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A New Approach in Printing Wool and Wool/Polyester Fabrics with Disperse Dye Nanoparticles

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ABSTRACT

The chemical wet processing of textiles continues to expand each year using new technologies. The driving force being the need for cleaner, cost-effective and value added textile products. It is well known that, wool fabrics can be colored easily with acid, basic, direct, metal complex and reactive dyes while disperse dyes are used to colorate polyester fabrics. In the present study, wool and wool/polyester fabrics are printed with disperse dye nanoparticles treated by refluxing the particles in nitric acid with two different concentrations for a certain period of time at different temperatures. This treatment results in hydrophobic carboxylic groups on dye nanoparticles surfaces which behave similarly to acid dyes used in printing wool fabrics. Various parameters and measurements involving this work are investigated in detail such as: K/S values, dye particle size, nitric acid treatment conditions, printing paste pH, steaming conditions of the prints, SEM, TEM and FT-IR analysis of dye nanoparticles as well as fastness properties of the prints.
KEYWORDS: nanoparticles, SEM, TEM, FT-IR, fastness properties.

1. INTRODUCTION

The concept of nanotechnology (NT) is not new. It was started over 40 years ago. Nanotechnology is defined as the utilization of structures with at least one dimension of nanometer size for the construction of materials, devices or systems with novel or significantly improved properties due to their nano-size. The unique and new properties of nanoparticles have attracted not only scientists and researchers but also businesses due to their huge economical potential [(1)].

A disperse dye is defined as a substantially water insoluble dye having an affinity for one or more hydrophobic fibers [(2)]. Disperse dyes are essentially non-ionic dyes [(3)], they are the most commonly employed dyes in the textile industry to colour synthetic fibers such as polyester, acrylic and acetate [(4)]. Disperse dyes have extremely low water solubility and to be applied from this medium, they must be milled to a very low particle size and dispersed in water using a surfactant (dispersing agent) or else a carrier must be added during textile coloration [(5)].

The actual mechanism by which a carrier accelerates textile coloration has been widely debated. Polyester fibers absorb the carrier and swell. This swelling can impede liquor flow in packaging causing unlevelness. The overall effect seems to be a lowering of the polymer glass transition temperature (Tg), thus promoting polymer chain movements and creating free volume. This speeds up the diffusion of the dye into the fibres. Alternatively, the carrier may form a liquid film around the surface of the fibre in which the dye is very soluble, thus increasing the rate of transfer into the fibre [(6)].

In one of our previous researches it was shown that; disperse dye nanoparticles in a nominal size of 23 nm are able to diffuse slowly into polyester fabric at a temperature above its glass transition temperature (Tg) with the assistance of ultrasound waves [(7)]. However, in case of printing wool and wool/polyester fabrics, dispersion of disperse dye nanoparticles in aqueous pastes and adsorption of the particles onto fibres' surface are critical. Traditional dyes employed in printing of fibres are water soluble and have intermolecular actions with the fibres. The intermolecular interactions between dyes and surfaces of fibres serve as a driving force for the adsorption, and the particles can enter into the fibres through diffusion consequently [(8)]. Disperse dye nanoparticles are hydrophobic in nature and tend to aggregate in printing pastes. Thus increasing hydrophilicity and dispersibility of the nanoparticles is the key to the nanoparticle printing of fabrics.

In the present work, wool and wool/polyester fabrics are printed with disperse dye nanoparticles treated with nitric acid with different concentrations and at different temperatures. This treatment is applied on dye nanoparticles in order to obtain an oxidative effect that generates carboxylic groups in

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disperse dye nanoparticle ends. Consequently, this treatment makes the modified nanoparticles self-dispersible in printing pastes and adsorptive to fibres that contain functional groups ready to interact with carboxylic groups. If both can be achieved, printing of wool and wool/polyester fabrics can be carried out successfully without the addition of neither dispersing agents nor carriers, provided that the size of nanoparticles is small enough to diffuse into fibres properly.

2. EXPERIMENTAL

2.1. Materials

2.1.1. Substrates

Two scoured, felted, steamed and chlorinated fabrics are used in the present study: 100% wool fabric having a weight of square meter of 245g and a twill 1/2 structure and, a 45:55 wool/polyester blended fabric having a weight of square meter 175g and a plain structure 1/1. The materials are purchased from El-Mahalla El Kobra Co. for Spinning and Weaving, Egypt. All the materials used are of analytical grade.

2.1.2. Thickening agent

Commercial Mypro Gum NP-16 (Meyhall), which is a non-ionic thickening agent based on modified plant seeds gum, is used as a thickener throughout the present work at a concentration of 8%.

2.1.3. Dyestuff

A disperse dye having the commercial name Dianix Yellow-Brown-HRSL-SE150 kindly supplied by Dystar Textilfarben, Germany was used throughout the present work.

2.2. Preparation of dye nanoparticles

The disperse dye is ground using an energy Ball Mill with a speed of 50 cycles/min. The dye powder is sealed in a hardened steel vial using hardened steel balls of a 6 mm diameter. Milling is performed using a ball : powder mass ratio of 4:1. The dye is milled at different intervals: 4, 6, 9 and 25 days. After each milling interval, the particle size of the resulted dye powder is measured. The smallest particle size chosen to be incorporated in the present study resulted from milling the dye powder for 25 days is 23 nm.

The milled dye particles (1 g) are dissolved in distilled water (100 cm^3) then mixed with two different concentrations of nitric acid (25ml) separately: 25% and 50%. The mixture was refluxed at 85°C for 4 hours.

2.3. Printing procedures

To investigate each factor of the present work, a printing paste having the following formula was applied on both substrates:

20 g stock dye mixture
80 g Mypro Gum
X ml water
1000 g total weight of paste

The pH is adjusted according to each required value using sodium carbonate. The printing paste is applied to fabric through flat screen printing technique then, the prints are left to dry at room temperature. Fixation of the dye is carried out via steaming at 120 and 125°C for 30 min. for both substrates wool and wool/polyester, respectively. The samples are finally washed off using 2g/l non-ionic detergent: Sera-Wash M-RK at a liquor ratio of 1:50. Washing is carried out at 60°C for 10 min.

2.4. Measurements and analysis

2.4.1. Colour strength

The colour strength of the printed specimens is evaluated by a light reflectance technique at maximum. The spectraphotometer used is of model ICS-Texicon Ltd., England.

2.4.2. Scanning Electron Microscope (SEM)

The untreated and treated samples with plasma are investigated by a Scanning Electron Microscope (SEM) Philips XL 30 attached with EDX unit; with accelerating voltage 30 K.V. magnifications range 1500-2000x and a resolution of 200 A. Before examinations, the fabric surface was prepared on an appropriate disk and coated randomly by a spray of gold.

2.4.3. Transmission Electron Microscopic analysis (TEM)

The observation of the particle shape and the measurement of the particle size distribution of the precipitate were performed using a JSM-5200 Scanning Electron Microscope (JEOL) using conductive carbon paint. Transmission electron microscope (TEM) is a good tool to study the particle size and morphology. TEM gives a good resolution down to a nanometer scale. Photographs were taken using JEOL-2010.

2.4.4. Fourier Transition Infrared Spectroscopy (FT-IR)

Fourier transition infrared spectroscopy (FT-IR) of the samples was recorded using a Brucker-FTIR. The method includes mixing few mgs. of a fine powder of the sample with KBr powder in agate

mortar. The mixture was then pressed by means of hydraulic press. The absorbance was automatically registered against wave number (cm^{-1}).

2.4.5. Fastness properties

Fastness properties of wool prints to rubbing, washing, perspiration, and light are assessed according to standard methods [9].

2.4.6. Tensile mechanical testing

The samples are cut into strips of dimensions – x – cm and every data point is the average of 3 tests. Tensile strength measurement is carried out using a Textile Tensile Strength tester No: 6202, 1987, type: Asano Machine MFG**,** Japan.

3. RESULTS AND DISCUSSION

3.1. Effect of printing paste pH

Disperse dye nanoparticles are hydrophobic and stay in aggregated form in aqueous environments, which makes it difficult to be adsorbed onto surfaces and diffused into most textile fibres, particularly the hydrophilic ones. Oxidative treatment of disperse dye nanoparticles is effective to increase the hydrophilicity of the dye due to the generation of carboxylic groups on particles' surfaces. Nitric acid, an oxidizing agent, can attack some imperfect areas of dye nanoparticles during refluxing, which leads to the formation of carboxylic acid groups (-COOH) on disperse dye nanoparticles surfaces [10].

It is established that, the rate of dye fixation into wool fibres increases by decreasing the pH value since at lower pH values, dye concentration increases on the available wool fibre surfaces by increasing the number of ammonium ion sites [6]. On the other hand, disperse dyes are sensitive to alkalis and so polyester is generally printed under weakly acidic conditions with the incorporation of the proper dispersing agent in the printing paste [11]. Moreover, refluxing disperse dye nanoparticles in nitric acid acquires the dye an extremely low pH value that reaches pH 2, which causes damage of the fabric. Subsequently, addition of sodium carbonate (an alkali) was essential to increase the pH level up to the neutral medium. Therefore, in the present work, we investigated the effect of applying different pH values in the printing paste used to print both wool and wool/polyester fabrics using disperse dye nanoparticles refluxed in two nitric acid concentrations (25, 50%) separately on the K/S values of the prints. The obtained results are shown in Figure (1).

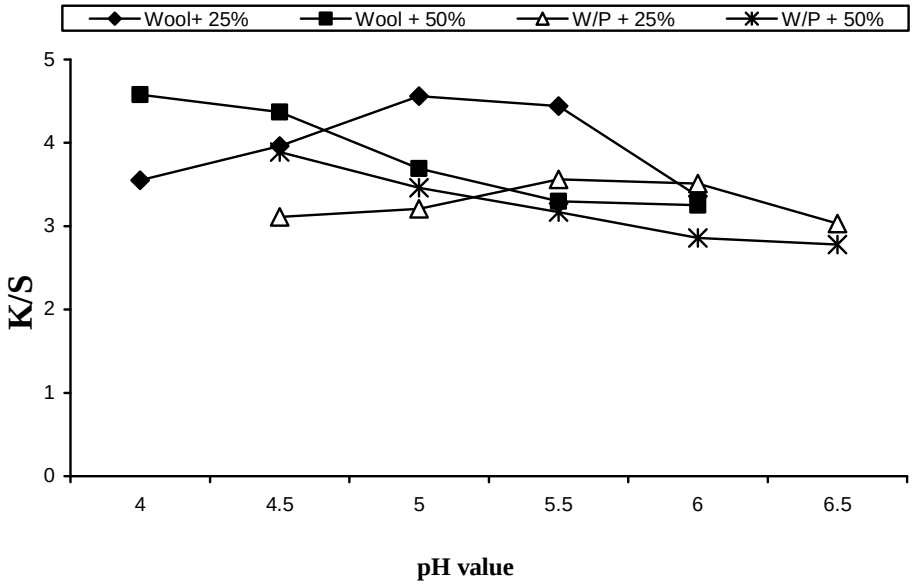

Fig. (1): Effect of pH on the K/S values of printed wool and wool/polyester (W/P) fabrics using disperse dye nanoparticles refluxed in 25 and 50% nitric acid.

It is obvious from the figure that, best K/S values of the prints can be obtained at pH 5 and 5.5 for wool and wool/polyester fabrics printed using disperse dye nanoparticles refluxed in 25% nitric acid, respectively. While on refluxing disperse dye nanoparticles using 50% nitric acid had a negative effect on the K/S values of the prints of both substrates. This result can be referred to the effect of using both: the severity of refluxing conditions, using high concentrated nitric acid (50%) at a very high temperature for a prolonged time, as well as using sodium carbonate to adjust the pH value. It can also

be concluded from the figure that, wool prints give higher K/S results than wool/polyester prints using same disperse dye nanoparticles refluxed in an identical concentration of nitric acid. These results may be referred to that, carboxylic groups on disperse dye nanoparticles significantly increase dye hydrophilicity and make them self dispersible in water. Thus, the dye becomes interactive with wool fibres and is potentially applicable to fibres that can be coloured by anionic dyes.

3.2. Effect of steaming conditions

It is well known that steaming causes increasing the colour strength of the prints due to its swelling effect on wool substrate, since moisture reduces the binding forces between polymer chains of wool leading to swelling which increases by increasing steaming temperature. Also, steam condenses onto the fabric raising its temperature to 100 °C which swells the thickener film printed on polyester resulting in the absorption (build up) of the disperse dye [12]. Thus, in this work we investigate the effect of steaming conditions on the K/S values of wool and wool/polyester fabrics printed with disperse dye nanoparticles refluxed in 25 and 50% nitric acid, different steaming temperatures and periods of time are used in the fixation process and the results are plotted in Figs. (2 & 3).

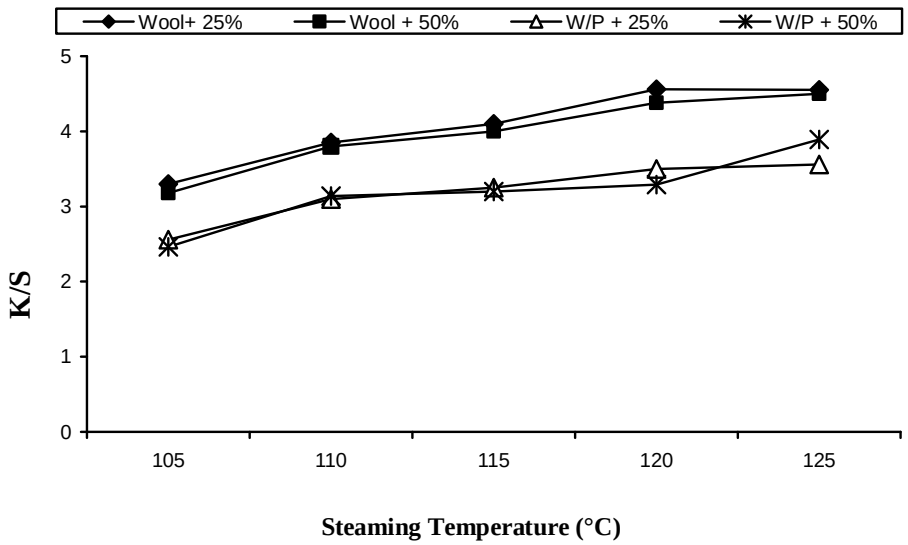

Fig. (2): Effect of steaming temperature on the K/S values of printed wool and wool/polyester fabrics using disperse dye nanoparticles refluxed in 25 and 50% nitric acid

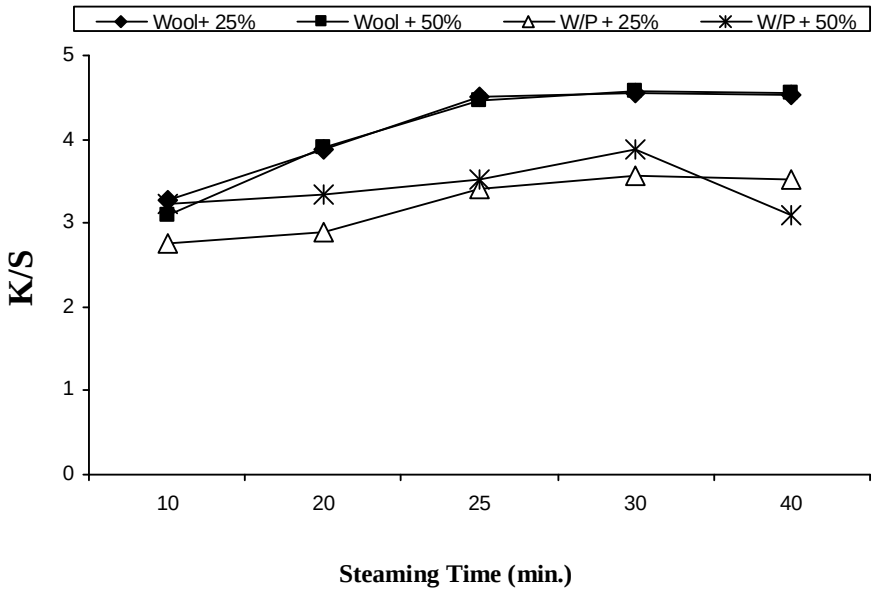

Fig. (3): Effect of steaming time on the K/S values of printed wool and wool/polyester fabrics using disperse dye nauoparticles refluxed in 25 and 50% nitric acid

It can be seen from both figures that, best K/S results can be achieved on steaming wool and wool/polyester prints using disperse dye nanoparticles refluxed in 25 and 50% nitric acid and fixed at 120 and 125°C for 30 min, respectively.

3.3. Scanning Electron Microscopic (SEM) and Transmission Electron Microscopic analysis (TEM)

The surface morphology, structure and particle size of dye samples milled at different durations are shown in Table (1) and Fig. 4 (a & b). Fig. 4a shows the SEM images of dyes which indicate that, the dye particles have different shapes like breaking dishes shape, spherical shape and tiny sprinkled dots. The TEM micrographs of the samples milled at 9 and 25 days compared with the un-milled sample are shown in Fig. 4b. The micrographs indicate uniform spherical dye nanoparticles, with a size lie in the range of 23 -200 nm in diameter. It was found that, the particle size decreases as the period of grinding increases. The difference in particle size after grinding is referred to their dissociation due to the impact of shear forces that act on dye particles in the ball mill that converted gradually the particle size from 0.2 µm (before milling) to 23
nm (after 25 days of milling).

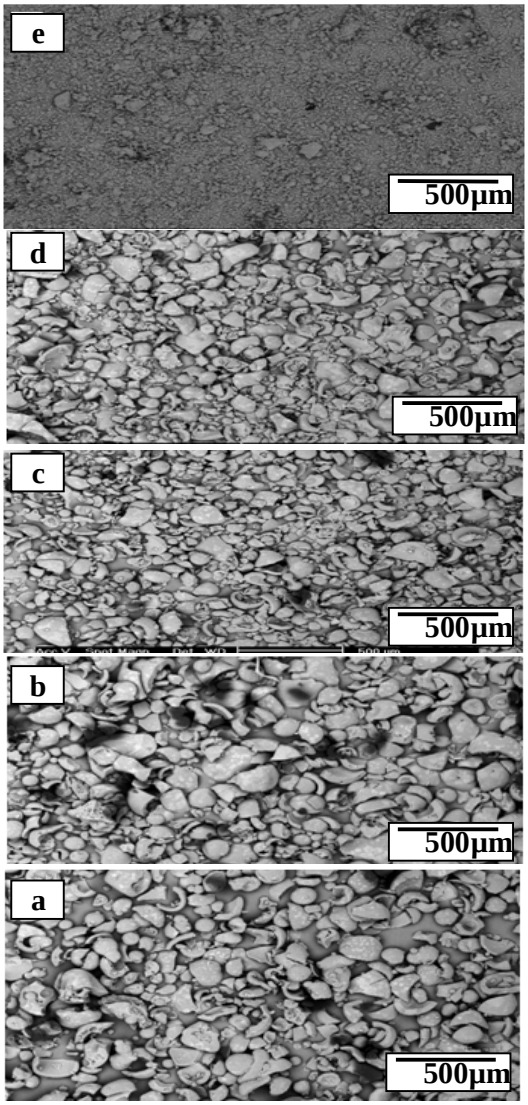

Fig.4 a: SEM images of disperse dye: a) before milling b) after 4 days c) after 6 days d) after 9 days e) after 25days

Table (1): Particle size obtained from TEM of dye samples

Sample	Particle size (TEM) (nm)
D_b	200
D_9	60
D_{25}	23

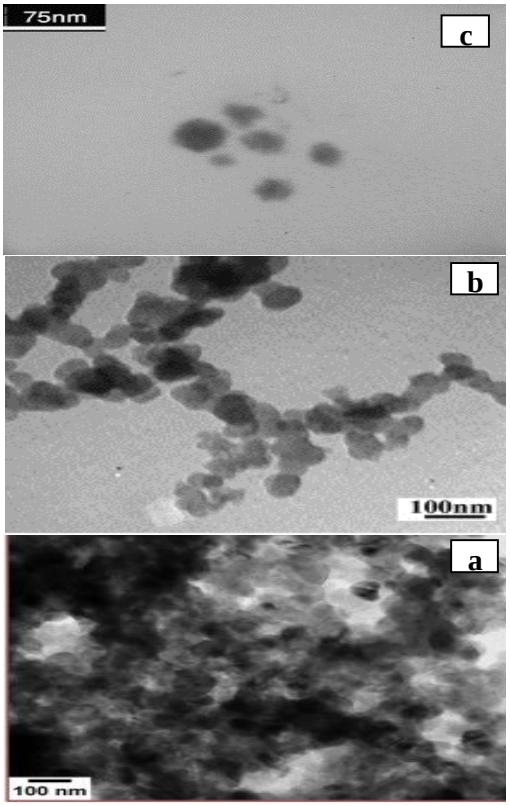

Fig.4 b: Representative TEM images of disperse dye: a) Before milling b) after 9 days c) after 25 days

It was observed that, the spectra of the two samples show also three peaks at about 827, 1348 and 1634 cm^{-1} with small blue shift in the treated sample. This suggests that, the effect of HNO_3 on the dye itself is very small while the main effect has been observed on the functional groups of the dye.

3.5. Fastness properties

The durability of printing wool as well as wool/polyester fabrics with disperse dye nanoparticles treated with 25 and 50% nitric acid (using overall optimum conditions) is evaluated in terms of washing, perspiration, rubbing, light, tensile strength (tenacity and elongation) and the results obtained are given in Table (2).

Table (2): Fastness properties of wool and wool/polyester fabrics printed with disperse dye nanoparticles treated with 25 and 50% nitric acid using overall optimum conditions.

Substrate treatment with nitric acid	Washing		Perspiration				Rubbing		Light	Tensile strength	
			Acidic		Alkaline						
	St.	Alt.	St.	Alt.	St.	Alt.	Dry	Wet		Tenacity(kg)	Elongation (%)
Wool treated with 25 % nitric acid	4-5	4	4-5	4	4-5	4	4-5	4	6	44	55
Wool treated with 50 % nitric acid	4-5	4	4-5	4	4-5	4	4-5	4	6	42	37
Wool/polyester treated with 25% nitric acid	4-5	4	4-5	4	4-5	4	4	4	5-6	50	47
Wool/polyester treated with 50% nitric acid	4-5	4	4-5	4	4-5	4	4-5	4	6	50	54

It can be concluded from the table that, all prints are characterized by very good fastness properties which proves that the dye is well penetrated and properly fixed in fibres despite being hydrophobic in its original structure.

4. Conclusion

Disperse dye nanoparticles have been oxidized via nitric acid having two concentrations (25 and 50 %). The resulted disperse dye is printed on wool and wool/polyester fabrics using acid medium without the addition of dispersing agents, using steaming for dye fixation. FT-IR analysis confirmed that, the resulted nanoparticles contain a certain amount of carboxylic groups. Best K/S results are obtained from wool prints using disperse dye nanoparticles refluxed in 25 % nitric acid at pH 5. All prints' fastness properties are very good which indicates that, dye particles penetrated and interacted properly into fibres.

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