Research Article

Evaluation of the Antimicrobial Activity for Wool Fibers Dyed with New Synthesized Reactive Dyes

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ABSTRACT

The purpose of this study is to evaluate the antimicrobial of synthesized reactive dyes which were used for dyeing wool fibers with conventional method and microwave method. It is found that the dyeing of wool fibers with microwave improve the dyeability of fibers and decrease the time of dyeing. Also, color strength (K/S) values were measured for dyed wool fibers and the fastness properties including light, washing and perspiration of dyed fibers. We measure antimicrobial activity for dyed and undyed wool fibers. The results indicate that microwave technique in dyeing is more effective than tradition technique and show good fastness properties. We used synthesized monofunctional reactive dye which bearing higher exhaustion, fixation values, colour yield and fastness properties. Also, these reactive dyes have higher reduction of bacteria and fungi count.

Keywords: Reactive dyes, microwave, Dyeing, Antimicrobial, wool fibers, fastness.

INTRODUCTION

An unconventional microwave energy source has been used for heating food materials nearly 50 years.[1] but now is being used for a variety of chemical applications such as organic synthesis[2-11] where in chemical reactions are accelerated by selective absorption of microwave radiation by polar and nonpolar molecules[12]. In recent years, dyeing of some materials and modifications has been conducted under microwave irradiation condition. The advantage of microwaves, which it is use much less liquid, they can exhaust or save dyes and leave no waste of liquid dye compared to conventional methods. Microwave dyeing has other advantage such as less power consumption, easy production of desired shades; some synthetic dyes are being banned by the Western countries due to their toxic, carcinogenic and polluting nature[13-15].

It became more important for antimicrobial finished textiles to protect the fabric from bacteria [16]. The control of microorganism such as, bacteria, mildews, yeasts, molds and viruses on textile fabrics extends into diverse area as environment and hospital. Neither synthetic nor natural fibers have resistance to microorganism so, antimicrobial finished have been developed for all types of textiles[17].

We have previously synthesized some disazo and bifunctionanal reactive dyes based on pyrazolopyrazole and studied their application on cotton, wool and silk fabrics[18-19]. The aim of our work is to prepare new monofunctional reactive dyes based on thiazole as chromophoric moiety and investigate their dyeing properties on wool fibers with conventional method and microwave technique, and then we evaluate the antimicrobial activity of these dyes towardsfungi and bacteria.

MATERIALS AND METHODS

Materials

Fabric

Wool fibers (10/2 g/m^2; El Mahalla company, Egypt)

Chemicals

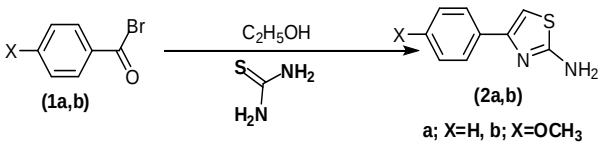
Cyanuric chloride (98 %) was brought from Merk-Schuchardt, also, thiourea, 4- aminobenzene-sulfonic acid and H-acid were obtained from FlukaChemite AG. .. All other chemicals used in our work were found in laboratory.

Synthesis

We are synthesized these dyes previously.[20]

Synthesis of intermediates

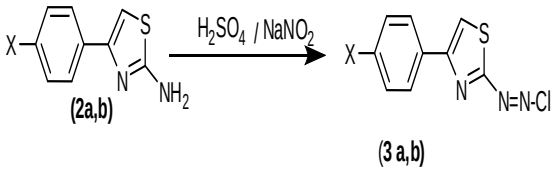
4-phenylthiazol-2-amine (2a) and 4-(4-methoxyphenyl) thiazol-2-amine (2b) A solution of 2-bromo-1-phenylethanone 1a or 2-bromo-1-(4-methoxy-phenyl) ethanone1b (1.99g, 0.01mol),(2.29 g, 0.01 mole) and thiourea (0.76 g, 0.01 mole) in ethanol (40 ml) was refluxed for 2h. After addition of pyridine (5 ml) and continued reflux for 5h., the solvent was removed under vacuum. The obtained product 2a and 2b respectively were collected and crystallized; melting points were determined with authentic samples and yellow precipitation was formed.[21] Scheme 1



Scheme 1

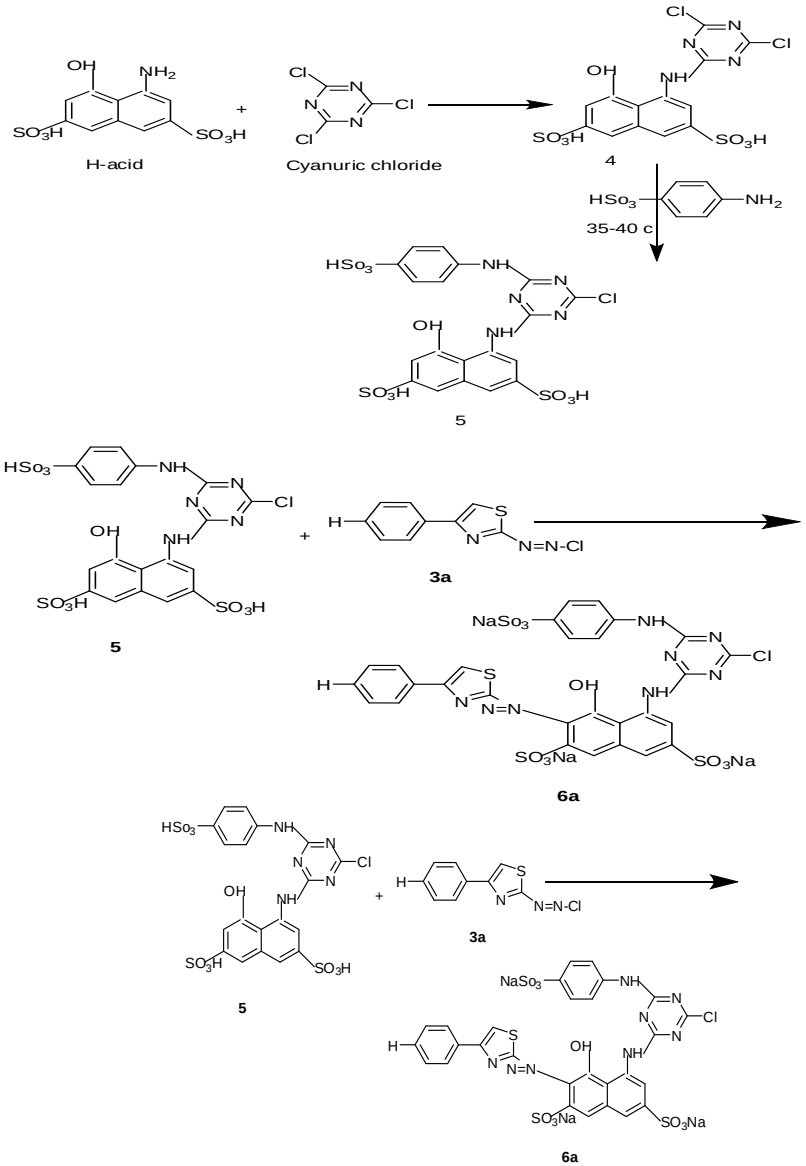
Synthesis of dyes

The synthesis of dyes were formed in two steps,the first is formed the coupling compound by a solution of cyanuric chloride(1.88g, 0.01 mol) was stirred in aceton(50 ml) containing a few amount of crushed ice at 0-5˚C in an ice /bath a solution of H-acid (3.19g, 0.01 mol) 1-amino-8-naphthol-3,6-disulphonic acid in water(30 ml) was adjusted at pH 6-7 by sodium carbonate solution was added, portion wise 4. The reaction mixture was stirred at 0-5˚C for 4 hour then a neutral solution of 4-aminobenzene-sulfonic acid(1.73g, 0.01 mol) in water was adjusted at pH 5 by sodium carbonate added drop wise to whole solution with adjusted pH of whole solution at pH 5 at room temperature **5**. The second were formed diazonium salts by the intermediate compounds 4-phenylthiazol-2-amine (2a) and 4-(4-methoxy-phenyl)thiazol-2-amine(2b)were dissolved in concentrated sulphuric acid(2.07 gm, 0.01 mol) followed by addition of sodium nitrite solution(0.70g, 0.01mol) to give the corresponding diazonium salts3a and 3b respectively. Scheme 2

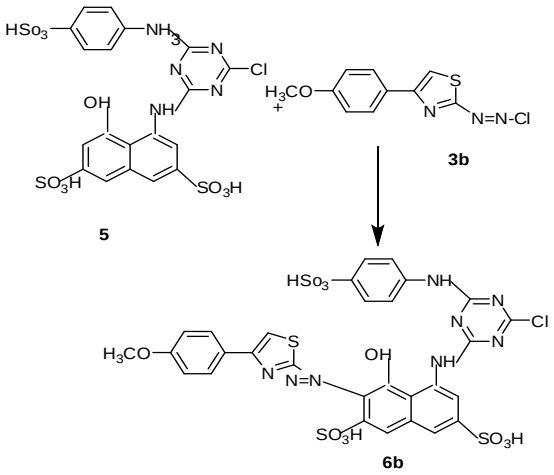


Scheme 2

Finaly, the diazonium salt solution was coupled with coupling compound which was formed in the first step by adding the coupler to the diazonium salt dropwise with stirring and adjusted pH at 5 until the pH is fixed. The resultant mono azo monfunction monchlorotriazine reactive dyes 6a, 6b were filtrated off and dried under vacuum at 50˚C Scheme 3, 4.



Scheme 3



Scheme 4

[(p-sodium sulphonato-4-phenylamino)-1,3,5-triazine-2-ylamino]-3,6-disodiumsulphonato-8-hydroxy7-napthylazo]- 4-phenylthiazol(6a)

Dye 6a: Blue, m.p.> 300˚C, yeild 89 per cent, λ_{max} (H_2O) 568 nm.

[(p-sodium sulphonato-4-phenylamino)-1,3,5-triazine-2-ylamino]-3,6-disodiumsulphonato-8-hydroxy7-napthylazo]-3-methoxy- 4-phenylthiazol(6b)

Dye 6b : Blue, m.p.> 300˚C, yeild 91 per cent, λ_{max} (H_2O) 570nm.

Dyeing procedures

Reactive dyes 6a and 6b were carried out by conventional method in an Ahiba dyeing machine at liquor ratio 50:1 and also dyeing wool fibers in microwave techniques. The microwave equipment used in this experiment was the Samsung M 245 with an output of 1,550 watts operating at 2450 MHz

Dyeing of wool

By conventional method

Reactive dyes were applied at various pH 3-7 using 5% o.w.f. ammonium sulphate and 1 g/l Albegal A. Each dyeing was performed at 50°C, allowing the temperature of the dye bath to raise to the boil over 30 min. The dyeing was continued at the boil for a further 60 min. At the end of the dyeing process, the samples were thoroughly rinsed and air-dried.

By microwave technique

The dyes were applied at different pH (3-7), 5% ammonium sulphate and 1g/l albegal for periods (1-5 minutes) in microwave.

The dyeing was carried out in distilled water, and then dyed samples were rinsed and extracted with 50 % DMF at boil for 15 minutes.

Measurements and testing

Dye exhaustion

For all dyeings, the dye exhaustion was measured by sampling the dye bath before and after dyeing. The dye concentration (g/l) of the dye bath was measured on Shimadzu UV-2401PC UV/Vis spectrophotometer at λ_{max} of the dye. The percentage of dye exhaustion (*%E*) was calculated using Eqn. (1):

%E= [1-C2/C1] x100(1)

where C_1 and C_2 are the concentrations of the dye bath be for and after dyeing.

Dye fixation

Measurement of the dye fixation was carried out by stripping the unfixed dye from the dyed material using 50 % aqueous dimethylformamide solution at the boil [22]. The stripping treatment was carried out repeatedly for 2 min periods using fresh aqueous DMF solutions until no further dye was removed. The percentage fixation (*%F*) of each dye was determined using Eqn. (2).

$$\%F=\frac{(C1-C2-C3)}{C1-C2}x100 \qquad (2)$$

where C_3 is the concentration of the extracted dye.

The total dye fixation (%T), which is the percentage of the dye chemically bound on the fabric relative to the total amount of dye used, was determined using Eqn. (3).

$$\%T=\frac{(\%\,E\;x\;\%F)}{100}(3)$$

Colour strength

The relative colour strength (K/S) and CIELAB coordinates (L* a* b*) of undyed and dyed cotton fabrics were determined using an Ultra Scan PRO spectrophotometer (Hunter Lab) with a D65 illuminant and 108 standard observer[23].

Fastness testing

The dyed samples were washed-off using 2 g/l nonionic detergent at 80°C for 30 min, and tested according to ISO standard methods (Methods of tests for colour fastness of textiles and leather, 5 th ed. Bradford: SDC, 1990). The specific tests were ISO 105-X12 (1987), ISO 105-C02 (1989), ISO 105-E04 (1989), and ISO 105-B02 (1988) corresponding to colour fastness to rubbing, washing, perspiration and light, respectively.

Antimicrobial activity

The method developed by Low and Webely (1959)[24] was used to study the microbial flora associated with textile fibers. The treated and untreated fibers (1 gm) were transferred to wide-mouth reagent bottles container 99 ml sterile distilled water in each. The bottles were shaken on a mecha mil shaker for 30 min. Serial dilution from 10^{-3} to 10^{-6} were made up by using tert tubes containing 9 ml DSW in each as a diligent. The plate count techniques according to Alen (1961)[25] was followed for determination of the total count of fungi and bacteria as follows: Total count of fungi: martins medium.

Allen was used for counting the total fungal count of at dilution of 10^{-3} to 10^{-4} . Plates were incubated at 27 ^{0}C. Fungal colonies were counted after 6-8 days in each treatment. The antifungal activities was expressed as the reduction of total count using the formula AFA = A-B / A X 100 [24].

Where AFA = antifungal activity

A=Total fungal count in control

B= Total fungal count in treatment

Total bacterial count

For the total bacterial count, 1 ml from eachat dilution of 10^{-5} to 10^{-6}

Was plated on Nutrient agar medium and incubated at 28-30 ^{0}C bacterial colonies were counted after 3-4 days.

The antibacterial activity was expressed as the reduction of of total count of bacteria using the formula ABA = A-B / B X 100

ABA =antibacterial activity.

1. Total bacteria count in control
2. Total bacterial count in treatment

RESULTS AND DISCUSSION

Figure 1, 2 indicated that the exhaustion and total fixation of wool at different pH (3-7) for dyes **1, 2** with conventional and microwave techniques respectively. The results prove that at low dyebath (pH3) the substantivity of the dyes is highdue to the electrostatic interaction existed between the protonated amino groups on the and anionic sites in the dyes. But at higher pH values (pH 4-5) the covalent bonding increase between nucleophilic substitution and b-attack mechanisms. In addition the exhaustion and fixation of wool with microwave technique is higher than conventional method. Also from results we observed that exhaustion and fixation of dye 1 is higher than dye 2 due to two factors, first, molecular weight of 1 is lower than 2 thus increase substantively to fiber. Second, the presence of aggregation of dye 2increases than dye 1 which decrease the solubility of dyes and thus decrease the exhaustion of dye 2than 1.

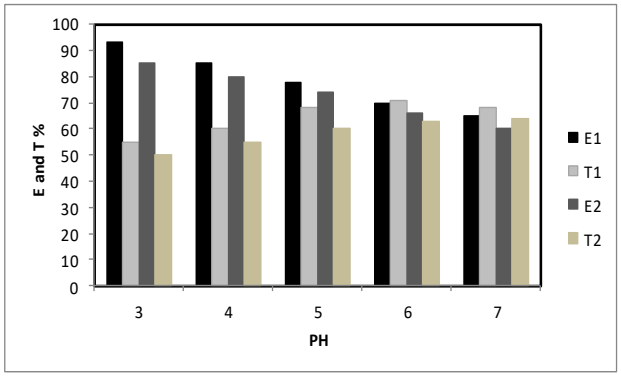


Figure 1: the exhaustion and total fixation 6a, 6b of wool at different pH (3-7) in conventional method.

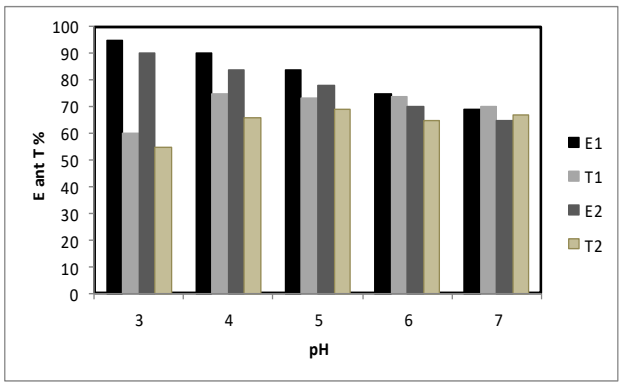


Figure 2: the exhaustion and total fixation 6a, 6b of wool at different pH (3-7) in microwave.

Figure 3 shows the relation between K/S and dyeing time for wool fibers dyed with microwave technique. The result prove that the highest value of K/S of wool recorded after 3 minutes due to using microwave which saving time .

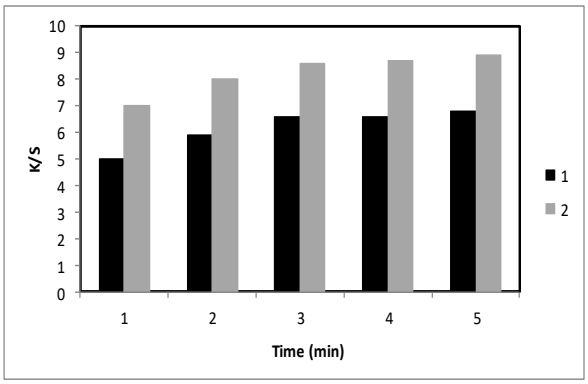


Figure 3: shows the relation between K/S and dyeing time of wool for dye 6a and 6b in microwave

Colour strength and fastness properties

Fastness properties and the colour yield of the reactive dyes**1,** 2 onto wool were evaluated. Table 1 indicate that colour fastness to rubbing, washing and perspiration of all dyes are excellent to good and are approximately the same; in microwave and in conventional method; because the dye was fixed.

Table 1: fastness properties of reactive dyes 6a, 6b on wool fiber

Dye No.	Fabric*	Fastness to rubbing		Washfastness**			Fastness to perspiration**						Light
							Alkaline			Acidic			
		Dry	Wet	Alt	SC	SW	*Alt*	*SC*	*SW*	*Alt*	*SC*	*SW*	
6a	W	4-5 4-5	4 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5
6b	W	4-5 4-5	4 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5	5

* C, Cotton; W, wool; S, silk; ** Alt = alteration; SC = staining on cotton; SW = staining on wool

Also the light fastness for all dyed fibers was found to be approximately the same for the two methods. In addition, the colourimetric CIE L*a *b*C*h data were measured for dyed wool fibers and were shown in table 2, 3.

Table 2: Colorimetric data of the dyed wool fiber using dyes **6a** and **6b** in conventional method

Dye	K/S	L*	a*	b*	h	▲E
6a	6.01	35.54	11,66	-15,60	306.77	56.06
6b	8	32	14.09	-17.76	308.57	60.1

Table 3: Colorimetric data of the dyed wool fiber using dyes 6a and 6b in microwave

Dye	K/S	L*	a*	b*	h	▲E
6a	6.56	36.92	13.10	-23.24	299.40	59.34
6b	8.6	32.63	10.13	-22	295.99	61.58

Antimicrobial activities of the reactive dyes under investigation

Table 4shows the reduction % of bacteria and fungi account for the dyed and undyed fibers. The results showed that, the dyed fibers have higher reduction % of bacteria and fungi than undyed fibers

Table 4: Show total reduction % of fungi and bacteria toward reactive dyes 6a, 6b

Sample	Total fungal count	Reduction%	Total bacterial count	Reduction%
1	$4.2x\ 10^3$	32.2	$2.4x\ 10^5$	36.8
2	$5.2x\ 10^3$	16.1	$3.0x\ 10^5$	21.0
3	$4.0x\ 10^3$	35.4	$2.2x\ 10^5$	42.1
4	$5.4x\ 10^3$	12.9	$3.2x\ 10^5$	15.7
Control	$6.2x\ 10^3$	0.0	$3.8x\ 10^5$	

CONCLUSION

We successfully prepare monofunctional reactive dyes previously based on thiazole moiety have antimicrobial activity toward bacteria and fungi. In addition, we dye wool fibers with conventional method and microwave techniques. The results of dyeing indicate that dyeing with microwave method exhibited higher results than conventional method. Microwave decrease dyeing time and safely to environment .The results of dyeing with microwave prove that the exhaustion and fixation values, colour yield and fastness properties are higher than with conventional methods. Also, we measure the efficiency of the dyed wool fibers as antimicrobial activity towards bacteria and fungi and it was found that, the dyed fibers have higher reduction % of bacteria and fungi than undyed fiber.

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