A Study on ZnO Nanorod Arrays Formed on the Surface of Polyester Fabric

Mina Emadi, Maryam Sharzehee, and Algy Kazlauciunas

***Abstract*— A feasibility study on the possible growth of rod-shaped nano size zinc oxide particles on the surface of polyester fabric was investigated. The nanoparticles were produced using a hydrolysis method, with a zinc compound being utilized as the starter material. Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDS) and X-ray diffraction (XRD) have been used to characterize the composition of the nano particles, as well as their shape, size and crystallinity. The application of nano zinc oxide onto the polyester fabric was carried out in aqueous solutions at high temperatures, using the exhaustion method. The effective parameters such as time, temperature, the concentration of the dispersed nanogel and also the rate of heating, were selected to study the adsorption of the nanoparticles on the polyester fabric. The elemental analysis technique SEM/EDS was used to evaluate the amount of zinc on each sample. Finally, the possible growth of nanorod zinc oxide on a prepared sample using the sol gel technique was tested and desirable results were confirmed by means of SEM analysis.**

***Keywords*: exhaustion method, nanorod, nano zinc oxide, polyester fabric**

I. Introduction

The modification of the surface and the interface of textile substrates by nanomaterials is well known. Today the multifunctional finishing performance of textile materials is increasingly demanded, and by using this technique antibacterial and self-cleaning properties, super-hydrophobicity, fire retardancy, anti-static properties, moth-proofing, electromagnetic shielding, electrical conductivity and also UV protection have been readily achieved [1-5]. The coatings that can modify the surface of textiles are usually composed of nanoparticles, a surfactant, other ingredients and a carrier medium [6]. Several methods have been utilized to make a nanocoating on textiles, including sol-gel technique, magnetron sputter coating, and cross-linking by polymers [2]. Of all these methods, padding is the most commonly used method [3-5]. The nanoparticles are attached to the fabric using a padder adjusted to suitable pressure and speed, followed by drying and curing.

In order to make semiconductor devices, especially by using textile materials, the growing of nanocrystals on the surface, which are close to an ideal crystal with only a small number of defects and are oriented in one direction, is required. ZnO is an important semiconducting and piezoelectric material with a wide variety of applications in the field of optoelectronics, piezoelectric sensors, transducers and resonators. It is a semiconductor with a direct wide band gap of 3.37 ev and a large excitation binding energy of 60 meVd. The non-central symmetry in wurtzite, combined with a large electromechanical coupling, causes ZnO to exhibit strong piezoelectric and pyroelectric properties, and is ideally suitable for application in sensors and actuators [7-11]. Finally, it is bio-safe and also biocompatible [12,13].

The growth of nanorod zinc oxide on textile materials has also been investigated. The deposition of nano zinc oxide as a seed layer on cotton fabric using a solution method was reported in a research [6], whereas in another work radio frequency magnetron sputtering was used [14]. The ZnO nanorod fabrication was achieved via a hydrothermal route, with a growth solution containing zinc nitrate and hexamethylene tetramine. The formation of nanorods on polyester and also polyamide fabrics using an electrolysis deposition technique was confirmed. The roughness structure generated from nanorods created super hydrophobic properties on polyester fabrics, with water contact angles exceeding 150◦ [15]. The same technique was also carried out on cotton fabric and proved to be highly reproducible, easy scalable, and cheap; thus allowing a wide range of applications [16]. Well-integrated zinc oxide nanorod arrays on a conductive fabric (woven Ni-plated PET fibre) were produced using cathodic electrochemical deposition on the ZnO seed layer coated on the surface of the substrate in an ultrasonic bath [17]. The photoluminescence property of the fabric surface with good crystallinity and high density was optimized at an external cathodic voltage of -2 V. FEG-SEM analysis of zinc oxide nanowires grown on carbon cloth has also been reported. Thermal vaporization and condensation was used to grow nanowires from a source mixture of ZnO and graphite powder in a tube furnace [18].

Polyester fibers are hydrophobic with an extremely crystalline structure without any polarity groups and therefore water is not able to swell the fibres. As a result, only a minimal amount of water is imbibed in the polymer system, so diffusion of dye and also nanomaterials take place slowly. In terms of polyester treatment in dying finishing, the exhaustion technique at high temperatures is recommended. However, in the finishing process, the application of various finishing agents onto the polyester fabric is required.

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II. Experimental

A. Materials

1) Characteristics of the fabric

Woven polyester fabric with a weight of 170 g m^{-2} was provided with a weft and warp density of 18 and 21 yarns per centimeter, respectively.

2) Chemical materials

The initial material zinc oxide was provided from Pars Company (Iran), with an estimated diameter of 0.8 µm. All other reagents were provided from Merck Company (Germany).

B. Physicochemical Characterization of Prepared Nano Zinc Oxide

1) X-Ray Diffraction

The crystalline structure of the nano zinc oxide sample was resolved using the X-ray diffraction method (XRD). The radiation intensity distribution curve $I = f(\theta)$ was achieved using a counting rate gauge, inter-operating with the counter and electronically coupled to a graphic recorder.

2) Energy dispersive X-ray spectrometry

The chemical stoichiometry of the ZnO nanoparticles was studied using secondary electron energy dispersive X-ray spectrometry (SEM/EDS). The utilized instrumentation was a Jeol JSM-6610LV scanning electron microscope, in conjunction with an Oxford Instruments INCA X-max80EDS system.

3) Surface area

The surface area of the zinc oxide nanoparticles was measured using laser diffraction particle size analysis. This was achieved using a Mastersizer 2000 from Malern Instruments. The diffraction patterns of a laser beam passed through any object from nanometers to millimeters in size is used to quickly measure the geometrical dimensions of a particle [19, 20].

4) Fourier transforms infrared spectroscopy

The chemical structure of the nanogel and the treated fabric samples were studied using FT-IR analysis. The utilized instrument was an Equinex 55 Infrared Fourier Transform Spectrophotometer from Bruker Company, using a diamond ATR attachment.

C. ZnO Nanogel Preparation

The nano-particulate re-dispersible zinc oxide gels were prepared using hydrolysis of the zinc compound in an alcohol/water mixture according to the procedure described by Womelsdoef [21]. A mixture of zinc oxide, glacial acetic acid and methanol was heated to 60 °C, with stirring. The hydrolysis process was carried out by adding a KOH/methanol solution to the mixture slowly. After 30 minutes, the final milky white solution was cooled in an ice bath for 20 minutes to reach a temperature of 16°C. Several times purification by introducing fresh methanol to the system was required to achieve the desirable size and purity of the particles. These were then centrifuged at 5500 rpm for 30 minutes, and then the supernatant liquor was decanted off. The gel weight was about 60% of the weight of the initial zinc oxide used through the process.

D. Application of ZnO Nanogel onto Polyester Fabric

The application of zinc oxide onto polyester fabric was studied in the exhaustion method at high temperatures. The thermodynamic adsorption properties of nano zinc oxide and the diffusion ability parameters in the exhaustion bath at various temperatures and concentrations were investigated during the course of the work. It has been indicated that the rate of heating affects the nano zinc oxide adsorption.

E. Growth of Nanorods on Polyester Fabric

Hexamethylenetetramine or methenamine ($C_6H_{12}N_4$), is a growth directing agent widely used for the synthesis of ZnO nanorods [22-25]. The polyester fabrics with an initial ZnO deposit were placed in the solution of a mixture of zinc nitrate and hexamethylenetetramine ($C_6H_{12}N_4$) in a 1: 1 volume ratio, at various times and temperatures, as indicated in Table I. The fabrics with grown ZnO nanorod arrays were washed with deionized water and dried at 110°C in air for 1 h, and then were characterized using scanning electron microscopy.

TABLE I
NANOROD GROWING CONDITIONS FOR SOME SAMPLES

Samples Code	Methenamine ($C_6H_{12}N_4$) molar	Zinc nitrate molar	Temperature (°C)	Time (min)
S(1)	1	1	90	20
S(2)	0.5	0.5	90	20
S(3)	1	1	70	20
S(4)	1	1	90	10

F. Dielectric Strength

The dielectric strength of an insulating material is defined as the maximum electric field that a pure material can withstand under ideal conditions without breaking down. The theoretical dielectric strength of a material is an intrinsic property of the bulk material and depends on the configuration of the material or the electrodes with which the field is applied [26]. In this study, further investigation on samples was carried out to find more information regarding electrical conductivity. The dielectric strength of nano-treated samples and other fabrics containing nanorod zinc oxide on them, was measured according to the standard method ASTM D149 [27], using Semi-Automatic Oil Test Set from the Italian company Ceast, which worked to a maximum of 60 kV.

III. Results and Discussion

A. X-ray Powder Diffraction

The X-ray powder diffraction technique was used to characterize the zinc oxide nanogel. The diffraction maxima observed at the 2θ angles of 31.8, 34.5, 36.3, 47.6, and 56.6 correspond to the characteristic ZnO planes of the wurtzite structure: (100); (002); (101); (102); (110) [28]. The X-ray diffraction spectrum of the nanogel sample is shown in Fig. 1.

The average crystal size ($D_{average}$) of the ZnO samples was estimated from the width of the lines in the XRD spectrum using the Sherrer equation:

$$D = K\lambda / \beta \cos\theta \quad (1)$$

where K is a unitless constant taken as 0.9, λ is the X-ray wavelength (1.5418 A°), β is the width of the line at half maximum intensity, and θ is half of the diffraction angle [27]. The $D_{average}$ values were calculated for the three highest intense peaks of the ZnO spectrum, which were (101), (110), (102), (002), and (100). This data is reported in Table II. The morphological structure of the prepared nanocrystals has a similar diffraction pattern compared with the data provided in other papers [28-31], except for the relative intensities due to its random orientation.

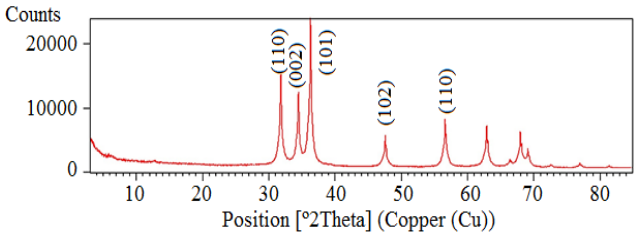

Fig. 1. XRD Diffraction pattern of the prepared nano zinc oxide.

TABLE II
CALCULATED AVERAGE CRYSTAL SIZE OF ZNO USING THE SCHERRER EQUATION

D(100) (nm)	D(002) (nm)	D(101) (nm)	D(102) (nm)	D(110) (nm)	$D_{average}$ (nm)
5.02	185.45	7.33	30.79	8.54	65.93

B. Energy Dispersive X-ray Spectrometry

The chemical stoichiometry of ZnO nano particles was investigated with the SEM/EDS technique, with the information provided in Table III. The atomic ratio of Zn:O was found not to be 1:1, however, in weight percentage the amount of zinc element is nearly twice that of the oxygen element. Environmental effects should be considered.

TABLE III
ELEMENTAL ANALYSIS OF PREPARED NANO ZINC OXIDE USING EDAX TECHNIQUE

Element	Element %	Atomic %
Zinc	58.96	25.74
Oxygen	29.68	52.32
Potassium	2.75	2.03
Carbon	8.63	20.22

C. FT-IR Analysis

In order to study the characteristic functional groups present at the surface of the ZnO sample, the prepared nanogel zinc oxide was subjected to spectroscopic analysis. The FT-IR spectrum taken from the sample after complete drying at room temperature is presented in Fig. 2. The spectroscopic analysis confirmed the previously published results [32,33]. The presence of the bands assigned to the stretching vibration of Zn-O is indicated at 511.58 cm^{-1}, 492.51 cm^{-1}, 463.19 cm^{-1}, and 431.41 cm^{-1}. The two weak bands at ~1390 and 1500 are studied further to find more information about the surface character of the ZnO sample. The FT-IR results from specific frequencies 600 to 4000 cm^{-1} are shown in Fig. 3.

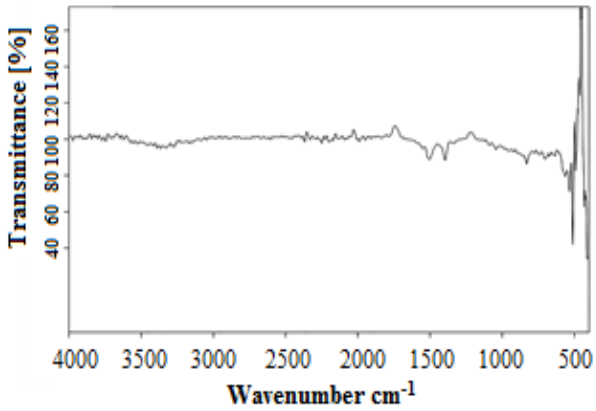

Fig. 2. FT-IR spectrum of the prepared nano zinc oxide.

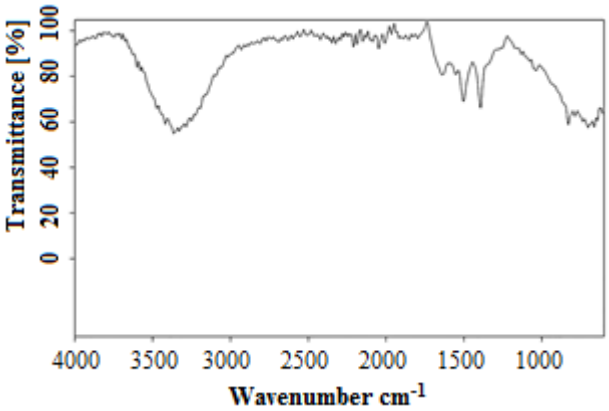

Fig. 3. FT-IR spectrum of the prepared nano zinc oxide. Only part of the spectrum is highlighted.

The OH stretching vibrations due to intermolecular interaction are indicated in a very wide band centered at 3364.8 cm^{-1}. The C-O stretching vibration and O-H deformation vibration of alcohol are identified at 1502.39 and 1393.03 cm^{-1}. In addition, the band at 828.95 cm^{-1} also presents the CH wagging vibration of a secondary alcohol, which was used as an initial material in the preparation of the zinc oxide nanoparticles [34].

D. Surface Area

The particle size distribution of the prepared nano zinc oxide is shown in Fig. 4. Some information regarding the size of the nanoparticles were indicated: the specific surface area of the sample was 18.4 m^2/g, the surface weighted mean was 0.334, and the volume weighted mean was 0.741 microns. It was found that the specific surface area of the sample prepared by the hydrolysis method was approximately 10 times higher compared to the conventional zinc oxide as the starting material.

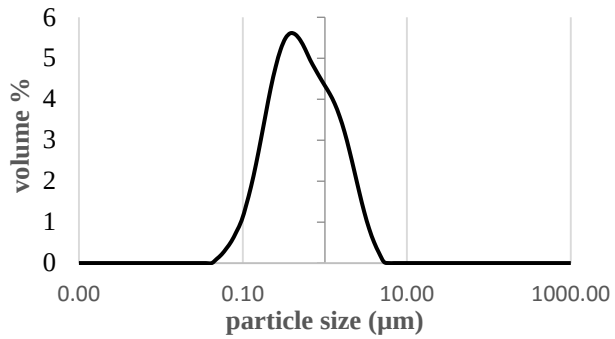

Fig. 4. The particle size distribution of prepared nano zinc oxide.

E. Application of Nano Zinc Oxide to Polyester Fabric

The growing of zinc oxide nanorod crystals on the surface of polyester fabrics was defined as the main aim of this work. A high performance fabric with a seed layer of deposited nano zinc oxide particles on it was required for this treatment. Applying the growth solution at a low temperature under atmospheric pressure is a simple, reliable and reproducible technique for growing crystals onto the textile substrate. The procedure of the treatment includes immersing the substrate into a deposition solution, containing zinc nitrate and methenamine, for a specific time and temperature. To facilitate a uniform crystal growth in a single ordered direction and to have high quality performance, the availability of nanoparticles as a seed layer on the fabric is required.

In this research the application of nano zinc oxide onto the polyester fabric using the exhaustion method was studied. The treatment was carried out in an aqueous solution containing nano-dispersed gel, dispersing agent and a piece of fabric. The variable conditions were selected to accelerate the rate of the nano zinc oxide adsorption onto the polyester fabric. The concentration of nano-dispersed gel, the time, and the temperature of the treatment were selected. A typical exhaust nanoparticle application sequence for polyester fabric is shown in Fig. 5. Two heating steps were performed: the first stage from room temperature to 50°C, and the second stage from 50°C to 130°C. Finally, after staying for 45 minutes at this temperature, the heating system was turned off to allow the temperature to cool to 70°C (~5 degree per minute).

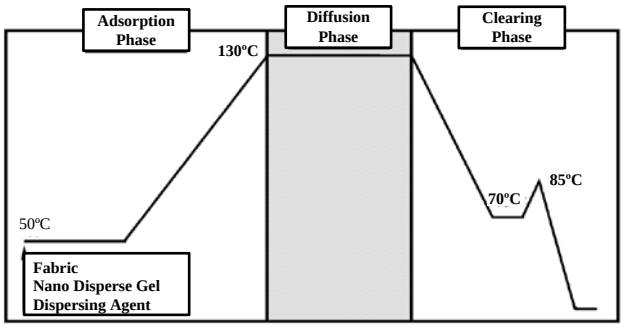

Fig. 5. Exhaust phase when applying nano zinc oxide to polyester fabric.

In the second stage of this experiment (the adsorption phase), the heating rates of 2, 3, 4 & 5 °C per minute were used. The SEM/EDS analysis provides sufficient information regarding the effect of the treatment conditions on the zinc oxide adsorption onto the polyester fabric. The treatment procedure for each sample is shown in Table IV. However, it must be mentioned that, following the treatment, the fabric samples were washed with water to remove any unattached material and then dried at room temperature. The SEM/EDS technique was used to evaluate the amount of zinc on each individual sample.

TABLE IV
SEM/EDS ANALYSIS DATA FROM THE NANO ZINC OXIDE TREATMENT OF POLYESTER FABRIC

Sample Code	Nanogel (owf)	Heating Rate (°c/min)	Time (min)	Temperature (°c)	Zinc	
					At%	Wt%
R(1)	250	2	45	130	9.09	1.98
R(2)	250	3	45	130	21.99	5.37
R(3)	250	4	45	130	13.95	3.16
R(4)	250	5	45	130	4.64	0.98
T(130)	100	3	45	130	7.37	1.59
T(120)	100	3	45	120	10.59	2.35
T(110)	100	3	45	110	8.78	1.74
T(100)	100	3	45	100	8.34	1.82
M(45)	100	3	45	130	7.27	1.59
M(30)	100	3	30	130	8.76	1.91
P(10)	10	5	30	130	1.12	0.23
P(30)	30	5	45	130	1.81	0.37
P(50)	50	5	45	130	2.12	0.44
P(70)	70	5	45	130	5.89	1.22
P(100)	100	5	45	130	9.48	2.07

The EDX analysis spectra for two of the examined samples T(120) and P(100) are presented in Figs. 6 and 8, respectively. In Figs. 7 and 9, the SEM electron micrographs of the two treated samples clearly indicate the homogeneous distribution of the nanoparticles coating the surface of the fibres. These figures also include the respective X-ray mapping images for the zinc element. These images support the electron micrographs in showing the homogeneous distribution of the nanoparticles.

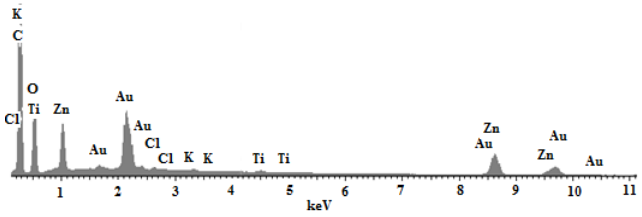

Fig. 6. Elemental analysis information of treated sample, code T(120).

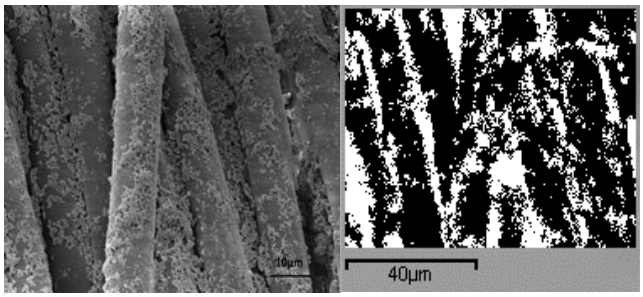

Fig. 7. SEM micrograph and zinc element mapping distribution (white dots) for nanoparticle treated sample, code T(120).

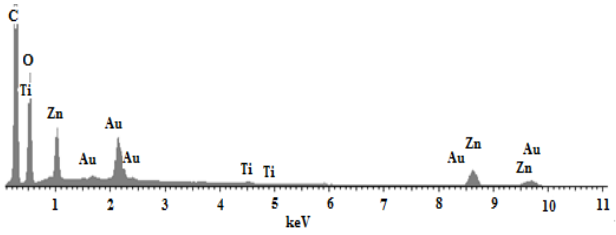

Fig. 8. Elemental analysis information of treated sample, code P(100).

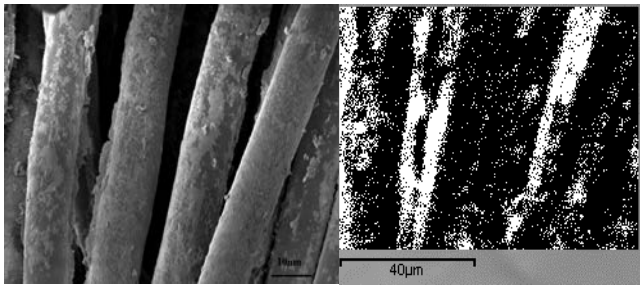

Fig. 9. SEM micrograph and zinc element mapping distribution (white dots) for nanoparticle treated sample, code P(100).

In the exhaustion process the three main phases of the transaction are the heating or adsorption phase, the high temperature or diffusion phase, and finally the clearing phase. The heating or adsorption phase is the most critical one in determining the level of nanoparticle adsorption onto the treated samples. Therefore, the rate of heating during this stage is an essential parameter in controlling the adsorption behavior. According to the results shown in Table IV, during the rapid heating cycles the time at the highest temperature is minimal, and it is even more critical to ensure that nanoparticles are applied in a uniform manner during the adsorption phase onto the fabric. However, during a slow treatment nano zinc diffusing takes place more rapidly. In this condition, at high temperatures, the mobility of the polymer chains in the amorphous regions is increased. Therefore the nanoparticles can easily penetrate into the surface of the fibers, with more entering and exiting, and only the depth penetrating particles are trapped within the interior structure of the fibers. Low levels of zinc oxide on the samples were determined for this treatment procedure, as shown in Table IV. In fact, the heating rate is appropriate to allow the controlled adsorption of nanoparticles onto the polyester fabric.

In the diffusion phase when the main processing temperature was indicated, the highest level of nano zinc oxide adsorption was reported at 120°C. The resting of the fabric in nano dispersed solution at 130°C for nearly 45 minutes had no effect on the rate of nano adsorption. Despite the time needed for the adsorption phase, it is largely influenced by the machine conditions and in the diffusion phase the time required at the highest temperature is directly related to the diffusion characteristics of the nano particles. The migration properties of nanoparticles due to their size may become a key factor if zinc oxide is applied unevenly during the adsorption phase, since the key parameter in the diffusion phase is the diffusion rate of the nanoparticles.

In the clearing phase, some particulate nano zinc oxide may still be occluded on the fibre surface but during the wash process with water at a high temperature, most of the contamination should be removed.

The concentration of applied nano zinc oxide was also varied. The amount of zinc oxide on some of the samples are comapred in Fig. 10. It is well estabilised that nano particle adsorption on the surface occurres by transfer of the particles from nano disperse solution into the fibre. Because of this, the linearity of the isotherm can be predicted.

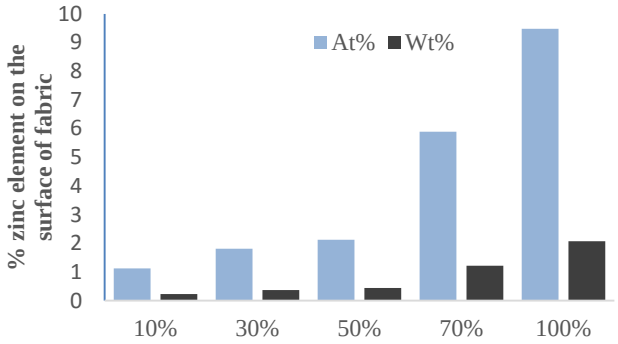

Fig. 10. Weight% and atomic % of zinc element on the treated samples with various nanogel concentrations in the exhusion method.

The amount of nanoparticle adsorption onto the fabric is relevant to the nano concentration in the bath. It has to be mentioned that, a high level of un-applied particles still remain in the dispersion, however, this dispersion can be reused for further treatement. To find the exact model of adsorption equilbrium of nano zinc oxide onto polyester fabric, further investigation is required.

F. The FT-IR Spectrum of Treated Fabric

The FT-IR spectrum of the treated samples in aqueous solutions containing various amounts of nanoparticles, indicate that the specific band at 626.85 cm^{-1} can be used to determine the amount of zinc element present on the surface of the fabric. The FT-IR spectra of an untreated sample and a treated one in a high concentration nano zinc oxide solution, 250% (OWF), are compared in Fig. 11.

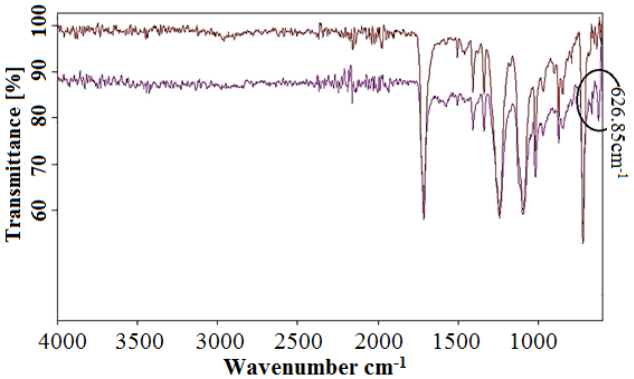

Fig. 11. FT-IR spectra of (a) untreated sample and (b) a treated sample with nano zinc oxide (code R2).

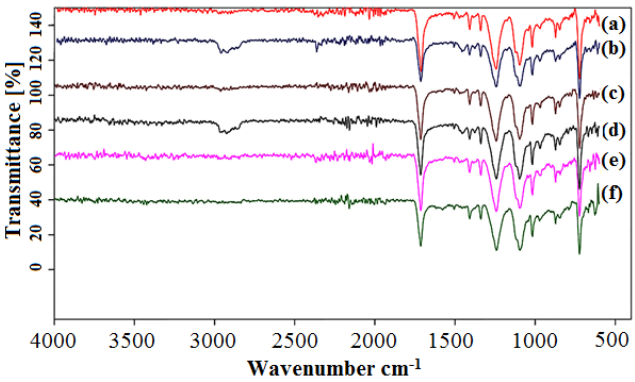

Fig. 12. FT-IR spectra of some treated samples in different nano zinc oxide concentration. The amount of zinc oxide on the samples is indicated (% weight) (a) 10%, (b) 30%, (c) 50%, (d) 70%, (e) 100% and (f) 250%.

TABLE V
A COMPARISON OF IR TRANSMITTANCE % OF TREATED SAMPLES WITH NANO ZINC OXIDE AT VARIOUS NANOGEL CONCENTRATIONS IN THE EXHAUSION METHOD

Sample Code	Nanogel (owf)	IR - Transmittance (%)	Zinc	
			At%	Wt%
P(10)	10	0.978	1.12	0.23
P(30)	30	0.970	1.81	0.37
P(50)	50	0.953	2.12	0.44
P(70)	70	0.951	5.89	1.22
P(100)	100	0.914	9.48	2.07
R(2)	250	0.899	21.99	5.73

The difference between the two samples is very clear. The FT-IR spectra of a number of treated samples using various zinc oxide concentrations are compared in Fig. 12.

As it has been indicated, increasing the zinc oxide on the surface of fabrics, can reduce the IR transmittance from the fabric, therefore the high level of infrared absorption can confirm it. This information indicated in Table V, is obtained from the FT-IR spectrum of a number of samples.

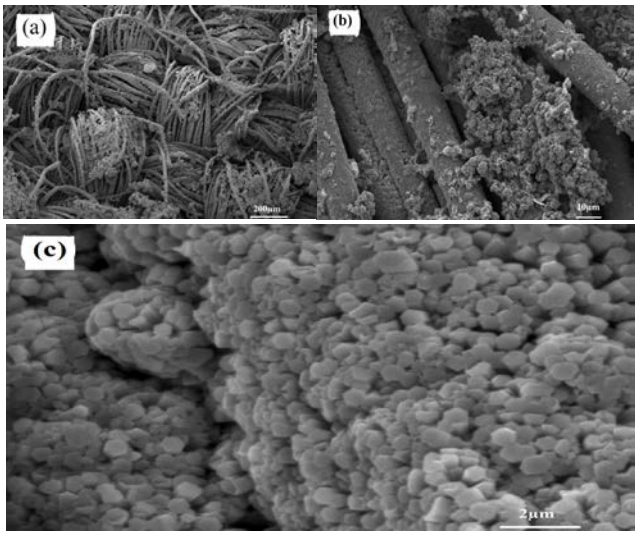

Fig. 13. SEM electron micrographs of a treated sample in 0.5 molar solutions for 20 hours at 90°C, at three different magnification, (a) x75, (b) x1000 and (c) x8000.

G. Growth of Nano Zinc Oxide on the Fabric

This part of the research was undertaken to determine the feasibility of the possible growth of nanorod structure on the surface of the polyester fabric. The process was carried out on the samples with nano zinc oxide, treated using the exhaustion method at 120°C for 30 minutes in the bath containing 100% (OWF) of nanogel. The heating rate selected for all samples was 3°C per minute. The test was undertaken on each sample using various molar ratios of the starting material, at different times and temperatures. The SEM electron micrographs of one of the treated samples are shown in Fig. 13.

The growth of nano zinc oxide particles packed close together and oriented in various directions was confirmed. The nanorod structure formation on polyester fabric needs to be in uniform orientation to improve the electrical conductivity of the fabric and therefore a low rate of the growth process in short time duration should be considered as the most appropriate condition.

H. Dielectric Strength of Treated Samples

The dielectric strength of a number of samples is indicated in Table VI. At a specific voltage, the fabric performed as a semi-conductor. Though, it has to be mentioned that the dielectric strength data obtained from this experiment are not the true values. During the test the fabric was immersed in oil and because of this some of the oil was adsorbed through the fabric and hence provided a barrier, which prevented accurate measurement.

TABLE VI
THE DIELECTRIC STRENGTH OF SOME TREATED SAMPLES[1]

Sample No.	Thickness (mm)	Current Intensity (mA)	The rate of voltage ($kV\ s^{-1}$)	Dielectric strength ($kV\ mm^{-1}$)
No crystal growing	0.048	30	1	18.3
S(1)	0.55	30	1	14.7
S(2)	0.56	30	1	14.0
S(3)	0.50	30	1	16.3
S(4)	0.51	30	1	15.1

[1] The nanorod zinc oxide growth conditions were different.

IV. CONCLUSIONS

The deposition of nano zinc oxide as a seed layer on the polyester fabric was performed using the exhaustion method at a high temperature. The durability of the treatment was confirmed with immersing the fabric in the growing solution to attain the nanorod structure on the surface of the fabric. Uniformity and high crystallinity of the nanorods is required to produce a conductive fabric. In a comparative investigation, it was clearly observed that the seed layer and the growing conditions played a key role in providing a uniform distribution of ZnO nanorods on the surface of the fabric. However, further investigation is required.

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