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Structural and Thermal analysis 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 for its structural characteristics through SEM, IR spectra, XRD and DSC. The SEM showed the uniform distribution of silica nano particles on fabric, IR spectra showed the presence of silica particles. The semicrystalline nature has been shown by XRD patterns. The enthalpy (ΔH) required to melt the polyester silica nanocomposite fabric has increased as compare to pure polyester fabric.

Keywords: SEM, FTIR, XRD, DSC, silica, nanocomposite, polyester.

I. 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 material 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 structural and thermal nature of the material have been analyzed using XRD and DSC.

IJARSE
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II. 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	Fabric Setting	Weave	GSM	Thickness (mm)
	Warp/Weft	Ends/cm, Pick/cm			
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.

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.

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.

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

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. X-Ray diffraction (XRD) Model X'Pert Pro PANalytical, Singapore for evaluation of crystalline/amourphous structure of prepared nanocomposite material. Differential scanning calorimeter (DSC) Model DSC 6000, Perkin Elmer, USA; for thermal analysis.

III. RESULTS AND DISCUSSION

Polymer silica nanocomposite fabrics were prepared by incorporating different concentrations of silica nano

IJARSE
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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 and thermal 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

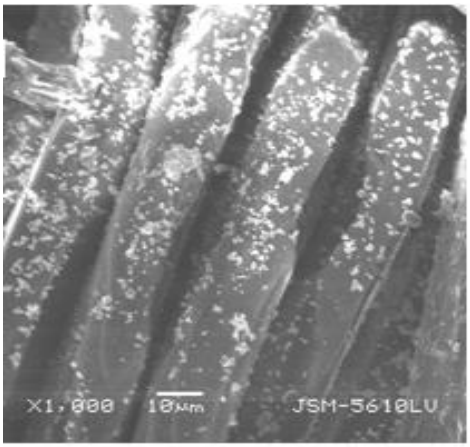

Figure 1: SEM microphotographs of polyester fabric treated with silica nano particle

The surface of the treated polyester fabric sample was observed on scanning electron microscopy. The result is shown in figure 1, 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

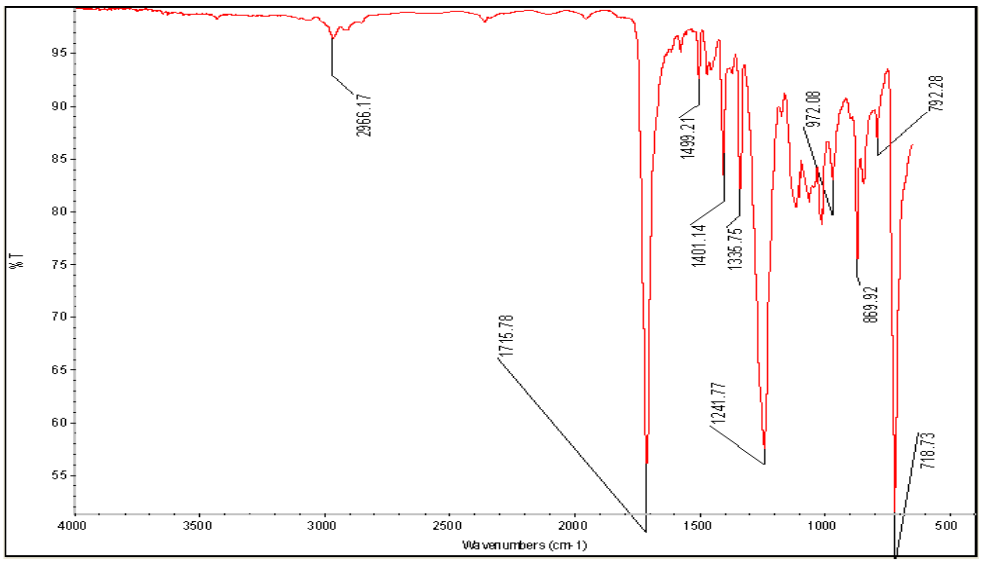

Figure 2: IR characterization absorption peak of pure polyester fabric

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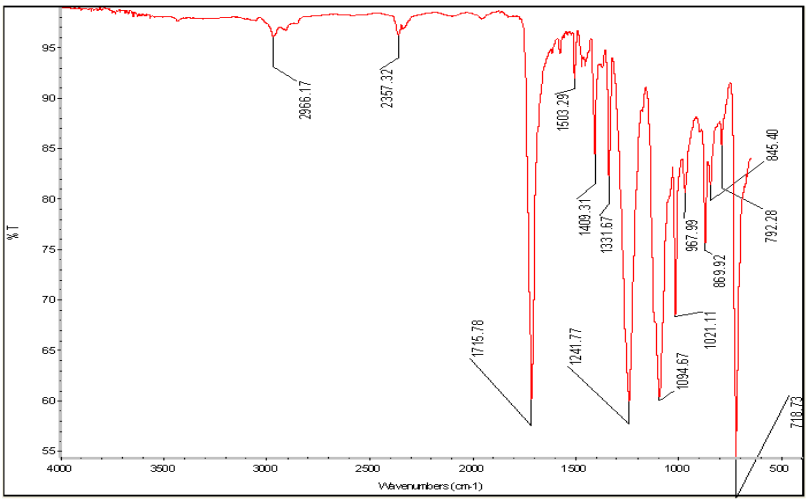

Figure 3: IR characterization absorption peak of 5 gpl nano silica treated polyester fabric

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.

3.1.3 Structural Analysis by XRD

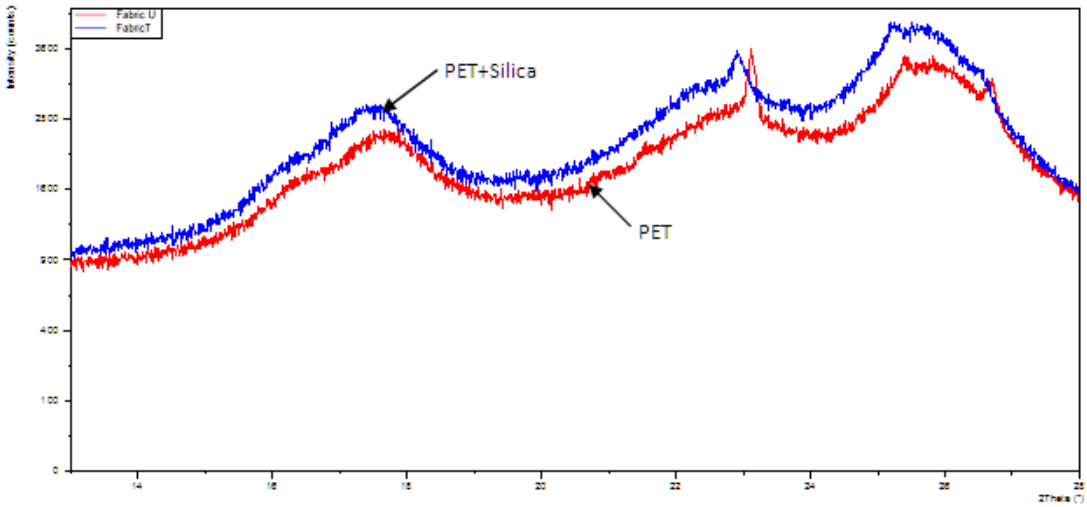

Figure 4: Combined XRD patterns of Pure Polyester and NPT3 samples

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Figure 4 shows the XRD pattern of pure polyester and nanosilica treated polyester fabrics, the pure polyester sample is showing semi crystalline state of the material. Four prominent features are observed. One distinct crystalline peak is observed due to which the semi crystalline nature of polyester fibre may be considered. Some broadness in peak is also observed in rest of the peaks, but they by and large denote the amorphous nature of fibre. The peak at 2θ value of 23.11° has the highest intensity followed by 25.89°, 26.6° and 17.66° and these peaks are indicative of polyester polymer[14].

The incorporation of silica nano particles lead to the development of some kind of force which drifts the atomic planes, such that the d-value increases. From the figure it can be seen that the intensities in the treated sample is on the lower side compared to the untreated samples. There is a shift in the peak 2θ values towards the lower side in general, indicating a change in d-values across the range, to the same extent. Hence, the presence of silica nano particles affects the structure of material in terms of atomic plane spacing. The SEM micrographs show the incorporation of silica nano particles well dispersed on the surface of fabric. The d-values for the observed diffraction peak of silica is in close agreement with those reported for corresponding standard samples as reported in JCPDS data file 84-0384. Here the peak corresponding to the d-value of 3.4224 Å is in agreement. Sosa et. Al[15] reported that as each crystalline material including the semi crystalline polymers as well as metal and metal oxide particles and layered silicate nano particles have a characteristic atomic structure, it will diffract X-ray in a unique characteristic diffraction order or pattern.

3.1.4 Thermal Analysis by DSC

Differential scanning calorimetry (DSC) was done to study the effect of incorporation of nano silica particles on thermal behavior of polyester fabric, which is shown in figure 5 and 6.

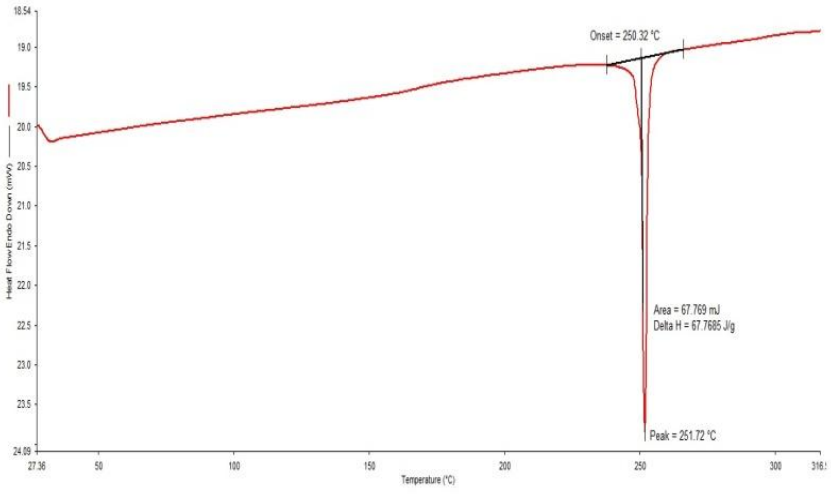

Figure 5: DSC curve of pure polyester fabric (PT)

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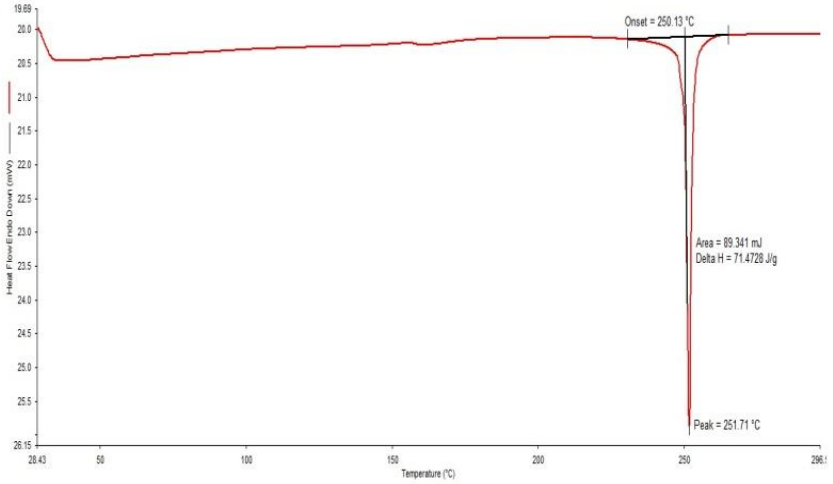

Figure 6: DSC curve of polyester fabric treated with 5gpl nano silica (NPT3)

Table 2 shows the melting temperature and enthalpy (ΔH) values of treated and untreated fabrics determined by DSC. Figure 4.43 shows the thermal behavior of pure polyester fabric. The melting temperature of polyester is 251.72°C and the enthalpy required to melt the pure polyester is 67.77 J/g. Figure 4.44 shows the thermal behavior of 5 gpl silica treated polyester silica nanocomposite fabric. The melting temperature of silica treated polyester fabric is 251.71°C, so there is no effect on melting temperature due to addition of silica nano particles. The enthalpy required to melt silica treated polyester fabric is 71.47 J/g. Due to nano silica treatment the rise in enthalpy is by 5.46%. This rise in enthalpy may be due to nanoscopic level of silica, hence inducing better thermal stability to the fabric. Altan et. Al[16] has made similar observation for polypropylene and polyester, being thermoplastic fibre may behave in the same manner due to the coating of nano SiO_2 particles. Similar observations are observed in earlier parts like in case of polyamide film and polypropylene filament, due to addition of silica nanoparticles the enthalpy required to melt the film/filament has also increased and there is no change in melting temperature due to addition of silica nanoparticles.

Table 2: Enthalpy (ΔH) of treated and untreated polyester fabric

Sr. No.	Concentration of nano silica (g/lt)	Melting Temperature °C	Enthalpy (ΔH) (J/g)
1	0.0	251.72	67.77
2	5.0	251.71	71.47

IV. CONCLUSION

The nano silica particles were successfully applied on polyester fabric using pad-dry-cure technique. The presence of silica on the fabric surface was observed in SEM micrographs further confirmed by the FTIR spectrum and XRD pattern. SEM micrographs indicates that the silica nano particles are of 100 nm diameter and spherical in shape, distributed uniformly on the surface of individual fibres of fabric. The chemical composition of treated fabric was confirmed by FTIR spectra. Further the structure of fabric was found semi crystalline in nature as per X-ray diffraction analysis. The enthalpy (ΔH) required to melt the

IJARSE
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polyester silica nanocomposite fabric has increased as compare to pure polyester fabric by 5.46%. The nano silica coating of PET fabric was done by padding technique, which could be the probable reason for less interaction between nano particles and PET polymer, which is also reflected by lesser changes in mechanical properties, however in FTIR analysis minor Si-O-Si peak was observed. This may also reflect in melting point which remains almost unchanged for coated as well as pure polyester fabric.

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