Eco-friendly coloration of silk and flax fabrics with natural dye enhanced by ultraviolet radiation

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Background and objectives
Plants are the main source of natural colorant as they are widely available and can be considered as zero cost dyes, as they are obtained from plants planted for other purposes. All the parts of plants are used for extracting natural color, and most of them have antimicrobial and antifungal values. The importance using of natural dye is not restricted only to its antimicrobial or other medicine value but also to its global benefit through elimination of environmental pollution, caused by usage of synthetic dyes. The disadvantage of coloring fabric with natural dye is its fastener properties, as natural dye has no affinity to the fabric.

The objective of the present work is to produce an eco-friendly colored fabric produced through use of safe materials (no dyeing salts or mordant) with the aid of ultraviolet (UV) radiation. The coloring component from *Chelidonium majus* was extracted using boiling water, and examination was done on its coloration effect on flax and silk fabrics before and after being exposed to UV radiation for both extracted dye and fabrics.

Materials and methods
Natural dye from UV-irradiated powder of *C. majus* was extracted using boiling water. The dye was used in coloration of flax and silk fabrics through dyeing process and screen printing. The effect of irradiated treatment time, dyeing bath conditions (time and temperature) as well as fixation type of printed samples has been studied. The effect of UV radiation on the morphology structure of both fabrics was illustrated by scanning electron microscope.

Results and conclusion
UV radiation improved the color strength (K/S) and fastness properties for both colored fabrics either dyed or printed. The optimum dyeing bath conditions used for silk and flax samples were 30°C for 30 min and 90°C for 1 h, respectively. The printed silk samples fixed by thermofixation and printed flax samples fixed using steamer have given better color strength. All dyed samples have antimicrobial properties.

Keywords:
antimicrobial, *Chelidonium majus*, flax fabric, silk fabric, ultraviolet radiation

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Introduction

Since the past decade, there has been a revival in the world to get back to all natural products (dye and fabric) owing to international awareness to environment and ecology preservation. Among the nature fabrics, flax and silk have gained attention owing to their properties. Flax like cotton is a cellulose polymer but has the freshness, comfort, and elegance of linen clothing. The crystalline structure of flax makes it stronger but with poor dyeing ability owing to the low penetration of dye molecules into the fiber [1,2]. To go beyond this problem, a number of modifications by cationic agent or polymer grafting have been carried out to improve the dye uptake and overall fastness properties [3,4]. Silk is the highest priced among all natural fabrics owing to its properties such as washability and wearability; however, its dye ability and color fastness properties are weak, and they should be improved. For this purpose, surface modification of silk by some physical and chemical techniques has been suggested [5].

Chelidonium majus belongs to Papaveracecae family and is broadly distributed across the world, as it is found in Europe, Asia, Northwest Africa, and North America. *C. majus* is known under different names, and more than 20 different *Chelidonium* alkaloids have been identified. The different groups of chemical molecules that are

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present in the herb of *C. majus* are as follows: benzylisoquinoline type (0.01–1); acids such as chelidonic, malic, citric, caffeic (0.4%) ferulic (0.02%), and *p*-coumaric (0.06%); and others such as flavonoids [6].

Physical treatments such as plasma and ultraviolet (UV) radiation are dry and clean processes, which make them good alternative to wet treatment. There are several types of radiations. However, UV radiation constitutes 5% of the total incident sunlight on earth surface (visible light 50% and IR radiation 45%). Even though its proportion is quite less, it has the highest quantum energy compared with other radiations [7]. Recently researchers carried out work on the effect of UV radiation on dyeing of cotton with reactive and nature dyes. The irradiation of fibers with UV increases the wet ability of cotton which improves the color shade and fastness properties [8,9].

This research studies the effect of UV radiation on both flax and silk fabric morphologies to improve their dyeing and printing ability with aqueous extracted natural dye of *C. majus* without addition of any mordant or salts. Different conditions of UV radiation treatment of silk and flax were studied, subjected to different dyeing bath conditions (time and temperature). The irradiated fabrics were printed using irradiated dye and subject to fixation by either steaming or thermofixation.

Materials and methods

Nature coloring matter

Coloring substance used in this work was extracted from *Chelidonium* plants.

Fabrics

Silk and flax fabrics, mill scoured and bleached, were kindly supplied by Misr El-Beida Dyers Company (Kafr El-Dawar, Alexandria, Egypt).

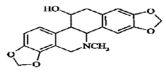
Radiation processes

UV radiation (245 nm, 180 W) was used for irradiating dye powder, silk, and flax fabrics for different time interval.

Extraction of the natural coloring matter

C. majus (Fig. 1) were crushed to the powder form, and the coloring matter was extracted using 1–5 g of the powder in 100 ml of water at the boil for 1 h. At the end, the solution was filtered off and left to cool down.

Figure 1



Chelidonium majus.

Textile coloration

Dyeing process

Fabric samples were dyed with the natural coloring matter extracted from *C. majus* at liquor ratio 1 : 40. Dyeing was carried out at pH 3–10. To observe the effect of dyeing time and temperature, the dyeing process was carried out at different period of time (20, 30, 40, 50, 60, and 70 min) and temperature (20–100°C). The effect of dye concentration was studied at 1, 2, 3, 4, and 5% of dye solution. The dyed samples were rinsed with cold water and washed for 30 min in a bath containing 3 g/l of nonionic detergent at 45°C. Finally, the fabrics were rinsed and air dried.

Textile printing

The UV-irradiated fabrics were printed by silk screen technique, and the fabrics samples were fixed by steaming and/or thermofixation.

Preparation of printing paste

The printing pastes were set by adding 3 g of the dye to 50 g of thickener suspension, and then the total weight of the whole paste were adjusted to 100 g. Thickener suspension was prepared by soaking 3 g of sodium alginate in little amount of water overnight at room temperature. All the printed samples were fixed by two different ways: thermofixation at 160°C for 10 min and steaming at 100°C for 15 min. The fixed samples were washed with cold running water, soaping (using 2 g/l nonionic detergent) at 45°C for 15 min, and at last, rinsing with cold water. After drying, the printed fabrics were assessed for color strength value (K/S) and overall fastness properties.

Analysis and measurements

Color measurements

The color strength (K/S) and Lab values was measured by reflection spectroscopy with a Hunter Lab UltraScan PRO (USA, 2007) spectrophotometer according to a standard method [10].

Tensile strength of fabric

The test was carried out according to the ASTM Standard Test method D 682 1924 on a tensile strength apparatus type FMCW 500 (VebThuringer Industries Work, Rauenstein, Germany) at 25±2°C and 60±2% relative humidity [11].

Morphology of the fabrics by scanning electron microscope

The untreated and treated fabrics were analyzed by scanning electron microscope (SEM), Topcon-Microscope (ATB-55), to investigate morphological changes of the surface structure.

Fastness testing

The dyed samples were subjected to rubbing, washing, perspiration, and light according AATCC test methods [12].

Antimicrobial activity

The microorganisms used were ATCC registered strains, except *Bacillus cereus*, which was a local isolate obtained from Agriculture Microbiology Department, National Research Centre, Egypt. The following microorganisms were included as test microorganisms: *Streptococcus pyogenes* (19 615), *Escherichia coli* (25 922), and *Aspergillus niger* (6275).

Results and discussion

Effect of ultraviole radiation time on fabric

The used fabrics (silk and flax) were exposed to UV radiation from 10 to 70 min, and dyed with the native dye powder for 30 min, 3% weight of fabric (w.o.f.) at pH 8 and liquor ratio (L.R) 1: 40. Dyeing was continued for 60 min at 100°C. Figure 2 show that irradiating fabrics exhibited better color strength (K/S) than nonirradiating fabric at all irradiation time regardless of the fabric type. However, the dyed silk samples had higher K/S, as its light weight makes its surface more affected by the UV rays. UV radiation increases the dye uptake by two ways: the oxidation process of cellulosic fabric, as the OH moieties of cellulose oxidize to carboxylic acid, which have more affinity to dye, and the photomodification of the fabric surface [13,14]. As can be seen from Fig. 2, the maximum color strength value was obtained at UV radiation for 30 min for silk and 60 min for flax.

Effect of ultraviole radiation time on dye powder

The extracted color product from *C. majus* in aqueous solution was dried and exposed to UV radiation for different time (20–80 min). The dyeing bath of UV-treated fabric (silk for 30 min and flax for 60 min) was subjected to dyeing conditions of pH 8, 3% (w. o. f.), and L.R 1 : 40. Dyeing was continued for 60 min at 100°C. The color strength (K/S) values of dyed washed samples were plotted in Fig. 3. The maximum color strength (K/S) value was obtained at 30 min of UV radiation for both silk and flax. Moreover, we can see that by increasing dye radiation time from 40 to 80 min, the color strength value decreased. UV radiation acts as a catalyst in the oxidation reaction of organic materials in the atmospheric air, forming peroxides from tannins and oils; this increases the coloring matters to some extent, but further radiation time could cause hydrolysis [15].

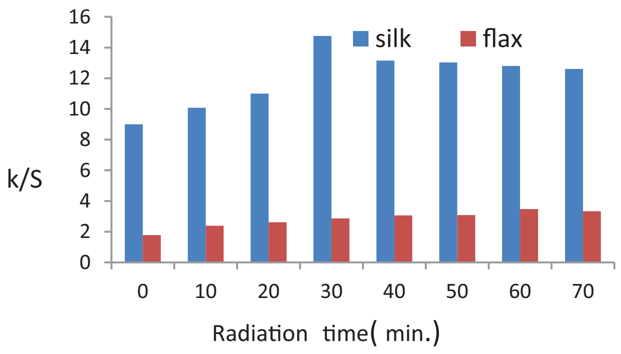
Effect of pH

To study the effect of pH on the dye uptake and color strength (K/S), the dyeing bath was set at different pH from 3 to 10 for dyeing the treated fabrics using irradiated dye powder at optimum UV radiation time. Figure 4 showed that the maximum color strength value was obtained at pH 9 which is a strong alkaline medium, whereas the lowest value can be observed at acid medium of pH 3.

Effect of different dyeing temperature

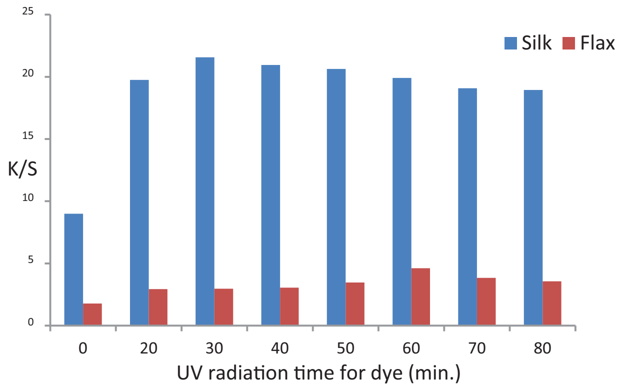
To study the effect of UV treatment of both fabric and dye powder on dyeing conditions such as temperature, the dyeing bath containing the irradiated fabric and dye powder at pH 9 was subjected to 20–100°C for 60 min.

Figure 2



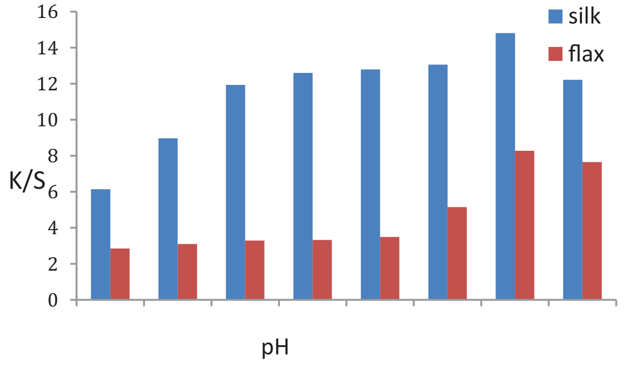
Effect of different UV radiation times of silk and flax fabric on K/S value of fabric dyeing, at pH 8, 3% (w. o. f.), 100°C, for 60 min and irradiation of the dye for 1 h. UV, ultraviolet.

Figure 3



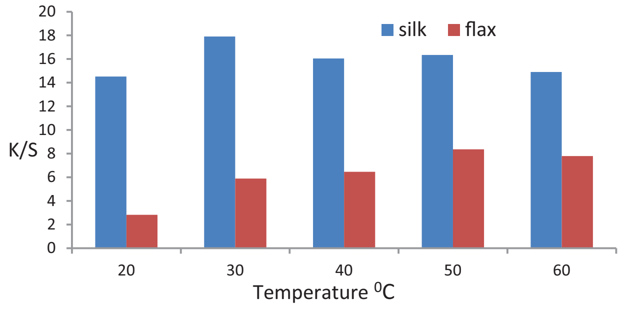
Effect of different UV radiation time of dye on K/S value of silk and flax fabrics. UV, ultraviolet.

Figure 4



Effect of different pH on K/S value of silk fabric dyeing at 100°C, 3% (w. o. f.), for 60 min, with fabric irradiation for 30 min and dye irradiation for 30 min.

Figure 5



Effect of different dyeing temperatures on K/S value of silk fabric dyeing at pH 9, 3% (w. o. f.), 100°C, for 60 min, with fabric irradiation for 30 min and dye irradiation for 30 min.

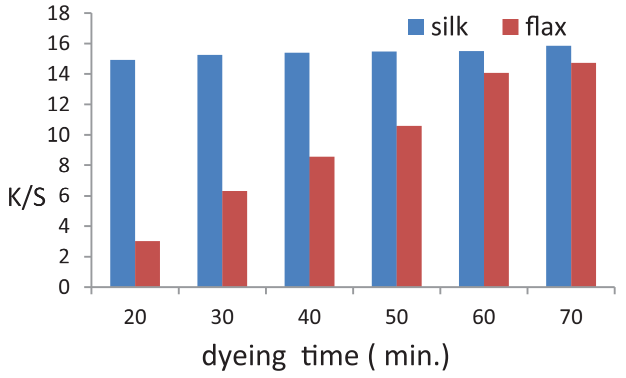
Figure 5 showed that UV radiation decreased the dyeing bath temperature for both fabrics. The optimum temperature of silk dyeing bath temperature was 30°C, which is low compared with conventional temperature, which reaches 100°C [16]. Recent research on silk dyeing using corona discharge and chitosan pretreatment stated that the dyeing temperature was 60°C, which is relatively high [17]. On the contrary, the optimum temperature of dyeing bath for flax was at 90°C, and this difference could be attributed to the nature of the fiber.

Effect of different dyeing time

The effect of dyeing time is as important as the effect of dyeing temperature, which was also studied. Treated fabrics and dye powder at optimum radiation were used at different time intervals (20–70 min) at pH 9 and at 30°C for silk and 90°C for flax fabric.

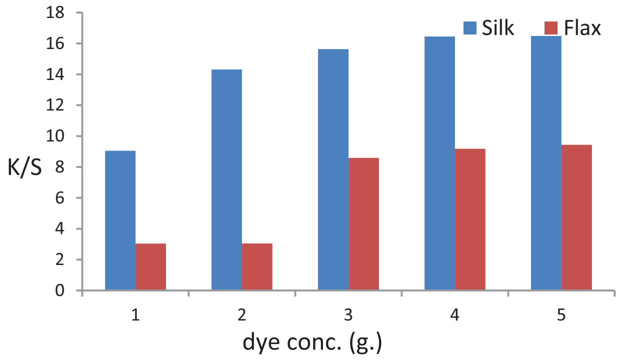
Figure 6 shows that increasing dyeing time from 20 to 70 min leads to increase in K/S value as the color gets adsorbed then absorbed on the fabric surface.

Figure 6



Effect of different dyeing time on K/S value of silk fabric dyeing at pH 9, 3% (w. o. f.), 30°C, with fabric radiation for 30 min and dye radiation for 30 min.

Figure 7



Effect of different dye concentrations on K/S value of silk fabric dyeing at conditions of pH 9, 30°C, for 20 min, with fabric IR-radiation for 30 min and dye irradiation for 30 min.

Further increase in the dyeing time after 30 and 60 min for silk and flax, respectively, has no significant effect on the color absorbance, as reflected in the K/S values. This points to an equilibrium between the dye molecule adsorb to fabric surface and that which leaves to the dye bath, and this differ according to the fabric type (henna).

Figure 6 shows that optimum dyeing time for silk and flax fabrics was obtained at 30 and 60 min, respectively.

Effect of different dye concentration

The irradiated fabrics (flax for 60 min and silk for 30 min) were dyed with irradiated dye (for 30 min) at pH 9. Dyeing was continued for 30 min at 30°C in case of silk fabric, whereas 60 min at 90°C for flax fabric. Figure 7 show that as the dye concentration increases from 1 to 4%, the color strength also increases. Above the 4% dye concentration, the color strength values nearly leveled off or a very slight improvement took place.

Scanning electron microscope study of fabric

The effect of UV radiation on the morphology structure of both silk and flax fabrics is shown in the Fig. 8. Silk fibers' backbone is fibroin, which is a highly crystalline fibrous protein, and the sericin, which constitutes ~30% in weight; however, all of sericin should be removed from the silk fibers before coloring [18]. Figure 8 shows the surface structure of untreated silk, and the presence of residual sericin covering the fiber filament is noted, and the surface is smooth with no cracks or pits. Figure 8 illustrate that after UV irradiation, the silk surface is clean, and we notice the appearance of nodes and cracks in the direction of filament axes, where the absorbance of water occurs, and that dyes and finishing agents often accumulate there [19]. Moreover, the filament compactness of the fabric increases the surface area pores allows the penetration of dye into the fiber core enhancing the color strength and rate of fastness properties. Multicellular flax fibers are connected to each other by pectin, and the outer surface of fibers consists of pectin, lignin, and waxy materials. As seen in the SEM image of untreated fibers (Fig. 9), the outer surface of the fiber filament is covered with impurities (noncellulosic materials), and there are almost no spaces present, which decreases its wettability. The photomodification by UV radiation cleaned the surface and made the fiber filaments creating more spaces where more dye could be accumulated, and as a result the interaction for dyeing flax fiber becomes more significant (Fig. 9).

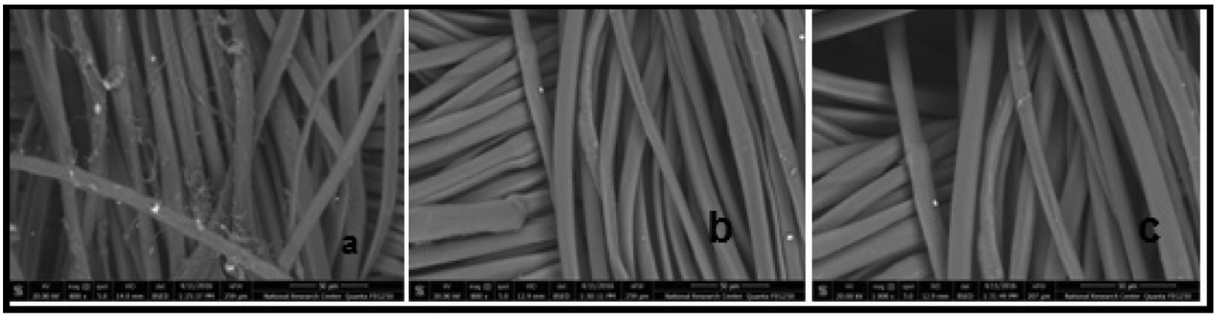
Textile printing

The irradiated printed fabric were printed with paste containing irradiated natural dye and fixed with steaming for 30 min and/or thermofixation at 150°C for 5 min. Parallel steps were applied by using untreated fabric and dye for comparison study. The fixation step is essential to the printed fabrics to have almost completely fixed dye before washing any default in this step has a dramatically effect on the samples. The type of fixation depends on the nature of fabric and dye. Table 1 shows that for the printed silk samples fixation by thermofixation is more suitable as the K/S raise from 8.19 to 12.73 for treated sample, and a value is positive point to that the sample has reddish yellow hue. As for flax printed samples, the steaming fixation of samples gave higher K/S (11.52) for irradiated samples whereas it was 5.93 by thermofixation. The steamer provides the heat and moisture necessary to swell the flax fiber and for dye diffusion to the fiber surface.

Evaluation of colored samples

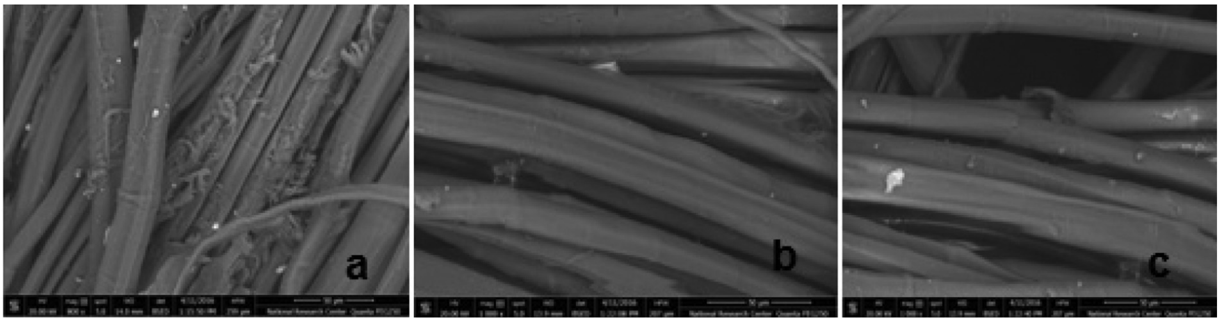
The results for color fastness to light, washing, and rubbing are given in Table 2. It is revealed that under optimum conditions of temperature, time, and pH,

Figure 8



SEM of untreated and UV-treated silk fabric. SEM, scanning electron microscope; UV, ultraviolet.

Figure 9



SEM of untreated and UV-treated flax fabric. SEM, scanning electron microscope; UV, ultraviolet.

Table 1 Effect of type of fixation on K/S of printed fabrics

Types of fixation	Sample	K/S	*l*	*a*	*b*	Δ*E*
Silk						
Thermofixation	Blank	8.19	73.01	−0.85	41.12	30.8
	Irradiated	12.73	70.69	1.11	50.43	40.31
Steaming	Blank	5.17	69.84	2.87	30.86	21.78
	Irradiated	5.64	68.49	2.53	30.24	21.87
Flax						
Thermofixation	Blank	4.52	71.14	3.26	33.12	23.49
	Irradiated	5.93	70.79	3.69	34.92	25.33
Steaming	Blank	7.73	72.53	−1.4	37.84	27.74
	Irradiated	11.52	70.08	0.27	46.89	36.98

“L” describes lightness; “a” measures redness or greenness and “b” measures yellowness or blueness.

Table 2 Fastness properties of colored silk and flax samples

Samples	Crocking		Acidic perspiration			Alkaline perspiration			Washing fastness			Light fastness
	Dry	Wet	Cotton	Wool	Alt	Cotton	Wool	Alt	Cotton	Wool	Alt	
Blank silk	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	3–4
UV-treated silk fabric	3–4	3–4	3	3	3	3	3	3	3	3	3	4
Blank flax	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	4–5
UV-treated flax fabric	4–5	4–5	4	4	4	4	4	4	4	4	4	5
Printed samples fixed with thermofixation												
Silk blank	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	3–4
UV treated silk sample	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3	3	3	4
Flax blank	4–5	4–5	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	5
UV treated flax sample	4–5	4–5	4	4	4	4	4	4	4	4	4	5
Printed samples fixed with steamer												
Silk blank	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	2–3	3–4
UV treated silk sample	3–4	3–4	3	3	3	3	3	3	3	3	3	4
Flax blank	4–5	4–5	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	3–4	5
UV treated flax sample	4–5	4–5	4	4	4	4	4	3–4	4	4	4	5

UV, ultraviolet.

irradiated flax and silk fabrics dyed with irradiated natural dye have good color fastness properties. The upgrading of fastness properties are because of the presence of benzene rings in dye molecules which show more affinity toward irradiated fabric and resistance toward factors such as detergent, heat, light, and rubbing [20]. The results shown in Table 2 regarding color fastness properties confirmed that UV irradiation had the ability to modify the surface of the fabric, which could improve the fastness properties.

Tensile strength

The UV radiation effect on the surface morphology of silk and flax fabrics, as is proved in the scan figures [17,18], becomes important through the measurement of tensile strength. The tensile strength of silk increases owing to the compactness of filament, whereas in flax, the tensile strength slightly decreases owing to filament swelling (Table 3).

Antimicrobial activity

The results presented in Table 2 show that the most potent antimicrobial effect was viewed by irradiated silk printed sample, represented by 42 mm as axial zone of inhibition against *S. pyogenes* followed by its blank, whereas for flax irradiated and blank samples, 23 and 17 mm as axial zones of inhibition against *S. pyogenes*, respectively, were seen. On the contrary, the antifungal effects against *A. niger* were showed by all the samples; the highest effect was given by the irradiated samples of either flax or silk giving 17 and 16 mm as axial zones of inhibition, respectively. The same phenomena was noticed with the antibacterial effect against *E. coli* by 18 and 17 mm as axial zones of inhibition, respectively. Latest reports on phytochemical and therapeutic perspectives of *C. majus* extract recognize two alkaloidal compounds, 8-hydroxydihydrosanguinarine and 8-hydroxydihydrochelerythrine, which possess antibacterial effect against strain of *Staphylococcus*

Table 3 Tensile strength measurement of flax and silk

Samples	Tensile strength
Flax blank	43
Flax treated	40
Silk blank	26
Silk treated	29

Table 4 Qualitative antimicrobial susceptibility tests estimated by samples

Samples	*Aspergillus niger*	*Streptococcus pyogenes*	*Escherichia coli*
Treated silk	16	42	18
Blank silk	5	30	6
Treated flax	17	23	17
Blank flax	3	17	2

aureus. Moreover, sanguinarine and chelerythrine also show effective antibacterial activity against *S. aureus*, *E. coli*, and *Aeromonas hydrophila* (Table 4) [21,22].

Conclusion

UV irradiation can be successfully applied to enhance the color strength as well as color fastness properties of colored silk and flax fabrics by improving their wettability. The samples treated at 30 and 60 min of UV-irradiated for silk and flax, respectively, have the highest wettability. The dyeing condition (temperature and time) has been effected by the UV treatment, as the silk fabric is dyed at 30°C for 30 min whereas for flax was at 90°C for 60 min in alkaline media (pH 9). All the UV-irradiated samples had antimicrobial properties.

Industrial importance: physical treatment of silk fabric by UV radiation is a very promising method, as it can improve the color strength and fastness parameters without adding any mordant or salts at low dyeing bath temperatures for a short time period (saving energy) which could be applied for other dyes.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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