Natural Dye from Red Onion Skins and Applied In Dyeing Cotton Fabrics for the Production of Women's Headwear Resistance to Ultraviolet Radiation (UVR)

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Abstract: Human exposure to ultraviolet radiation has important public health implications. Wear protective clothing represents one of the most important measures to protect against exposure to UV that are recommended by the WHO. The ultraviolet properties of textiles dyed with synthetic dyes have been widely reported in literature. However, limited studies have investigated the ultraviolet properties of natural fabrics dyed with natural colorants. The research was carried out to investigate the dying and ultraviolet protection properties of cotton fabric using an aqueous extract of red onion skin as the natural dye. Different factors affecting dyeing ability were also thoroughly investigated and applied for the production of Women's headwear resistance to ultraviolet radiation (UVR). A cotton fabric dyed in a solution containing the OSD showed a shade of yellowish white-brown. It can be observed that the K/S values increase with an increase of dye concentration. In all cases copper sulfate mordant yielded the best dyeing results, and the next good result was obtained in the order of alum and stannous chloride. Comparison of three mordanting techniques showed that the pre-mordanting gave the highest depth of shade on cotton fabric (K/S, 0.89) and UPF (33.2). Thus, pre-mordant was the best technique during mordanting method of dyeing and UVPC. The mordant activity and UPF of the three sequences was as follows: pre-mordant > Simultaneous mordant > Post-mordant > undyed in cotton fabric. It was observed that the ultraviolet protection factor (UPF) and UV absorbance values rated as very good for the cotton fabric. In addition, a darker colour, such as that provided by a copper sulfate mordant, gave better protection because of higher UV absorption. All previous data have been used in the production of women's headwear resistance to UVR. Such clothing supplements could represent a particularly important for people who are exposed to ultraviolet rays whether from natural and/or industrial sources.

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Key words: onion skin dye, dyeing property, mordanting techniques, color strength, UV protection

1. Introduction

Ultraviolet radiation (UVR) is part of the spectrum of electromagnetic radiation emitted by the sun. The UVR covers the wavelength range 100-400 nm and is arbitrarily divided into three bands of different wavelength although the exact wavelength at which the divisions are made differ for different disciplines (Diffe, 2002). The divisions first proposed by the Second International Congress on Light in 1932 were as follows: UVA 400-320nm, UVB 320-290nm and UVC 290-200 nm. UVC is totally absorbed by atmospheric ozone, has minimal penetration to the surface of the Earth and thus has little effect on human health. Ninety percent or more of UVB is absorbed by atmospheric ozone, while UVA passes through the atmosphere with little change (Roy ***et al.***, 1998). Thus, the solar ultraviolet radiation of importance to human health consists of UVA and UVB.

Human exposure to solar ultraviolet radiation has important public health implications. Many studies have been demonstrated that excessive exposure to UV radiation is associated with different types of skin cancer, sunburn, reduces the effectiveness of the immune system, accelerated skin ageing, cataract and other eye diseases (Capjack *et al.,* 1994; Frost *et al.,* 1998 and Robyn *et al.,* 2006). While UVA penetrates the human skin more deeply than UVB, action spectra for biological responses indicate that it is radiation in the UVB range that is absorbed by DNA – subsequent damage to DNA appears to be a key factor in the initiation of the carcinogenic process in skin (Horneck, 1995 and http://www.who.int/uv/uv_and_health/en/index.html). Therefore, the National Toxicology Program, U.S. Department of Health and Human Services has classified UVR as a known human carcinogen (http://ehp.niehs.nih. gov/roc/toc10.html).

Wear protective clothing represents one of the most important measures to protect against exposure to UV that are recommended by the WHO (http://www.who.int/mediacentre /factsheets/fs305/en/index.html). Many studies have proven that clothing made from natural and non-dyed fibers like wool, silk, cotton and flax with limited effectiveness in UV protection. Our previous studies with other indicated that the dyeing of these fibers by natural dyes have led to the increase of such effectiveness (Sarkar, 2004; Mongkholrattanasit *et al*., 2011; Samanta and Konar, 2011 and Hussein and Elhassaneen, 2013). Additionally, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to the worldwide environmental consciousness.

Onions (*Allium cepa* L.) are the second most important horticultural crop worldwide, after tomatoes, with current annual production around 74 million tones (http://faostat. fao.org/site/567/ DesktopDefault.aspx? PageID=567#ancor). Lately, there has been an increase in demand for processed onions which has led to an increase in waste production. Accordingly more than 450,000 tonnes of onion waste are produced annually in the European Union **(**Benítez *et al.,* 2011). The major by-products resulting from industrial peeling of onion bulbs are brown skin, the outer two fleshy leaves and the top and bottom bulbs. That onion skin is one of the most important sources of natural dye that gives yellowish to brown colorants. The major coloring components of the dye are including flavonols (86.86%), flavones (1.04%) and anthocyanidines (12.10%) which similar in part with the synthetic disperse dyes and considered very useful substances during the dying process because of their ability to fix dyes within fabrics (Hussein and Elhassaneen, 2013). Therefore, the research was carried out to investigate the dying and ultraviolet protection properties of cotton fabric using an aqueous extract of red onion skin as the natural dye. Different factors affecting dyeing ability were also thoroughly investigated.

2. Materials and Methods

2.1 Materials

2.1.1 Chemicals

Mordant's [Copper sulfat, Copper sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$; potassium alum, hydrated potassium aluminium sulfate, $KAl(SO_4)_2 \cdot 12H_2O$ and Tin(II)chloride, Tin(II) chloride dihydrate $SnCl_2.2H_2O$ were purchased from Sigma-Aldrich Chemical Co., St. Louis, Mo. All solvents and buffers, analytical grade, were obtained from Al-Ghomhoria Company for Chemicals, Drugs and Medical Instruments, Cairo, Egypt.

2.1.2 Onion skin:

The outer dry layers of red onion *(Allium cepa* L.) variety Giza-20, used in this study was obtained from a local onion exporting station, Mahallat Rooh, Tanta, El-Gharbia Governorate, Egypt.

2.1.3 Cotton fabric

Undyed cotton fabric used in this study was purchased from El-Nasr Company for Spinning and Weaving, AlMahalla El-Kobra City, El-Gharbia Governorate, Egypt. The fabric characterization parameters have been measured in our previous study (Hussein and Elhassaneen, 2013) as follow: fabric structure, plain weave 1/1; weight, 124 ± 2.11g/m^2; thickness, 0.041 ± 0.002 cm and thread count, 210 ± 0.00 per inch.

2.2 Methods

2.2.1 Dye extraction

Red onion skins were Pre-cut using special scissors (Singer, Japan), crumbled using an electrical blender (Model Mx-900, ElAraby Co. Egypt) and used as the raw material for dye extraction, which was achieved by the reflux technique as follow: 70 g of crumbled onion skin was mixed with one liter of distilled water and refluxed for 40 min. The aqueous extraction of the corresponding dye solution is double filtered in fine mesh nylon cloth and sintered glass crucible and the filtrate is evaporated using a vacuum oven (Model 200 Vacuum Prier, Poland) at lower temperature (70°C) to a semi-dried solid mass and the same is then dried under reduced pressure using a rotary evaporator (Model Rot AVC Evaporator, Czech Republic). The crude dye extract of the onion skins was then crumbled with a blender (Moulinex, French) and used for the dying process.

2.2.2 Dyeing process

Dyeing process was done in stainless steel canisters (Al-Ahram Co., Egypt) using of onion skin dye (1.5- 6%) on weight of fabric. The liquor-goods ratio was 40:1. Fabrics were introduced into the dyeing solutions at room temperature. Temperature was raised to the boil and dyeing continued at the boil for 40 minutes. For dyeing process optimization including dye concentration, mordanting techniques, type of mordant's, mordant concentration and mordanting time were tested. The liquor ratio was 1:40 and mordant concentration was 10% on weight of the fabric. After mordanting, fabric was squeezed thoroughly, rinsed in water, washed using a non-ionic detergent and air-dried. Three replications were done for each tested treatment.

2.2.3 UV absorbance, UPF and Color strength measurement

UV absorbance through a fabric is the crucial factor determining the UV protection of textiles was measured according to Hoffman *et al.,* (2001). Ultraviolet protection factor (UPF) is the scientific term used to indicate the amount of Ultraviolet (UV) protection provided to skin by fabric. It was measured *in vitro* using ultraviolet absorbance analyzer (SPECTRO UV-VIS Spectrophotometer (D2 lamp power supply), LaboMed, Inc. USA) according to standard AS/NZ 4399 (AS/NZ, 1996). Fabrics were classified with UPF values ‹ 15, 15 – 24, 25 – 39 and 40 or greater equal UV protection Class No Class, Good, Very good and Excellent, respectively. Measured UPF values were also correlated to the color strength of the dyed fabrics. Color strength was evaluated using K/S values such as mentioned in Sarkar, (2004). Higher the value of K/S greater is the color strength. All parameters were measured on triplicates and the values were presented as mean ±SD.

3. Results and Discussion

3.1 Dyeing property of cotton fabric dyed with onion skin extract by using padding techniques by varying quantity of dye concentrations

Table (1) shows the ultraviolet protection factor (UPF) and colour values of cotton fabric dyed with varying quantity onion skin dye (OSD) concentrations. It can be observed that the ultraviolet protection factor (UPF) and the *K/S* values increase with an increase of dye concentration. It is clear that the onion skin dye gives a yellowish white color which maximize in depth with the increasing of that dye concentration, yield brown color with the dye concentration 6 g/L . The results also show that UPF values for OSD applied at higher concentrations gave higher UPF values. For example, the UPF of the cotton fabric at a 1.5% onion skin dye on weight of fabric was 17.8 and that increased to UPF 36.9 at a concentration of 6%. Samples of dyed cotton fabrics with onion skin colorant could be classified as having good UV protection (UPF values between 15 and 24) to very good UV protection (UPF values between 25 and 39). It is reported that UPF values are dependent on a multitude of fabric construction factors such as pores in the fabric, thickness, and weight in addition to processing parameters such as dyeing and finishing (Sarkar and seal, 2003). Regarding the K/S values of the dyed fabrics which are a measure of color depth, it is reviewed that when the K/S value of the cotton dyed samples with onion skin colorant increased from 0.46 to 1.19 the UPF values rose from 17.8 to 36.9. In similar study, Sarker, (2004) reported that higher K/S increases UPF values. Also, Gies *et al.,* (1994) indicated that dyeing fabrics in deeper shades and darker colors improves sun protection properties.

Table (2) shows the ultraviolet protection factor (UPF) and colour values of cotton fabric dyed with onion skin dye (OSD) and different metal mordants. Cotton fabric dyed with OSD in the absence mordant showed yellowish white shades. Comparison of three metal mordants showed that the copper sulfate metal mordant gave the highest depth of shade on cotton fabric (K/S, 0.93) and UPF (34.7). Thus, copper sulfate was the best mordant during mordanting method of dyeing and UVPC . The mordant activity and UPF of the three sequences was as follows: $CuSO_4$ > $AlK(SO_4)_2$ > $SnCl_2$ > without mordanted in cotton fabric, the absorption of colour by cotton fabric was enhanced by using metal mordants.

From the results, it is clear that copper sulfate mordant is well known for its ability to form coordinate complexes and in this experiment all readily chelated with the dye. As the coordination number of copper sulfate is 4, some co-ordination sites remained unoccupied when it interacted with the fiber. Functional groups such as hydroxylic groups on the fiber can occupy these sites. Thus this metal can form a ternary complex on one site with the fiber and on the other site with the dye (reviewed in Kumbasar, 2011). Stannous chloride and alum metals formed weak coordination complexes with the dye, they tend to form quite strong bonds with the dye but not with the fiber, so they block the dye and reduce the dye interaction with the fiber (Bhattacharya and Shah, 2000).

Table (3) shows the colour values and UPF of cotton fabric dyed with OSD by varying quantity of mordant concentrations. It can be seen that the K/S values and UPF increase with an increase of mordant concentration. The dyed uptake values were greater at the higher mordant concentration. This could be attributed to the darkening and dulling of shades due to mordant effect.

Table (4) shows the ultraviolet protection factor (UPF) and colour values of cotton fabric dyed with onion skin dye (OSD) and different mordanting techniques. Cotton fabric dyed with OSD in the absence mordant showed yellowish white shades. Comparison of three mordanting techniques showed that the pre-mordanting gave the highest depth of shade on cotton fabric (K/S, 0.89) and UPF (33.2). Thus, pre-mordant was the best technique during mordanting method of dyeing and UVPC . The mordant activity and UPF of the three sequences was as follows: pre-mordant > Simultaneous mordant > Post-mordant > undyed in cotton fabric.

From the results, it is clear that copper sulfate mordant is well known for its ability to form coordinate complexes and in this experiment all readily chelated with the dye. As the coordination number of copper sulfate is 4, some co-ordination sites remained unoccupied when it interacted with the fiber. Functional groups such as hydroxylic groups on the fiber can occupy these sites. Thus this metal can form a ternary complex on one site with the fiber and on the other site with the dye (reviewed in Kumbasar, 2011). Stannous chloride and alum metals formed weak coordination complexes with the dye, they tend to form quite strong bonds with the dye but not with the fiber, so they block the dye and reduce the dye interaction with the fiber (Bhattacharya and Shah, 2000). In similar study, Mongkholrattanasit *et al.,* (2011) reported that alum and ferrous sulfate were the best mordant during simultaneous mordanting method of wool fabric dyed with eucalyptus leaf extract. However, copper sulfate showed the best mordant during simultaneous mordanting and pre-mordanting method of dyeing.

Table 1. Ultraviolet protection factor (UPF), UV protection class (UVPC) and color strength (K/S) of cotton fabric dyed with different onion skin extract dye concentrations and using 10g/L $CuSO_4$ as a mordant

Dye conc. (g/L)	UPF	UVPC	K/S	Dyed sample
Undyed	4.1 ± 0.23	No class	-------------	
1.5	17.8 ± 0.98	Good	0.46 ± 0.06	
3	20.1± 1.09	Good	0.58 ± 0.08	
4.5	33.2 ± 1.77	Very good	0.91 ± 0.05	
6	36.9 ± 2.10	Very good	1.19 ± 0.09	

Table 2. Ultraviolet protection factor (UPF), UV protection class (UVPC) and color strength (K/S) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10g/L mordant

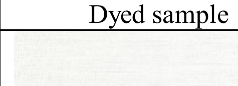
Mordant	Dye conc. (g/L)	UPF	UVPC	K/S	Dyed sample
-----	Undyed	3.67 ± 0.20	No Class	-------------	
Without	4.5	18.3 ± 0.81	Good	0.49 ± 0.06	
$SnCl_2$	4.5	30.5± 0.27	Very Good	0.83 ± 0.02	
$AlK(SO_4)_2$	4.5	34.4 ± 1.32	Very good	0.85 ± 0.07	
$CuSO_4$	4.5	34.7 ± 1.57	Very good	0.93 ± 0.11	

Table 3. Ultraviolet protection factor (UPF), UV protection class (UVPC) and color strength (K/S) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using different concentration of $CuSO_4$ mordant

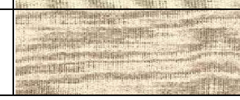
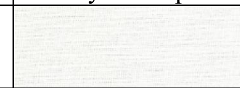
$CuSO_4$ mordant concentration (g/L)	UPF	UPFC	K/S	Dyed sample
Undyed	3.98 ± 0.21	No class	-------------	
5	29.8 ± 2.01	Very good	0.80 ± 0.09	
10	34 .3± 1.13	Very good	0.93 ± 0.10	
15	35.4 ± 0.99	Very good	0.95 ± 0.08	
20	36.1 ± 2.12	Very good	1.11 ± 0.06	

Table 4. Ultraviolet protection factor (UPF), UV protection Class (UVPC) and Color strength (K/S) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10 g/L $CuSO_4$ mordant by pre-, simultaneous and post mordanting techniques

Mordanting techniques	UPF	UVPC	K/S	Dyed sample
Undyed	3.45 ± 0.59	No Class	-------------	
Post- mordanting	29.1 ± 0.88	Very Good	0.80 ± 0.06	
Pre-mordanting	33.2± 2.31	Very Good	0.89 ± 0.12	
Simultaneous mordanting	32.0 ± 2.63	Very good	0.86 ± 0.05	

The effect of mordanting time on colour strength (*K/S)* value and UPF, was evaluated by dying a sample of cotton fabric with OSD and copper sulfate as mordant (Table 5). The samples were processed only by mordanting by using 10 g/L of $CuSO_4$ for 10, 20, 30 and 40 minutes. It is clear that the *K/S* values and UPF increase with increase in the mordanting time in cotton fabric. A study of table (5) reveals that the high colour strength values (K/S,1.03) and UPF (35.8) were achieved for the cotton fabric on mordanting for 40 minutes.

Table 5. Ultraviolet protection factor (UPF), UV protection class (UVPC) and color strength (K/S) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10 g/L $CuSO_4$ mordant for different times

Time (min)	UPF	UVPC	K/S	Dyed sample
Undyed	3.61 ± 0.22	No class	-------------	
10	32.1 ± 2.01	Very good	0.89 ± 0.06	
20	33.2± 1.85	Very good	0.93 ± 0.04	
30	33.9 ± 1.11	Very good	0.94 ± 0.11	
40	35.8 ± 2.63	Very good	1.03 ± 0.04	

3.2 Spectral analysis [UV protection properties] of cotton fabric dyed with onion skin extract

To investigate the UV-protection property of onion skin dye (OSD), UV absorption spectra of the cotton fabric with or without dyeing were compared and shown in Figure (1). The results show significantly different between the dyed and undyed fabrics, which yields a low UV absorbance. The UV absorbance of the undyed cotton was in the range of about 1.211- 1.014 in the UV-A band, 1.014-0.900 in the UV-B and about 0.900-0.938% in the UV-A band. This indicates that the resistance of undyed fabrics to ultraviolet ray was very poor while the UV absorbance of cotton fabrics dyed by OSD appeared to be higher in all UV regions. The results also show that OSD applied at higher concentrations gave higher UV absorbance values. For example, the UV absorbance of the cotton fabric at a 1.5% OSD on weight of fabric was ranged 1.327-1.157 (UV-C band), 1.157-1.086 (UV-B band) and 1.086-1.053 (UV-A band) and that increased to UV absorbance 1.602-1.389 (UV-C band), 1.389-1.339 (UV-B band) and 1.339-1.230 (UV-A) at a concentration of 6%. Generally, the UV protection property of fabrics is evaluated as good when the UV transmittance is less than 5% (absorbance, 1.301) (Feng *et al.,* 2007). The finding of this study, maximum absorption of prepared dyed cotton fabric occurs in the UVC region and UVA region, is an important requirement for those at the dangers of this kind of harmful rays. The effect of mordanting with different metals mordants on the UV protection properties of cotton fabric dyed with onion skin dye (OSD) are shown in figure (2). Cotton fabric dyed with OSD in the absence mordant showed a poor UV absorbance ranged 1.373-1.091. For the samples mordanted with $SnCl_2$, $AlK(SO_4)_2$ and $CuSO_4$, FeSO4 the UV absorbance values were in the range of 1.550-1.163, 1,561-1.169 and 1.572-1.207, respectively. It is clearly seen that the values of the spectral absorbance are increased with all of the tested mordants and different mordants had different effects on the spectral absorbance of the fabric dyed. Such data are in accordance with that obtained by Feng *et al.,* (2007). Figure (3) shows the UV protection properties of cotton fabric dyed with onion skin dye (OSD) by varying quantity of mordant concentrations. It can be seen that the UV absorbance values increase with an increase of mordant concentration. The effect of mordanting techniques on the UV protection properties of cotton fabric dyed with OSD is shown in figure (4). Comparison of three mordanting techniques showed that the pre-mordanting gave the highest UV absorbance on OSD dyed cotton fabric (1.564-1.234) followed by simultaneous mordant (1.427-1.190 and post-mordant (1.294-1.119). Figure (5) shows the UV protection properties of cotton fabric dyed with onion skin dye (OSD) by varying mordanting time. UV absorbance values are increased with increase in the mordanting time in cotton fabric.

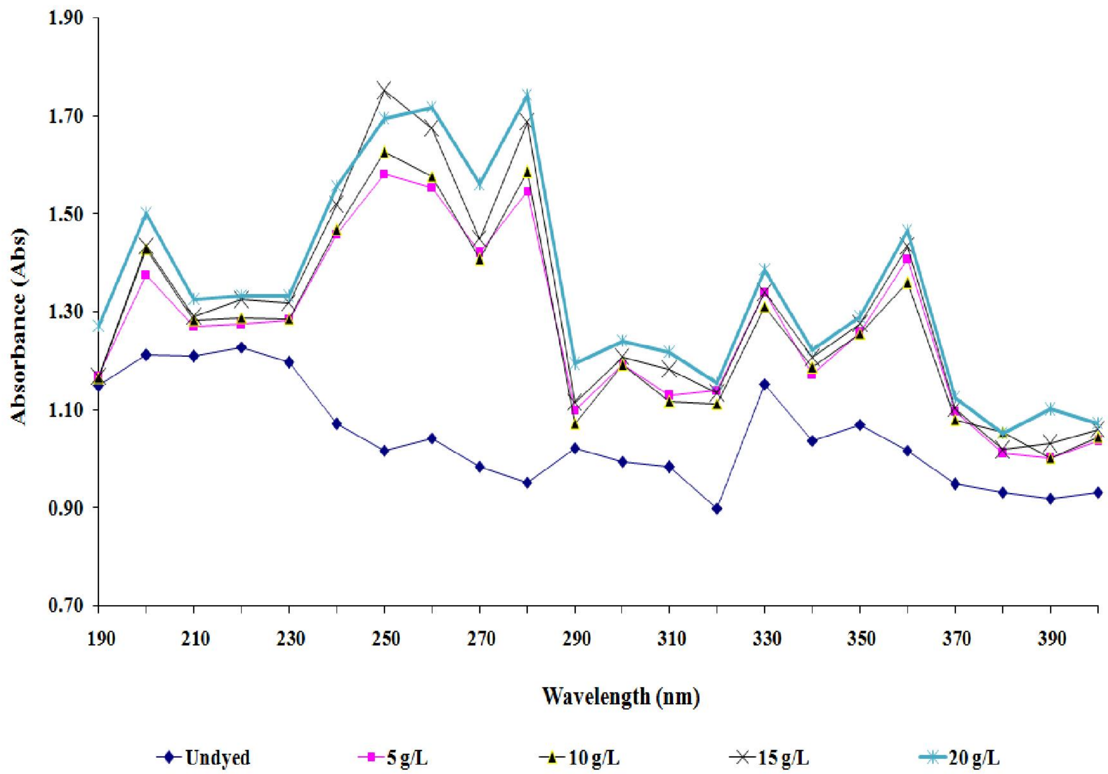


Figure 1. Ultraviolet absorption (Abs) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using different concentration of $CuSO_4$ mordant

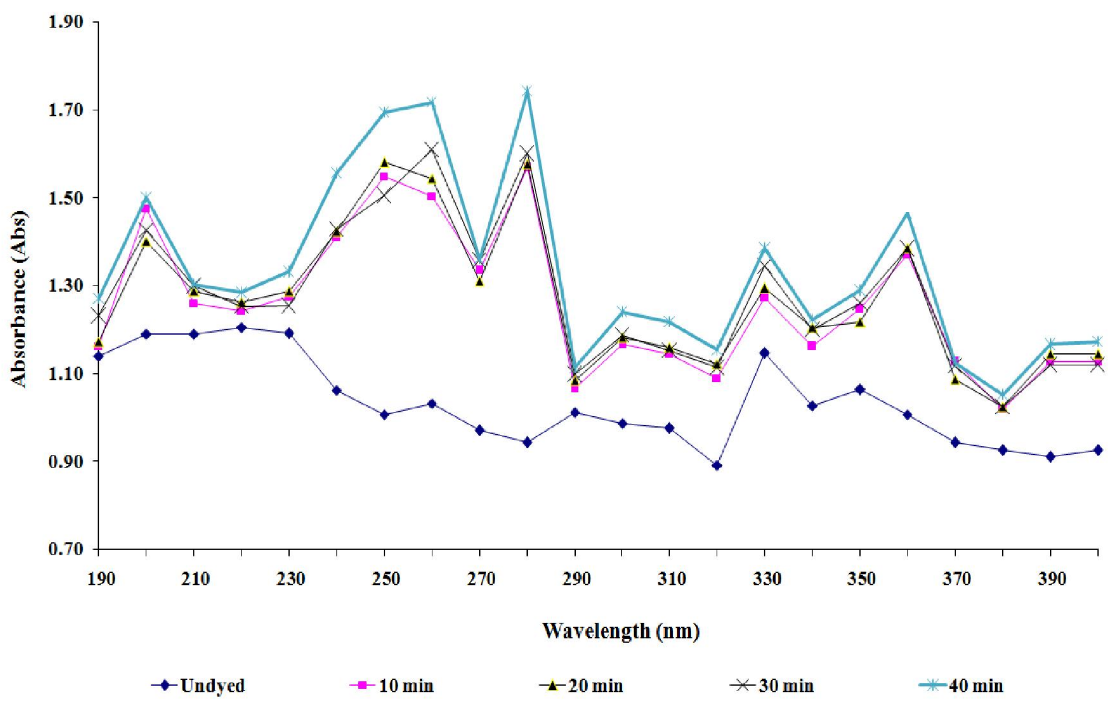


Figure 2. Ultraviolet absorption (Abs) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10 g/L CuSO4 mordant for different times

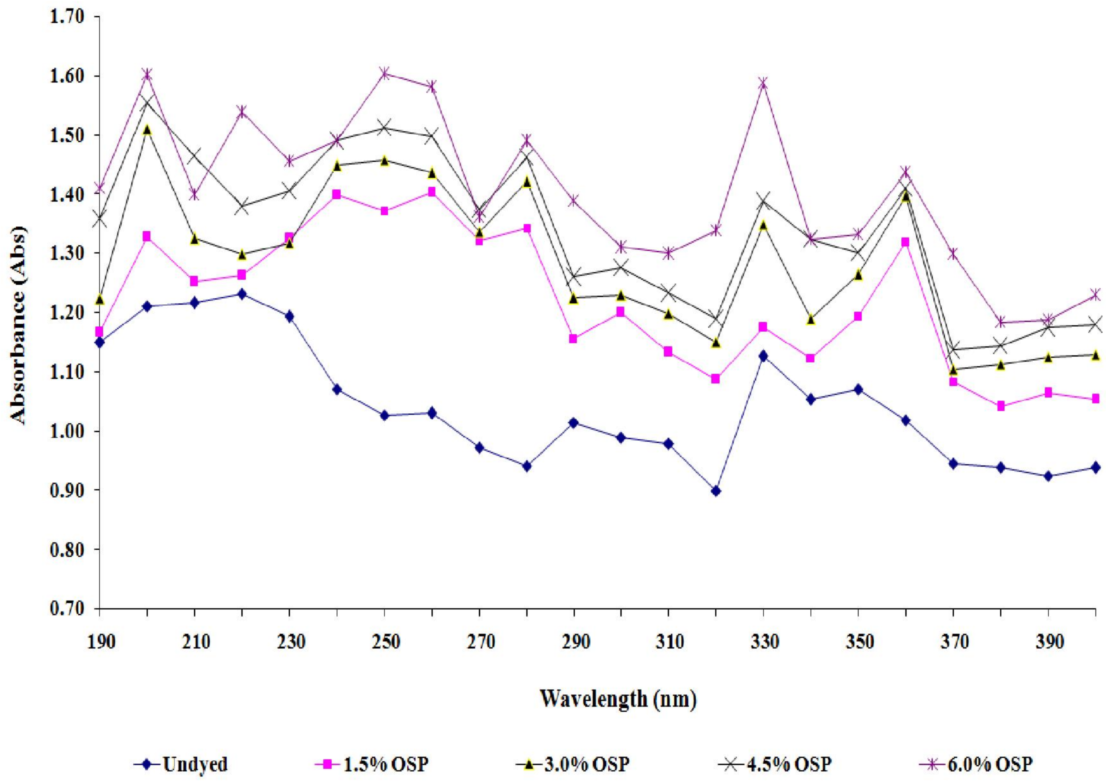


Figure 3. Ultraviolet absorption (Abs) of cotton fabric dyed with different onion skin extract dye concentrations and using 10g/L $CuSO_4$ as a mordant

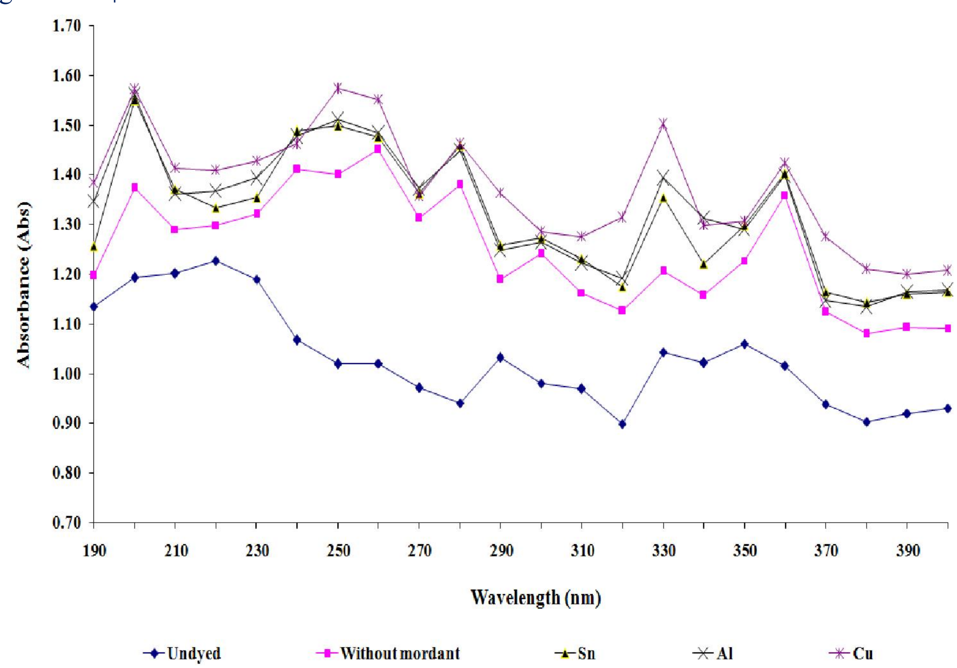


Figure (4). Ultraviolet absorption (Abs) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10g/L mordant

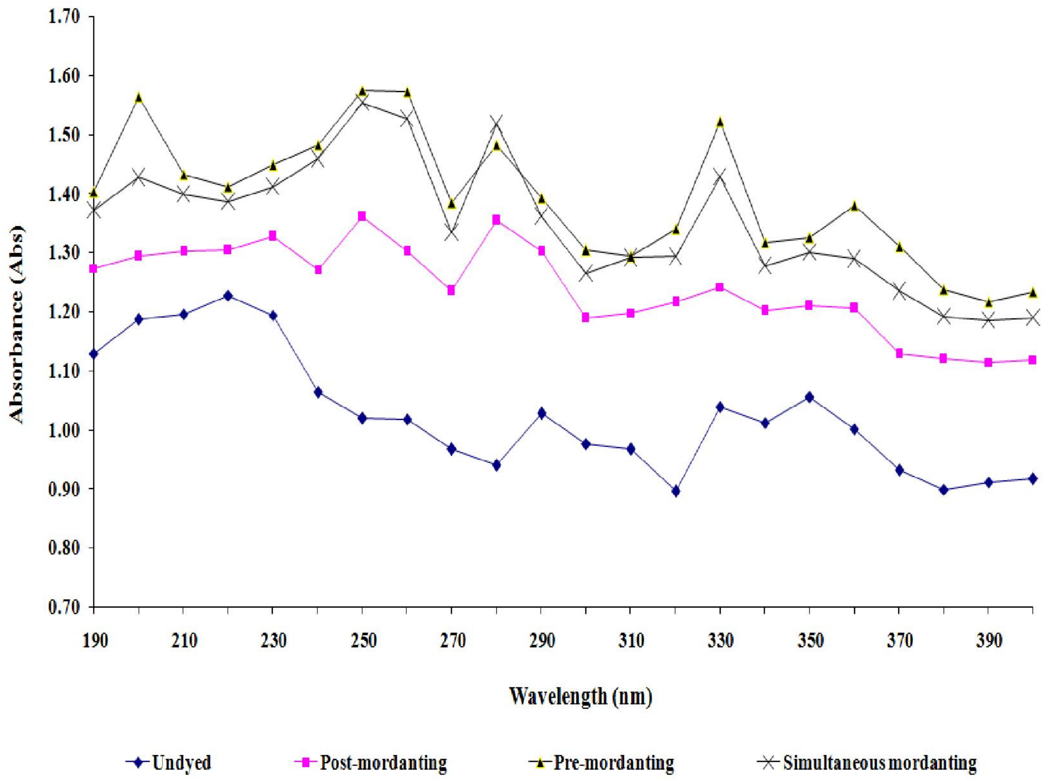
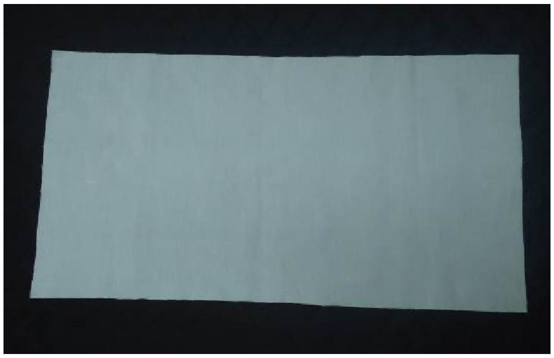


Figure 5. Ultraviolet absorption (Abs) of cotton fabric dyed with 4.5 g/L onion skin extract dye solution and using 10 g/L $CuSO_4$ mordant by pre-, simultaneous and post mordanting techniques

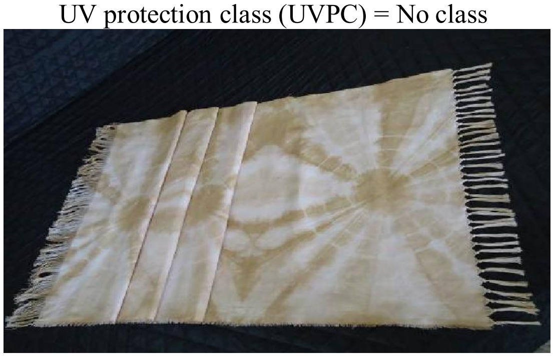
3.3 Production of women's headwear from cotton fabric dyed with OSD

Previous results achieved in the present study have been applied in producing of women's headwear resistance to ultraviolet radiation (UVR) as follow: Cotton fabrics were prepared for different shapes (shawls and scarves) by using nodding and binding style, pre- mordanting with $CuSO_4$ (10g/L) for 40 min at 100 ^{0}C, dyeing with OSD solution (4.5 g/L for 10 min at 100^{0}C, washing in tap water three times to remove the access dye, drying in natural air under the room temperature (25±5^{0}C for 12 hrs), iron (vapor machine, Japan) and finally raveling the edges manually. The final parts were wearing by twenty women (working in Clothing unit, Faculty of Home Economics, Minoufiya University) choosing by randomization process for satisfaction testing. Satisfaction rate including the color, general shape, comfortable, acceptance, appearance, functional relevance and fitness climate have a percent of 89.9, 87.6, 96.8, 88.3, 85.3, 90.4 and 97.2% respectively. Figure (1) shows the ultraviolet protection factor (UPF) of women's headwear manufactured from cotton fabric dyed with OSD. It can be observed that the ultraviolet protection factor (UPF) of the manufactured women's headwear (shawls and scarves) record 25.01-36.09 and could be classified as having very good UV protection (UPF values between 25 and 39). Like of these clothing supplements could be used successfully by the women who are exposed to solar UV radiation.



Undyed cotton fabric
Ultraviolet protection factor (UPF) = 3.57 ± 1.98
UV protection class (UVPC) = No class

Dyed cotton fabric
Ultraviolet protection factor (UPF) = 36.09 ± 2.04
UV protection class (UVPC) = Very good



Shawl No. 1
Ultraviolet protection factor (UPF) = 28.34 ± 3.15
UV protection class (UVPC) = Very good

Shawl No. 2
Ultraviolet protection factor (UPF) = 26.42 ± 4.12
UV protection class (UVPC) = Very good

Scarve No.1
Ultraviolet protection factor (UPF) = 25.01 ± 1.98
UV protection class (UVPC) = Very good

Scarve No.2
Ultraviolet protection factor (UPF) = 26.80 ± 3.57
UV protection class (UVPC) = Very good

Figure 6. Women's headwear (shawls and scarves) manufactured from cotton fabric dyed with OSD and ultraviolet radiation (UVR) protection properties.

4. Conclusions

A cotton fabric dyed in a solution containing the OSD showed a shade of yellowish white-brown. It can be observed that the *K/S* values increase with an increase of dye concentration. In all cases copper sulfate mordant yielded the best dyeing results, and the next good result was obtained in the order of alum and stannous chloride. Comparison of three mordanting techniques showed that the pre-mordanting gave the highest depth of shade on cotton fabric (K/S, 0.89) and UPF (33.2). Thus, pre-mordant was the best technique during mordanting method of dyeing and UVPC . The mordant activity and UPF of the three sequences was as follows: pre-mordant >

Simultaneous mordant > Post-mordant > undyed in cotton fabric. It was observed that the ultraviolet protection factor (UPF) and UV absorbance values rated as very good for the cotton fabric. In addition, a darker colour, such as that provided by a copper sulfate mordant, gave better protection because of higher UV absorption. So, dyeing technique properties explained in the present study can be considered as best suitable for small scale industries or cottage dyeing of red onion skins. On the other side, UV-protective properties of clothing made from cotton fabric dyed with natural colorants such OSD could represent a particularly important for people who are exposed to ultraviolet rays whether from natural and/or industrial sources.

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