



Surface Modification of Blended Fabrics by Silica Nanoparticles to Improve Their Printability

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THE sol-gel technique offers an effective way of modifying fabrics, and has many advantages. In this paper, silica nano particles in the solid form were prepared from TEOS precursor; further application of these silica nanoparticles to polyester / wool, and polyester/cotton blends as well as polyester and cotton fabrics is carried out. Improved fabrics printability, as well as acquiring antibacterial properties were achieved. The basic idea for using sol-gel technology to modify fabric blend surfaces in order to solve their coloration problems and to attain solid shades using a single set of dyes.

Keywords: Silica nano particles, Surface modification, Blend fabrics, Hybrid materials.

Introduction

In the last two decades, material science and engineering has come out with some excellent nanomaterials exhibiting novel features due to their unique physio-chemical properties (1,2). Recently, silica nanoparticles (SNPs) have drawn widespread attention due to their applications in many emerging areas because of their tailorable morphology. A number of techniques available for preparing SNPs namely, flame spray pyrolysis, chemical vapor deposition, micro-emulsion, ball milling, sol-gel etc. However, among these sol-gel process remains the most popular one, due

to its ability to control the size, distribution and morphology of the particles through systematic monitoring of their action parameters (3).

The manufacturing of nanomaterials can be classified as bottom-up and top-down approaches Fig. (1). The bottom-up approach involves building up from atomic or molecular scale and top-down approach involves making smaller sizes through etching or grinding from the bulk materials. Sol-gel, for the preparation of SNPs, is an extensively used bottom-up approach. This approach has some advantages such as low cost, higher productivity, environmentally benign etc.

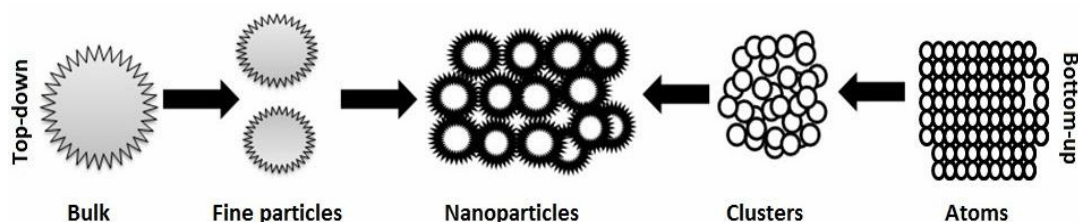


Fig. 1. Approaches for the preparation of nanoparticles.

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A pioneer works on the synthesis of spherical and mono-dispersed silica particles were reported by Stöber *et al.* (4, 5, 6). Silica particles with the size ranging from 5 to 2000 nm from aqueous alcohol solutions of silica alkoxides in presence of ammonia as catalyst (basic condition) have been produced. Following that many contemporary research works describing the synthesis of nanosilica particles are indeed evolved from the Stöber method. The main advantage of Stöber method is the ability to form mono-dispersed spherical silica particles compared to the acid-catalyzed systems which usually produce gel structures.

Models proposed for the synthesis of SNPs

Hydrolysis and polymerization of silicon alkoxides in the presence of ammonia lead to the formation of a stable suspension of particles. Two models have been proposed to describe the growth mechanism of silica namely monomer addition (7,8) and controlled aggregation (9).

Coating of textile materials with silica nanoparticles

Extensive research has recently been conducted to use silica nanoparticles for the functional textiles, including improved stability against mechanical or thermal destruction, improved water, oil and soil repellence, changing of light absorption of textiles, improved electric conductivity of textiles, immobilization and controlled release of bioactive species, oils and flavors (10).

For example, Jain Swapnal *et al.* (11) treated polyester blended fabrics with the silica nanoparticles and commercially available water repellent agent to impart super hydrophobicity. Hongxia Wang *et al.* (12) produced super hydrophobic surfaces with water contact angles over 170 degrees by a particulate silica sol solution of co-hydrolyzed TEOS/fluorinated alkyl silane with $\text{NH}_3 \cdot \text{H}_2\text{O}$ on various substrates, including textile fabrics (e.g. polyester, wool and cotton), electrospun nanofibre materials, filter papers, glass slides, and silicon wafers.

In this work, silica nanoparticles in the solid powder form were prepared from TEOS precursor, further application of these silica nanoparticles to the textile surface is carried out. Then previously modified fabrics were printed by screen and transfer printing techniques. The work aimed at improving the fabrics printability as well as acquiring antibacterial antimicrobial properties.

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Experimental

Materials

Fabrics

- Polyester/wool (PET/W)

Polyester/wool [55: 45] knitted fabric of 165 g/m², with plain 1/1 weaving structure, supplied by a private sector company.

- Polyester/cotton (PET/C)

Polyester/cotton [35: 65] knitted fabric of 122 g/m², with plain 1/1 weaving structure, supplied by a private sector company.

- Polyester.

Polyester (PET) knitted fabric of 150 g/m², supplied by a private sector company.

- Cotton

Cotton knitted fabric of 158 g/m² with plain 1/1weaving structure, supplied by Private Sector Company.

-Silane precursors

Tetraethoxysilane (**TEOS**) was used.

Dyestuffs and auxiliaries

Dyestuffs

It is clear from Table (2) that the synthesized dye possess good inhibition of the bacteria growth against the tested Gm positive and Gram negative bacteria and the inhibition zone diameters obtained are in the range of (21) mm which is quite good compared to control value of 0 (20).

Auxiliaries

• **Thickeners**

Two types of thickeners were used

-Commerical sodium alginate thickener with medium viscosity supplied by a pharmaceutical chemicals company.

-Commerical synthetic thickener (Leuco print) supplied by BASF company.

• **Binder.**

Commerical binder SME-2 from DAICO for Chemicals Industries Company.

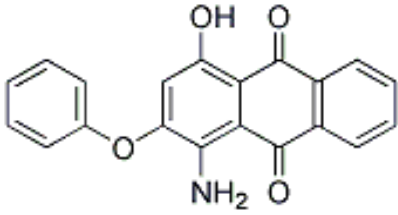
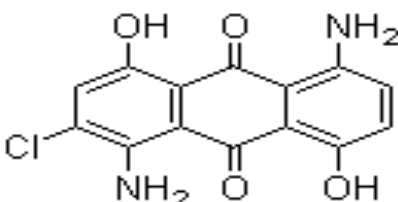
• **Urea.**

Commerical urea from El-Nasr Pharmaceutical Chemicals Company.

• **Solvents.**

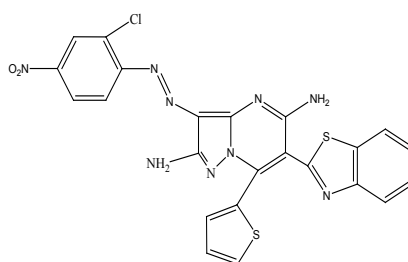
Commerical solvents (acetic acid, ethanol and ammonia solution from pharmaceutical chemicals company were used.

TABLE 1. Dyestuffs used:

Dye name	λ_x	Chemical structure	Supplier
C.I. Disperse Red 60	525		Sun colors® company
C.I. Disperse Blue 56	585		Sun colors® company

Synthesized dyes *

Synthesized Dye
 3-(2-(2-chloro-5-nitrophenyl) diazenyl 5 (benzo [d] isothi -azol-3-yl)-7-(thiophen-2 yl) pyrazolopyrimidin-2-amine. Brown crystals, m.p. $\geq 300^\circ\text{C}$; yield: (85%)



* The synthesized dye was prepared earlier ⁽²⁰⁾, and were found to posses antibacterial properties which were evaluated and found to be as follows in Table II.

TABLE 2. Inhibition zone diameter of the synthesized dye against gram positive and gram negative bacteria, expressed as (mm):

Dye sample	Inhibition zone diameter (mm)	
	EsherichiaColi	Staphylococcus Aureus
	(G-)	(G+)
Tetracycline	30	30
Control	0.0	0.0
Synthesized dye	21	21

Methods

Preparation of silica nanoparticles

Silica nanoparticles were prepared by adding a mixture of ethanol (2.7 mol) and ammonia solution (0.75 mol) dropwise to TEOS (0.17 mol) precursor. Then, the final mixture was stirred overnight, centrifuges and decanted, and the solid particles were dried at 70°C (4,5).

Coating with silica nanoparticles

For coating the blend fabrics with silica nanoparticles, the fabric samples were soaked for 10 min in 1% dispersion containing (3% synthetic binder) along with synthesized silica nanoparticles. Then, the fabrics were padded to wet pick-up of 80 %. The padded fabric samples were then dried at 60°C for 10 min and then cured at 150 °C for 5 min, followed by rinsing and drying.

Characterization of modified fabrics:

Fourier-Transition Infrared Spectroscopy (FTIR)

Infrared spectroscopy is used to identify chemical compounds, mixtures, extent of reaction, and molecular structure. Different chemical compounds absorb infrared radiation at frequencies corresponding to their own molecular vibrational frequencies(13). The modified fabrics samples were examined by a Pye-Unicam spectra-1000 machine to determine the functional groups on the surface of the samples. Potassium bromide (KBr) disc was used.

Scanning electron microscope (SEM)

Fabric samples were located on copper coated carbon tap double face, and then coated by the gold layer by vaporization of gold in argon atmosphere using sputter coater. The surfaces of samples were scanned using (Quanta FEG250). The magnification was set at 200,400 to 1700. All samples were scanned at room temperature.

Printing of modified fabrics

In this work, two techniques of textile printing were used :

- Silk screen printing.
- Transfer printing.

Silk screen printing

- Print paste recipe.

The printing paste was prepared according to the recipe shown in Table 3

Fixation technique

Print fixation was performed by steaming for 20 min at 102 °C in a high- temperature steamer.

Transfer printing

Printing paste as in Table 3

Transfer paper

Uncoated paper of 80 g/m². The transfer paper was manually printed with the previous formulation using silk screen and then air dried.

Characterization methods of printed fabrics

Color strength measurements.

The color strength (K/S) of each printed sample was measured using a Data Color SF 600 plus Colorimeter using a measured area with diameter of 9mm.

Antibacterial test

The antibacterial properties were quantitatively evaluated by Bacterial colony count according to the AATCC test method 100-1999. Two non-spore forming bacteria were used, one Gram-positive *Staphylococcus aureus* (and one Gram negative *Escherichia coli*). The colonies of the bacterium on the agar plate were counted and the reduction rate of bacteria by the modified fabrics is calculated using the following equation.

$$\text{Reduction rate (R)} = \frac{B - A}{B} \times 100$$

Where R is the % reduction of bacterial colonies, B is the number of the bacterial colonies of the unmodified fabric (control) and A is the number of bacterial colonies after 18 hours in contact with modified fabric (14,15).

TABLE 3. Disperse dye printing paste:

Dye	3 g
Synthetic thickener (leuco print)	3 g
Water	94 g
	100 g

Fastness properties

Washing, rubbing, Perspiration and light fastness are evaluated for unmodified and modified printed fabrics.

Results and Discussion

Characterization of silica nanoparticles surface modified blend fabrics:

FTIR:

Fig. 2 shows the FTIR spectrum of the synthesized silica particles. It shows absorption bands arising from asymmetric stretching vibrations of Si-O-Si (1125 cm^{-1}), and asymmetric vibration of Si-OH (850 cm^{-1}). The absorption band 2950 cm^{-1} (CH₃) group and 2870 cm^{-1} (CH₂)

can be used to identify the presence of unreacted TEOS in the silica particles.

The FTIR spectra of the unmodified and silica particles modified PET|cotton and PET|wool fabrics were done and are represented by Figs (3,4).

The FTIR spectra of the unmodified, modified PET|cotton and PET| wool fabrics with silica particles were done to determine the chemical changes that could have occurred as a result of silica particles coating, Fig. 3 shows the FTIR spectra of the unmodified and modified PET|cotton fabrics with silica particles.

Fig. (3) shows the FTIR spectra of the unmodified PET/ cotton fabrics with silica particles.

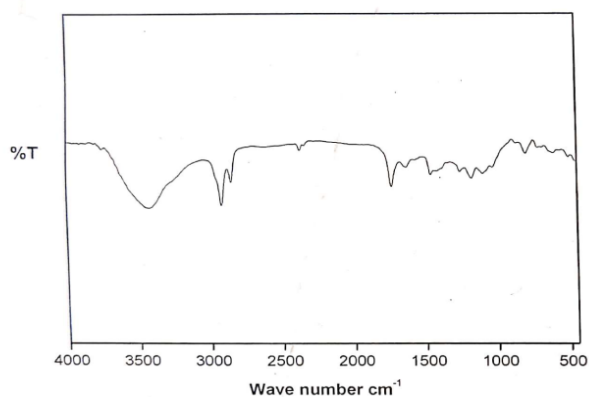


Fig. 2. FTIR spectra of silica particles obtained with TEOS prepared by Stöber method.

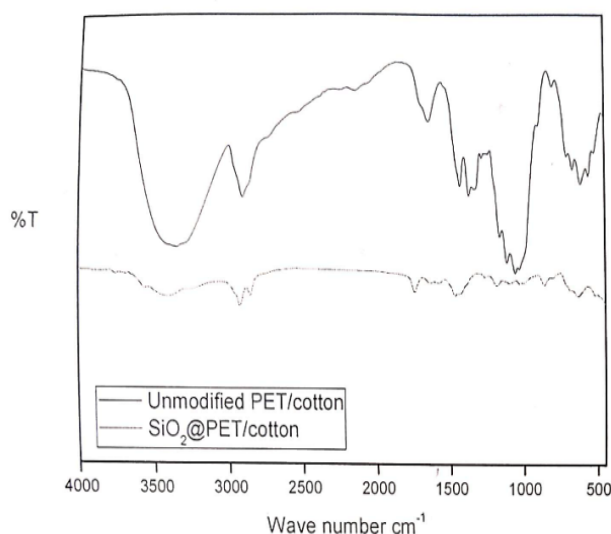


Fig. 3. FT-IR spectra of the unmodified and modified PET|cotton fabrics with synthesized silica particles.

All the absorption peaks are labeled with the wave number values, the spectrum of the unmodified PET/ cotton fabric was very complex because it included absorption bands characteristic of both cellulose and polyester. In the polyester component spectrum, the following bands were observed; an absorption band at 1720 cm^{-1} due to the C=O symmetric stretching vibrations of carbonyl group of the ester bond; and bands at $850, 793$ and 720 cm^{-1} caused by the C-H and C-C vibrations of the benzene ring. The absorption bands at $1370, 1338, 1240$ and 1095 cm^{-1} , belonging to the C-OH and C-O-C vibration of the polyester fibers, were blurred by the absorption bands characteristic of the cellulose component fingerprint region. Interference of the absorption bands of cellulose also occurred in the $2850\text{-}2600\text{ cm}^{-1}$ spectra region due to the stretching vibrations of CH_2, CH_3 and C-H, as well as in the absorption band region of $3500\text{-}3250\text{ cm}^{-1}$ characteristic of O-H stretching and of adsorbed water molecules.

Chemical changes that occurred on polyester and the wool components of fabrics by silica particles coating process were measured using FTIR spectra analysis of the unmodified and modified PET/ wool fabrics with silica particles. Fig. (4) represents FTIR spectra of the unmodified and modified PET/ wool fabrics with silica particles.

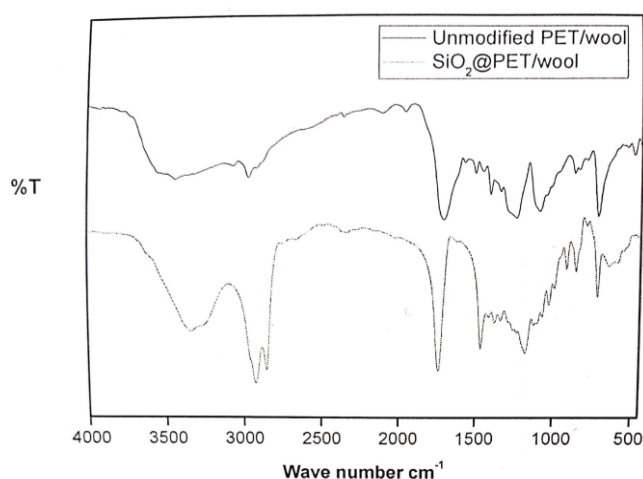


Fig. 4. FT-IR spectra of the unmodified and modified PET/wool fabrics with synthesized silica particles.

In the PET/wool spectrum, the strongest peaks occurring at around the following bands were observed. Absorption bands at 1690 cm^{-1} and 1511 cm^{-1} are attributable to primary and secondary amide (-CONH) respectively. It was also found that, the obvious new band at 1125 cm^{-1} assigned to Si-O-Si confirms the successful loading of silica particles onto PET/cotton and PET/wool.

Scanning Electron microscope

The morphology of the synthesized particles was investigated in terms of dimension, shape, and aggregation state by means of scanning Electron Microscope (SEM). Figure (5) shows the SEM image of the synthesized silica nanoparticles prepared by stöber synthesis method. The particles were almost smooth spheres with diameters about 150 nm.

Figure (6) shows the morphological changes induced by the treatment of PET/cotton, PET/wool with prepared silica particles compared with the untreated fabrics. It can be observed that, the micrographs show silica particles deposited on surface of the different fabrics.

Printing of silica nanoparticles modified fabrics

In the process of formation of silica coatings on the fabrics the SiOH in a silica particle form network containing silanol and siloxane groups which are usually interconnected to the fabrics by thermodynamic interaction, e.g. dipolar-dipolar (16) and hydrogen bond interaction (17).

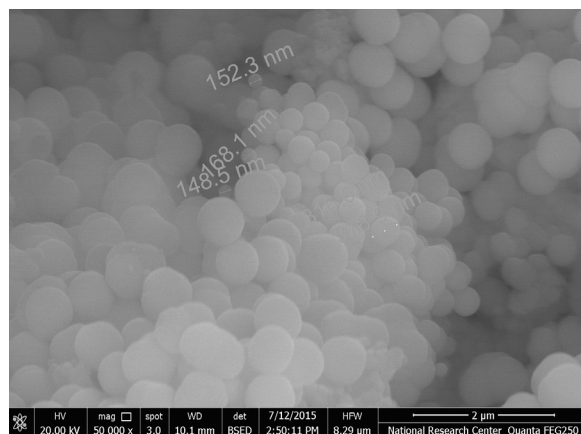


Fig. 5. Scanning electron micrograph of silica nano particles obtained from TEOS prepared by stöber synthesis.

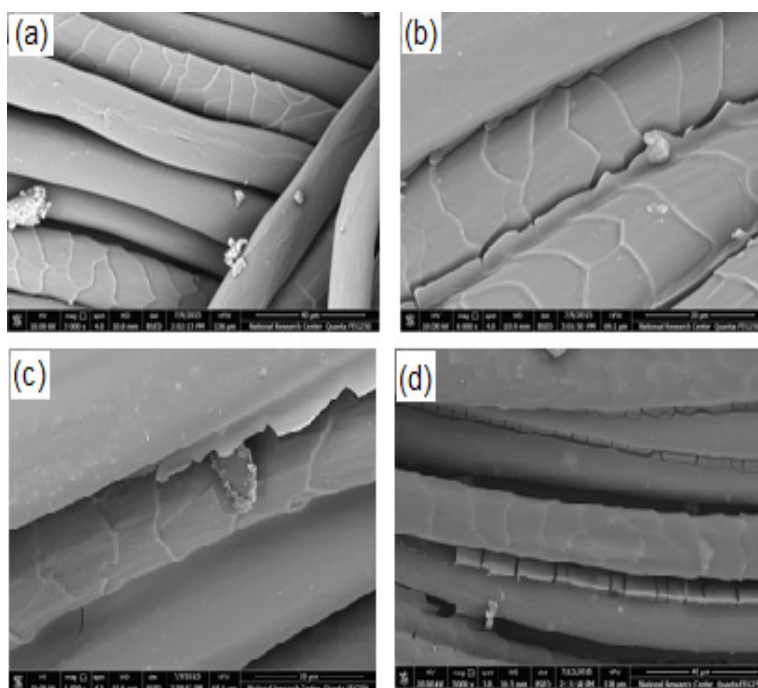


Fig. 6. Scanning electron micrographs of unmodified PET/cotton (a), modified PET/cotton (b), unmodified PET/wool (c), and modified PET/wool with silica particles.

Consequently stronger adhesion of the coating to the fabric will result from stronger interaction. Although cellulosic fibers are composed of polar molecular chain, there are some hydrogen bond interactions between the molecules in the cellulosic chains which will decrease to some extent the rate of thermodynamic interaction of the silica gel with the cellulose fabrics. In case of polyester fabrics with polar chain structure, they have a much higher hydrogen bond and dipolar-dipolar interactions (16, 18, 19).

Silk screen printing

Two disperse dyes were used (Disperse Red 60 and Disperse Blue 56) .

Figs (7, 8) represent the color strength of unmodified and modified screen printed PET/W and PET/C silica nanoparticles using Disperse Red 60 and Disperse Blue 56 respectively.

The color strength results of the unmodified, screen printed PET/W and PET/C fabrics are remarkably lower when compared to those of the modified fabrics by the silica nanoparticles for both dyes highlighting the effect of modification treatments.

The results show that, there are some similarities, to a certain extent in the transfer

printing behavior of PET/W and PET/C fabrics. An explanation of the high color strength values for prints of nanoparticles modified PET/W and PET/C blends could be due to that the nanoparticles have large surface area and they can intrude more easily into the interior of cotton and polyester fabrics.

It can also be seen that the color strength results of unmodified and nanoparticles modified

screen printed cotton and polyester fabrics using disperse red 60 and disperse blue 56 Figs. (9,10) have similar trends to a certain extent with the blend fabrics, with the exception that, the prints of unmodified polyester fabrics possess much higher color strength values when compared to the unmodified prints of cotton fabrics, this is of course due to the known affinity of disperse dyes for polyester and almost no affinity for cotton.

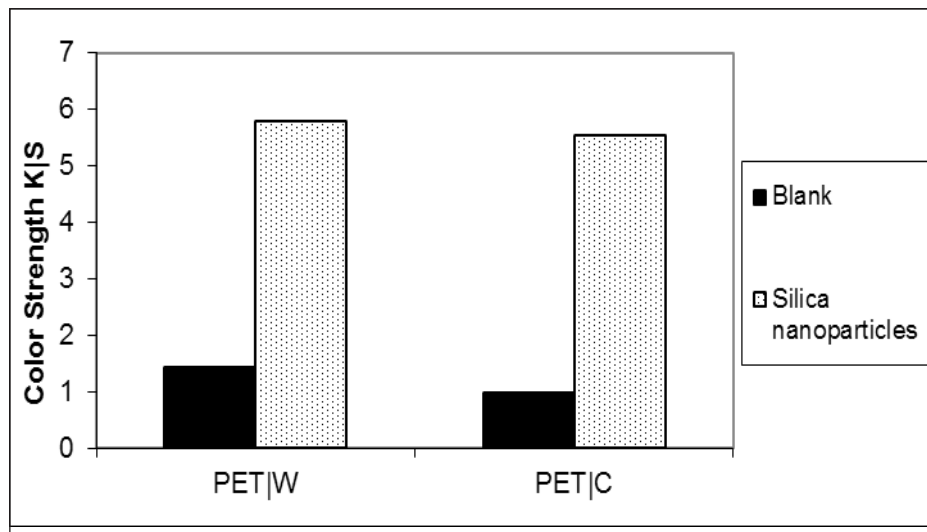


Fig. 7. Color strength of “Silica nanoparticles” modified, then screen printed* blend fabrics.

*Using disperse red 60

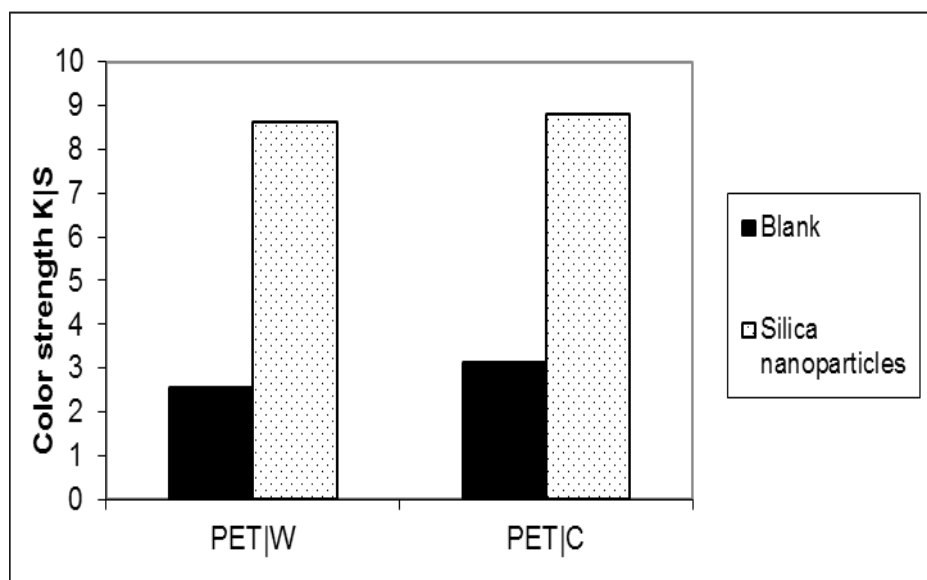


Fig. 8. Color strength of “Silica nanoparticles” modified, then screen printed* blend fabrics

*Using disperse blue 56.

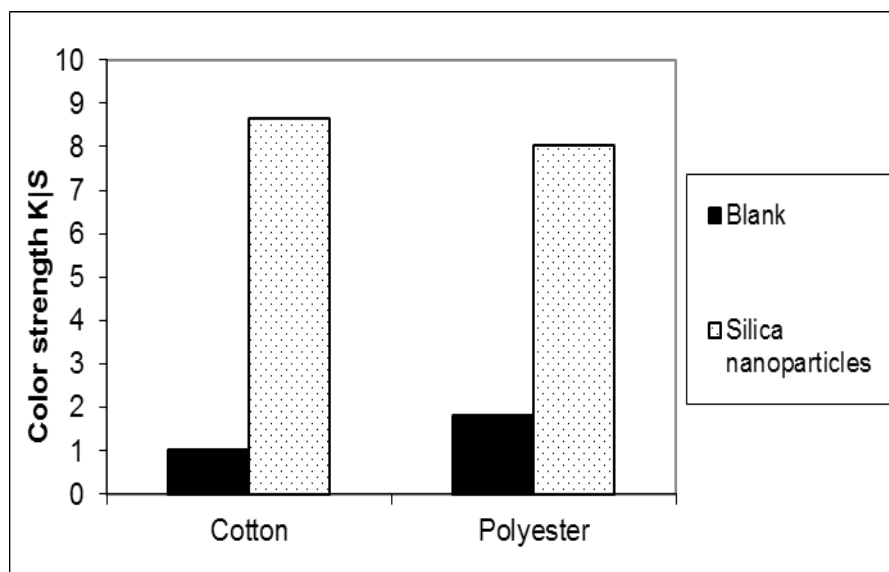


Fig. 9. Color strength of “Silica nanoparticles” modified, then screen printed* single fabrics.

*Using disperse red 60.

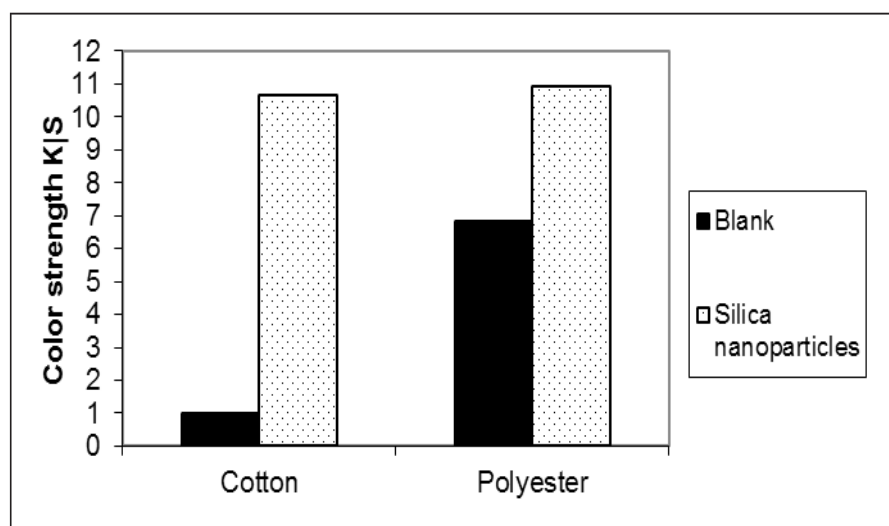


Fig. 10. Color strength of “Silica nanoparticles” modified, then screen printed* single fabrics.

*Using disperse blue 56.

Transfer printing

The same two disperse dyes were used to transfer print the unmodified and modified PET/W, PET/C, cotton and polyester. Fabric modification was carried out using silica nanoparticles prepared from TEOS precursor.

Figs. (11-14) represent the color strength of unmodified and silica nanoparticles modified, transfer printed PET/W, PET/C, cotton and polyester using disperse red 60 and disperse blue

56 respectively. Transfer conditions of 190°C and 30 sec were used. Again, similar trends to those obtained with the corresponding modified, screen printed fabrics were obtained.

The modified fabrics usually undergo curing treatment before the printing process. A trial to omit the curing step and conducting simultaneous printing and curing in one step by either the screen or the transfer printing techniques using disperse red 60 was carried out.

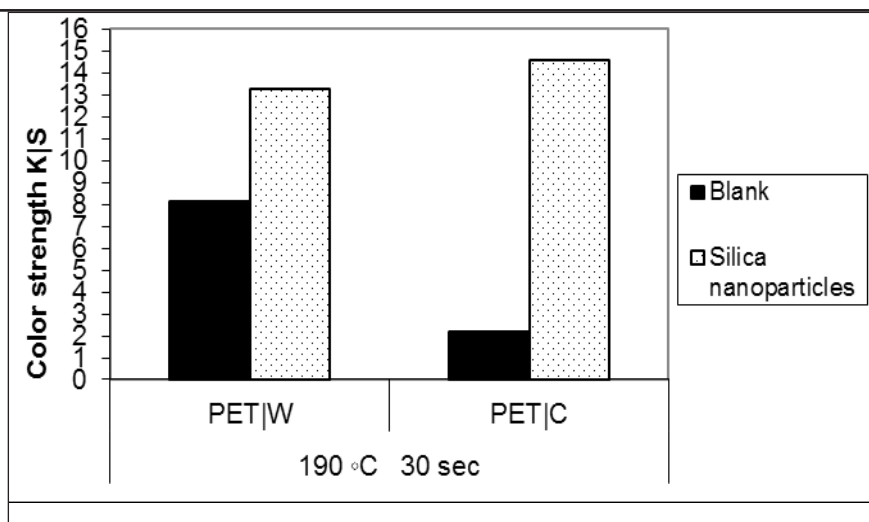


Fig. 11. Color strength of “silica nanoparticles” modified, then transfer printed*blend fabrics.

*Using disperse red 60.

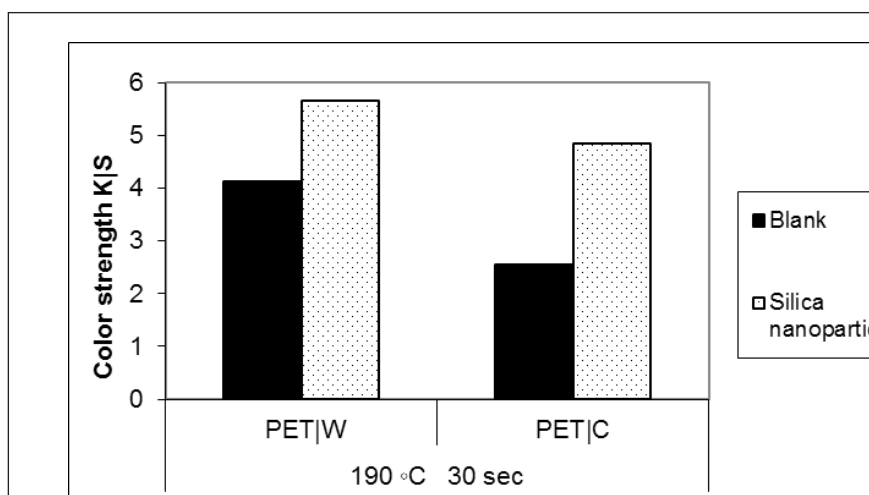


Fig. 12. Color strength of “silica nanoparticles” modified, then transfer printed*blend fabrics.

*Using disperse blue 56.

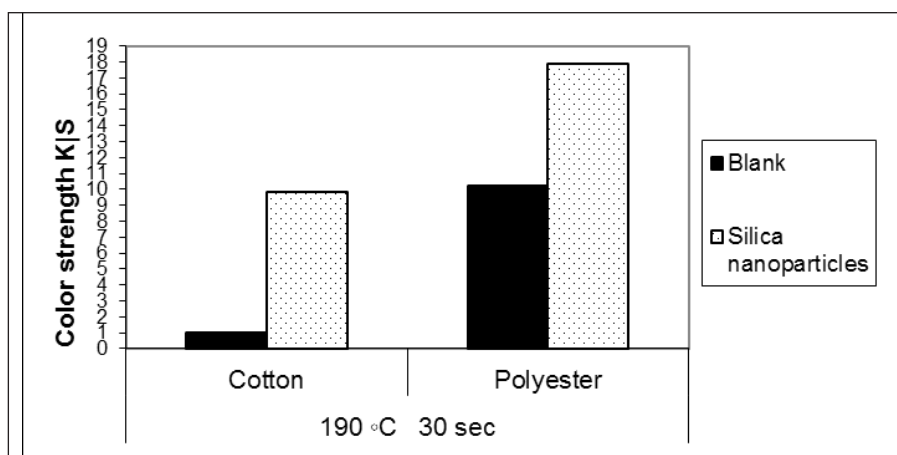


Fig. 13. Color strength of “silica nanoparticles” modified, then transfer printed*single fabrics.

*Using disperse red 60.

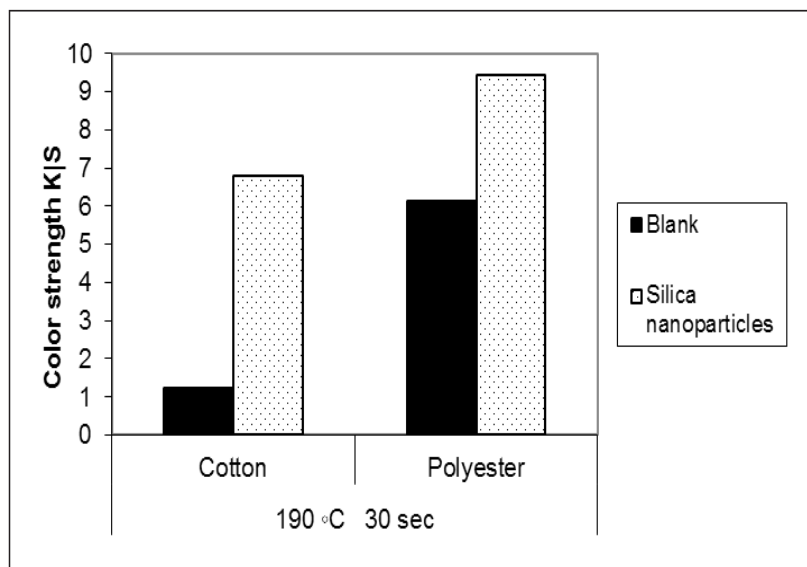


Fig. 14. Color strength of “silica nanoparticles” modified, then transfer printed*single fabrics.

*Using disperse blue 56.

Printing of modified, uncured and cured fabrics with silica nanoparticles using disperse dyes

The color strength results of modified, uncured and cured screen printed PET/W, PET/C, cotton and polyester fabrics with silica nanoparticles using disperse red 60 are represented by Figs (15, 16).

It can be seen that, curing of all the modified fabrics prior to the screen printing operation did not, almost affect the color strength results implying that, simultaneous curing and printing could occur in one step, resulting in time and energy saving.

Similar trends were observed in the case of simultaneous curing and transfer printing PET/W, PET/C, Cotton and polyester fabrics with silica nanoparticles in one step using disperse red 60 Figs. (17, 18).

Transfer printing of (silica nanoparticles) modified fabrics with synthesized antibacterial dye

PET/W and PET/C as well as cotton and PET fabrics were modified with nanoparticles from TEOS and subsequently transfer print them with a previously synthesized disperse dye ⁽²⁰⁾ having antibacterial properties in order to achieve modified fabrics with antibacterial behavior.

Evaluation of the antibacterial behavior of transfer printed modified fabrics with nanoparticles prepared from TEOS

Several standard methods to assess antibacterial activity on textiles exist. These standard methods are classified on the basis of the type of evaluation of microorganism inhibition (qualitative and quantitative). Qualitative methods are fast when many samples have to be tested. Quantitative methods are based on the reduction of the microorganism population ^(21,22). They are time consuming methods.

The antibacterial activity of silica nanoparticles modified, transfer printed PET/W and PET/C fabrics was quantitatively evaluated. The antibacterial performance of these fabrics is investigated against Gram-negative bacterium *Escherichia Coli* and Gram-positive *Staphylococcus aureus*.

The Results of the antibacterial activity gained by PET/W and PET/C modified fabrics are presented in tables 2. They show the bacterial growth reduction rate of (silica nanoparticles modified), transfer printed PET/W and PET/C fabrics against both *Escherichia Coli* and *Staphylococcus aureus*, in comparison with their unmodified ones.

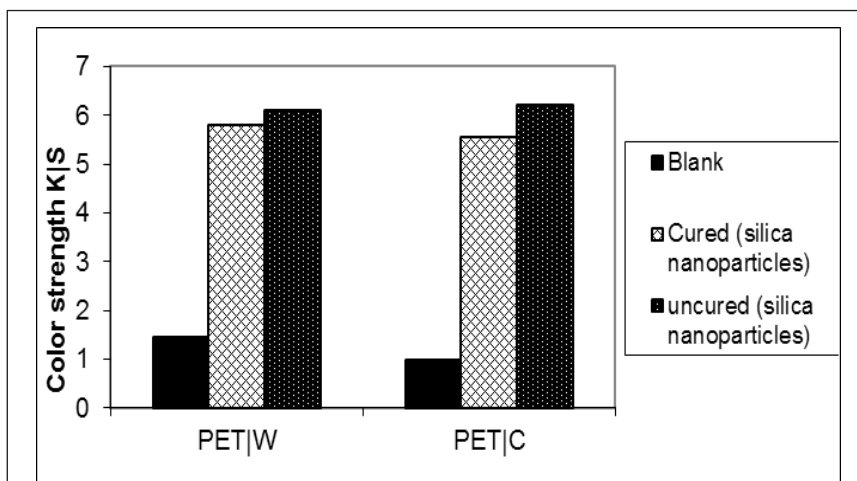


Fig. 15. Color strength of cured and uncured (silica nanoparticles) modified, then screen printed * blend fabrics

*Using disperse red 60

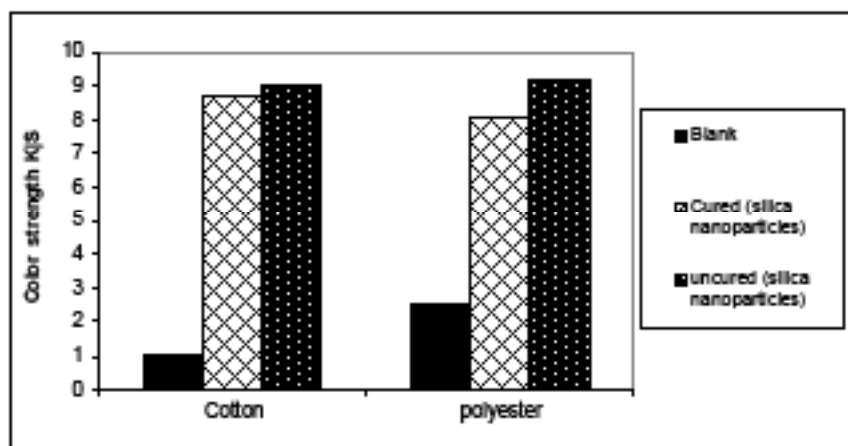


Fig. 16. Color strength of cured and uncured (silica nanoparticles) modified, then screen printed * single fabrics.

*Using disperse red 60

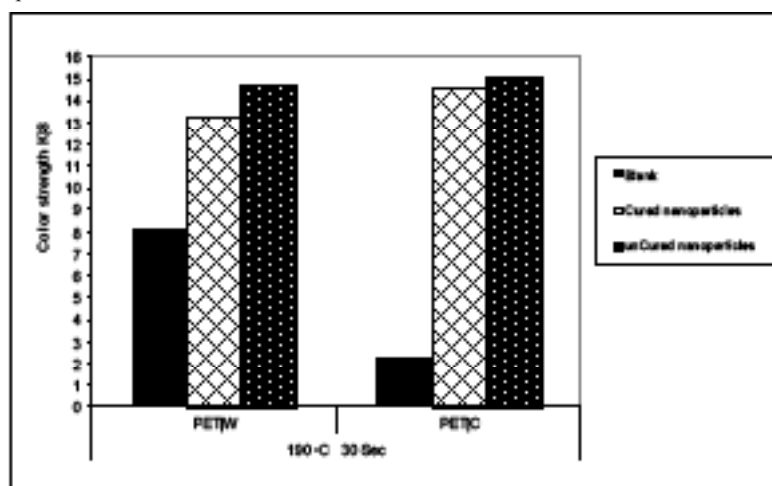


Fig. 17. Color strength of with cured and uncured (silica nanoparticles) modified, then transfer printed * blend fabrics.

*Using disperse red 60

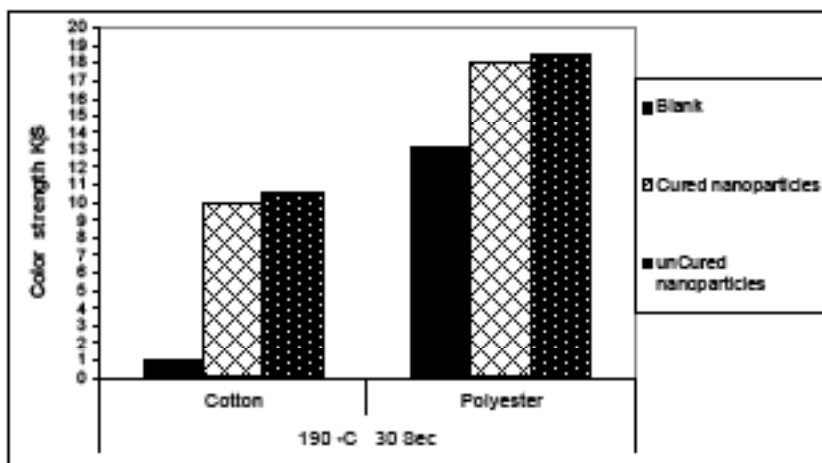


Fig. 18. Color strength of with cured and uncured (silica nanoparticles) modified, then transfer printed *single fabrics .

*Using disperse red 60

Figs (19,20) represent the color strength of modified, transfer printed PET/W, PET/C, cotton and polyester fabrics with silica nanoparticles respectively. Transfer conditions of 190°C and transfer time of 30 secs were selected as optimum.

It could be concluded that, the adhesion of silica nanoparticles coating on the polyester substrates is due to thermodynamic affinity by dipolar-dipolar and hydrogen bond interaction. In case of cellulose substrates, covalent bonds form between the hydroxyl groups on cellulose and the end groups of silica precursor, producing a durable silica coating. It is also certain, that the adhesion of these coats on the different fabrics surface is influenced principally by their structure and chemical composition.

Fastness properties

One of the main concerns of the textile dyers & printers is the fastness properties of the dyestuffs in the market. Therefore, evaluation of dye fastness properties modified, and subsequently printed fabrics in comparison with the corresponding unmodified and printed fabrics is considered of at most importance.

The fastness properties of unmodified and silica nanoparticles modified, Transfer printed PET/W and PET/C fabrics with disperse red 60 and disperse blue (selected as examples) are represented by Tables 4 and 5 respectively.

In general, the fastness properties of the modified and printed blends of PET/W, PET/C, fabrics range from good to excellent ratings with disperse dyes used as compared to the corresponding values for unmodified and printed fabrics which range from moderate to very good in some cases.

Conclusion

Surface modification of textile blend fabrics as well as cotton and polyester fabrics by silica nanoparticles via the promising, simple and valuable sol-gel technique using metal alkoxide precursor (TEOS) produced modified fabric blends (PET/W and PET/C) that could be printed successfully with one class of dyes (disperse dye) using the proper selected printing technique, as well as modified, printed cotton and polyester fabrics with increased printability properties. Multi-functional dyes with antibacterial properties could also be used providing functionality to those fabrics.

The fastness properties of all the modified, printed fabrics (light, washing, rubbing, perspiration) range from very good to excellent ratings. The above achievements help in solving blend fabrics coloration problems.

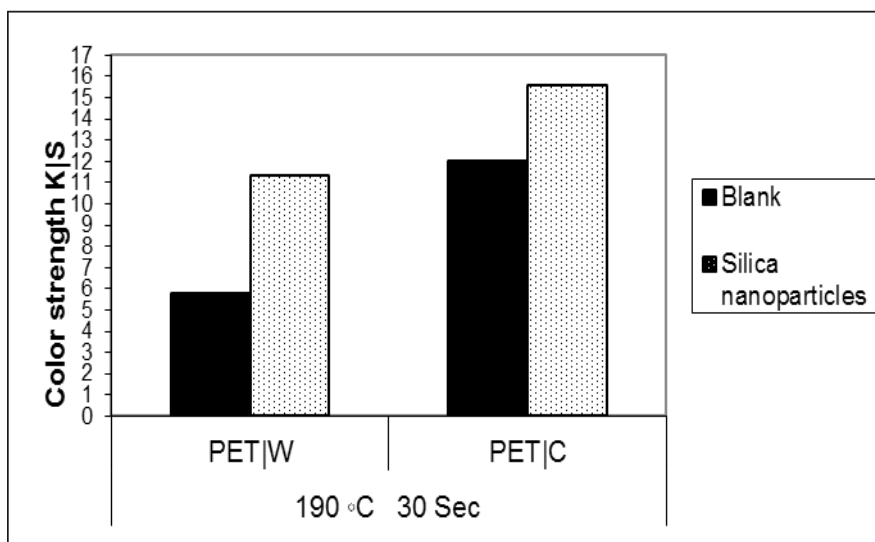


Fig. 19. Color strength of (silica nanoparticles) modified, then transfer printed * blend fabrics .

* Synthesized dye.

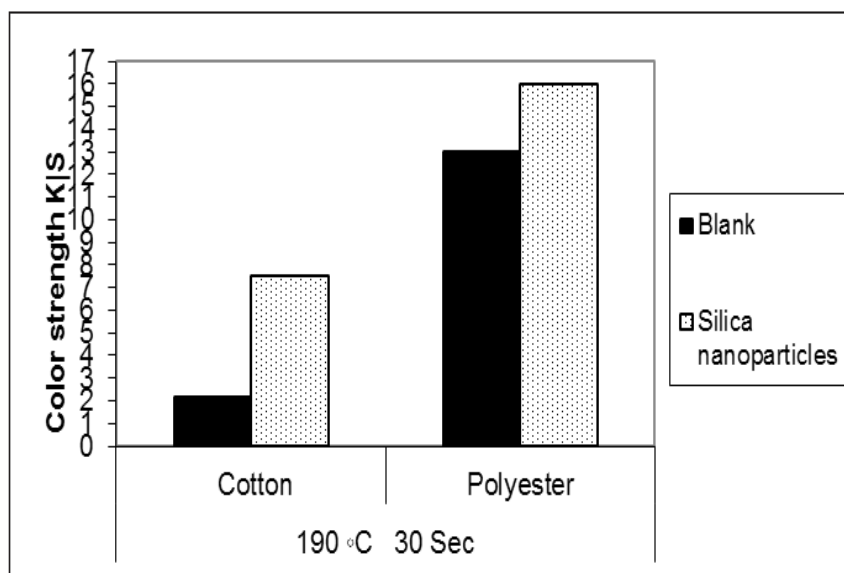


Fig. 20. Color strength of (silica nanoparticles) modified, then transfer printed * single fabrics .

* Synthesized dye.

TABLE 4. Fastness properties and color strength of unmodified and silica nanoparticles modified, (Transfer printed * PET/W fabrics).

		Rubbing fastness		Washing fastness		Perspiration fastness				Light fastness
		Dry	Wet	Alt	St	Alkali		Acid		
						Alt	St	Alt	St	
Disperse red 60	Silica nanoparticles modified	4	4	3-4	4	4	3-4	4	4	3-4
	Unmodified	3-4	3	3	2-3	2-3	3	3-4	3	4
Disperse blue 56	Silica nanoparticles modified	4	4	4	4	5	5	3-4	4	4
	Unmodified	4	3-4	3-4	3	3-4	3	3-2	3	3-4

*Using Disperse red 60 and disperse blue 56.
Transfer printing temperature 190 °C for 30 Sec.

TABLE 5. Fastness properties and color strength of unmodified and (silica nanoparticles), (Transfer printed * Polyester/ cotton fabrics).

		Rubbing fastness		Washing fastness		Perspiration fastness				Light fastness
		Dry	Wet	Alt	St	Alkali		Acid		
						Alt	St	Alt	St	
Disperse Red 60	Silica nanoparticles (modified)	4	4	4	3-4	4-5	5	5	5	5
	Unmodified	3-4	3	3	2-3	2-3	3	3-4	3	4
Disperse blue 56	Silica Nanoparticles (modified)	4	3-4	5	5	5	5	5	5	4
	Unmodified	4	3-4	3-4	3	3-4	3	3-2	3	3-4

*Using Disperse red 60 and disperse blue 56.
Transfer printing temperature 190 °C for 60 Sec.

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التحوير السطحي لبعض الأقمشة المخلوطة عن طريق جزيئات السيليكا النانوية لتحسين خواص الطباعية

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توفر تقنية السول-جل طريقة فعالة لتعديل الأقمشة ، ولها العديد من المزايا. في هذه الورقة البحثية ، تم تحضير جزيئات السيليكا نانو في الشكل الصلب منماده TEOS (Tetraethoxysilane) ؛ يتم تنفيذ المزيد من تطبيق هذه الجسيمات النانوية السيليكا على الأقمشة المخلوطة من الصوف والبوليستر والأقمشة القطنية والبوليستر. حيث يظهر التعديل تحسناً في قابلية طباعة القماش ، بالإضافة إلى الحصول على خصائص مضادة للبكتيريا. الفكرة الأساسية هي استخدام تقنية السول-جل لتعديل أسطح الأقمشة المخلوطة من أجل حل مشاكل تلويثها والوصول إلى أقمشة طباعية باستخدام نوع واحد الأصباغ.