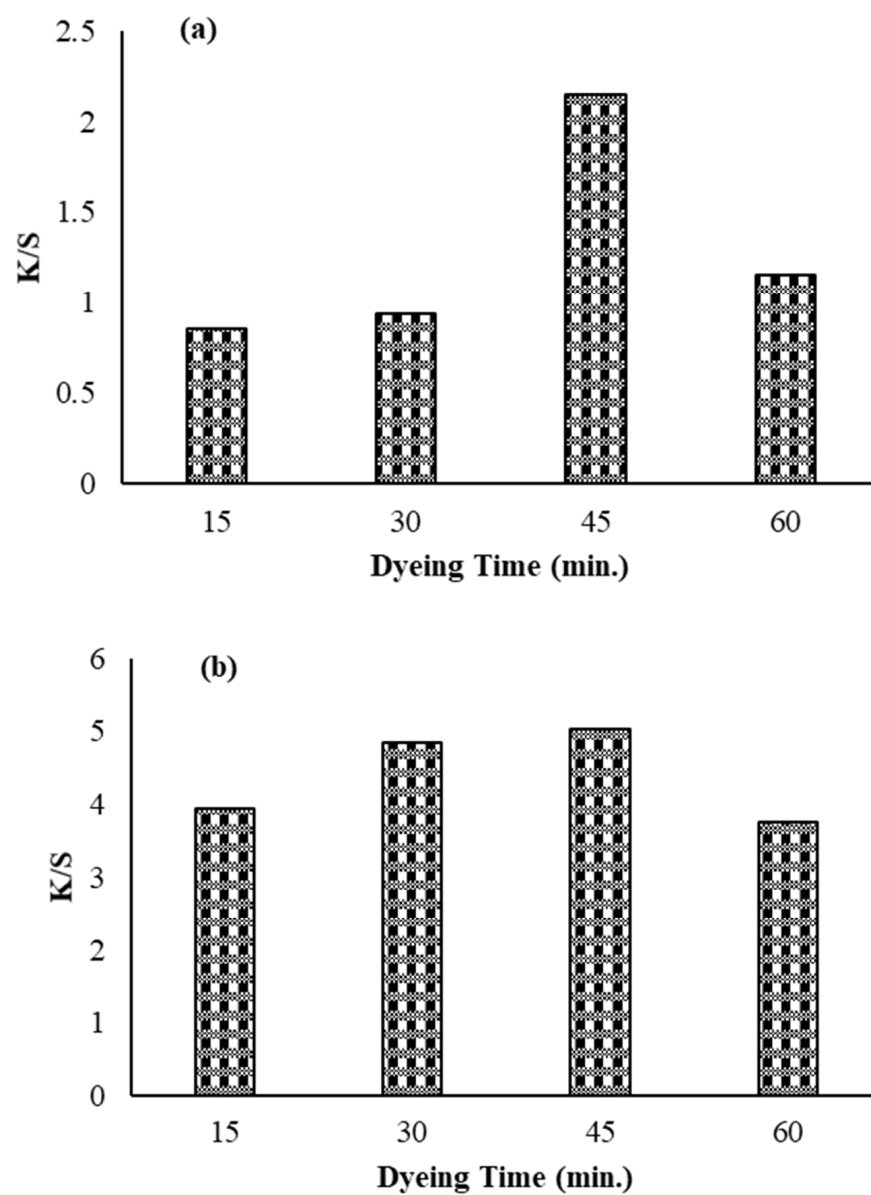


Figure 3. Effect of dyeing time on the color strength of dyed polyamide fabric using optimized extract of saffron (a) safflower (b).

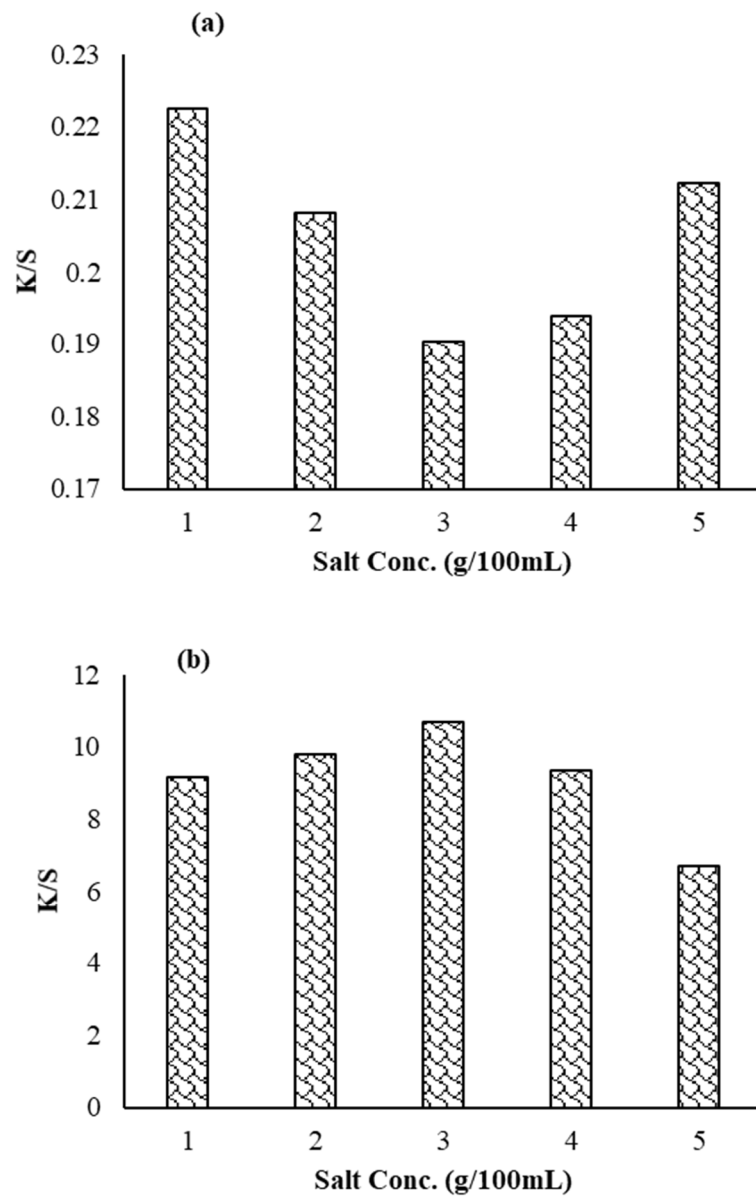


Figure 4. Effect of table salt on the color strength of dyed polyamide fabric using optimized extract of saffron (a) safflower (b).

Mordanting behavior of synthetic fabrics particularly polyamide fabrics has been observed using sustainable chemicals and biomolecules [33]. The results given in Figure 5a,b show that saffron 9% of Fe before dyeing and 7% of Fe salt after dyeing have given a high yield. For safflower extract, 5% of Fe before and after dyeing of irradiated polyamide fabric has produced a high yield. The color coordinates given in Table 2 reveal that mostly mordanted fabrics have given much brighter shades with a reddish and yellower tone, but 5% of Fe salt before (pre) the dyeing of irradiated nylon fabric using irradiated safflower extract has given a brighter shade ($L^* = 67.16$) with a reddish hue and yellower tone ($a^* = 4.20$; $b^* = 29.85$). Using Al salt for saffron, the application of 1% Al before and after dyeing has given a high yield, whereas for safflower, 1% of Al before dyeing and 3% of Al after dyeing has given a high yield (Figure 6a,b). The color coordinates given in Table 2 show that when using Al mordant, fabrics have mostly given darker shades with a less red and more yellow tone, but 1% of Al salt before (pre) the dyeing of irradiated nylon fabric using irradiated safflower extract has given a darker shade ($L^* = 57.08$) with a reddish and yellower tone ($a^* = 7.34$; $b^* = 31.81$). Overall utilization of Fe and Al for safflower has given good results.

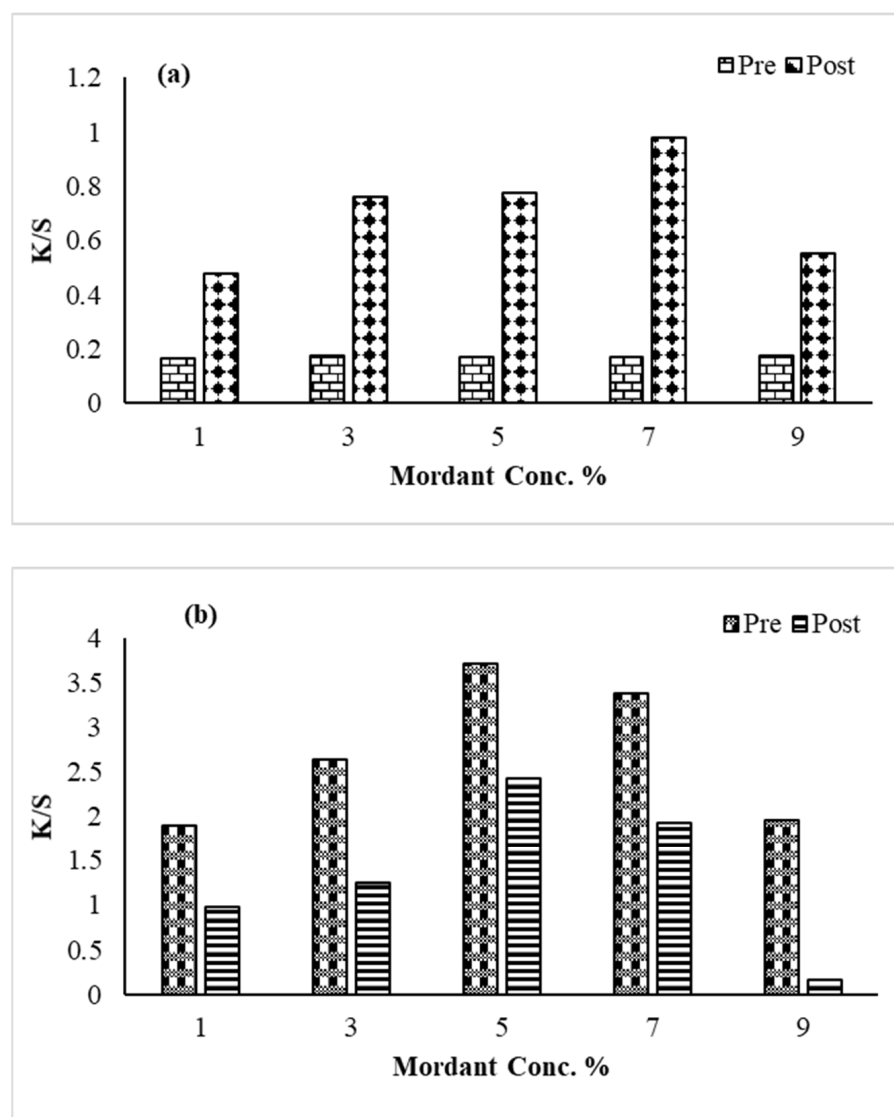


Figure 5. Effect of Fe-electrolyte on the color strength of polyamide fabric with optimum extract of saffron (a) and safflower (b) as pre- and post-mordant.

Table 2. Colorfastness rating of chemical and bio-mordanted polyamide fabric dyed using acid solubilized extract of saffron powder.

Mordants Used	L^*	a^*	b^*	Pre-Mordanting						
				Light Fastness	Washing Fastness	Rubbing Fastness		Dry Cleaning	Perspiration	
						Dry	Wet		Acid	Base
Without-mordant	80.42	2.85	45.11	3/4	3	3/4	3	3/4	3/4	3/4
Fe 9%	93.83	2.25	17.55	5	4/5	4/5	4/5	4/5	4/5	4/5
Al 1%	76.42	3.05	19.74	5	4/5	4/5	4/5	4/5	4/5	4/5
Pomegranate 7%	90.24	1.73	31.86	5	4/5	4/5	4/5	4/5	4/5	4/5
Turmeric 7%	82.70	12.34	99.12	4/5	4	4/5	4	4/5	4/5	4/5
				Post-mordanting						
Fe 7%	87.70	−1.67	44.09	5	4/5	4/5	4/5	4/5	4/5	4/5
Al 1%	94.91	−2.41	9.74	5	4/5	4/5	4/5	4/5	4/5	4/5
Pomegranate 7%	79.82	15.22	92.95	5	4/5	4/5	4/5	4/5	4/5	4/5
Turmeric 5%	82.28	1.94	39.02	4/5	4	4/5	4	4/5	4/5	4/5

L^* = lighter/darker shade, a^* = redder/greener tone and b^* = yellower/greenish huse.

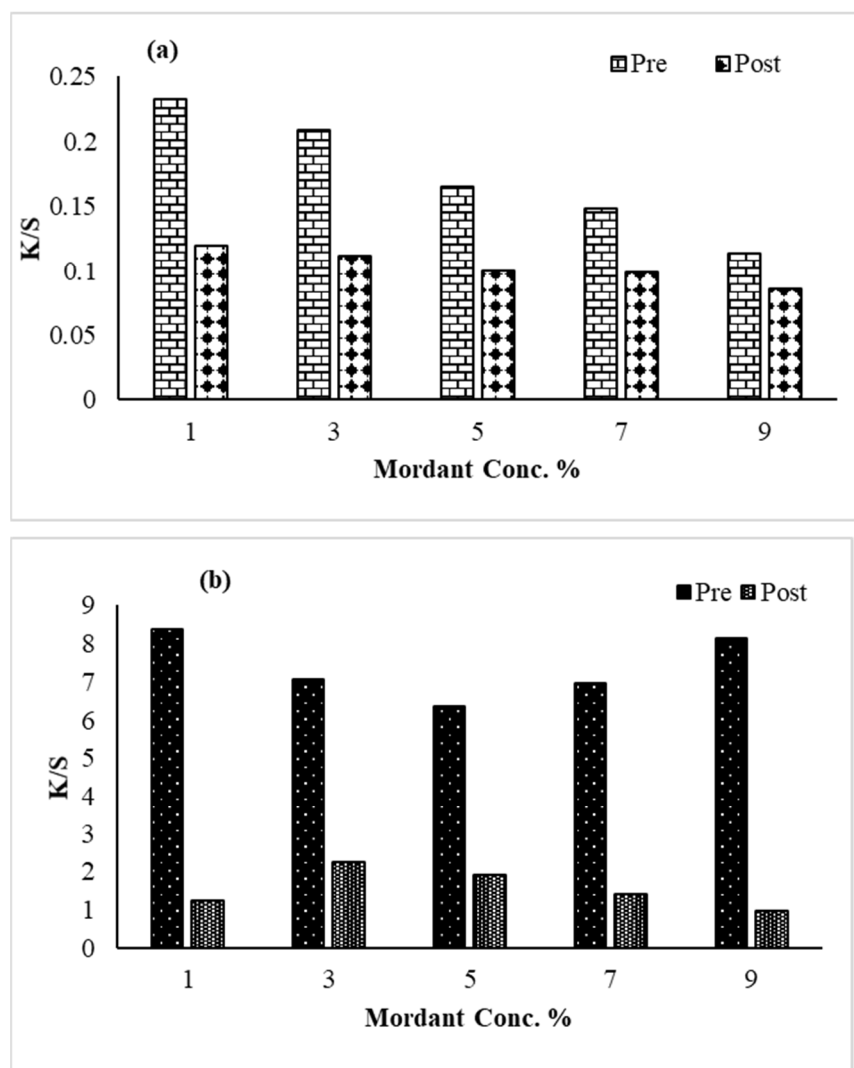


Figure 6. Effect of Al-electrolyte on the color strength of polyamide fabric with optimum extract of saffron (a) and safflower (b) as pre- and post-mordant.

In the case of biomolecules, their behavior has changed the color depth of dyed fabrics more promisingly: using 7% of pomegranate extract before and after dyeing has given a high color yield using saffron extract (Figure 7a). For safflower extract, 5% of pomegranate before dyeing and 3% of pomegranate after dyeing has given a high yield (Figure 7b). The color coordinates given in Table 3 reveal that MW treatment of fabric dyed with acidified extract before and after bio-mordanting has given brighter shades with reddish yellow hues, but using saffron extract fabric dyed before the application of 7% of pomegranate extract has given brighter shades with a more reddish and bluer tone ($L^* = 79.82$; $a^* = 15.22$; $b^* = 92.95$). Turmeric contains curcumin that furnishes more yellow colorants with an excellent yield. Using an acidic saffron extract, 7% of turmeric extract before dyeing and 5% of turmeric extract after dyeing of polyamide has produced a high yield (Figure 8a). Similarly using safflower extract, 5% turmeric extract before dyeing and 7% of turmeric extract after dyeing of irradiated polyamide fabric has given a promising yield (Figure 8b). The application of 5% of turmeric extract before dyeing (pre) has also given a darker shade ($L^* = 36.51$) with the redder but the yellower tone ($a^* = 13.42$; $b^* = 42.87$) (Table 3). It can be seen that bio-mordants have $-OH$ group as functional sites which form extra H-bonding with $-OH$ group of crocin from saffron and carthamin from safflower and $(-NH, -CO)$ amide linkage of fabric due to which firm, fast, and stable shades have been found [39]. Similarly, using the salt of Al and Fe, the metal interacts with $-OH$ of Crocin (saffron) or with $-OH$ of

carthamin (safflower) and amido linkage (NHCO) of polyamide to form a stable metal dye complex [26]. This is due to the reduction power of metal color, which is playing its role in furnishing firm and stable shades.

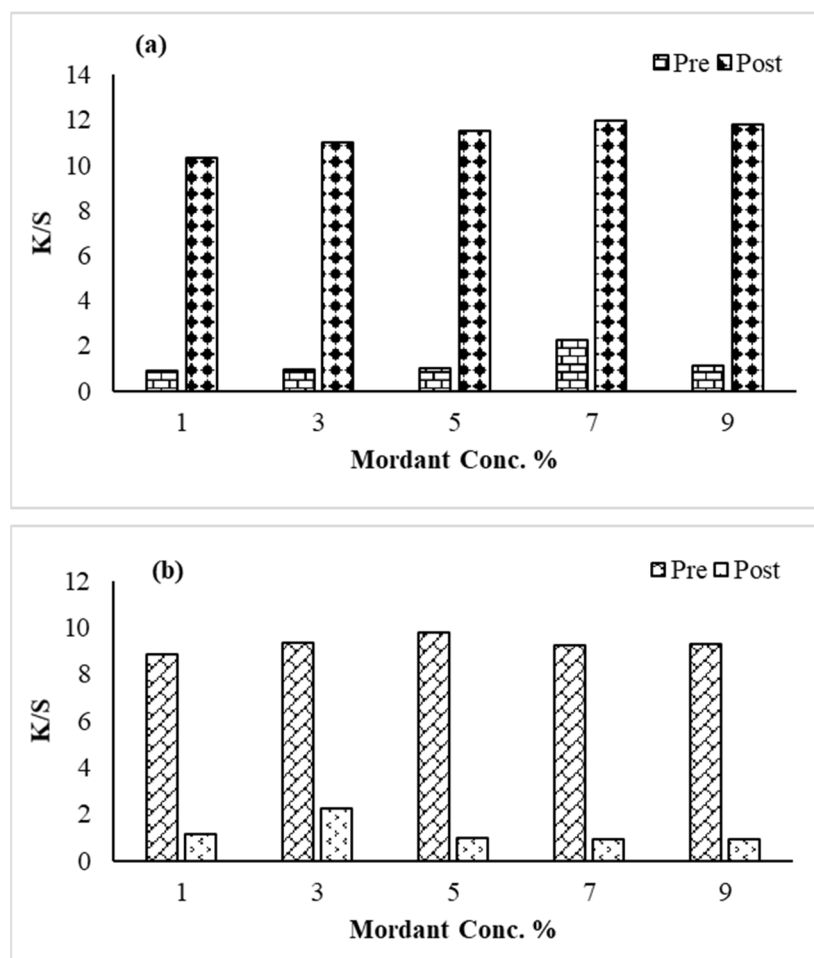


Figure 7. Effect of pomegranate on the color strength of polyamide fabric with optimum extract of saffron (a) and safflower (b) as pre- and post bio-mordant.

Table 3. Colorfastness rating of chemical and bio-mordanted polyamide fabric dyed using acid solubilized extract of safflower powder.

Mordants Used	L^*	a^*	b^*	Pre-Mordanting						
				Light Fastness	Washing Fastness	Rubbing Fastness		Dry Cleaning	Perspiration	
						Dry	Wet		Acidic	Basic
Without-mordant	52.96	14.36	46.65	3/4	3	3/4	3	3/4	3/4	3/4
Fe 5%	67.16	4.20	29.85	5	4/5	4/5	4/5	4/5	4/5	4/5
Al 1%	57.08	7.34	31.81	5	4/5	4/5	4/5	4/5	4/5	4/5
Pomegranate 5%	59.45	11.39	37.80	5	4/5	4/5	4/5	4/5	4/5	4/5
Turmeric 5%	36.51	13.42	42.87	4/5	4	4/5	4	4/5	4/5	4/5
				Post-mordanting						
Fe 5%	72.14	5.12	30.11	5	4/5	4/5	4/5	4/5	4/5	4/5
Al 3%	71.71	2.86	29.65	5	4/5	4/5	4/5	4/5	4/5	4/5
Pomegranate 3%	62.03	8.30	33.64	5	4/5	4/5	4/5	4/5	4/5	4/5
Turmeric 7%	28.94	11.88	29.34	4/5	4	4/5	4	4/5	4/5	4/5

L^* = lighter/darker shade, a^* = redder/greener tone and b^* = yellower/greenish huse.

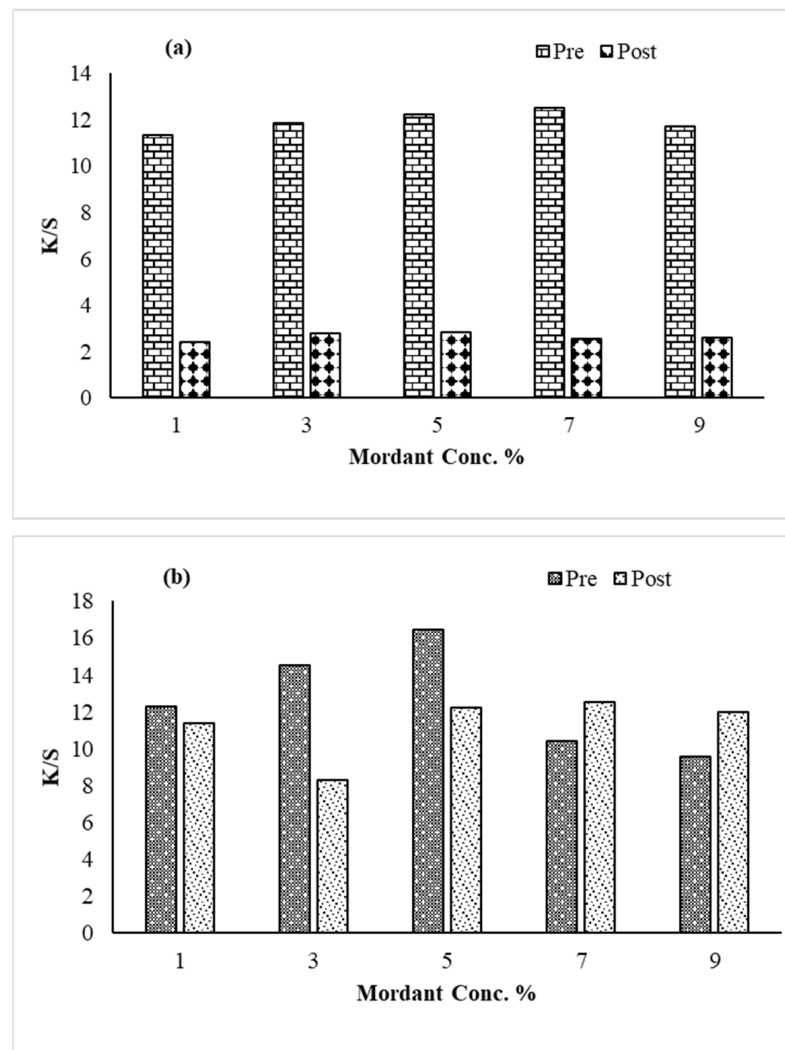


Figure 8. Effect of turmeric on the color strength of polyamide fabric with optimum extract of saffron (a) and safflower (b) as pre- and post-bio-mordanting.

The fastness properties shown in Tables 2 and 3 are obtained after the assessment of dyed fabrics showing that bio-mordants and chemical mordants have improved the ratings. During chemical mordanting before and after dyeing, depending upon the concentration of mordants, the formation of the metal dye complex at the surface of the modified polyamide fabric has formed the firm brighter or darker shade with reddish-yellow hues. The extent of shade always depends upon the nature of the metal, the concentration used, and the nature of the fabric functional active sites, whereas the presence of functional moiety in the plant material, via extra H-bonding with the functional group of the colorant and the functional site of the fabric, determines the extent of the shade and its hues [40].

4. Conclusions

The hazardous effect of synthetic dyes has forced people to utilize bio-dyes in textiles. According to the products, saffron and safflower is a great plant with a high coloring potential for textiles. The use of MW radiation as a tool for extracting crocin from the saffron and carthamin from the safflower has made the process more environmentally friendly. The application of sustainable biomolecules and chemical mordants has given colorfast shades of better quality. It is concluded that, on account of economy, time, energy, and labor effectiveness, modern tools such as microwave radiation can be used to add more value to the sustainable extraction of natural dyes.

Author Contributions: F.U.R. is the supervisor, and S.A. is the co-supervisor who made the plan of work R.B. and M.A. have conducted the experimental work, whereas W.H., B.A. and R.M. analyzed the data scientifically and technically. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Department of Applied Chemistry, Govt. College University Faisalabad 38000, Pakistan and Louisiana State University (Baton Rouge, LA, USA).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: Data are contained within the article. Additional data are available on request to the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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