

Antibacterial activity and dyeing characteristics of some azo-pyrazole disperse dyes using eco-friendly ultrasound energy for PET fabric.

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Abstract— The objective of this research was to use ultrasound as ecofriendly technique to enhance dyeing performance and antibacterial behaviors of PET dyed with some Azopyrazole disperse dyes. The use of ultrasound imparted greater color strength and higher antibacterial activity. The color strength expressed as k/s of the dyed samples was evaluated in addition to CIE lab coordinates (L*, a* and b*) were also measured. Washing, rubbing and light Fastness properties were evaluated for both conventional and ultrasound dyeing techniques. Scanning electron microscope was used to investigate the morphological characteristics of the dyed samples.

Index Terms —Pyrazoles, Azo disperse dye, Ultrasound, Polyester fabric, Antibacterial activity, Color, SEM, ecofriendly. dyeing

1 INTRODUCTION

Textile wet processing consumes a large amount of energy. These processes involve the use of chemicals assisting, accelerating, or retarding their effectiveness. In addition, it must be carried out at elevated temperatures to transfer mass from processing liquid medium across to the surface of the textile substrate in reasonable time^[1] The present day scenario in the textile processing calls for the conservation of energy or wage for a low amount of energy. One of the methods to achieve that goal is the use of ultrasound energy, which has taken an undeniable place in chemical and physical activities of the process industries as a very effective and eco-friendly technique^[2].

Azo dyes containing heterocyclic rings lead to brighter and often deeper shades than their benzene analogues and they are still very important for applications such as disperse dyes for polyester fibers^[3]. In this context, pyrazole derivatives that used in this work are important intermediates in organic synthesis and possess a range of interesting biological and antimicrobial properties. Azopyrazole dyes constitute a fascinating class of azo-dyes exhibiting a variety of novel dyeing properties. Changes in their structure have offered a high degree of diversity of molecules that may be endowed with better dyeing properties. The usage of many pyrazole derivatives has undoubtedly created considerable attention in developing many different synthetic procedures in pharmaceuticals, agrochemicals, dyestuff. The recent developments in the synthetic routes and the chemistry of pyrazoles have been thoroughly reviewed^[4-11]

Synthetic fibers have acquired an important part of the

current textile industry. A great disadvantage of some of the synthetic fibers is their low surface energy. This causes poor wettability and dyeability. Surface modification techniques mainly carried out to remove loosely bound materials, foreign particles/impurities and to improve hydrophilic nature and thereby improving their dyeability^[12].

The main task of the current work is to study the effect of ultrasound power on the dyeing performance of a set of azopyrazoledisperse dyes as well as its antibacterial activity.

2 EXPERIMENTAL

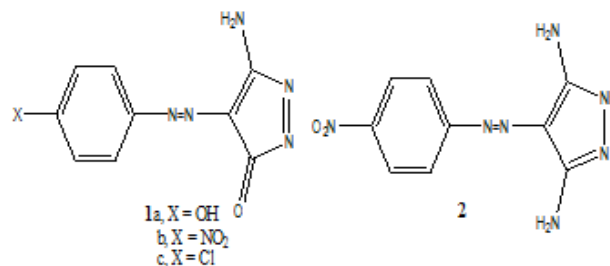
2.1 Materials

2.1.1 Fabrics

Polyester (polyethylene terephthalate) (PET). Washed and unfinished polyester fabric having 1/1 plain weaved and 122 g/m² was purchased from Mahalla factory for spinning and weaving, Egypt.

2.1.2 Disperse dyes

The disperse dyes under study were prepared according to the method described in literature.^[5,6]



Disperse dyes

2.2 Dyeing Procedures

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2.3.1 Dyeing of polyester fabrics using conventional method

Polyester fabric samples (1.7g each) were dyed with the disperse dyes (1a-c, 2) at liquor ratio 1:50. Dyeing was carried out at different concentrations of dye (1,2,3% respectively). The dye dissolved in ethanol and added drop wise with stirring to a solution of an anionic dispersing agent (2g/dm³), then the dye precipitated in a fine dispersion ready for use in dyeing. The dye bath, containing 5g/dm³ carrier, 4% acetic acid at PH 5.5, brought to 60°C. The polyester fabric entered and run for 15 min., then the fine dispersion of the dye added, and the temperature raised to boiling within 45 min. Dyeing continued at boiling temperature (120°C) for about 1h, then the dyed material was rinsed and soaped with 2% nonionic detergent.

2.3.2 Dyeing of polyester fabrics using ultrasonic method

Polyester fabric samples (1.7g each) treated with ultrasound waves using ultrasonic device (Water bath, Falc-Italy, Model LBS), This operation take place by adding samples in frequency (59 KHZ), temperature (70°C), tank temperature (46°C), power (100%), and time (30 min). Modified polyester fabric samples were dyed with the disperse dyes under study at liquor ratio 1:50. Dyeing was carried out at different concentrations of dye (1, 2, 3 % respectively). The dye dissolved in ethanol and added drop wise with stirring to a solution of (2g/dm³) an anionic dispersing agent, then the dye precipitated in a fine dispersion ready for use in dyeing. The dye bath (1:50 liquor ratio), containing 5g/dm³ carrier, 4% acetic acid at PH 5.5, was brought to 60°C. The polyester fabric was introduced and run for 15 min. The fine dispersion of the dye was added, and the temperature was raised to 90°C within 45 min, dyeing was continued at (90° C) for about 15 min, then the dyed material was rinsed and soaped with 2% nonionic detergent.

2.4 Measurements

2.4.1. Antibacterial activity

In the current study, the antibacterial activity of the synthesized series of pyrazoles dyes against common pathogenic bacteria examined as well as the Ultrasound treated polyester samples using qualitative methods. The test specimen and an untreated control pressed into intimate contact with an agar culture medium inoculated with the test bacteria solution. If antibacterial activity is present, it will be possible to observe a clear zone around the treated sample comparing to the zone of bacterial growth around and over the untreated control sample after the same contact time [15].

2.4. Color strength

The color yields of the dyed samples were determined by using the light reflectance technique performed on a Perkin-Elmer (Lambda 3B) UV/VIS Spectrophotometer.

The color strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation [16] as follows:

$$K/S = [(1-R)^2 / 2R] - [(1-R_0)^2 / 2R_0]$$

Where R = decimal fraction of the reflectance of the dyed fabric; R₀= decimal fraction of the reflectance of the undyed fabric; K = absorption coefficient; S = scattering coefficient

2.5 Analysis

2.5.1. Assessment of color fastness

Fastness to washing and rubbing tested according to the reported methods [17].

2.5.1.1. Fastness to washing

A specimen of dyed polyester fabric was stitched between two pieces of undyed cotton fabric (all fabric pieces had the same diameter) and then washed at 50°C for 30 min. The staining of the undyed adjacent fabric was assessed using grayscale (1, poor; 2, fair; 3, moderate; 4, good; 5, excellent).

2.5.1.2. Fastness to rubbing

The dyed polyester fabric placed on the base of a crock meter, so that it rested flat on the abrasive cloth with its long dimension in the direction of rubbing. A square of white testing cloth forced to slide on the tested fabric back and forth twenty times by turning the crank ten complete turns. For the wet rubbing test, the test square was thoroughly wetted in distilled water. The rest of the procedure was the same as the dry test. The staining on the white testing cloth was assessed using grayscale (1, poor; 2, fair; 3, moderate; 4, good; 5, excellent).

2.5.1.3. Fastness to light

Light fastness was determined by exposing the dyed polyester on Axenotest 150 (original hanau, chamber temperature 25-30°C, black panel temperature 60°C, relative humidity 50-60%, and dark glass (UV) filter system) for 40h. The changes in color were assessed using standard blue scale (1, poor; 3, moderate; 5, good; 8, very good).

2.5.2. SEM analysis

A scanning electron microscope (Joel scanning electron microscope, Japan) used to examine the surface morphology of conventionally and ultrasonically dyed polyester fabrics.

3. RESULTS AND DISCUSSION

3.1. Factors affecting disperse dyeing of polyester fabrics

3.1.1. Dye concentration:

Figure 1 shows the effect of the dye concentration on the extent of dye uptake in all dyes (1a-c, 2) for treated and untreated fabrics, expressed as the K/S value, of the dyed polyester substrates

As is evident, increasing the dye concentration from 1% to 3% brings about a noticeable increase in the K/S value.

This is due to the fact that more amount of dye diffuse into and react with the polyester fiber.

Increasing K/S of the ultrasound treated fabric than untreated fabric may be attributed to the fact that, ultrasound waves consist of expansion and compression cycles. Compression cycles exert a positive pressure on the liquid and push molecules together, while expansion cycles exert a negative pressure and pull molecules apart. Cavities generated during the expansion of a sound wave cycle with sufficient intensity, that the distance between the molecules exceeds the critical molecular distance necessary to hold the liquid together. A bubble in irradiated liquid continually absorbs energy from alternating compression and the expansion cycles of the sound wave. This causes the bubbles to grow in expansion cycles and contract in compression cycles. The implosion of cavities creates an unusual environment for chemical reactions. The vapor and gases inside the cavity extremely compressed during cavity collapse. Large increases in temperature and pressure are generated [18]. Cavitation and all the results of this phenomenon (release of temperature and pressure, intense shear forces, shock waves, micro jets) have important beneficial effects on chemical reactions.

Dispersion effect i.e. breaking up of micelles and high molecular weight aggregates into uniform dispersion in the dye bath. Degassing by the removal of dissolved or entrapped gas molecules or air from fiber capillaries and interstices at the cross over points of the fabric into liquid. Thereby facilitating a dye-fiber contact and accelerating the rate of diffusion of the dye inside the fiber by breaking the boundary layers covering the fiber and accelerating the interaction between dye molecules and fibers.

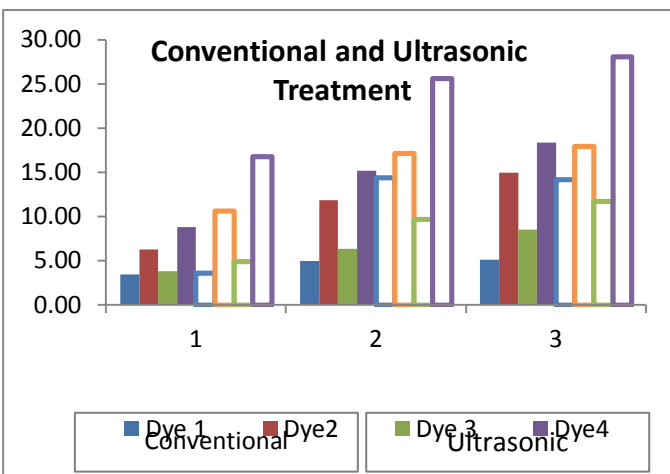


Fig.1. Effect of dye concentration

3.2 Color assessment

The following CIELAB coordinates were measured, (L^*) lightness, (a^*) value represents degree of redness (positive) and greenness (negative) and (b^*) represents the degree of yellowness (positive) and blueness (negative).

The measured K/S given by the reflectance spectrometer directly correlated to the dye concentration on the dye substrate.

The color coordinates listed in table 1 indicate that the dye has good affinity to polyester fabric and tend to give the following

- i- The dyes under study showed good affinity to polyester fabrics at the given temperature and gave generally bright and intense hues ranging from yellow to red.
- ii- The color hues of the dyed samples treated with ultrasonic power shifted to the yellowish direction on the yellow-blue axis according to the positive values of b^* . While, the same samples are shifted to the reddish direction on the red- green axis as indicated from the positive value of a^* except for the sample dyed with dye 2 shifted greenish direction according to the negative value of a^* .

Table 1. Effect of ultrasonic treatment on color assessment

Dye	Conventional Dyeing				Ultrasonic Dyeing			
	L^*	a^*	b^*	k/s	L^*	a^*	b^*	k/s
1a	29.72	41.07	-68.18	18.39	53.48	55.12	65.96	28.08
1b	62.02	18.38	47.45	8.48	58.61	28.62	53.07	11.68
1c	43.46	24.38	28.32	14.96	47.09	27.17	43.12	17.94
2	67.67	-2.50	51.78	5.10	50.86	-1.29	73.69	14.17

3.3. Antibacterial activities

The results indicated that all the samples (treated-untreated) exhibit antibacterial activity. It is clear that treated samples (1a-c & 2) exhibit relatively high antibacterial activity than untreated samples.

Table 2. Diameter of the zones of inhibition of the tested disperse dyes against Gram positive, Gram negative bacteria

Dye	Conventional Dyeing		Ultrasonic Dyeing	
	G(+ve)	G(-ve)	G(+ve)	G(-ve)
1a	14.5	13.0	19.5	17.0
1b	19.2	16.0	22.0	24.5
1c	24.5	23.0	20.5	22.0
2	18.5	10.5	18.5	19.0

3.4. Fastness properties

Washing fastness assessment of obtained colors on polyester fibers have shown good levels (4-5). The good results of treated and untreated polyester fibers might be attributed to the fact that these disperse dyes are mainly dispersed within the pores in the polyester fabrics and are held in their places by different forces including Van Derwaal forces. Electron-attracting groups enable stronger Van Derwaal forces and hydrogen bonding with the dyed fabrics that increases their stability to washing.

The results obtained also showed that dyed fabrics (treated-untreated) have excellent fastness levels to rubbing

(wet, dry) especially wet rubbing of ultrasonic treated fabrics because ultrasound increase penetration.

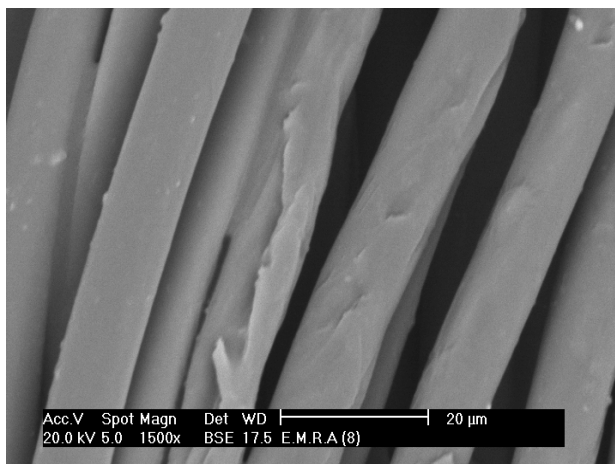
The light fastness results are good and acceptable showing little fading of light shades in both the conventional and ultrasound dyeing, however the ultrasonic treated samples showed excellent light fastness.

Table 3. Fastness properties of Polyester dyed fabrics.

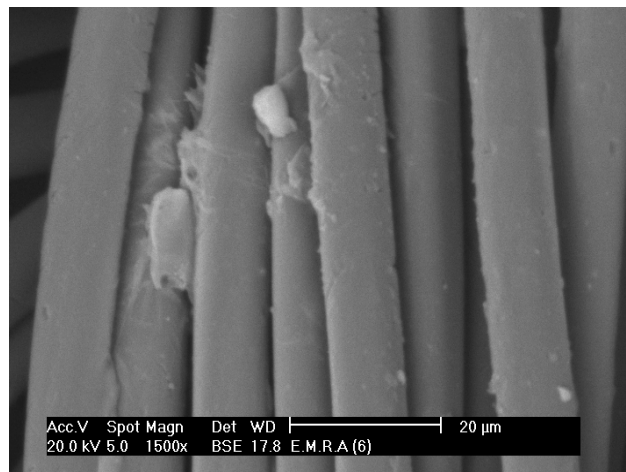
Dye	Fastness to washing	Fastness to dry rubbing	Fastness to wet rubbing	fastness to light
1-T	4-5	4	4	6
1-U	4-5	4	3-4	5
2-T	4-5	4	4	5
2-U	4-5	4	3-4	5
3-T	4-5	4	4	6
3-U	4-5	4	3-4	5
4-T	4-5	4	4	6
4-U	4-5	4	3-4	6
T(Treated) U(Untreated)				

3.5. SEM analysis

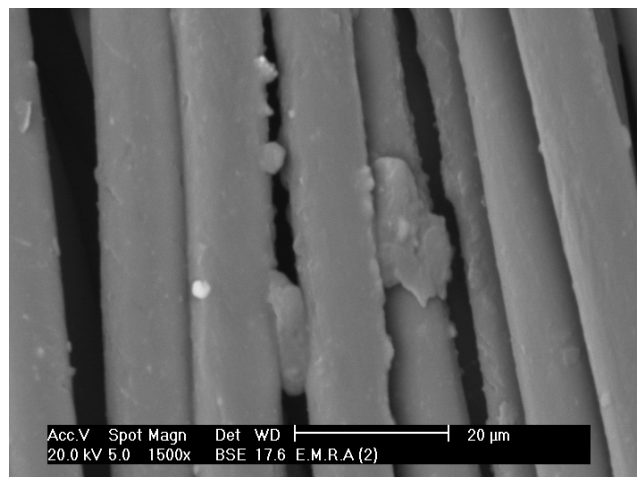
It is clear that the smoothness of the fiber decreased due to the action of ultrasonic and consequently dye uptake increased.



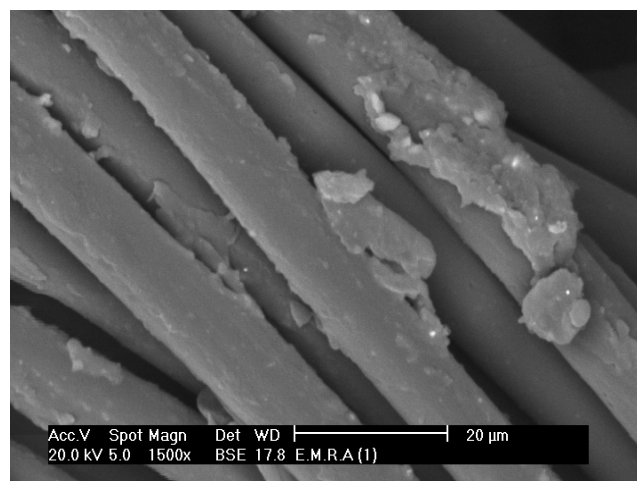
Untreated PET dyed with 5-Amino-4-(p-hydroxyazobenzene)-pyrazole-2-one (1a). (X1500)



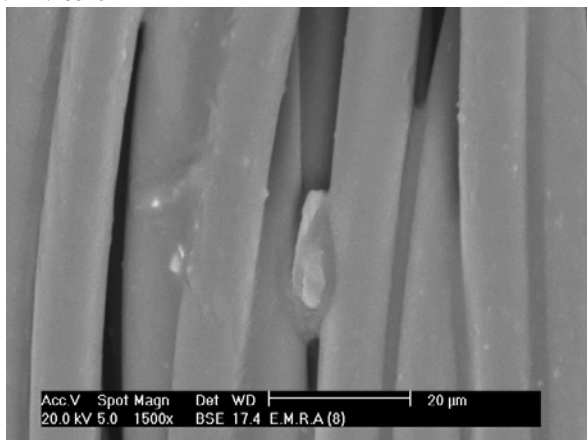
Treated PET dyed with 5-Amino-4-(p-hydroxyazobenzene)-pyrazole-2-one (1a). (X 1500)



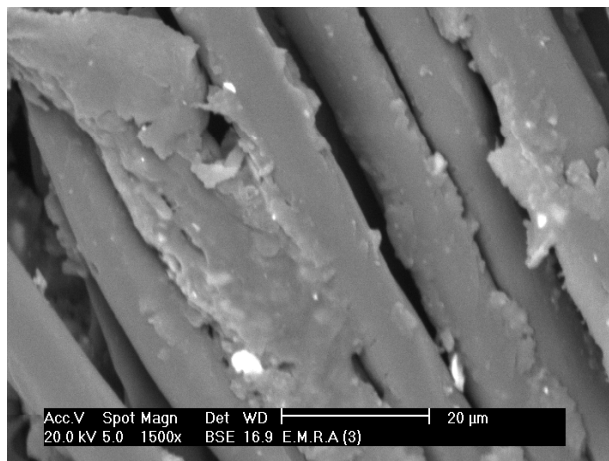
Untreated PET dyed with 5-Amino-4-(p-chloroazobenzene)-pyrazole-2-one (1c). (X 1500)



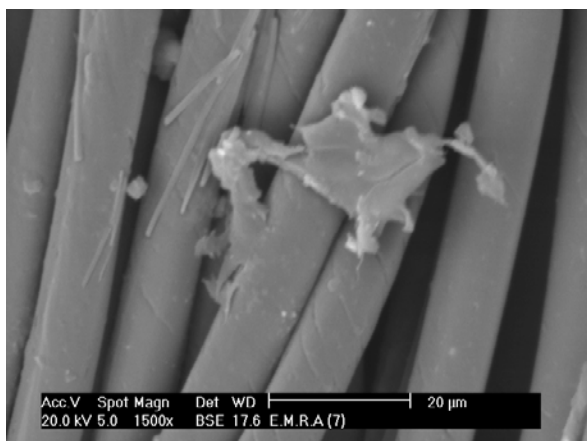
Treated PET dyed with 5-Amino-4-(p-chloroazobenzene)-pyrazole-2-one (1c). (X1500)



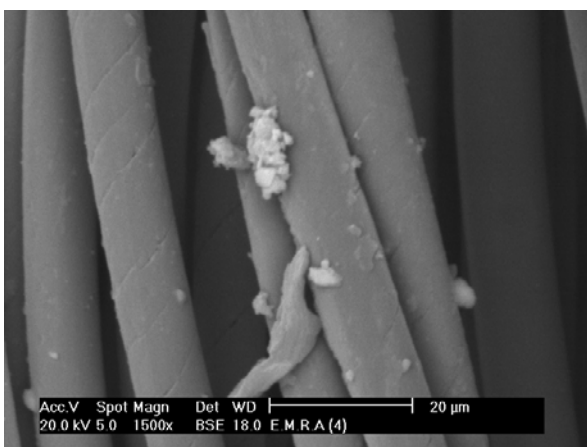
Untreated PET dyed with 5-amino-4-(p-nitroazobenzene)-pyrazole-2-one (1b). (X1500)



Treated PET dyed with 3,5-Diamino-4-(p-nitroazobenzene)-pyrazole (2). (X 1500)



Treated PET dyed with 5-amino-4-(p-nitroazobenzene)-pyrazole-2-one (1b). (X1500)



Untreated PET dyed with 3,5-Diamino-4-(p-nitroazobenzene)-pyrazole (2). (X1500)

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4 CONCLUSION

Samples dyed after treatment with ultrasonic power showed higher antibacterial behavior towards both gram positive and negative bacteria as shown with the larger inhibition zones than those conventional ones. Color strength and CIE Lab parameters showed a significant increment by using ultrasonic assisted dyeing. The results obtained in this work also, exhibited high fastness properties to washing, rubbing and light. Ultrasonic treated fabric have better fastness when compared to the conventional dyeing .

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