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Research Paper

Study on Structural, Mechanical and Functional Properties of Polyester Silica Nanocomposite Fabric

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Abstract: *Polyester silica nanocomposite fabrics were prepared by incorporating different concentrations of silica nano particles to the fabric by pad-dry-cure method. The prepared composite fabrics were analyzed in terms of change in their mechanical, structural as well as their functional properties. The surface morphology showed the uniform distribution of silica nano particles on fabric through SEM, FTIR showed the presence of silica particles. Some improvement in mechanical properties observed like tensile, tearing and crease recovery angle. Improvement in functional properties observed like antimicrobial property, self cleaning, electrical surface resistivity and dyeing behavior. The most significant part of the study was that the treatment improves performance of polyester fabric without affecting the COD, BOD and TDS values.*

Keywords: SEM, FTIR, mechanical property, structural property, functional property, environmental aspects.

1. Introduction:

Nanotechnology is the art and science of the design, characterization, production and application of structures, devices and systems by controlling shape and size on the nanoscale[1]. The nano-structured materials is the most emerging field of science and engineering. It includes metals, metal oxides, silicates, carbon products like graphite and carbon nano-tubes (CNTs). These materials can be used either as filler to obtain nano-composite fibers or deposited onto the surface. Metals and metal oxides are important classes of nano-materials [2, 3, 4].

It has been established in recent years that polymer-based composites reinforced with a small percentage of strong fillers can significantly improve the mechanical, thermal and barrier properties of the pure polymer matrix[5-8]. Moreover, these improvements are achieved through conventional processing techniques without any detrimental effects on process ability, appearance, density and aging performance of the matrix. Now-a-day's hybrid polymers, which are the hybrid structure of inorganic-organic nano-composite materials are being used to impart the combination of scratch resistance with dirt-repellent effect, high transparency, special barrier properties or antimicrobial function to the material [9-12].

From the available literature it has been observed that the interest in using nanotechnology in the textile industry is increasing. Earlier, we have prepared polyamide silica nano composite film for high performance application[13]. In this work an attempt has been made to apply SiO_2 nano particles to polyester fabric by pad-dry-cure technique. The chemical transformations have been analyzed using Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The change in mechanical properties has been evaluated in terms of tensile strength, tearing strength and crease recovery angle. The nano silica treated polyester fabric has been evaluated for change in its functional properties i.e. antibacterial, self cleaning, electrical surface resistivity and dyeing with disperse dye. Finally, the washing effluent of treated and untreated fabric was analyzed in terms of Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and Total Dissolved Solid (TDS).

2. Materials and Methods:

2.1 Materials

2.1.1 Fabric

The fabric was mild scoured using 5% soap i.e. Lissapol L and 2% soda ash at boil for 15 minutes before nano silica application. The specifications of polyester fabric are given in table 1. The fabric was procured from local textile market.

Table 1: Fabric specifications

Sample	Material Specification				
	Denier Warp/Weft	Fabric Setting Ends/cm, Pick/cm	Weave	GSM	Thickness (mm)
100% Polyester	128d/146d	36/28	Plain	109.7	0.21

2.1.2 Chemicals

Silica (SiO_2) nano particles with average size less than 100 nm, Polyacrylamide, Lissapol L and all other chemicals were of LR grade were used without further purification. Disperse dye (Disperse Nevy blue 3G) was used to dye the polyester fabric. Antimicrobial test chemicals: Gram-positive Staphylococcus aureus (Lab collection) was used in antimicrobial test as per ASTM E-2149 METHOD. Environmental impact test chemicals (BOD, COD, TDS): Standard $K_2 Cr_2 O_7$ (0.25 N), H_2SO_4 reagent, Standard ferrous ammonium sulfate, Mercury sulfate crystals and Ferroin indicator solution. Self-cleaning effect: Coffee (Nescafe).

2.2 Experimental Methods

2.2.1 Application of Nano SiO_2 on Polyester Fabric

Application of silica nano particles in different concentrations was done on polyester fabric by pad-dry cure method.

a) Preparation of silica nano padding liquor: Nano silica solution was prepared using 1gpl, 2.5 gpl, and 5 gpl concentrations. For 1 gpl solution, 0.1 gm nano particle was added with 5 gm Lissapol L surfactant and 10gm polyacrylamide binder. The mixture was then stirred using magnetic stirrer at 250 rpm for 30 minutes at 60^{o}C temperature. Likewise all concentration solution was prepared.

b) Application to fabric: Polyester fabric was immerged in padding liquor at room temperature for 10 minutes and then passed through a two bowl laboratory padding mangle, which was running at a speed of 15 rpm with a pressure of 1.75 Kg/cm^2 using 2-dip 2-nip padding sequence at 70% expression. The padded substrate was dried at 80^{o}C for 5-6 minutes and cured in a preheated curing oven, at 140^{o}C temperature for 3 minutes.

2.2.2 Testing and Analysis

a) Evaluation of surface morphology and structural compositions: Scanning electron microscopy (SEM) model JSM-5610 LV Japan with Oxford Inca software and Fourier transform infrared spectroscopy (FTIR) model Nicolet iS10 FT-IR spectrometer (Thermo Scientific, Japan) were used to study the surface morphology and structural compositions.

b) Measurement of mechanical properties: Lloyd LRX tensile strength tester, UK, Elmendorf tearing tester and crease recovery tester were used to study the mechanical properties.

c) Analysis of functional properties: For testing functional properties like antimicrobial, self cleaning, electrical surface resistivity and dyeing behavior of treated and untreated polyester fabric, the instruments used were Incubator cum oven; Laminar air flow model HIPL-042, Autoclave equitron, Shaker incubator, UV spectrophotometer, Spectra scan 5100 (RT) premier color scan instrument, KEITHLEY-614 Electrometer MET/K/16B, USA.

d) Testing of environmental effect: Environmental aspect was studied through COD, BOD and TDS test equipments like condenser, titration equipments.

3. Results and Discussion:

Polymer silica nanocomposite fabrics were prepared by incorporating different concentrations of silica nano particles to polymeric fabric i.e. polyester fabric by pad-dry-cure method. The prepared composite fabric was analyzed in terms of change in their mechanical, structural, thermal as well as functional properties and their comparison was done with the pure polyester fabric.

3.1 Effect on Surface Morphology and Structural Compositions

3.1.1 Surface Morphology by SEM

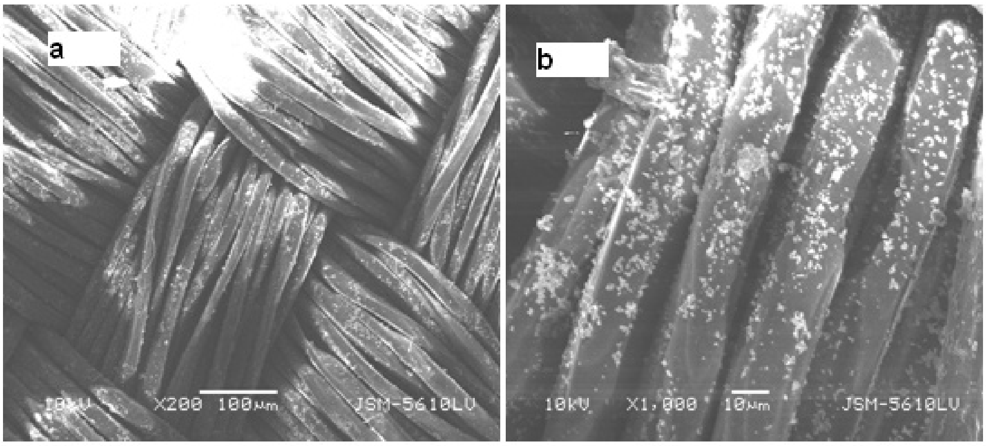

Figure 1: SEM microphotographs of polyester fabric treated with silica nano particle

The surface of the treated polyester fabric sample was observed on scanning electron microscopy. The result is shown in figure 1 (a, b), the nano scale silica particles can be clearly seen well distributed on the surface of polyester sample. The particle size plays a primary role in determining their adhesion to the fibre. It is reasonable to expect that the largest particle agglomerates will be easily removed from the fibre surface, while the smaller particle will penetrate deeper and adhere strongly into the fabric matrix.

3.1.2 Structural Compositions by FTIR

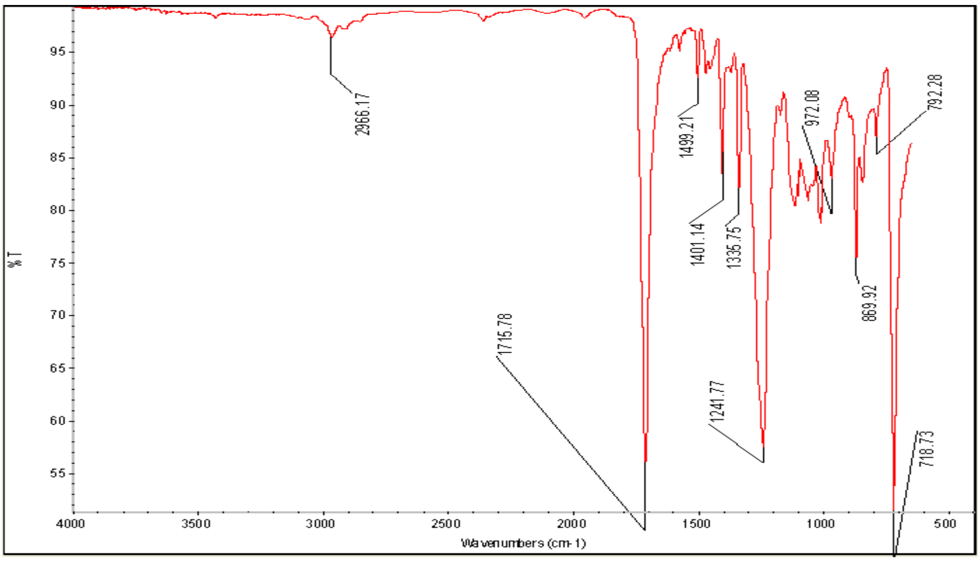

Figure 2: IR characterization absorption peak of pure polyester fabric

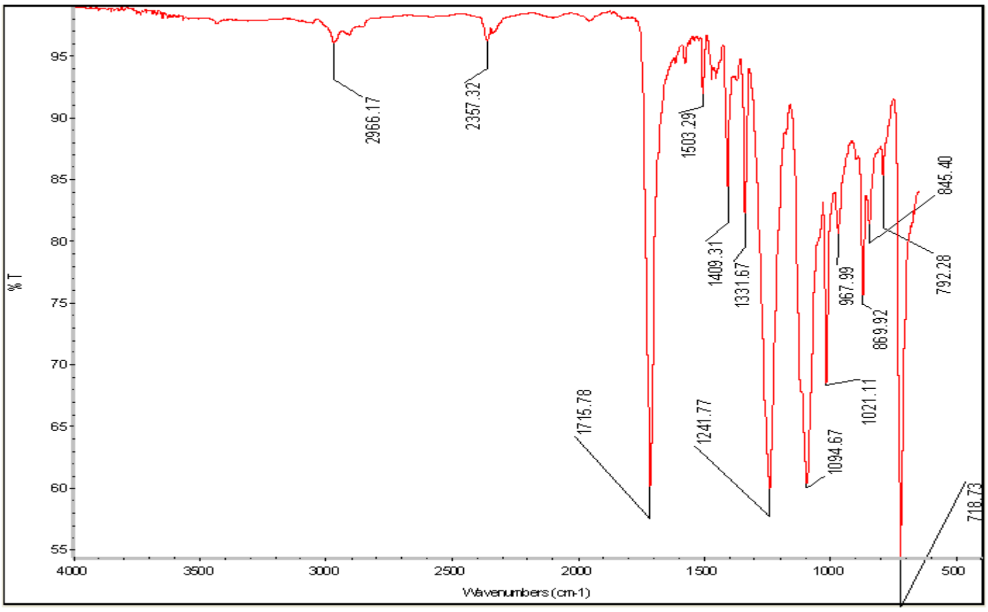

Figure 3: IR characterization absorption peak of 5 gpl nano silica treated polyester fabric

The peaks in the IR spectra of the polyester treated and untreated fabric as shown in figure 2 and 3 appeared in the range of 600-4000 cm^{-1}. The 1715 cm^{-1} shows C=O vibration, 1409 cm^{-1} of aromatic ring, 1331 cm^{-1} & 1021 cm^{-1} shows carboxylic ester or anhydride, 1021 cm^{-1} indicates the presence of O=C–O–C or secondary alcohol, 967 cm^{-1} is of C=C stretching, 869 cm^{-1} peak shows five substituted H in benzene. The main structure of the polyester sample had ester, alcohol, anhydride, aromatic ring and heterocyclic aromatic rings. Alcohol was able to react with anhydride and produce ester groups. This is a reason that there is still alcohol and anhydride as residual reactants left in the polyester.

The carboxyl, ester, anhydride and alcohol groups showed the polyester fabric was not pure PET. The peak at 1409 cm^{-1} corresponded to the aromatic ring which is a stable group. It was the characteristic absorption peak of PET. The peak at 1715 cm^{-1} was assigned to the ester group. The two main characteristic peaks of Si-O-Si bonds vibration modes were detected around 845 &1094 cm^{-1}, which are attributed to Si-O bending vibration band and Si-O-Si asymmetric stretching vibration band respectively in treated polyester sample, so it indicates that the silica nano particles are present in fibre matrix.

3.2 Effect of Nano Silica Treatment on Mechanical Properties

3.2.1 Tensile Strength

Table 2: Tensile strength of polyester fabric

Concentration of nano silica (grams/liter)	Tensile strength (Kgf)		% Strain	
	Warp way	Weft way	Warp way	Weft way
Untreated sample	99.84	73.86	31.46	34.2
1.0	101.7	77.66	29.61	39.89
2.5	103.4	82.15	30.72	40.67
5.0	104.9	85.04	31.20	36.74

The treated and untreated polyester samples were tested on Lloyd LRX tensile tester and the results are shown in table 2. The results show that the introductions of silica nano particle into the structure of the fibre cause an improvement in the load bearing capacity of the polyester fabric. This may be due to binding caused by introduction of silica nano particles into the polymer matrix. The warp way strength is found higher than the weft way tensile strength which may be due to high tension is kept during weaving in the warp yarns as compare to the weft yarns. The tensile strength is found increasing in both the directions as the concentration of silica nano particles increases. The percentage strain of warp and weft way has not affected much due to the nano silica treatment. But the overall strain percentage of weft way direction is higher than the warp way strain. This may be due to the less tension in weft yarns during weaving which may cause high crimp into the weft yarns.

3.2.2 Tearing Strength

The treated and untreated samples were tested on Elmendorf tearing tester. Table 3 shows result of tearing strength of treated and untreated polyester fabric. Polyester fabric sample show minor improvement in tearing strength compare to untreated sample. Also it is observed that as the concentration of silica nano particles increases, there is only 1 to 2 percentage increase in tearing strength, which is very minute. So, it can be interpreted that incorporation of nano silica particles does not affect the tearing strength of fabric. This may be due to the smaller size of the particle which may not be interfering the fiber matrix.

Table 3: Tearing Strength of polyester fabric

Concentration of nano silica (grams/liter)	**Tearing Strength (gf)**	
	Warp way	**Weft way**
Untreated sample	4352	3840
1	4388	3856
2.5	4412	3880
5	4458	3902

3.2.3 Crease Recovery Angle

The result given in table 4 shows, minor improvement in crease recovery angle of the treated samples. The silica nano particles because of their small size can enter in between the polymer molecules and perhaps act as filler or cross linking agent which also contributes to the load sharing phenomenon during load application to the material. Unlike chemical cross linking which cause an improvement in crease recovery angle at the cost of imparting some rigidity in the material to an extent depending on the extent of cross linking. The incorporation of nano silica particle remains quite gentle in this regard. There was little improvement in the property which proves that the particles penetrated in between the polymer chain molecules, do not interfere much to the polymer flexibility. Also the crease recovery property of fabric improves with increase in the concentration of silica nano particles.

Table 4: Crease recovery angle of polyester fabric

Concentration of nano silica (grams/liter)	**Crease Recovery Angle (^{o})**			
	Warp way		**Weft way**	
	30 Sec	**60 Sec**	**30 Sec**	**60 Sec**
Untreated sample	140	147	105	110
1	142	147	109	114
2.5	145	148	110	116
5	147	151	113	119

3.3 Effect of Nano Silica Treatment on Functional Properties

3.3.1 Antimicrobial Efficiency

Table 5: Antibacterial efficiency of polyester fabric

Concentration of nano silica (grams/liter)	Optical density of sample with S.aureus bacteria	Reduction of bacterial growth (%)
Untreated sample	0.882	Nil
2.5	0.802	9
5	0.750	15

The antimicrobial property of polyester treated and untreated samples was tested against S.aureus bacteria and the table 5 shows % reduction of bacterial growth. The result shows improvement in antimicrobial property of treated sample compared to untreated sample. It can also be observed that the antimicrobial property is increased with the increase in the concentration of silica nano particles.
The enhancement in the resistance towards bacterial attack may be attributed to the fact that the metallic ions and metallic compounds display a certain degree of sterilizing effect. It is possible that SiO_2 particles may get attached to the surface of the microbes cell membrane, enter inside the cell and destroy their metabolic function. Smaller SiO_2 particles having larger available surface area for interaction provides high bactericidal effect than larger SiO_2 particles.

3.3.2 Self Cleaning Property

Table 6: Self cleaning action of polyester fabric

Concentration of nano Silica (grams/liter)	*K/S* of unexposed sample	*K/S* of exposed sample
Untreated sample	0.4291	0.3671
1	0.3731	0.2985
2.5	0.3945	0.2558
5	0.5165	0.3140

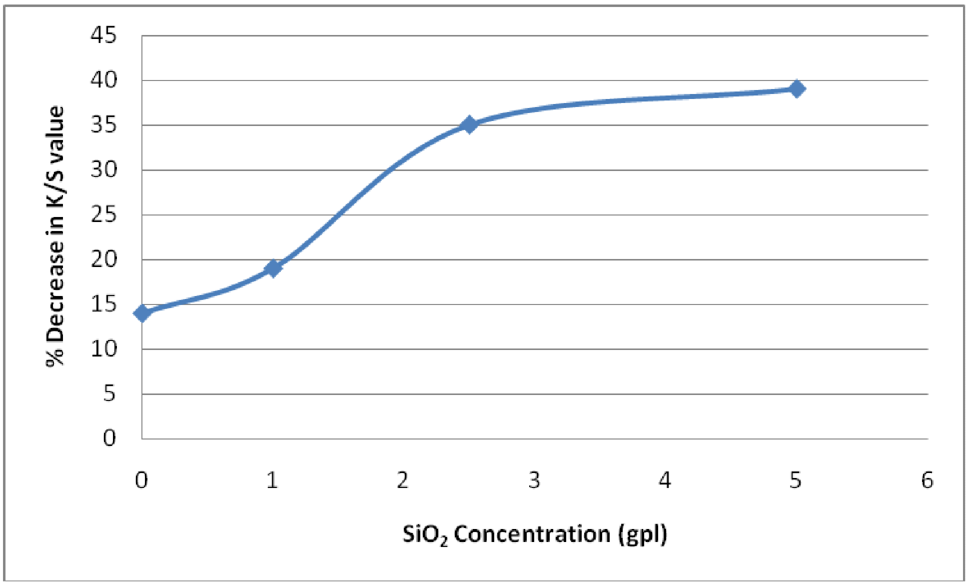

Figure 4: Decrease in K/S value of untreated and treated polyester fabrics

The pure polyester and nanocomposite polyester fabric samples were treated with 6% coffee solution then samples were exposed to sunlight to determine their *K/S* value. The table 6 and figure 4 show the results of self cleaning action of all fabrics.

From the results, it can be seen that higher the concentration of nano silica, higher is the self cleaning action i.e. at 5 gpl concentration, 178% improvement was observed compare to the untreated polyester fabric. This may be attributed to the presence of silica nano particles, which act as catalyst for the breakdown of dirt molecules by providing electrons, which ionize oxygen molecules in the surrounding air. The electrons are free from the silica via the photoelectric effect and these electrons must be reacting with oxygen atoms, which then be reacting with dirt particles.

3.3.3 Electrical Surface Resistivity

Table 7: Electrical surface resistivity of polyester fabric

Concentration of nano Silica (grams/liter)	Electrical Surface Resistivity (Ω)
Untreated sample	200×10^9
1	190×10^9
2.5	164×10^9
5	148×10^9

The Electrical surface resistivity of treated and untreated polyester fabric samples was analyzed on KEITHLEY614-Electrometer and the results are given in table 7. The incorporation of silica nano particles decreases the electrical surface resistivity and also the increase in the concentration of silica nano particles in fabric decreases the electrical surface resistivity further. The reduction in the electrical resistivity of treated fabric may be due to the semi-conductor property of silica.

3.3.4 Dyeing Behaviour

Polyester fabric is non ionic in nature can be dyed with non ionic dye i.e. disperse dye; the dye was fixed on fabric by mechanical means. The dyeing of polyester fabric with disperse dye was done by exhaust dyeing method. Result in table 8 shows the difference between dyeing yield which is measured by Kubelka munk equation to calculate *K/S* value of dyed fabric using different process. *K* is the absorption of light and *S* is the scattering of light. The colour strength of fabric treated with silica nano was found higher than untreated fabric, it may be due to the presence of nano silica on fabric, which may be getting attached with disperse dye through hydrogen bonding.

Table 8: Dyeing behavior of polyester fabric

Polyester fabric sample	Dyeing behavior (*K/S* value)
	Disperse dye (2 %)
Untreated sample	2.1626
Treated with nano silica (5 gpl)	2.5668

3.3.5 Effect of Nano Silica Treatment on Environment

Today, nano-particles are used worldwide, with much research being carried out into developing new synthetic procedures to improve their application characteristics. However, because of their high surface energy, nano-particles can cause great effluent problems for environment. It is clear from table

9 that the BOD, COD and TDS values of washing effluent of nano silica treated polyester fabric does not increase much than untreated polyester fabric. Also these values are comparatively lower than National environment quality standards for municipal and liquid industrial effluent. From the lower values of TDS it may be safely interpreted that nano particles can provide high durability to the desired textile function/s. By virtue of its small size and high surface energy, nano particles are bound/adhere to the fabric surface very strongly which gives it a reasonable wash fastness.

Table 9: BOD, COD and TDS of washing effluent of treated and untreated polyester fabric

Sample	Washing effluent	BOD (mg/l)	COD (mg/l)	TDS(mg/l)
Polyester fabric	Without silica nano	Nil	15	232
	With silica nano (5 gpl)	Nil	18	228

Note: National environment quality standard for municipal and liquid industrial effluent: BOD: 80 mg/l, COD: 400 mg/l, TDS: 3500 mg/l

4. Conclusion:

The silica nano particles with a diameter around 100 nm can be observed from the SEM images, which also indicate that silica nano particles are of spherical shape and are uniformly distributed on the surface of individual fibres of fabric. The FTIR spectrum of the treated polyester fabric confirms the presence of silica.

The treatment enhances the physical properties such as tensile strength, tearing strength and crease recovery angle of treated polyester fabric as compared with the untreated fabric. It also improves with increase in the concentration of silica nano up to 5 gpl.

The colour strength of disperse dye on polyester is improved with the incorporation of silica nano particles in polyester fabric.

The treatment with nano silica also improves the electrical surface resistivity, antimicrobial effect and self cleaning properties of treated polyester fabric.

From environmental point of view the silica nano is found to be less hazardous to the environment and its effluent toxicity is lower than standard parameter.

At last Nanotechnology holds an enormous, promising future for textile. The development in functional properties is endless with nano technology and at present, the application of nanotechnology in textile has merely reached the straight line. The new concept explored the nano material application area in textiles for further research and development.

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