Egypt. J. Chem. **Vol. 62**, No.1 pp. 119 - 130 (2019)

Dyeing Properties of Wool Fibers Dyed with Rhubarb as Natural Dye via Ultrasonic and Conventional Methods

N.F. Ali[1], E.M. El- Khatib[2], R.S.R. El- Mohamedy[3], S.H.Nassar[1], N.S.El-Shemy[1]*

[1]*Department of Dyeing, Printing, and Auxiliaries, National Research Centre, Dokki 12311, Egypt.*

[2]*Department of Proteinic and Man-made Fibres, National Research Centre, Dokki 12311, Egypt.*

[3]*Plant Pathology Department, National Research Centre, Dokki 12311, Egypt.*

THE utilization of ultrasonic waves in textile coloration processing possesses many hopeful advantages. Conventional method in dyeing processes consumes a large amount of dye and thermal energy. The dyeing processing is exclusively carried out at higher temperatures. Ultrasonic dyeing is a novel technique to save time, cost, energy and provides high value of dye uptake. The aim of this study was to investigate the dyeing behavior of pre-treated wool fibers with chitosan, then dyed with rhubarb as natural dye by conventional (Con) and ultrasonic (US) methods. Factors affecting the dye extraction and dyeing processes such as dye concentration, temperature, time as well as pH was studied. The results indicated that the pre-treated wool fibers with chitosan get higher color strength (K/S), and all over color fastness properties values if it compared with the untreated fibers. Pre-treated wool fibers can be dyed at lower temperatures in ultrasonic method as compared with conventional method. In this study the antimicrobial activity with some kinds of Bacteria and Fungi were verified. The results demonstrated that the pre-treated fibers exhibit higher inhibition percent than the untreated one. As a result, the utilization of ultrasonic waves in dyeing procedure helps to decrease the dyeing temperature, thereby reducing energy consumption and maintaining the extracted and dyeing quality.

Keywords: Rhubarb natural dye, Wool fibers, Chitosan, Ultrasonic energy, Conventional.

Introduction

Rhubarb (C.I. Natural Yellow 23) is a fellow of the family Polygonaceae, as are Japanese Indigo and Dock, and is indigenous to Asia.

Rhubarb is a recurrent plant, which has lengthy and bulky triangular leaves, these leaves can be utilized as a mordant. Rhubarb has a dense petiole and small greenish white flowers, which grows from underground rhizomes. Its roots contain two main coloring matter named gallic acid and tannins. The roots of the common eatable rhubarb as well as those of ornamental varieties produce lightfast shades of yellow and orange and the roots are an important source of dye [1, 2].

Textile manufacture is one of the main and oldest manufactures present all over the world [3, 4]. This industry doesn't require special skills that in turn support a major part in providing employment in poor countries. So, it plays a necessary role in the increase of total regional product value of these countries [5]. From ancient times, natural dyes are known for their use in food dyeing, leather; wood, as well as, dyeing and printing of natural fibers. Nowadays, the use of non-toxic and environmentally friend natural dyes to dyeing textiles has become an important source to increase environmental sensibility and to avert some severe of synthetic dyes. However, use of natural dyes for the textiles has fundamentally been limited to textual, small scale exporters and reproducers cooperation with high valued eco-friendly textile production [6-8].

There is an urgent need for novel technologies for textile industry to keep the industry competitive. Ultrasound or ultrasonic energy, promises significant rewards for the textile industry. The advancements of ultrasound improved quality as well as processing speed. Solving the environmental impact problems by

*Corresponding author: nanaelshemy@hotmail.com

DOI:10.21608/ejchem.2018.2935.1251

reducing in total use of energy, water, and hazard chemicals.

Ultrasound radiates the frequencies of 18 kHz-10 MHz beyond human hearing. This interval is divided into three categories; low (20-100 kHz), medium (300-1000 kHz) and high (2-10 MHz) frequency ultrasound. In the textile uses low frequencies are utilized. Ultrasound waves are high frequency longitudinal waves that propagate into the materials. Ultrasound waves have a significant effect on the rate of dyeing [9-11]. During ultrasound application, the ultrasound waves cause a rapid movement of the liquor in a dye bath this is due to variation of sonic pressure which subjects the liquor to compression, rarefaction and finally to micro steaming. Simultaneous formation and deflation of micro bubbles result in an increase in pressure and temperature in the dye bath at microscopic level [12, 13]. This induced heat is generally enough for dye processing and thus decreases the need for extra heating.

The main aim of the present work is to investigate the ability to use rhubarb as natural dye in dyeing of the wool fibers as eco-friendly dyeing. The use of ultrasonic waves in dyeing process is to reduces the processing time, energy consumption and improve the product quality

Experimental

Material

Mill, scoured wool fibers, which is found comprehensive use field in daily life, were used in this study. Chitosan (low molecular weight) (Aldrich) and all chemicals used in this study were of laboratory grade

Dyes

Clean, dry, ground rhubarb plant was used in this experimental as a natural dye. It was supplied from commercial market. Its properties and its chemical structure are listed in Table 1

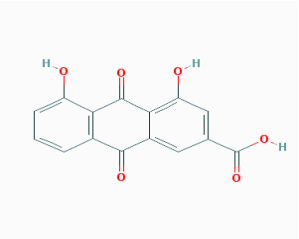
Couper sulphate and ferrous sulphate were used as mordents; sodium carbonate and glacial acetic acid were used to adjust the pH values. All other chemicals used in this study were of laboratory reagent grade.

Dye Extraction

Conventional extraction

Conventional extraction was carried out in 100 ml boiled distilled water using varying amounts of the dye materials (0.5-2.0%) for different time intervals (30-60 min) and temperature (40-100ºC). After filtration and certain dilution, the optical density of the dye liquor at λmax435 nm was measured

TABLE 1. Characteristics of Rhubarb Dye Used

Name	Latin name	Dye index	Dye component	Chemical structure
Rhubarb [16] (Dolu)	Rheum emodi	Natural Yellow 23	1-Chryosophanic Quinone (Anthraquinone) Mordant/Disperse Yellow	

Ultrasonic extraction

Ultrasonic extraction was carried out as described above in 100 ml distilled water using varying amounts of the dye materials (0.5-2.0%) at different temperatures(`40-80ºC) using different ultrasonic powers (50-80 Watt) and for different time intervals (20-60 min). After filtration and certain dilution, the optical density of the dye liquor at λmax 435 nm was measured [14].

Mordanting methods

The three methods of mordanting namely pre-mordanting (before dyeing), post-mordanting (After dyeing), and simultaneous mordanting (dyeing and mordant together in one bath) were carried out.

Pre-mordanting

In pre-mordanting method, the fibers were immersed in an aqueous solution of mordants for 30 min at 90ºC, in water bath and the other samples were immersed in mordant solution for 30min., at 80°C and 80 watt in ultrasonic. Then all samples were rinsed thoroughly with water. The mordanted fibers were then dyed at 80°C for 30 min in traditional heating while it dyed at 70°C for 30 min. at 70 watt for treated fibers and at 80°C for 40 min. and at 80 watt for untreated one.

Post mordanting

In case of post- mordanting, the dyeing was carried out in the absence of mordants at 70º-80ºC for 30 min. by using traditional heating for treated and untreated, respectively, and at 70-80°C for 30-40 min at 70-80 watt for treated and untreated yarn respectively, when using ultrasonic heating. All dyed yarn were rinsed with water, then mordanted in a separate bath at 80ºC for 30 min. at 90°C by using traditional heating and at 80°C for 30 min. at 80 watt when using ultrasonic heating.

Simultaneous mordanting

In simultaneous mordanting, the samples were immersed in a bath containing both mordant and dye solution together. The dyeing temperature was kept at 70º-80ºC for 30min.

Pretreatment with chitosan

Chitosan (high molecular weight) solution was freshly prepared by dissolving (2.0 g/l) of chitosan in distilled water containing acetic acid (4g/l) and (2.0 g/l) of chitosan in distilled water containing citric acid (4g/l) (Shin et al., 2005). The wool fibers were immersed in this solution at a liquor ratio 20:1 and treated at microwave for 5 minutes. Fibers were then squeezed and air dried.

Dyeing processes

In a dye bath containing different amounts of dye with liquor ratio 30:1, wool fibers were dyed using conventional heating at different pH values (3-9) for different durations (30-60 min.) and at different temperatures (50-90ºC). For comparison, the same condition of dyeing was made using ultrasonic dyeing (US) with different sonic power of (40-80 Watt). The dyed samples were rinsed with cold water, washed in a bath of liquor ratio 60:1 using 3 g/L non-ionic detergent (Hostapal CV, Clariant) at 50ºC for 30 min., then rinsed and finally dried at ambient temperature.

Measurements

Colour strength

An Ultra Scan PRO spectrophotometer was used to measure the reflectance of the samples and hence, the K/S was measured spectrophotometrically at wave lengths (λ max 435 nm). The K/S of untreated and pretreated wool fibers with chitosan and dyed with Rhubarb was evaluated.

Fastness Properties

The dyed samples were tested according to ISOstandard methods. The specific tests were: ISO 105-X12(1987), color fastness to rubbing; ISO 105-C02 (1989), color fastness to washing; ISO 105-E04 (1989), colorfastness to perspiration; and ISO 105-B02 (1988), colorfastness to light (carbon arc).

The antimicrobial activity

The antimicrobial activities of wool fibers dyed with Rhubarb as natural dye and pre-treated with chitosan under different conditions of dyeing such as ultrasonic and conventional methods were evaluated using serial dilution method.

Chitosan treatment wool fibres and dyed by different dyeing methods as ultrasonic and conventional under different conditions i.e., time of exposure and pH was expressed as reduction percent in total count (density) of fungi and bacteria in 1 gram of treated fibre as follows:

The serial dilution blanks were prepared in bottles containing 99 ml distillate water and marked sequentially starting from 10−1 to 10−5 dilution and autoclave sterilized. 1.0 gm of each fabric sample was added in 99 ml solution i.e. 10−1 dilution. 1 ml from this was then transferred to 9 ml of the 10−2labeled test tube i.e. 10−2 dilution, using a fresh sterile pipette; and this was repeated for each succeeding step till 10−5. Nutrient peptpneAgar media was used for counting of bacterial strains and for the counting of fungal strains potato dextrose agar (PDA) media was used. From 10−3, 10−4, and 10−5 dilution tubes, 0.1 ml of dilution fluid was then spread on sterilized petriplates in triplicates using the standard spread plate technique, for both bacterial and fungal strain isolation. The LB agar plates were then incubated at 37 °C for 24 h and the PDA plates were incubated at 27 °C for 72 h. After successful growth of microorganisms, characteristics of each distinct colony, e.g., shapes, color, transparency, etc. were determined. Gram stain was performed to observe the cellular morphology and gram reaction of the bacteria [8]. The number of bacterial and fungal colonies in the fibre samples was counted and the density was

expressed as Colony Forming Units (CFU).

Result and Discussion

Color Extraction

Dye amount

Figures 1 & 2 and Table 2 & 3reveals the effect of dye amount on K/S and CIELAB value of rhubarb by using conventional method (at 100°C) as well as ultrasonic heating (80°C)at 80% watt, for 30 min in both heating methods. Figure 1 reveals that as the dye amount increase the K/S also increases when using both types of heating with much higher K/S value at all dye amount in US case as expected. This may attribute to grinding and sonication effect on rhubarb natural dye particles. Grinding increases the specific surface area of the grinded particles due to particle size reduction [15]. Cavitation collapse sonication in solids leads to shock-wave-impacts on the surface together with interparticle collisions, which can result in particle-size reduction [16]. We can notice also that the k/s of treated wool fibers is much higher than untreated one.

The measurement of chromaticity values such as L*, a*, b* was then used to evaluate the dyed wool obtained by Con. as well as US waves. The chromaticity values shown in Tables 2 were comparable for all dyed fibers. The value of a* which represent the redness effect for dyed fabric was significantly higher at US compared to those at other Con. volume. This indicates that, the dyed fibers by using US was reddish orange whereas the rest of other dyed fibers were more towards yellowish orange.

TABLE 2. Effect of dye amount on untreated and treated wood fibers with chitosan by conventional (Con.) and ultrasonic methods (US):

Dye concentration	Con.				US			
gm/100 ml	K/S	L*	a*	b*	K/S	L*	a*	b*
Untreated								
0.5	18.97	51.00	10.23	50.2	20.00	56.08	11.63	44.14
1.0	22.34	52.21	13.2	50.21	25.12	54.15	13.83	48.75
1.5	25.1	43.67	13.61	50.2	26.00	53.02	14.72	50.73
2.0	22.3	40.23	13.21	50.0	24.99	48.77	13.80	47.65
Treated								
0.5	23.20	61.00	11.34	50.96	32.03	59.19	15.00	45.30
1.0	30,2	53.09	14.4`7	50.96	34.63	57.54	12.63	50.72
1.5	26.49	53.89	15.00	52.29	37.88	57.08	14.26	52.29
2.0	26.49	50.82	13.69	51.45	37.88	50.82	12.66	52.26

Extraction condition: pH 4.5, time 30 min., 100°C for WB and 80% watt for US at different concentration.

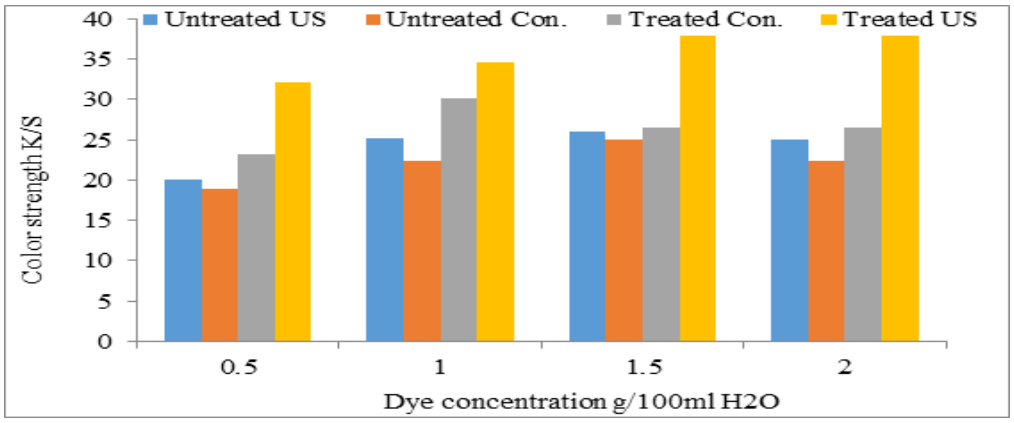


Fig. 1. Effect of extracted dye concentration on color strength of treated and untreated dyed wool fibers by using (Con.) as well as (US) methods.

Table 2 also shows that, the colorimetric data (L*, a* and b*) of different fibers dyed with different extracted dye concentration. From these data we can be concluded that increasing of the extracted dye concentration, accompanied by decreasing of L* values and thus color of samples got darker by growing the dye concentration, a* and b* values increased in the positive direction [17] The color of dyed wool fibers turned to more reddish yellow color and became darker with increasing the dye concentration from 0.5% to 2% and thereafter a* values and b* values slightly decreased. From Fig. 1 and 2 we can conclude that the best concentration for dye extraction is 1.5g/100ml, for untreated when using both of heating type, while it was 1.0 and 1.5 g/100 for treated fibers when using con and US respectively.

Effect of Extracted time

Dye extraction of rhubarb by using Con. and US were carried out for different time (10-50 min). Figure 2 reveals the effect of extracted time on dye uptake of untreated and treated wool fabric by using Con. as well as US waves. Figure 2 and Table 3 demonstrate that the dye uptake by US is higher than Con. to reveal maximum dye extraction after 40, 30 min for untreated and treated wool fibers when using Con. and US respectively. It is worth notice that the prolonged extraction time by using US may lead to dye degradation as revealed by decreasing K/S for dyed fibers.

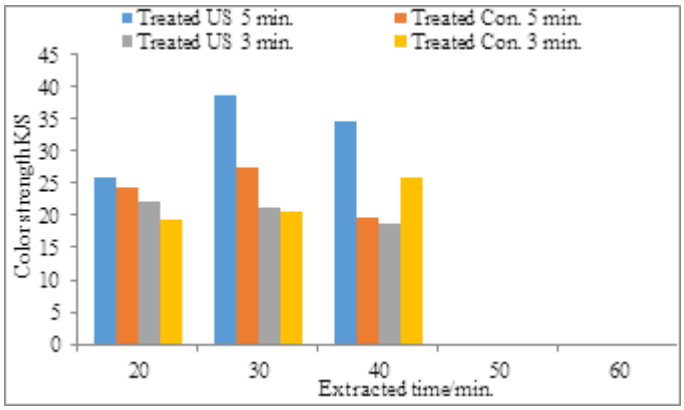


Fig.2. Plots of dye extraction versus dyeing time of Wool fibers using Con. and US waves condition.

TABLE 3. Effect of dye extraction time on K/S of untreated and treated dyed wool yarn by using Con and US wave.

Dyeing time/min.	WB				US			
	K/S	L*	a*	b*	K/S	L*	a*	b*
				Untreated				
10	18.52	50.71	11.00	54.77	25.06	52.51	11.83	53.34
20	19.55	53.58	11.78	53.25	30.13	56.97	13.12	57.77
30	33.90	52.48	14.75	56.14	52.96	54.72	12.53	58.48
40	34.25	57.67	11.75	50.88	40.26	62.46	14.87	52.54
50	26.95	48.16	12.62	49.84	33.95	54.83	8.00	52.52
				Treated				
10	23.92	68.56	8.19	57.31	28.28	68.46	12.88	61.21
20	29.67	64.01	9.26	56.55	42.11	67.11	11.89	57.94
30	36.77	61.75	9.96	54.91	60.88	62.53	9.00	54.84
40	35.73	63.62	10.03	55.50	55.27	61.34	8.78	54.01
50	29.51	61.40	9.37	53.46	45.38	62.97	6.55	52.23

Effect of extracted dye bath pH

The effect of extracted dye bath pH on the extractability of rhubarb was investigated at different pH (3-8). The color strength of dyed wool using US and Con. techniques tabulated in Table 4 and Fig. 3. From Fig. 3 we demonstrated that as the pH increases the absorption of extracted dye increases until 5, 7 and 7, 5 for untreated and treated of dyed wool fibers when using Con. and US respectively.

Effect of ultrasonic power

Ultrasonic power is the important parameter that effect on dyeing absorption, so this research involves the study of their effects on the quality of dye dispersion, change in dye solubility and its absorption by wool fibers. From Fig. 4 we can notice that the dye extraction increases with increasing the power level until 70 and 60 for untreated and treated wool respectively. This may be attributed to the effect of ultrasonic power which assists much dye extraction from origin resource. The effect of ultrasonic wave on dye extraction was suggested to have three effects: (a) Dispersion, (b) Degassing, and (c) Diffusion, i.e. accelerate the rate of dye diffusion inside the fiber and accelerating the chemical reaction between dye and fiber

Ultrasound can improve a wide assortment of chemical and physical procedures, principally by producing cavitation in fluid medium. The sonicator utilized is of 20 kHz frequency [18] which is observed to be reasonable for prompting cavitation. It is outstanding that cavitation which causes development and breakdown of

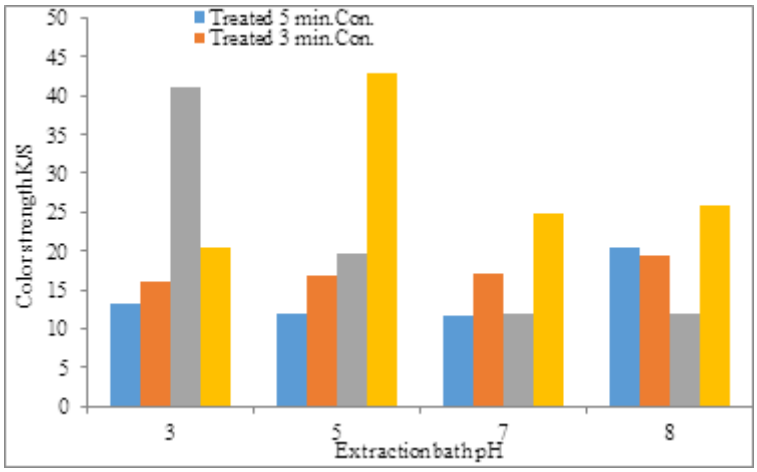


Fig. 3. Effect of extraction bath pH on color strength of dyed wool fibers by using Con. as well as US

TABLE 4. Effect of extracted bath pH on color strength of untreated and treated wool yarn when using Con and US wave.

Extracted bath pH	WB				US			
	K/S	L*	a*	b*	K/S	L*	a*	b*
				Untreated				
3	18.85	53.03	20.48	52.09	20.71	66.54	8.25	59.63
5	22.27	52.00	23.25	49.18	24.22	57.89	12.50	49.22
7	21.25	52.62	27.26	37.06	27.46	58.62	15.56	44.32
8	18.85	45.33	18.60	30.42	19.81	55.75	29.38	34.08
				Treated				
3	23.62	69.36	5.56	54.62	29.71	53.62	11.05	51.76
5	25.98	66.85	20.69	52.42	62.65	47.30	13.50	46.88
7	38.78	64.34	16.72	36.95	55.18	51.92	15.44	35.84
8	34.74	46.36	23.62	29.80	48.34	44.55	21.23	30.79

microbubbles is best effective for better color uptake. The micro bubbles which are unstable slowly develop during the process of oscillation. At the end they implode savagely, subsequently creating momentary localized high pressures and temperature. This initiated state causes compound response between the fibers and the color by forming stun waves and serious shear power equipped for breaking chemical bonds.

To compare we study the effect of extraction temperature on dye absorption from Fig. 5 we can notice that as the extraction temperature increase the dye absorption increase until 90°C. From Fig. 4 and 5, we can notice that the dye absorption is much higher when using US technique than that when using Con technique.

Mordant effect

To study the effect of mordant type on the dyeing properties, different mordanting method was studied. The mordanting method tried are (pre, post and simultaneous) involving treatment of wool fibers with metal salt such as copper and ferrous Sulphate. Different dye baths were prepared as follow:

- Untreated wool (Con.): 1.5g/100 ml, 100°C, 40 min at pH 5
- Treated wool (Con.): 1 g/100 ml, 100°C, 40

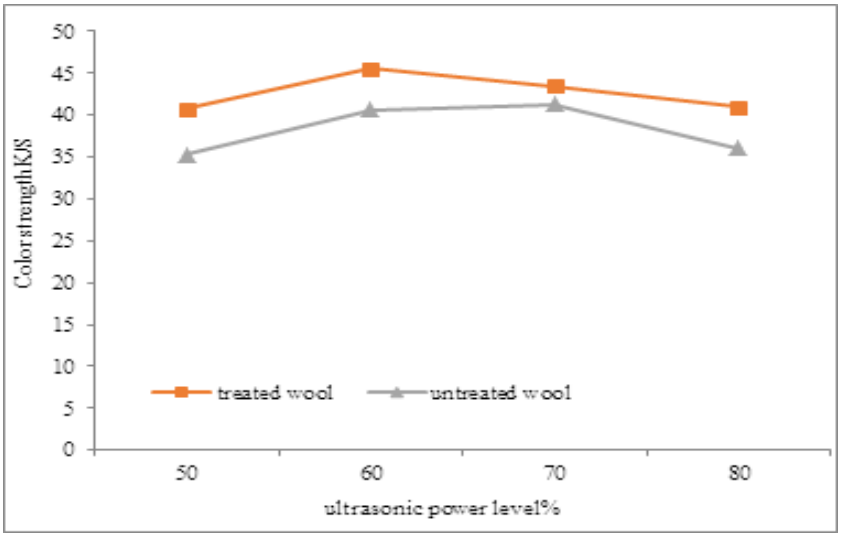


Fig. 4. Effect of ultrasonic power level on dye extraction.

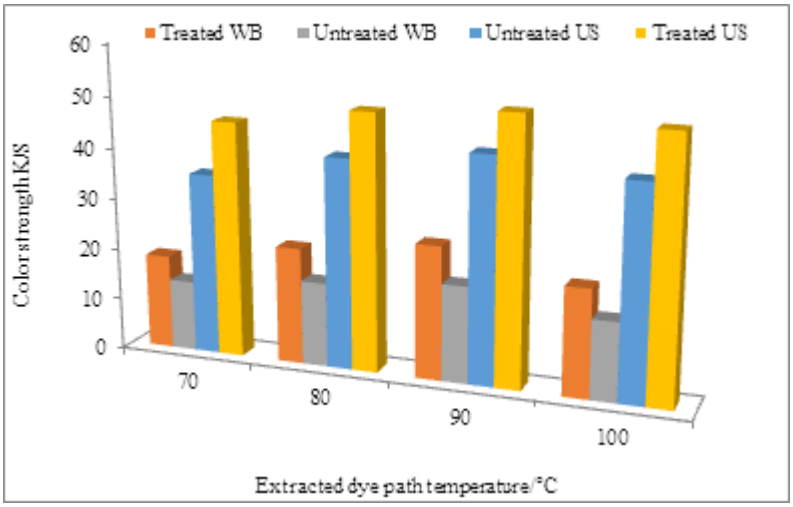


Fig. 5. Effect of extracted bath temperature on dye extraction for wool and silk fibers.

min at pH7.

- Untreated wool (US): 1g/100 ml, 70% watt, 40 min at pH 7
- Treated wool (US): 1.5 g/100 ml, 60% watt, 30 minat pH 5

Antimicrobial activities of wool fibers

In recent years antimicrobial textiles have gained interest from both academic research and industry because of their potential to provide high-quality life and safety benefits to people. Textile products are prone to host micro-organisms responsible for diseases, unpleasant odours, colour degradation and deterioration of textiles. Antimicrobial textiles can be used to produce many goods such as sportswear, outdoor apparels, undergarments, shoes, furnishings, upholstery, hospital linens, wound care wraps, towels and wipes. Self-sterilizing

TABLE 5. Effect of mordant on wool fibers dyed by simultaneous method.

Mordant type	K/S	L	A	b	C	H	ΔE
			Untreated Con.				
0	20.74	46.31	2.20	36.33	36.39	86.55	58.90
CU	21.44	57.60	13.08	53.34	54.92	76.22	79.59
Fe	37.69	43.94	13.76	32.15	34.97	66.83	56.61
			Treated Con.				
0	17.55	62.43	10.25	47.62	48.71	77.86	79.19
CU	23.88	44.70	1.74	20.95	21.02	94.75	49.39
Fe	13.00	56.57	6.22	31.43	31.95	78.77	64.96
			Untreated US				
0	27.22	51.81	16.46	51.88	54.13	73.40	74.93
CU	39.97	38.80	13.43	36.01	38.43	69.54	54.61
Fe	26.47	34.34	1.51	24.38	24.43	86.46	42.14
			Treated US				
0	24.90	54.59	11.22	46.13	47.48	76.33	72.35
CU	43.82	35.83	13.70	32.45	35.22	67.11	50.24
Fe	27.39	29.83	1.58	23.78	23.83	86.20	38.18

TABLE 6. Effect of mordant on wool fibers dyed by post; mordant method.

Mordant type	K/S	L	A	B	C	H	ΔE
			Untreated Con.				
0	16.2	50.12	15.3	47.23	33.13	70	72.4
CU	36.78	33.05	10.85	22.85	25.29	64.59	41.62
Fe	29.13	28.43	2.01	13.16	13.32	81.34	31.40
			Treated Con.				
0	20.3	44.87	12.31	45.5	30.4	66.7	55.5
CU	35.93	29.41	9.64	18.35	20.73	62.28	35.98
Fe	29.84	26.91	46.20	11.73	11.84	82.13	26.40
			Untreated US				
0	20.41	51.2	15.3	48.45	34.21	73.4	70.2
CU	25.02	43.60	10.07	31.64	33.21	72.35	54.81
Fe	24.76	40.03	2.76	24.44	14.60	83.55	46.98
			Treated US				
0	20.2	50.32	5.67	33.5	30.6	88.4	58.3
CU	19.78	48.59	1.90	29.16	29.22	86.27	56.70
Fe	22.43	48.43	8.87	32.66	33.84	74.81	59.08

TABLE 7. Effect of mordant on wool fibers dyed by pre-mordant method.

mordant type	K/S	L	A	B	C	H	ΔE
			Untreated Con.				
0	30.2	47.8	15.4	42.4	45	75.3	65
CU	34.31	46.86	11.67	39.95	41.62	73.72	62.68
Fe	29.02	41.31	7.96	37.29	38.13	77.96	00.00
			Treated Con.				
0	33.41	340.2	12.4	38.5	43	75.21	56.87
CU	32.24	39.60	11.79	37.23	39.05	72.42	55.61
Fe	28.87	40.26	5.17	31.08	31.51	80.55	51.13
			Untreated US				
0	12.4	50.4	7.3	20.23	21.33	72.34	66.4
CU	11.92	49.39	6.45	19.62	20.64	71.80	63.52
Fe	12.02	41.73	1.58	9.68	9.81	80.73	42.86
			Treated US				
0	15.52	47.21	7.34	21.23	22.37	75.41	52.36
CU	14.65	45.48	6.52	20.15	21.17	72.07	50.16
Fe	13.27	40.58	1.40	11.13	11.21	82.84	42.10

fabrics could have potential benefits to reduce disease transfers among hospital populations, bio warfare protection and other applications. Several test methods have been developed to determine the efficacy of antimicrobial textiles. The tests to evaluate the antimicrobial properties generally fall into two categories: dynamic shake test (quantitative method such as serial dilution and plate count method) and agar diffusion test (qualitative method such as halo method) [19, 20].

Antimicrobial activities of chitosan may be attributed to the chelation of metals, suppression of spore elements and binding to essential nutrients to microbial growth. Chitosan oligomers diffuse inside hyphae interfering on the the enzyme's activity responsible for the fungus growth. Chitosan molecules in bacteria surrounds might complex metals and blockage some essential nutrients to flow, contributing to cell death [21]. The positive charge on the N atom of, chitosan below pH 6.0 is more soluble and has a better antimicrobial activity by interfering with the negatively charged residues of macromolecules exposed on the fungal cell surface, and thereby changes the permeability of the plasma membrane [22-25]. It is found that the pretreatment with chitosan, effectively enhanced the antifungal activity of chitosan against many pathogens [26-29] for both wool and silk fibers as shown in Fig. 6 and 7. The effect of the antimicrobial activity depends on the time of dyeing and the pH of the dye bath.

Conclusion

Ultrasonic dyeing is a novel technique to save time, cost, energy and provides high value of dye uptake. In this study wool fibers pretreated with chitosan, then dyed with rhubarb as natural dye by conventional (Con) and ultrasonic (US) methods. Factors affecting the dye extraction, dye concentration, temperature, and time of extraction as well as pH were studied. The results indicated that fibers pretreated with chitosan exhibited higher color strength (K/S) and fastness properties than the untreated fibers. Pretreated wool fibers can be dyed at lower temperatures in ultrasonic method as compared with conventional method.

The measurement of chromaticity values such as L*, a*, b* was then used to evaluate the dyed wool fibers obtained by Con. as well as US waves. The chromaticity values were comparable for all dyed fibers. The value of a* which represent the redness effect for dyed fibers was significantly higher at US compared to those at other Con. This indicated that, the dyed fibers by using US was reddish orange whereas the rest of other dyed fibers were more towards yellowish orange.

The antimicrobial activity against bacteria and Fungi were tested. The results indicated that the fibers pretreated exhibited higher reduction percent than the untreated fibers.

Acknowledgment

This project was supported financially by the Science and Technology Development Fund (STDF), Egypt, Grant No 10390.

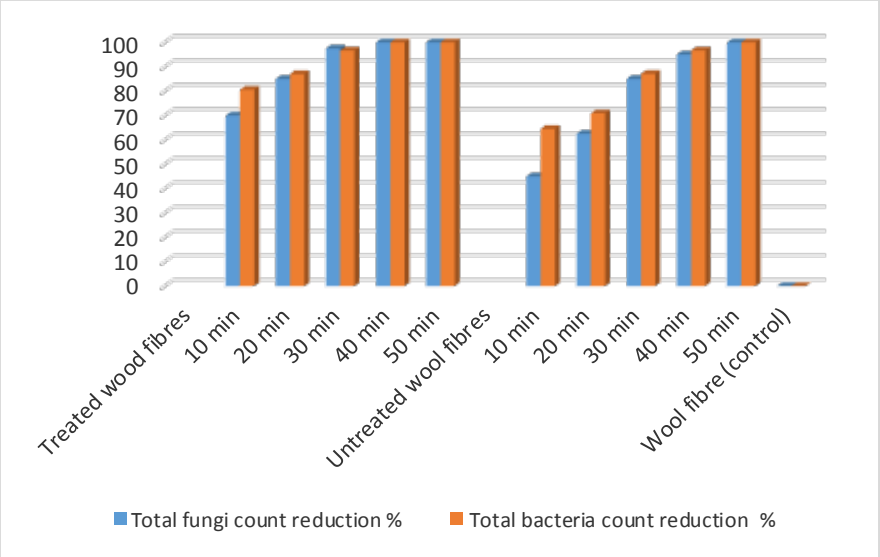


Fig. 6. Effect of time on Antimicrobial activities on wool fibres treated with chitosan and dyed with Rhubarbnatural dye expressed as reduction percentage of microbial (fungi and bacteria) total count.

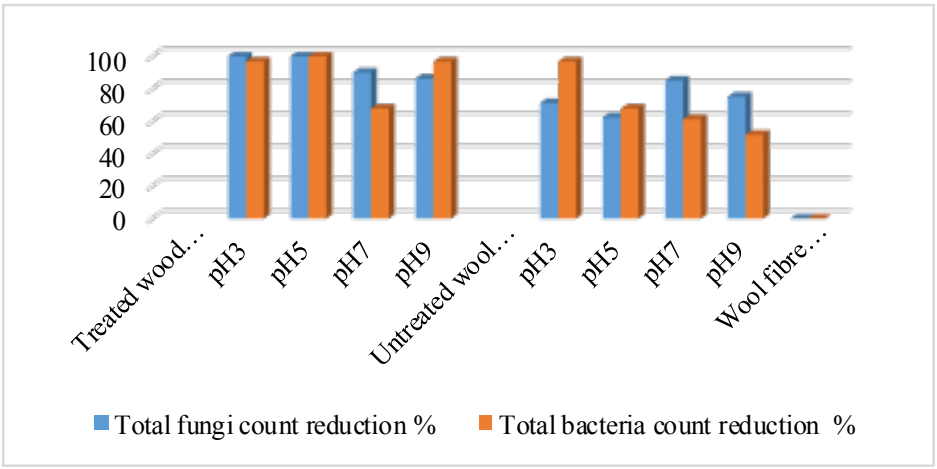


Fig. 7. Effect of pH on antimicrobial activities of wool fibres treated with chitosan and dyed with hubarbnatural dye expressed as reduction percentage of microbial (fungi and bacteria) total count.

References

1. Ali, N.F and El-Mohamedy R.S.R., Microbial decolourization of textile waste water. *Journal of Saudi Chemical Society,* **16**, 117–123 (2012).
2. Ali, N.F and El-Mohamedy R.S.R., Eco-friendly and protective natural dye from red prickly pear (Opuntia Lasiacantha Pfeiffer) plant, *Journal of Saudi Chemical Society,* **15**, 257–261(2011).
3. Gereffi, Gary. Outsourcing and changing patterns of international competition in the apparel commodity chain. In *Responding to Globalization: Societies, Groups and Individuals Conference, Boulder,* Colorado. (2002).
4. Ghaly, A. E., R. Ananthashankar, M. V. V. R. Alhattab, and Ramakrishnan V. V., Production, characterization and treatment of textile effluents: a critical review. *J Chem Eng Process Technol* **5** (1), 1-18 (2014).
5. Keane, Jodie, and Dirk Willem teVelde. *The Role of Textile and Clothing Industries in Growth and Development Strategies.* Overseas Development

Institute 7 (2008).

6. Samanta, Ashis Kumar, and Priti Agarwal. *Application of Natural Dyes on Textiles.* (2009).

7. Bechtold, Thomas, and Rita Mussak, eds. *Handbook of Natural Colorants.* John Wiley & Sons, (2009).

8. Vankar, Padma S. *Handbook on Natural Dyes for Industrial Applications.* National Institute of Industrial Re, (2007).

9. Gerald Harvey, Weidlinger Associates Ltd., Glasgow, Scotland; Anthony Gachagan, Tapiwa Mutasa, "Review of high-power ultrasound-industrial applications and measurement methods, *Browse Journals & Magazines, IEEE Transactions on Ultrasound,* **61**, 3 (2014).

10. Nagla. S Elshemy, Ahmed G. Hassabo, Zain M Mahmoud, and Karimah Haggag, Novel synthesis of Nano Emulsion Butyl Methacrylate/Acrylic acid via micro emulsion polymerization and and Ultrasonic waves, *JTATM,* **10**, 1 (2016).

11. Anasari, A.A and Thakur,B.D., Extraction, characterization of natural dye: The ecofriendly textile colorant, *Colurage*, **47**(2), 15-20 (2000).

12. Cai, Y., Sun, M., Xing, J., and Corke, H. Antioxidant phenolic constituents in roots of Rheum officinal and Rubia cordifolia: structure-radical scavenging activity relationships, *Journal of Agricultural and Food Chemistry*, **52**(26), 7884–7890 (2004).

13. Chvan, R.B., Revival of natural dyes – a word of caution to environmentalists, *Colourage*, **42**(4), 27 (1995).

14. Riad S.R. El–Mohamedy and Aboelfetoh M. Abdallah, Antifungal activity of Moreingaoleferaoleiferaoil and seed extract against some plant pathogenic fungi, *Middle East Journal of Agriculture Research*, **3**(2), 242-249 (2014b).

15. Franco, F., L.A. Perez-Maqueda and T.L. Perez-Rodriguez, The Effect of Ultrasound on Particle Size and Structural Disorder of a Well-Ordered Kaolinite, *Journal of Colloid and Interface Science,* **274**, 107-117 (2004).

16. Peters, D., Ultrasound in Materials Chemistry, *Journal of Material Chemistry,* **6**, 1605 (1996).

17. Youssef, Y.A., Magda M. Kamel, Taher, M.S., N F. Ali and Saadia A. Abd El - Megied, Synthesis and application of disazo reactive dyes derived from sulphatoethylsulphone pyrazolo[1,5-a]pyrimidine derivatives, *Journal of the Saudi Chemical Society,* **18**, 220–226 (2014).

18. Abramov, V. O., Abramov, O. V., Sommer, F., Gradov, O. M., and Smirnov, O. M. Surface hardening of metals by ultrasonically accelerated small metal balls. *Ultrasonics*, **36**(10), 1013-1019 (1998).

19. Singh R, Jain A, Panwar S, Gupta D, Khare SK, Antimicrobial activity of some natural dyes, *Dyes and Pigments,* **66**, 99-102 (2005).

20. Shin Y., Yoo, Dl. JANG, J., Molecular weight effect on antimicrobial activity of chitosan treated cotton fabrics, *J. of Applied Polymer Sci.,* **80**, 2495-2591 (2010).

21. Rejane C. Goy, Douglas de Britto, Odilio B. G. Assis, Review of the Antimicrobial Activity of Chitosan, *Polímeros: Ciênciae Tecnologia,* **19**(3), 241-247 (2009).

22. Rabea EI, Badawy MET, Stevens CV, Smagghe G, Steurbaut W., Chitosan as antimicrobial agent: Applications and mode of action. *Bio Macromolecules,* **4**, 1457-1465 (2003).

23. Avadi., M.R, Sadeghi, A.M.M. Tahzibi, A., Bayati, K. Pouladzadeh M. and Zohuriaan-Mehr, M.J., Diethyl methyl chitosan as an antimicrobial agent: Synthesis, characterization and antibacterial effects, *Eur. Polym.*, **40**, 1355-1361 (2004).

24. Rabea EI, Badawy MEI, Steurbaut W, Stevens CV., In vitro assessment of N-(benzyl) chitosan derivatives against some plant pathogenic bacteria and fungi, *Eur. Polym. J.,* **45**, 237-245 (2009).

25. Badawy MEI and Rabea EI., Characterization and antimicrobial activity of water-soluble N-(4-carboxybutyroyl) chitosan against some plant pathogenic bacteria and fungi, *Carbohydrate Polymer.* **87**, 250-256 (2012).

26. Hebeish A.A.1, N.F. Ali. and J.I. Abd El-Thalouth3, Green Strategy for Development of Antimicrobial Printed Textile Fabrics, *Research Journal of Textile and Apparel,***16**, 77-81 (2012).

27. Abdalla, Evaluation of antifungal activity of Moreinga olefera extracts as natural fungicide against some plant pathogenic fungi In-vitro, *Journal of Agricultural Technology,* **10**(4), 963-982 (2014a).

28. Rejane C. Goy, Douglas de Britto, Odilio B. G.

Assis. Review of the Antimicrobial Activity of Chitosan, *Polímeros: Ciênciae Tecnologia,* **19** (3), 241-247 (2009).

(*Received* 13/4/2018;
accepted 6/9/2018)

استخدام الطاقة بالموجات فوق الصوتية تتمتع بالعديد من المزايا خصوصاً في مجال النسيج والصباغة والتجهيز. ومن المعروف ان استخدام الطريق التقليدية في عمليات الصباغة تستهلك كمية كبيرة من الصبغة والطاقة وكذلك الوقت. حيث تتم عمليات الصباغة في درجات حرارة أعلى. اما الصباغة بالموجات فوق الصوتية فهي تعد من التقنيات الحديثة والتي تعمل علي توفير كلا من الوقت، التكلفة وكذلك الطاقة كما انها تعمل علي زيادة امتصاص الصبغة. وتهدف هذه الدراسة الي تحسين امتصاص الصباغات ألياف الصوف المعالجة مسبقا بالكيتوزان، والمصبوغه بصباغات الراوند كصبغه طبيعيه باستخدام طريقتان مختلفتان في التسخين (الطرق التقليدية، والموجات فوق الصوتية). وقد تمت دراسة العوامل المختلفة المؤثرة على استخلاص الصبغة وعمليات الصباغة مثل تركيز الصبغة، درجة الحرارة، الزمن ودرجة الاس الهيدروجيني لحمام الصباغة. هذا وقد أظهرت النتائج أن الألياف المعالجة مسبقا بالكيتوزان تؤدي إلى زيادة الشدة اللونية (K / S)، وتحسين خواص الثبات أذا ما قورنت بمثيلتها الغير معالجة. كمل أوضحت النتائج ان ألياف الصوف المعالجة مسبقا يمكن أن تمت صباغتها عند درجات حرارة أقل عتد استخدام طريقة الموجات فوق الصوتية أذا ما قورنت بمثيلتها المصبوغة بالطريقة التقليدية. كما تمت دراسة اختبار النشاط المضاد للميكروبات مع بعض أنواع البكتيريا والفطريات. وأشارت النتائج إلى أن الألياف المعالجة مسبقا تظهر نسبة تثبيط أعلى من تلك الألياف الغير المعالجة. ونتيجة لذلك، فإن استخدام الطاقة بالموجات فوق الصوتية في عملية الصباغة يساعد على تقليل درجة حرارة الصباغة، وبالتالي تقليل استهلاك الطاقة والحفاظ على جودة المنتج.