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Novel green printing of cotton, wool and polyester fabrics with natural safflower dye nanoparticles

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Abstract

Hiren safflower dye nanoparticles were successfully prepared by using a simple ball milling technique at room temperature. UV-vis. absorption, XRD, TEM, FT-IR spectroscopy, and SEM were used to characterize safflower dye nanoparticles. The prepared safflower dye nanoparticles were used as active ingredients for printing cotton, wool, and polyester fabrics via dye printing technique and pigment printing technique. Factors of printing process were studied such as Mordanting of Substrates, thickeners type, urea concentration and Printing Paste pH for first paste and urea concentration, Printing Paste pH and binder concentration for second paste. Results show fabrics printed with safflower dye nanoparticles via mentioned two methods show very good to excellent fastness properties with a full green method. These data indicated that printed samples have high quality for colour strength without any environmental hazards compared with other conventual and nanotechnological aspects.

Keywords: Novel green printing, cotton, wool and polyester fabrics, ball milling, safflower, natural dye, nanoparticles

1. Introduction

Please read these instructions carefully and print Natural dyes were replaced by synthetic dyes due to their ability to easily match the desired color, increasing variety, high purity, cheap price, and easy processing. However, through the production and application of synthetic dyes, producers and consumers have observed several ecological and biological problems [1-9].

Safflower (*Carthamus tinctorius* L.) is a member of the family Composite or Asteraceae. Plant extract chemical constituents such as flavonoids, phenylethanoid glycosides, coumarins, fatty acids and steroids have been isolated from different parts of the plant. The flowers also contain carthamidin, isocarthamidin, quercetin, kaempferol, 6-hydroxykaempferol and its glycosides chalcones including hydroxysafflor yellow A, safflor yellow A, safflamin C, safflamin A and safflomin A. [10-12].

Mordants have affinity for both textile fabrics and dyes, thus they used to link the dyestuff to the fiber [3, 7, 13]. Therefore, they can be used for improving dye uptake and fixation causing change in color shade and fastness properties. [10, 12, 14, 15]. The metal ions of these mordants can act as electron donors to form coordination bonds with the dye molecules, making them insoluble in water [4, 5, 16-18].

Nanotechnology is a new field of research dealing with nanomaterials with particles size from 1-100 nm. Regarding the textile industry, nanotechnology becomes a new and promising tool to develop new textile materials for technical and smart use [19-22]. Nanoparticles have a high surface-to-volume ratio, which gives them new interesting physical and chemical properties in the textile field [8, 11, 23-30].

Ball milling is a top-down technique to form micro to nano scale materials, by inducing heavy cyclic deformation in materials. Ball milling is nowadays widely used for the preparation of nanoparticles because of its simple operation, use of relatively inexpensive equipment and its applicability to essentially all classes of materials [31-36].

In the present work we prepare nanoparticles from safflower dye via ball milling technique for printing some selected natural and

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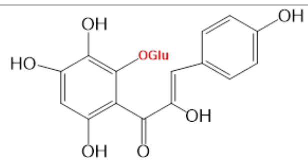
synthetic fabrics such as wool, cotton and polyester. The prepared nanoparticle dye was characterized by using transmission electron microscopy, UV-vis., spectra and X-ray diffraction. Several factors were studied to optimized the printing process such as (Mordanting of Substrates, thickeners type, urea concentration and Printing Paste pH for first paste and urea concentration, Printing Paste pH and binder concentration for second paste). the printed fabrics were characterized by using colorimetric measurements.

2. Materials and Methods

2.1 Materials

Fabrics: Mill-scoured wool fabric (100%) was supplied by EL-Nasr Company for Spinning, Weaving, and Dyeing, El-Mehalla Elkubra, Egypt. Mill desized, scoured, and bleached 100% cotton fabric was supplied by EL-Nasr Company for Spinning, Weaving, and Dyeing, El-Mehalla Elkubra, Egypt, and polyester 100%, kindly supplied from Artex Apparel, Egypt.

Natural dye: Clean, dry, ground safflower plant, was purchased from the Agricultural seeds Medicinal and Medical plant company (Harraz), Cairo, Egypt, having the following specifications

Botanical name	Class	Colour index	Part used	Chemistry
Carthamus tinctorius L.	Benzoquinone	Natural red 26	Flower	

Thickening agents: Sodium alginate used at concentration 8%. Meypro gum used at concentration 8%. Carboxymethyl cellulose (CMC) used at concentration 3%. DEL THICKNER P used at concentration 3%.

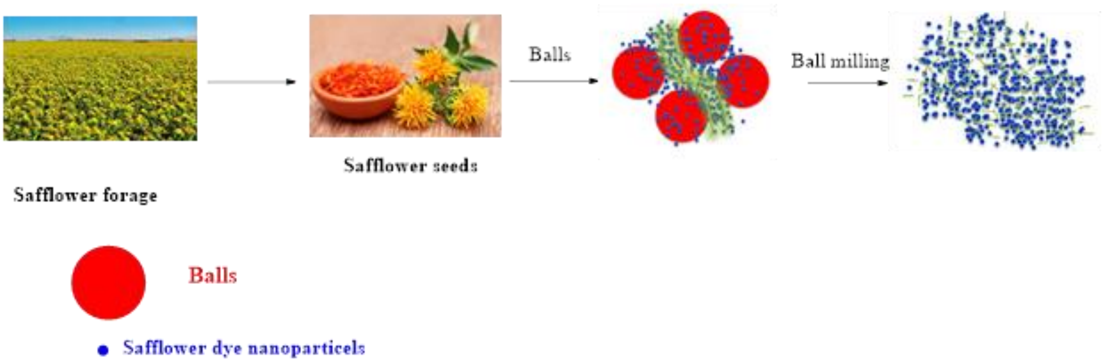
Mordant: Tartaric acid (2,3-dihydroxybutanedioic acid)

Other chemicals: Urea $[(NH_2)_2CO]$. Hostapal® CVL-ET (nonionic wetting agent based on alkyl aryl polyglycol ether, Clariant). Sodium carbonate Na_2CO_3. Diammonium phosphates $(NH_4)_2HPO_4$. Astroglitter binder® based on, nonionic/ anionic acrylic resin compound.

2.2. Methodology

2.2.1. Preparation of Dye Nanoparticles

The safflower dye powders were milled for 15 h in a planetary ball mill type. The milling conditions were; a) ball to powder weight ratio (BPR) equal 10:1, (b) 10 mm balls diameter, and (c) 400 rpm rotating speed. It is worth to mention that the milling was done in a cycle of 3 h and paused for 2 min. A stock solution of safflower dye nano particles was prepared by using the milled dye particles of a concentration of 3% (3g of dye powder was dispersed in 97 cm^3 of distilled water). The suspension was irradiated afterwards with ultrasound waves (53 mega Hz) and stirred at 60 ^oC for 90 min **(Scheme. 1).**



Scheme 1. Schematic diagram of safflower dye nanoparticles production by ball milling

2.2.2. Fabric Mordanting: wool is mordanted by two ways. The first way is prior to printing process. The second way is post to printing and fixation process. The mordanting bath is set with tartaric acid, separately on weight of fabric at L.R. 1:40. Mordanting is carried out wool at 50-60°C for 30 min after which the samples are washed with distilled water and air-dried.

2.2.3. Printing Procedures: To investigate each factor of the present work, two printing paste having the following formula was applied on all substrates:

The first Paste recipe (as a dye); (g /Kg paste)		The second Paste recipe (as a pigment); (g /Kg paste)	
Natural dye	50g	Natural dye	50g
Thickener*	600 g	Thickener*	600 g
Urea	Xg	Diammonium phosphate	x g
PH Adjusting	y g	Binder	y g
Water	z g	Urea	z g
		Balance	G
Total	***1000g***	***Total***	***100 g***

*** Thickener stock concentration is 3 wt.%**

The pH is adjusted according to each required value using Sodium carbonate and citric acid for first paste and Diammonium phosphate for second paste. 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 first paste is carried out via steaming at 105°C for 20 min. for wool and 125◦C for 45min. for polyester. The second paste is carried out with thermofixation at 160◦C for 4 min.

2.2.4. Washing

After fixation process, the samples were rinsed in cold water washed with 2 g/l non-ionic detergent (Hostapal CV-ET) at a liquor ratio of 1:50. for 15 min. at 40ºC for wool fabrics and at 60ºC for cotton and wool and 90 ºC for polyester.

2.3. Testing and analysis

2.3.1. Color measurements and fastness properties

The printed samples were tested according to AATCC and ISO standards [37, 38]. The colour strength values (K/S) were determined using CIE Lab: D-65 10 standard. The ISO-CO6 D1M was used to determine the colour-fastness to laundering. The color fastness to laundering, color fastness to perspiration and color fastness to rubbing was measured using AATCC-15, AATCC-8 and AATCC-16 standards, respectively. The test specimen and the two adjacent fabrics (cotton and wool) were compared using the gray scale. The rating scale of washing fastness for color change was from 1 (very poor), 2 (poor), 3 (fair), 4 (good) to 5 (excellent). The rating scale of light fastness was from 1 (very poor), 2 (poor), 3 (fair), 4 (moderate), 5 (good), 6 (very good), 7 (excellent), to 8 (outstanding).

2.3.2. FT-IR spectra

The FT-IR spectra of the samples were recorded by using an FT- IR spectrophotometer (JASCO FT-IR-6100) using the KBr pellet disk method for transmittance measurements, in the region of 4000 - 400 cm-1 with spectra resolution of 4 cm^{-1}.

2.3.3. Tensile strength

The tensile strength of the fabric sample was determined by the ASTM Test Method D5035. A Q-Test 1/5 tensile tester. Three specimens for each treated fabric were tested in the warp direction and the average value was recorded to represent the fabric breaking load (Lb).

2.3.4. Ultraviolet-visible (UV-vis) spectra

UV-Vis spectra have been used to confirm the formation of safflower dye nanoparticles. The spectra were collected over a range of 250-800 nm.

2.3.5. Transmission Electron Microscopy (TEM)

The shape and size of safflower dye nanoparticles were practically obtained by using TEM; JEOL-JEM-1200. Specimens for TEM measurements were prepared by placing a drop of colloidal solution on 400 mesh copper grids coated by an amorphous carbon film and evaporating the solvent in air at room temperature. The average diameter of the prepared safflower dye nanoparticles was determined from the diameter of 100 nanoparticles found in several arbitrarily chosen areas in enlarged microphotographs.

2.3.6. Scanning Electron Microscopy (SEM)

Microscopic investigation on safflower dye powder before and after milling were carried out by using a Philips XL30 scanning electron microscope (SEM) equipped with a LaB6 electron gun and a Philips-EDAX/DX4 energy-dispersive spectroscopy (EDS). Images were taken at different magnifications (from 1509 to 30009), using scanning electron microscope (SEM) in accordance with the clarity of the images. Fabric samples were fixed with carbon glue and metalized by gold vapor deposition to record images.

2.3.7. X-ray diffraction

X-ray diffraction patterns of samples were recorded on an STOE STADI P Transmission X-ray powder diffractometer system by monitoring the diffraction angle from 10 to 80 (2h) using monochromatized Cu Ka (k = 1.54051 Å) radiation.

3. RESULTS AND DISCUSSION

3.1. Preparation and characterization of Dye Nanoparticles

The colours before and after ball milling was no difference change. Fig.1a. shows that safflower dye shows broad band peaks in UV-vis. absorption while, safflower dye nanoparticles shows sharp band peak due to nanostructure of safflower dye nanoparticles.

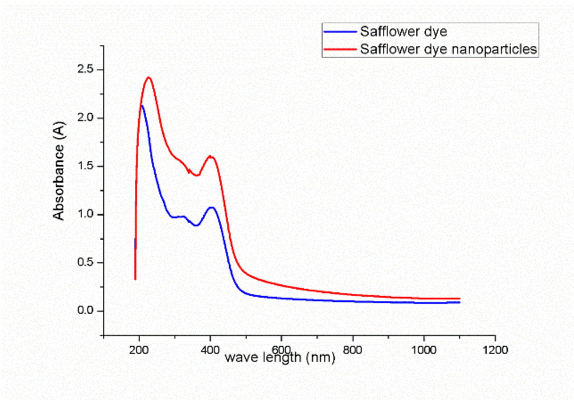


Fig. 1a. UV-vis. spectra of safflower dye and safflower dye nanoparticles

Fig. 1b., shows that FT-IR spectra of safflower dye nanoparticles have the same band peaks of safflower dye but its band peaks decreased compared with safflower dye itself

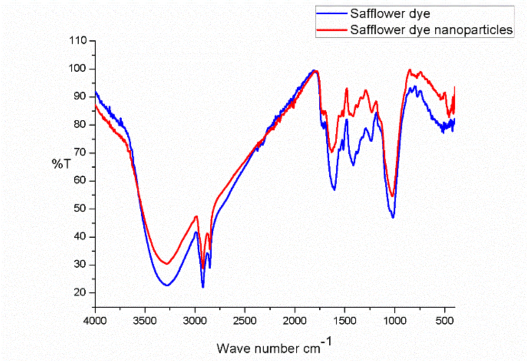


Fig. 1b. FT-IR of safflower dye and safflower dye nanoparticles

To compare crystalline structure of unmilled and milled safflower dye, XRD analysis was performed and its results are shown in Fig. 1c. XRD of both milled and unmilled safflower dye are similar except there is more broader bands in safflower dye nanoparticles than unmilled safflower dye.

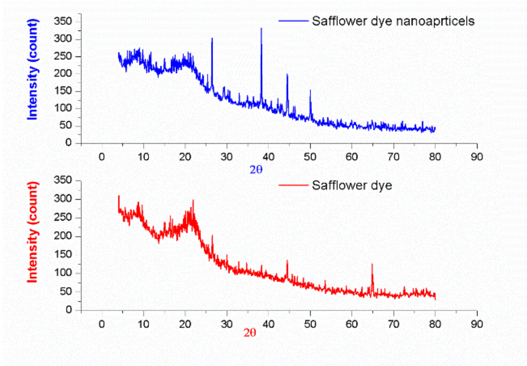


Fig. 1c. x-ray diffraction (XRD) of safflower dye and safflower dye nanoparticles

Transmission electron microscopy (TEM) illustrates the shape and size of safflower dye nanoparticles as shown in Fig. 1d. safflower nanoparticles formed from safflower powder via ball milling show well disperse and semi spherical shapes. From the histograms in Fig. 1d, it was indicated that particles size ranged from (20 - 60nm) with a major diameter range (30-40 nm). These results data agreed with UV-vis spectral data.

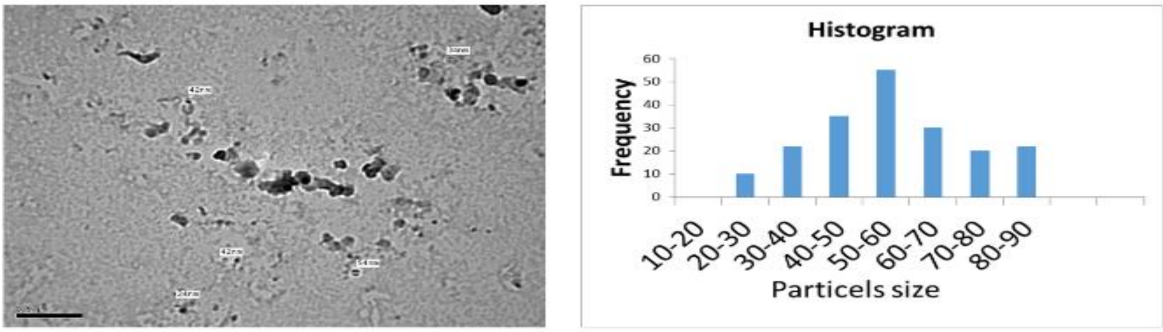


Fig. 1d. TEM image of safflower dye nanoparticles prepared via ball milling and histogram of its nanoparticle size

Fig. 2. shows scanning electron microscopy of safflower dye before and after milling. SEM images shows the conversion of particle size of the safflower dye from microform to nanoform. In addition, SEM images shows that surface particles size changed from 107-360 µm of safflower dye to 85-100 nm of safflower nanoparticles which confirm formation of nanoparticles via green method.

In addition, SEM images illustrate the safflower morphology and its conversion to more uniform nanoparticles with green method.

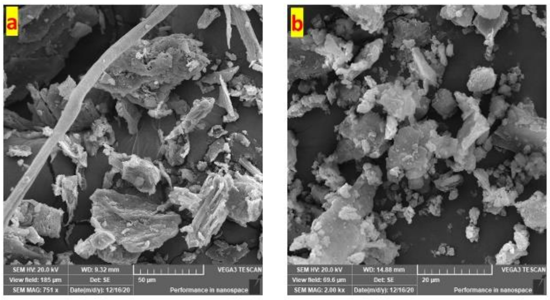


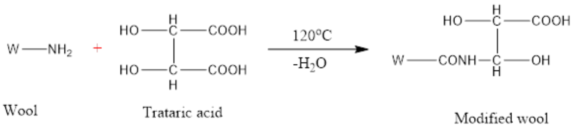
Fig. 2. scanning electron microscopy images of safflower dye a and safflower dye nanoparticles b.

3.2. Printing of cotton, wool and polyester fabrics with safflower dye nanoparticles

The main goal of the present study is printing of cotton, wool and polyester fabrics with a green printing paste consists of green thickeners such as carboxymethylcellulose, sodium alginate, Meypro gum and DEL THICKNER P and natural safflower dye nanoparticles. Natural fabrics such as wool were pretreated first with tartaric acid as a mordant to increase their dye accessibility (scheme 2).

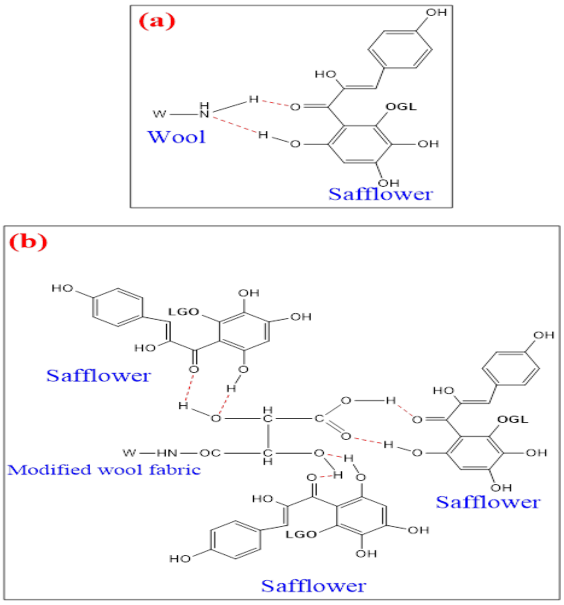
Mordanting of wool fabric and its effect on printed fabrics

wool fabric can be modified through its reaction with tartaric acid to form the corresponding ester and amide [39, 40] derivative as shown in **scheme 2.** The reaction will be takes place in physical absorption at 50-60 ^{0}C, then chemical reaction takes place in steam fixation process at 105°C for 20 min. for wool. this process create more reactive site e.g. hydroxyl and carboxylic groups in addition of native groups in wool fabrics.



Scheme 2. Modification of wool fabric via reaction with tartaric acid

Scheme 3 shows the how mordant have great effect of wool fabric compared with unmodified fabric. The main difference that unmodified fabrics have one accessible site form strong H. bond with one mole safflower dye where the mordanted fabrics have three accessible sites can form stronger H. bonds with three mols safflower dye. In addition, we can find that reaction fixation of wool fabrics comes from amide bond formation, this will reflect the difference in colour strength and fastness properties of the printed fabrics.



Scheme 3. Reaction of safflower dye with unmodified wool fabrics (A) and tartaric acid modified wool fabrics (B)

To investigate the effect of fabric mordanting on colour development of the used safflower dye nanoparticles, different concentrations (0, 40, 60,80,100 and 120 g/l) of tartaric acid, is used in wool fabrics' treatment, separately, prior to printing process and the results are illustrated in **Fig. 3.**

It is shown from the **Fig. 3** that, an increasing of mordant concentration results in increasing of K/S values until 100 g/L concentration of tartaric acid for wool, whereas the value of K/S increase by 59.284 % for pretreated wool prints and 51.01 % For post treated wool respectively, pretreated and post treated with tartaric acid compared with the untreated prints. from compare the results of pretreated and post treated found that pretreated is better than post treated.

These results are referred to the effect of both grinding and ultra-sonication of safflower natural dye particles on colour strength of wool fabrics expressed in K/S values. Grinding process increases the specific surface area of the safflower dye nanoparticles due to particle size reduction [41]. A feasible technique for particle-size reduction is ultrasonic. Ultrasonication's of solids leads to microjet and shock-wave-impacts on the surface, together with interparticle collisions, which can result in particle-size reduction [42]. Besides, comparing the results. Therefore, fabrics wool pretreated with 100 g/L tartaric acid as mordant used to improve printing of these fabrics with safflower dye nanoparticles.

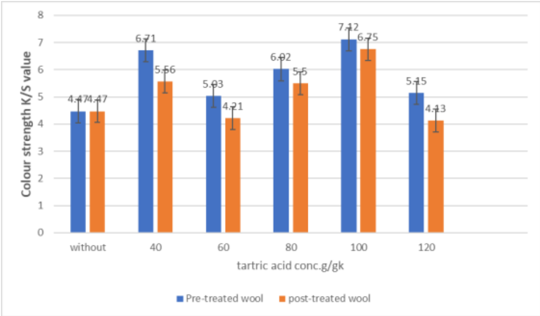


Fig. 3 Effect of concentration of mordant on K/S values of pretreated and post treated wool fabrics printed with safflower nanoparticles.

3.3. Factors affecting printing of wool fabrics with safflower dye nanoparticle for the first recipe

Several factors have been studied to illustrate the great role of using safflower dye nanoparticles as a new material prepared by green method compared with conventional dye.

3.3.1. Effect of thickener type

Different thickeners, carboxymethyl cellulose (CMC), sodium alginate, Meypro gum and DEL THICKNER P have been used in the printing pastes to investigate the effect of thickener type. Colour change of printed fabrics express in K/S values were showed in Fig. 4 for wool fabrics. Different types of thickeners were used in the printing past to study the effect of thickener type on printing. These thickeners were sodium alginate, carboxymethylcellulose (CMC), Meypro gum and DEL THICKNER P. The results of different thickeners were showed in Fig. 4. It is clear from Fig.4 that the type of thickener has a remarkable effect on the K/S of the printed samples. In most of the cases, the highest K/S value was obtained by using DEL THICKNER P as thickener whereas the value of K/S increase by % for wool prints respectively, printed fabrics with safflower dye nanoparticles compared with printed fabrics with safflower dye.

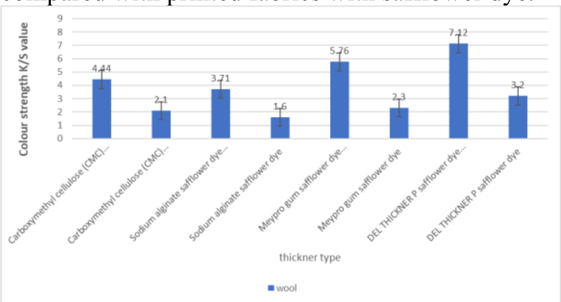


Fig.4 Effect of thickener type on K/S values of woolfabrics printed with safflower dye and safflower dye nanoparticles.

3.3.2. Effect of urea concentration

Urea considered an essential auxiliary in most printing pastes because of its ability to swell the fabrics that accelerate penetrating dye inside the fabrics [43]. In addition, it acts as a solvent for the dye i.e., used as moisture-absorbing agent and accelerates the migration of dye from the thickener film into the fabrics. The influence of urea concentration on colour strength of wool printed fabrics with safflower dye nanoparticles was studied by using different urea concentrations to the printing pastes (0, 10,30,50,70 and 150 g/kg) and the results were plotted in Fig. 5. In addition, Fig. 5 indicates that,150 g/gk can be considered as best urea concentration to be added to printing pastes with safflower nanoparticles on wool regardless of enhancements in colour yields by 24.657 % occurred in wool (pretreated with tartaric acid) print respectively, compared with the same printed fabrics without urea addition to their recipes. In addition, the treated fabrics higher than their corresponding untreated fabrics for wool, whereas the value of K/S increase by 26.829 % for treated wool print, respectively pretreated with tartaric acid compared with the untreated prints. These results come from that the urea has solvation and disaggregation effects on the dye molecules in the printing paste and enhances solubility of dyes in the paste.

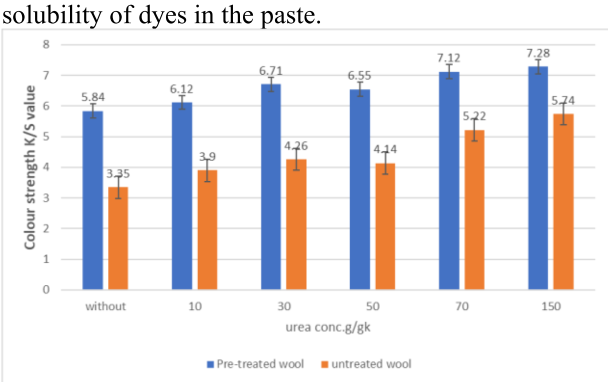


Fig. 5 Effect of urea concentration on K/S values of treated and untreated wool all fabrics printed with safflower dye nanoparticles.

3.3.3. Effect of pH of the printing paste

The pH of printing paste has an important role for dye fixation rate. The rate of dye fixation has been increased as the pH of printing paste of wool decreased because of increased of dye concentration and ammonium ion sites numbers at lower pH values [44]. The influence of printing paste pH on the K/S values of printed wool (pretreated with tartaric acid) substrates with safflower nanoparticles is studied, through using different values (5, 5.5, 6,6.5 and 7) as shown in Fig. 6. The optimum values of K/S were found at pH value of 5.5 for wool print, respectively.

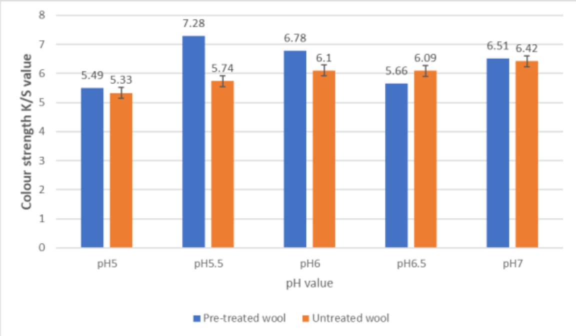


Fig. 6 Effect of pH value on K/S values of pretreated and untreated wool and polyester all fabrics printed with safflower dye nanoparticles.

3.4. Factors affecting printing of cotton, wool and polyester fabrics with safflower dye nanoparticle for the second recipe (as a pigment)

Effect of Diammonium phosphates concentration

Printing paste pH is considered as an effective factor in colour variation and subsequently, the influence of printing paste pH on colour intensity of the prints is studied by applying values (5,5, 6, 6.5 and 7) and the results are exhibited in Fig. 7. It is clear from Fig. 7 that, maximum K/S values can be obtained at pH 6 for Cotton, wool and polyester substrate printed with safflower nanoparticles respectively.

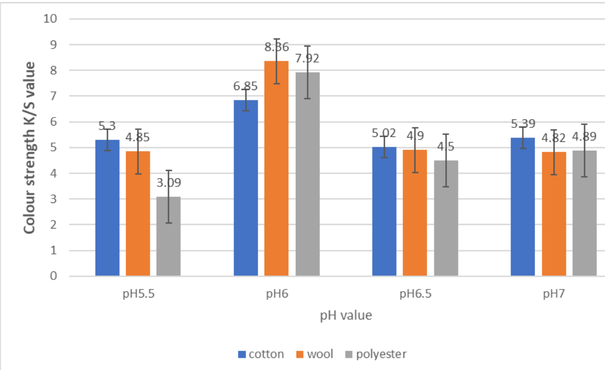


Fig. 7 Effect of pH value on K/S values of cotton, wool and polyester fabrics printed with safflower dye nanoparticles.

3.4.3. Effect of urea concentration

Different concentrations of urea (0, 10,20,40,60 and 80 g/kg) were used to study its effect on colour strength of cotton, wool and polyester printed fabrics with safflower dye nanoparticles. Fig. 8 shows that printed cotton, wool and polyester fabrics without urea has the highest K/S values. This may be because of swelling properties of urea that helps fixation of dye. So, the fixation here depends binder in the presence of diammonium phosphate as a catalyst. Diammonium phosphate catalyst can promote the crosslinking reaction, leading to the fixation of the binder to fabric. Diammonium phosphate at 160 ºC for 4 min., adjust the pH of the medium at 6. At this value binder can be chemically crosslinked on the fabrics and dye.

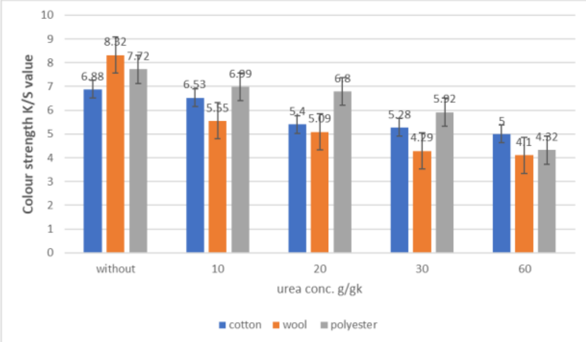


Fig.8 Effect of urea concentration on K/S values of cotton, wool and polyester all fabrics printed with safflower dye nanoparticles.

3.4.4 Effect of binder concentration

To investigate the effect of binder concentration on the K/S values of printing goods, using different mounts of safflower dye nanoparticles, viz. 80,100, 120, 140, 160. It is clear from Fig. 9, that the concentration of binder has a remarkable effect on the K/S of the printed samples. In most of the cases, the highest K/S value was obtained by using 140 g/kg

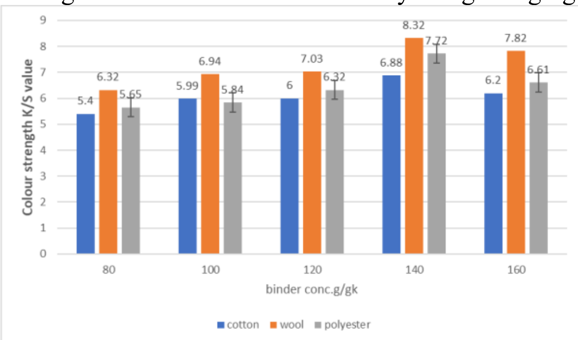


Fig.9 Effect of binder concentration on K/S values of cotton, wool and polyester fabrics printed with safflower dye nanoparticles.

3.5. Fastness properties of the printed fabrics with first Paste recipe

Table 1 shows the fastness characteristics of the printed cotton, wool and polyester fabrics using safflower dye nanoparticles for first recipe (as dye) and second recipe (as pigment). The printed fabrics reveal very good to excellent fastness properties using safflower dye nanoparticles as dye (first recipe) and very good to excellent fastness properties using safflower dye nanoparticle as pigment (second recipe). Light fastness properties were very good for all printed fabrics, indicating the suitability of safflower dye nanoparticles for better fabrics printed (dye or pigment) as a result of chemical bonds between the fabrics and the safflower dye nanoparticles molecules. Table 1shows that colour strength expressed in K/S values has higher values for all printed fabrics with second recipe as pigment than that fabrics printed with the first recipe as dye. In addition, mechanical properties (tensile strength and elongation at breaks show very good data compared with untreated and unprinted fabrics for all fabrics but the values of mechanical properties for fabrics printed with the first recipe as dye is slightly higher than that for fabrics printed with second recipe as pigment.

Table 1. Colour strength (K/S), fastness properties and mechanical properties of printed wool, cotton and polyester fabrics at optimum values for both first (as dye) and second (as pigment) recipes

	k/s		Washing fastness		Rubbing fastness		Perspiration fastness						Light fastness	Mechanical properties	
		St.	St*	Alt.	Dry.	Wet.	Acidic			Alkaline				TS (KgF)	EB (mm)
							St.	St*	Alt.	St.	St*	Alt.			
Pretreated wool printed with safflower dye	3.2	4	4	4	4	3	4-5	4-5	4-5	4-5	4-5	4-5	5-6	40	45
Pretreated wool printed with safflower dye nanoparticles	7.28	4-5	4-5	4-5	4	3	5	5	5	4-5	5	4-5	6-7	39	45
Untreated wool printed with safflower dye nanoparticles	5.74	4-5	4-5	4-5	4-5	3-4	4-5	4-5	4-5	4-5	4-5	4-5	5-6	40	45
prostrated wool printed with safflower dye nanoparticles	6.75	4-5	4-5	4-5	4	3	5	4-5	4-5	4-5	4-5	5	5-6	40	42
polyester printed with safflower	2.89	4	4	4	4	4-5	4-5	4-5	4-5	5	4-5	4-5	6	37	42
polyester printed with safflower dye nanoparticles	8.32	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5	4-5	4-5	6	37	42
Wool printed with safflower dye as pigment	1.46	5	5	5	5	4	4	4	4	5	4-5	5	4	35	40
Wool printed with safflower dye nanoparticles as pigment	7.72	5	5	5	5	4	4-5	4-5	4-5	5	4-5	5	5	36	40
Polyester printed with safflower dye as pigment	1.18	4	4	4	4-5	4	4-5	4-5	4-5	5	4-5	4-5	4	29	28
polyester printed with safflower dye nanoparticles as pigment	6.88	4	4	4-5	4-5	4	4-5	4-5	4-5	5	4-5	4-5	4	29	28
cotton printed with safflower dye as pigment	3.2	4	4	4	4	3	4-5	4-5	4-5	4-5	4-5	4-5	5-6	40	45
cotton printed with safflower dye nanoparticles as pigment	7.28	4-5	4-5	4-5	4	3	5	5	5	4-5	5	4-5	6-7	39	45

TS. Tensile Strength; EB. Elongation at Break; * Data are expressed as Mean ± S.D. for n=3; ** Tensile strength for wool, cotton and polyester blank fabrics were 36.75 ± 0.750, 45.25 ± 1.750 and 34.75 ± 0.250 kgf respectively; *** Elongation at break for wool, cotton and polyester blank fabrics were 49.00 ±1.000, 23.50 ± 1.500 and 36.50 ± 0.500 mm respectively

4. Conclusion

An ecological printing process of cotton, wool and polyester fabrics with safflower natural dye nanoparticles using green print Paste. Ball milling is an effective process for producing safflower dye nanoparticles in one step at ambient temperature and pressure. The prepared safflower dye nanoparticles have been confirmed via UV-vis, XRD, TEM and SEM. Safflower dye nanoparticles were used to print cotton, wool and polyester fabrics with dye printing and pigment printing techniques. The printed samples reveal very good to excellent fastness properties. Hence, the present process of printing cotton, wool and polyester fabrics with green print paste formulation by using safflower as dye and pigment may find wide application in textile coloration. Therefore, Using the dyes in the first method as a dye that gives a medium colour strength but preserves the texture of the material and gives good washing and friction fastness properties, while printing with natural dyes in the second method as a pigment gives high color strength, but the degree of stability to wet or dry friction is somewhat low compared to the materials printed with natural dyes using the first method. Thus, whoever wants high color depth and does not need high stability of friction can choose the second printing method.

Conflicts of interest

There is no conflict of interest to declare

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