

## Modeling and optimization of electromagnetic and saturated magnetic properties of polyester fabrics coated with Ag/kaolin/silica nanocomposites

E.Zarrinabadi<sup>1</sup>, R.Abghari<sup>1\*</sup>, A.Nazari<sup>2</sup>, M.Mirjalili<sup>3</sup>

<sup>1</sup> Department of Textile Engineering, Yazd Branch, Islamic Azad University, Yazd, Iran,,

<sup>2</sup>Department of Art and Architectural, Yazd Branch, Islamic Azad University, Yazd, Iran,

<sup>3</sup> Department of Textile Engineering, Yazd Branch, Islamic Azad University, Yazd, Iran,

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In this study, the photo-reduction method is used to synthesize a three-component nanocomposite containing Ag, Kaolin, and Silica. The values of these three components are optimized to make a central composite design. Through ultrasound and UV photo reduction, virgin polyester fabrics coated with different ratios of Silica/kaolin/Ag are evaluated for their electromagnetic and saturated magnetic properties. The fabrics are analyzed by electromagnetic reflection and electromagnetic transmission techniques as well as the vibrating sample magnetometry test, and their properties are optimized according to the response surface methodology. Detective analytical methods such as field emission scanning electronic microscopy, energy dispersive x-Ray analysis, and infrared spectroscopy are implemented on the triple nanocomposite-treated polyester fabrics. The experimental results show that the electromagnetic and saturation magnetic properties of such polyesters are significantly improved when a coating layer of the triple nanocomposite with proper concentrations of silica, kaolin and Ag is applied on the fabric surface.

**Keywords:** Electromagnetic properties, Composite, Silica/Kalolin/Silver, Polyester fabrics, Saturation magnetization

### INTRODUCTION

In recent decades, polyester has been of wide application in textile industries [1]. Therefore, researchers have paid significant attention to improvement of its properties. For this purpose, certain techniques and procedures have been applied, including enhancement of ultra violet protective properties using aluminum-doped zinc oxide thin films [2], deposition of silver nanoparticles on fiber surface using ultrasound waves [3], introduction of self-cleaning features through photo reduction [4, 5], coating by the direct current method in presence of Ag/ZnO hybrid nanoparticles [6], improvement of dye uptake [7] and using antibacterial as well as antifungal properties on fibers [8, 9]. Coating of polyester fabrics with nanoparticles was conducted by sonochemistry [10], UV photo reduction [11] and in-situ synthesis [12]. It has been possible to create and improve different properties in polyester fabrics by coating them with a combination of various nanoparticles [13]. Hybrid nanoparticles such as SiO<sub>2</sub>/TiO<sub>2</sub>, TiO<sub>2</sub>/ZnO<sub>2</sub> and SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> have been often used to enhance antibacterial, mechanical and UV protection features. Multifunctional polyester fabrics can be made with two- or three-component nanocomposites [14- 17]. In the last decade, researchers focused on application of three-component nanoparticles to modify textiles. There are reports in the literature about development of

three-component TiO<sub>2</sub>/Fe<sub>3</sub>O<sub>4</sub>/Ag nanocomposite with mechanical, magnetic and antibacterial properties [18] and kaolin/silica/Poly (vinyl chloride) with optical features [19]. The silica/kaolin/Ag nanocomposite has also been made by photo reduction, which has different properties such as reflection and transmission of electromagnetic waves [20- 21]. So far, no scientific and comprehensive research has reported the application of three-component nanocomposites of silica/kaolin/Ag to improve the saturated magnetic and electromagnetic properties of polyester fabrics.

In this research, a three-component nanocomposite is synthesized based on different proportions of silica, kaolin and Ag. To this end, central composite design (CCD) is used, and then the fabricated nanocomposites are coated on polyester fabrics with different concentrations. The saturation magnetization and the electromagnetic properties of the blank and the treated fabrics are compared and analyzed using the response surface method.

### EXPERIMENTAL

#### Materials

The nanoclay particles (kaolinite, commercially known as Sillitin N85) were supplied from Haffman Co. (Germany). They are neutral particles containing amorphous and crystalline silica and a kaolin layer with the chemical formula SiO<sub>2</sub>-Al<sub>2</sub>[(OH)<sub>4</sub>Si<sub>2</sub>O<sub>5</sub>]. The density of these particles was 2.6 g/cm<sup>3</sup>. Silver nitrate was obtained from Sinchem Co. (South Korea) and Citric Acid, Cetyl-trim ethyl-ammonium

\*) To whom all correspondence should be sent:

E-mail: abghariramin@iauyazd.ac.ir

bromide (CTAB) and Sodium Hypophosphite (SHP) were purchased from Merck Chemical Co. (Germany). Completely raw polyester filament fabrics with a twill structure (0.36 gr/m<sup>2</sup> warps and 0.26 gr/m<sup>2</sup> wefts, yarn count of 150 Denier, warp/weft density of 22/16 cm<sup>-1</sup>) were produced on a Rapier Sulzer Ruti machine (2005 Sulzer Swiss Co.). The polyester fabrics were coated by Osram UV 400 nm lamps. Ultra violet irradiation was applied to the nanocomposites by means of a Swiss-made Camag, model 02209110, and infrared spectroscopy was done with a Tensor 27 Device (Bruker Co-Germany).

## METHOD

### *Fabrication of three-component nanocomposites*

The 3.0 g SiO<sub>2</sub>-kaolinite (commercially named Silitin N85) was dispersed in 100 mL of deionized water for an hour. Then, 30.00 mL of a silver nitrate (0.4 N) solution was added to the kaolinite solution, and the mixture was stirred with a mixing machine for an hour at 25°C. The ultra violet irradiation was simultaneously applied at a standard ambient humidity (20±2 C and 65±2%) for 72 hours. The UV chamber used to make the nanocomposites had an ultra violet lamp of 325 nm. The resulting samples were centrifuged and washed with deionized water in a filter paper for 10 repeats. The powders were dried in an oven at 110.°C for three hours.

### *Coating of polyester fabrics*

The polyester fabrics were scoured with 0.5 % w/v based on the weight of the fabric (O.W.F) non-ionic detergent with a 40:1 (L: G) solution at 50°C for 15 minutes in a washing machine. The synthesized nanocomposite was fixed on the polyester samples at four successive stages according to the procedure that follows. Firstly, as presented in Table 1, various dispersions of the nanocomposite including the polyester sample, CTAB and water were mixed based on the Design Expert Software (to be discussed later). The ratio of the fabricated nanocomposite to CTAB in all the experiments was constantly 1:2. Secondly, in order to reduce the particle size and prepare stable colloids, the samples were placed in an ultrasound (Euronada Co.) bath at 50°C for two hours. Then, 8.0 % (O.W.F) CA and 5.0 % (O.W.F) SHP were added to the dispersions at 40°C for 20 minutes. The prepared samples were washed and padded at an uptake rate of 85%. Afterwards, the treated fabrics were dried in an oven at 60°C for four minutes. In the next place, the cross-sectional linkage and fixation of the fabrics were conducted under UV irradiation at room temperature, as in Table 1. The

irradiated fabrics were rinsed with deionized water in a washing machine for three times until the additional and unfixed materials such as nanocomposite, CA and SHP were removed. Finally, the treated fabrics were fully dried at 40 °C for further detective analysis.

### *Field emission scanning electronic microscopy (FESEM)*

FESEM is an analytical technique used in science to investigate the molecular surface structure and electronic properties of materials. Researchers in technology, chemistry and physics employ field emission scanning electron microscopy (FESEM) to observe the small structures on the surface of cells and material [22,23]. The morphologies of fibers were analyzed by a Mira 3-XMU (Czech Republic) scanning electronic microscope. The surface of the samples was coated with a thin gold layer under vacuum. The presence of the nanocomposite on the fibers was confirmed by EDAXs.

### *Fourier transform infrared spectroscopy*

Fourier Transform infrared Spectroscopy (FTIR) is a technique which is used to obtain the infrared spectrum of the absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This confers a significant advantage upon a dispersive spectrometer which measures intensity in a narrow range of wavelengths at a time [24]. FTIR was employed for evaluation of the blank and treated polyester samples.

### *Characterization of electromagnetic and saturated magnetic features*

EMR, or EMF, is a general name for all types of electromagnetic radiation [25]. TES-593 (Malaysia), which is a device with the capability of determining waves at the precision of three integers and one decimal, was used to determine the electromagnetic properties of the treated polyester fabrics. To measure the saturated magnetic features of the treated samples, a VSM analyzer (Germany) which was mounted in a laboratory in Birjand, Iran, was used.

### *Modeling by central composite design (CCD)*

In this study, the design expert software of the response surface methodology was used to optimize conditions for synthesis of fabrics. This involved three factors including Silitin N85 concentration (3.0-9.0 g), silver nitrate (30-60 mL) concentration and UV irradiation time (30-60 min). The factors were chosen as independent variables, and their

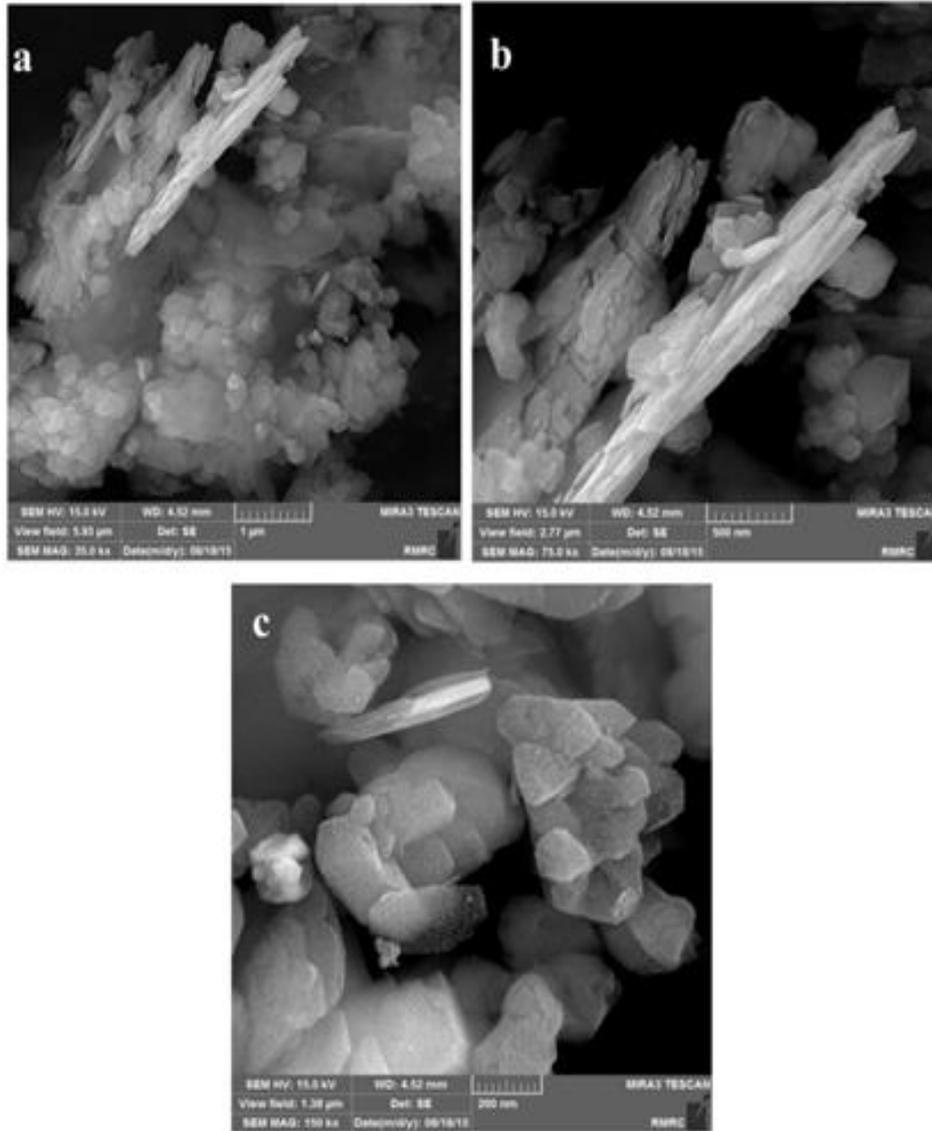
effects on the saturation magnetization response of the treated polyester fabrics were evaluated.

## RESULTS AND DISCUSSION

### *FESEM Analyses*

FESEM is a proper tool for studying the morphology of kaolinite inorganics. Figure 1 shows the morphology of the Ag/Kaolin/silica

nanocomposite. As it can be seen, kaolinite is accumulated with Ag nanoparticles in some areas. Due to UV irradiation, some particles are adhered and clustered together. Because of this adherence, the particle size is 50-70 nm due. The FESEM images of the treated and the blank polyester fabrics are given in Figures 2-6.



**Fig. 1.** FESEM image of the Ag/Kaolin/silica nanocomposite, (a) 35 kX, (b) 75 kX and (c) 150 kX.

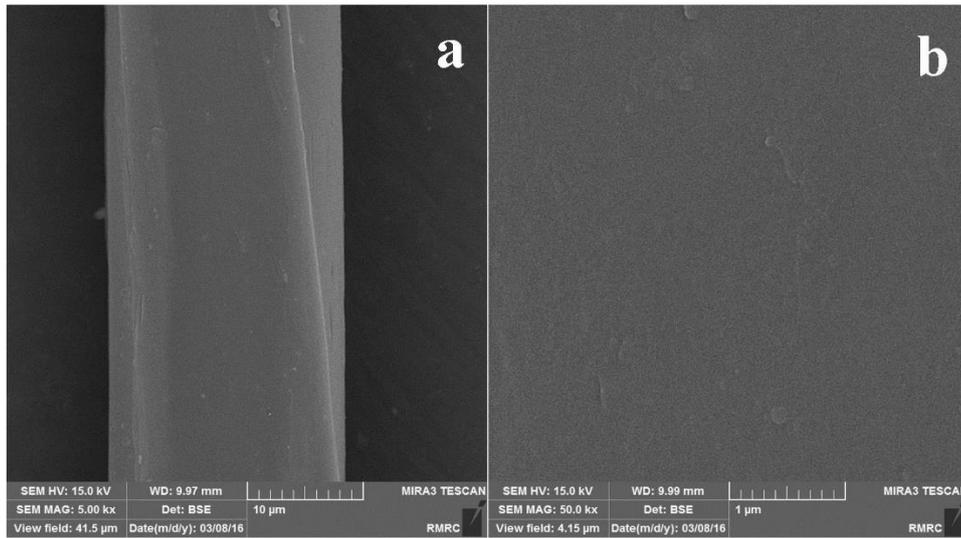


Fig. 2. FESEM images of the untreated polyester fiber (a: in 5.00 kx and b: 50.0 kx)

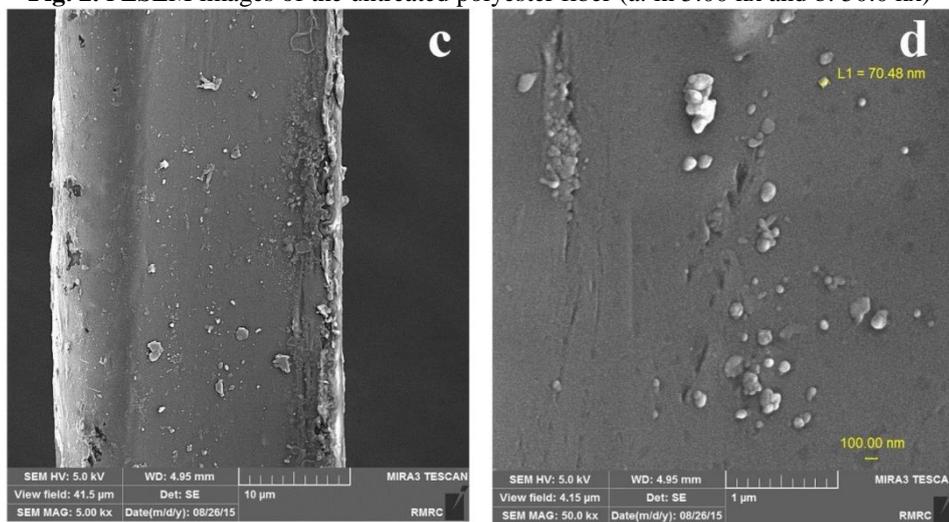


Fig. 3. FESEM images of the polyester treated with 60 mil of silver nitrate, 3.00 gr of Silitin N85, and 60-min UV radiation (c: in 5.00 kx and d: 50.0 kx)

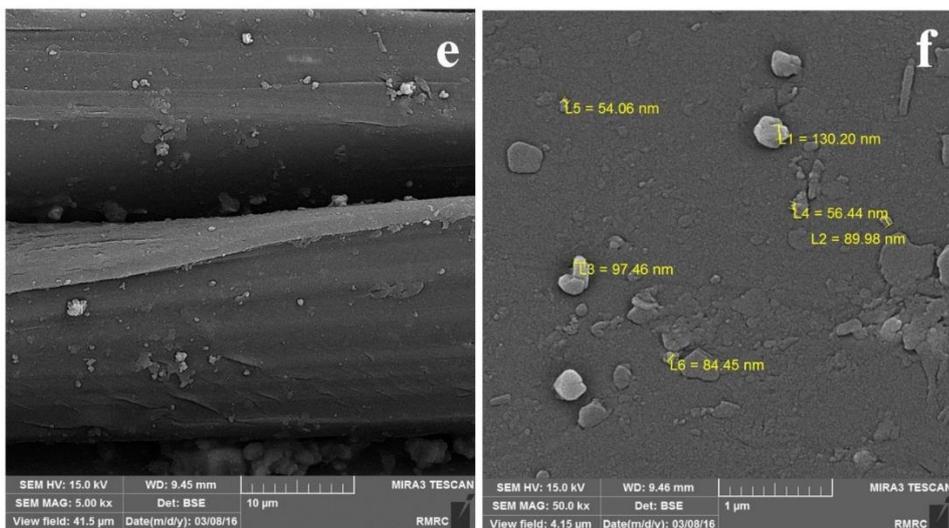
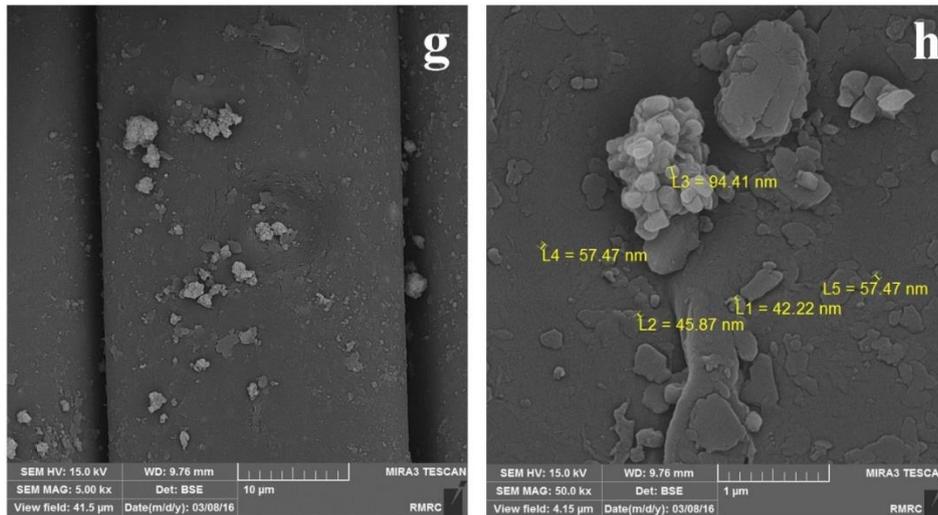
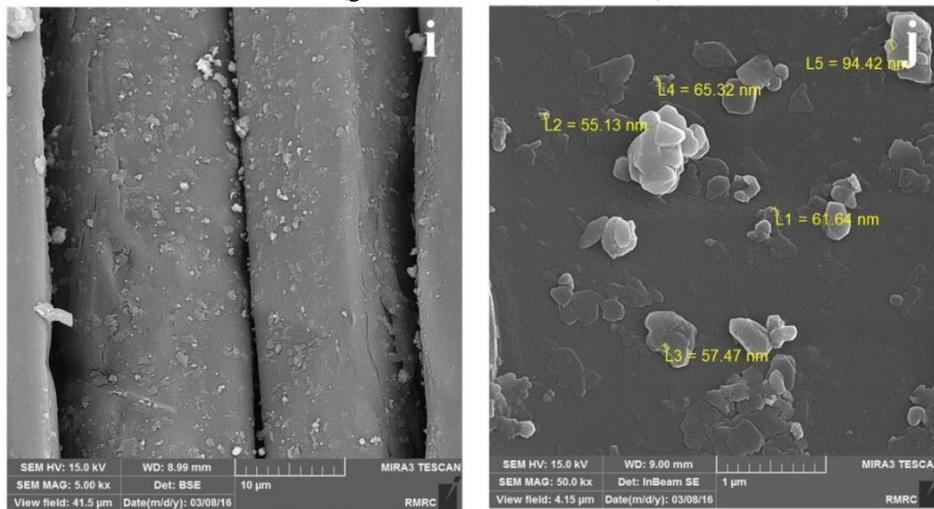


Fig. 4. FESEM images of the polyester treated with 60 mil of silver nitrate, 3.00 gr of Silitin N85, and 30-min UV radiation (e: in 5.00 kx and f: 50.0 kx)



**Fig. 5.** FESEM images of the polyester treated with 45 mil of silver nitrate, 6.00 gr of Silitin N85, and 45 min UV radiation (g: in 5.00 kx and h: 50.00 kx)



**Fig. 6.** FESEM images of the polyester treated with 60 mil of silver nitrate, 9.00 gr of Silitin N85, and 60-min UV radiation (i: in 5.00 kx and j: 50.00 kx)

The FESEM images of four treated polyester fabrics confirm the deposition and fixation of the Ag/kaolin/silica nanocomposite on the polyester fibers.

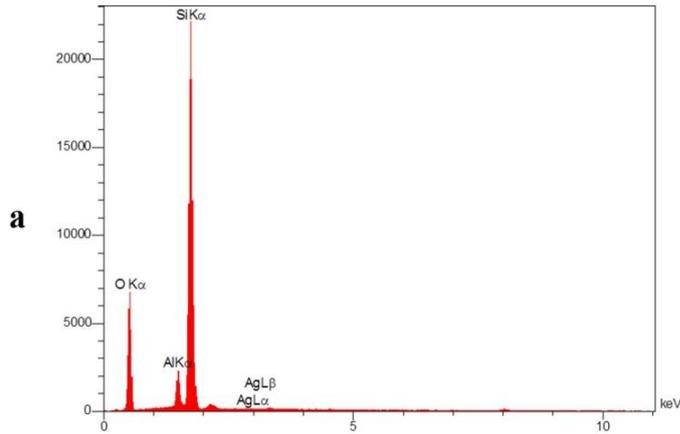
#### Scanning electron microscopy and EDX analyses

EDX is a robust tool for studying kaolin compounds and Ag/kaolin/ silica. The results of using the tool in this study are presented in Figure 7.

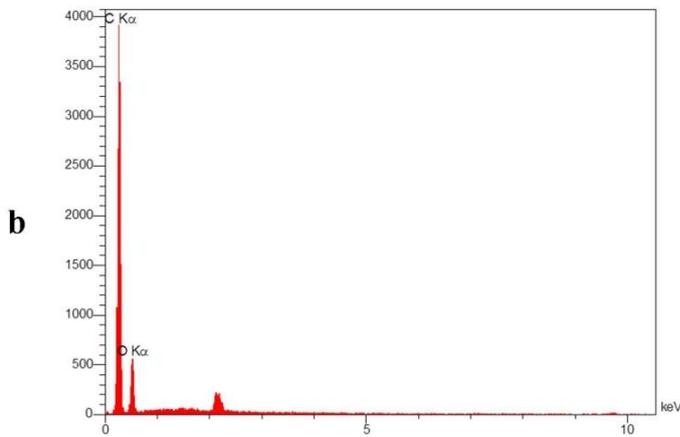
As it can be noted, the presence of elements such as K, Ti, Ca, Mg, Si, Al and Fe is confirmed.

#### Structural information obtained from the FTIR spectra

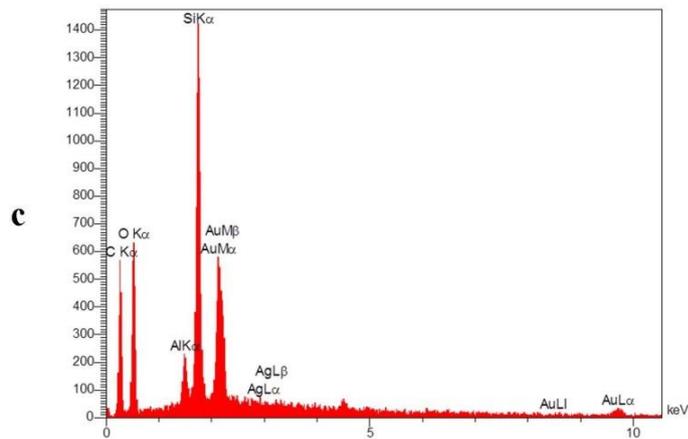
The FTIR analyses of four polyester fabrics coated with the Ag/kaolin/silica nanocomposite are shown in Figures 8-12. From these analyses, it can be concluded that a three-component nanocomposite has been deposited on all the studied fabrics.



Elements	W %	A %
O	60.52	47.17
Al	2.81	3.59
Si	30.50	43.04
Ag	6.17	6.20
Total	100.00	100.00



Elements	W %	A %
O	79.56	74.51
Al	20.44	25.49
Total	100.00	100.00



Elements	W %	A %
C	58.97	37.63
O	31.96	27.16
Al	0.56	0.80
Si	6.04	9.02
Ag	0.09	0.51
Au	2.38	24.87
Total	100.00	100.00

**Fig. 7.** EDX analysis of (a) the Ag/kaolin/silica nanocomposite, (b) the control, (c) the polyester fiber coated with the Silica/Kaolin/Silver nanocomposite

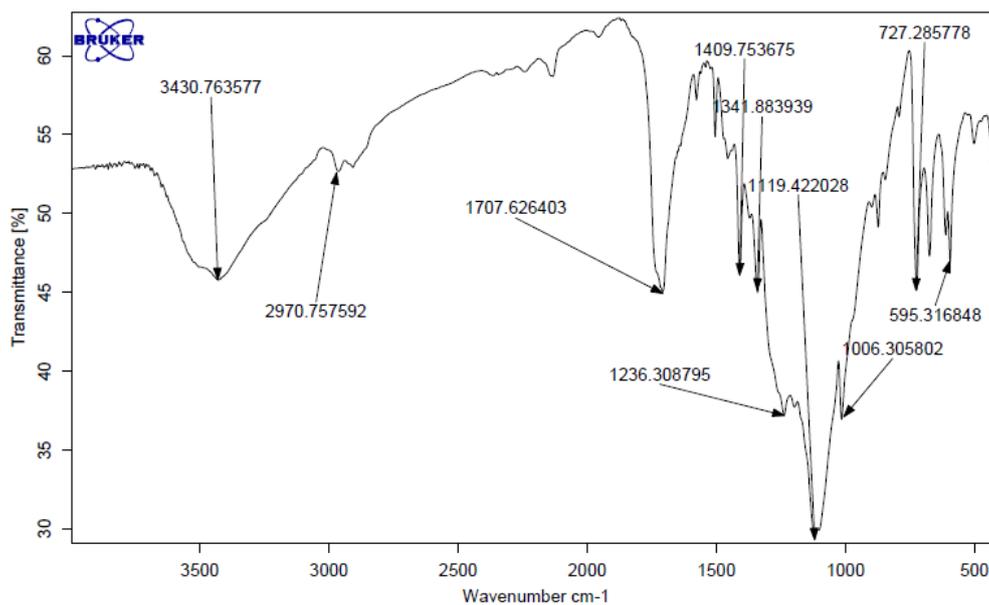


Fig. 8. FTIR of the untreated polyester fiber

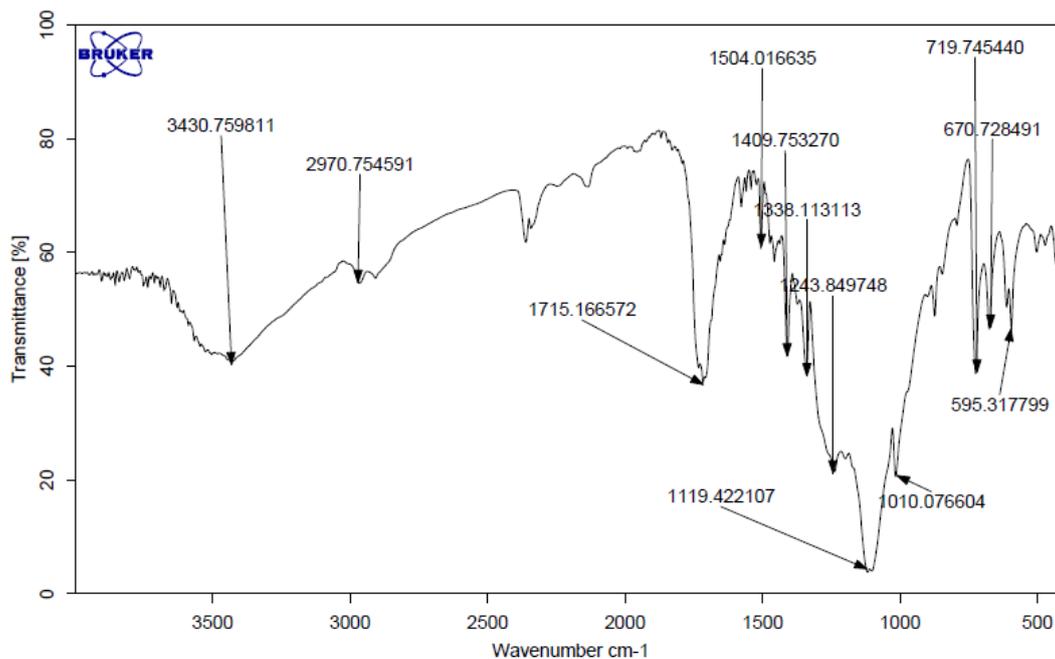


Fig. 9. FTIR of the polyester treated with 60 ml of silver nitrate, 3.00 gr of Silitin N85, and 60-min UV radiation

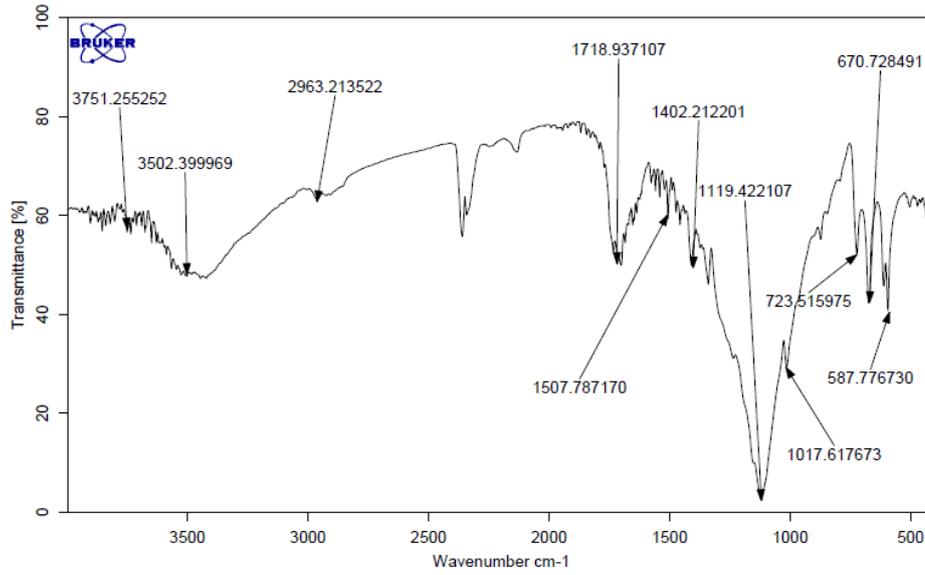


Fig. 10. FTIR of the polyester treated with 60 ml of silver nitrate, 3.00 gr of Silitin N85, and 30-min UV radiation

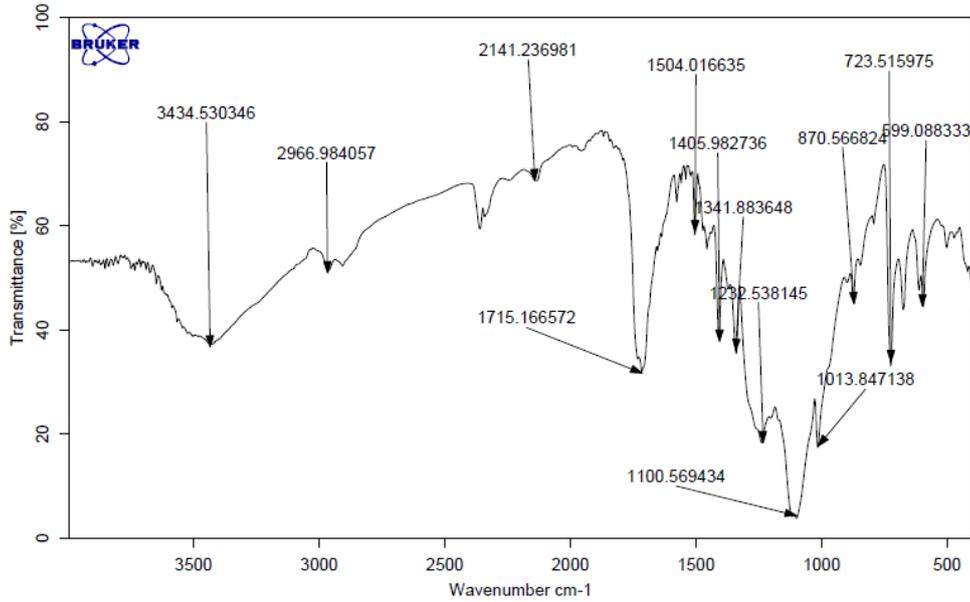
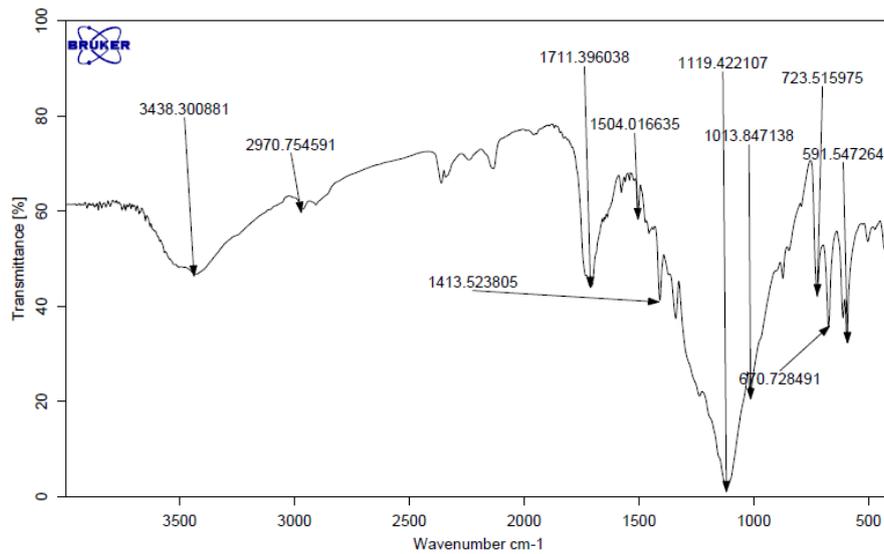


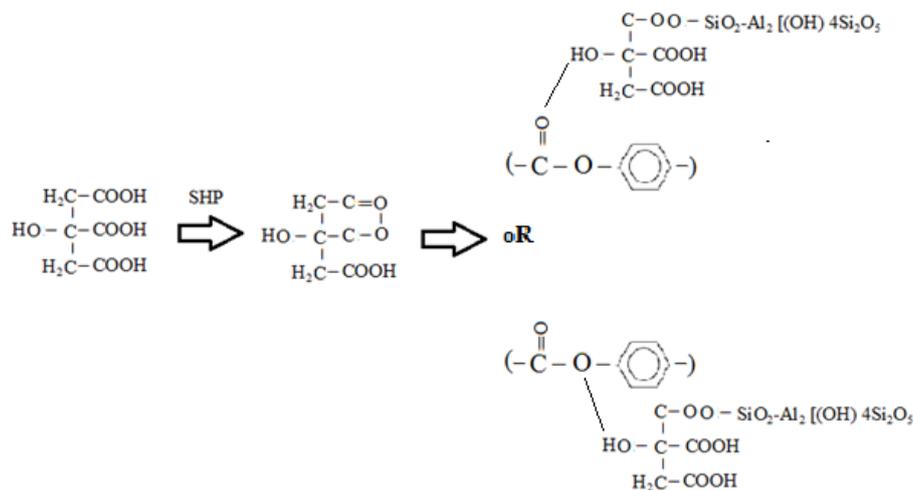
Fig. 11. FTIR of the polyester treated with 45 ml of silver nitrate, 6.00 gr of Silitin N85, and 45-min UV radiation



**Fig. 12.** FTIR of the polyester treated with 60 mil of silver nitrate, 9.00 gr of Silitin N85, and 60-min UV radiation

Various parameters such as the size, mobility and functional groups of the nanoparticles as well as the molecular structure of the fabrics and particles affected the stability of the nanoparticles on the fabric surface. The FTIR confirmed the interaction between the polyester and CA-free radicals. It seems that these interactions happened at a high level on the polyester fabric, and the nanoparticles were well

deposited on the samples (Figure 13). The size of the nanoparticles coated on the polyester fibers was estimated to be about 70 nm. The FESEM image clearly shows that the synthesized nanocomposite was thoroughly coated on the fibers. The presence of the three-component nanocomposite, i.e. Silica/kaolin/Ag, was also confirmed by EDAX.



**Fig. 13.** The suggested mechanism for coating of polyester fibers with CA cross-linking agents and nanocomposites

#### Saturation magnetic properties of the coated polyester fabrics

The highest and the lowest saturated magnetic values were obtained in experiments 10 and 6 respectively. It was shown that silitin N85, which contains Silica and kaolinite, in comparison with the other two parameters, has the greatest influence on saturated magnetic properties. In addition, in experiment 16, with 70 mL of silver nitrate, a higher

saturated magnetic value was achieved than in experiment 11, with 19.77 mL of silver nitrate. It can be concluded that silver nitrate has a very significant influence on the saturated magnetic value of samples. Regarding the changes in the UV irradiation time in experiments 18 and 3, it can be said that irradiation duration, too, has an influence on saturated magnetic properties, but its intensity is lower than that of the other two factors.

**Table 1.** The proposed experiment for preparation of electromagnetic polyester fabrics

Factor1				Response
Run	Silver Nitrate(ml)	silitin N85 (gr)	UV(min)	Ms(emu/g)
control	0	0.00	0	<b>0.02524</b>
1	45	6.00	45	<b>0.38829</b>
2	45	6.00	45	<b>0.37989</b>
3	45	6.00	19.7	<b>0.30446</b>
4	60	3.00	30	<b>0.31491</b>
5	30	9.00	30	<b>0.39785</b>
6	45	0.95	45	<b>0.25967</b>
7	45	6.00	45	<b>0.3525</b>
8	45	6.00	45	<b>0.38966</b>
9	45	6.00	45	<b>0.37988</b>
10	60	9.00	30	<b>0.42568</b>
11	19.77	6.00	45	<b>0.27866</b>
12	60	9.00	60	<b>0.41144</b>
13	30	9.00	60	<b>0.37426</b>
14	30	3.00	30	<b>0.29573</b>
15	30	3.00	60	<b>0.28497</b>
16	70.23	6.00	45	<b>0.39625</b>
17	45	11.05	45	<b>0.41384</b>
18	45	6.00	70.23	<b>0.32163</b>
19	60	3.00	60	<b>0.31225</b>
20	45	6.00	45	<b>0.38639</b>

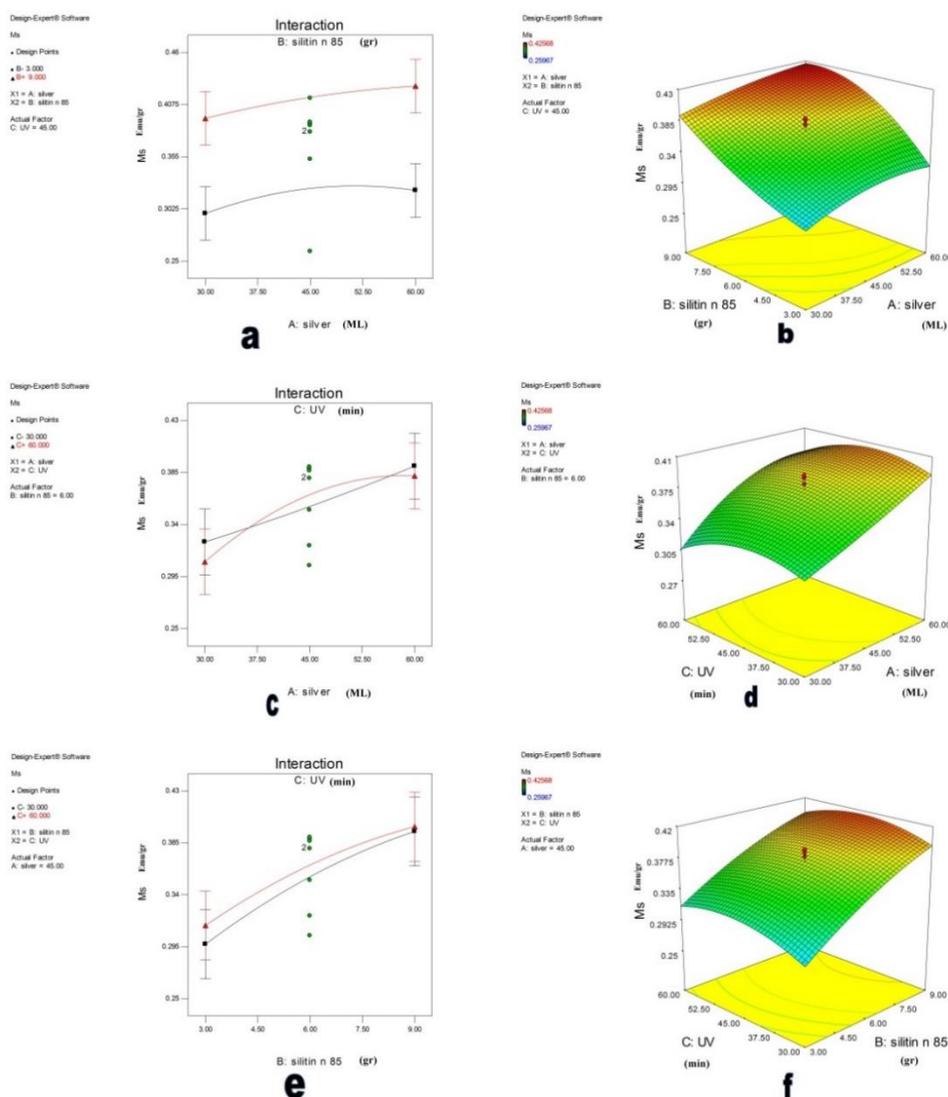
**Table 2.** The ANOVA results for the saturated magnetic data obtained from the treated polyester fabrics

ANOVA for response surface cubic model					
	Sum of squares	df	Mean Square	F Value	p-value
Source					Prob > F
Model	0.047343	13	0.003642	6.972619	0.0127
A(silver nitrate(ml))	0.006914	1	0.006914	13.23724	0.0109
B(silitin n 85(gr))	0.011884	1	0.011884	22.75394	0.0031
C(UV(min))	0.000147	1	0.000147	0.282226	0.6143
AB	4.3E-05	1	4.3E-05	0.082354	0.7838
AC	3.81E-05	1	3.81E-05	0.072877	0.7962
BC	7.45E-05	1	7.45E-05	0.142604	0.7187
A^2	0.001255	1	0.001255	2.403581	0.1720
B^2	0.001323	1	0.001323	2.532741	0.1626
C^2	0.00465	1	0.00465	8.903745	0.0245
ABC	1.95E-07	1	1.95E-07	0.000374	0.9852
A^2B	6.23E-05	1	6.23E-05	0.119296	0.7416
A^2C	0.000439	1	0.000439	0.840662	0.3946
AB^2	0.001465	1	0.001465	2.804872	0.1450
Residual	0.003134	6	0.000522		
Lack of Fit	0.002177	1	0.002177		
Pure Error	0.000957	5	0.000191	11.3688	0.0199
Cor Total	0.050476	19			

There was a significant difference among the variables in terms of how they affected the saturated magnetic properties. Based on the validity of the test designed by RSM, the P value was lower than 0.05. Therefore, it can be concluded that applying Ag, silitin N85, and UV irradiation time had significant effects on the optimum saturated magnetic features of the polyester fabrics coated with Ag/kaolin/silica nanocomposites.. In addition, given that the F value of silitin N85 is higher than that of the proposed model, silitin N85 has the highest influence on

saturated magnetic properties. The optimal formulation in terms of the amount of Ag, silitin N85 and the irradiation time of the UV 400 source to achieve the highest saturated magnetic conditions in the polyester fabric is presented in Table 4. Figure 15 indicates the saturated magnetization of the prepared samples in optimum conditions. The maximum saturated magnetization in optimum conditions was 42.195E-3 emu/g. The mathematical model presented for estimating the saturated magnetic features in the polyester fabric is given as follows.

$$M_s = +0.38 + 0.035 * A + 0.046 * B + 5.105E-003 * C + 2.319E-003 * A * B + 2.181E-003 * A * C - 3.051E-003 * B * C - 9.333E-003 * A^2 - 9.581E-003 * B^2 - 0.018 * C^2 + 1.563E-004 * A * B * C + 4.336E-003 * A^2 * B - 0.012 * A^2 * C - 0.021 * A * B^2 \quad (1)$$



**Fig. 14.** a) The effect of Ag and siliting N85 on the saturated magnetic properties in the coated polyester samples, b) 3D graphs for the response surface of the saturated magnetic properties in the polyesters coated with Ag/kaolin/silica and affected by Ag and silitin N85, c) effect of Ag and UV irradiation time on the saturated magnetization of the coated polyester, d) 3D graphs for the response surface of the saturated magnetic properties in the polyester coated with Ag/kaolin/silica affected by Ag and UV irradiation time, e) effect of silitin N85 and UV irradiation time on the saturate magnetic properties in the coated polyester, d) 3D graphs for the response surface of the saturated magnetic properties in the polyester coated with Ag/kaolin/silica affected by silitin N85 and UV irradiation time.

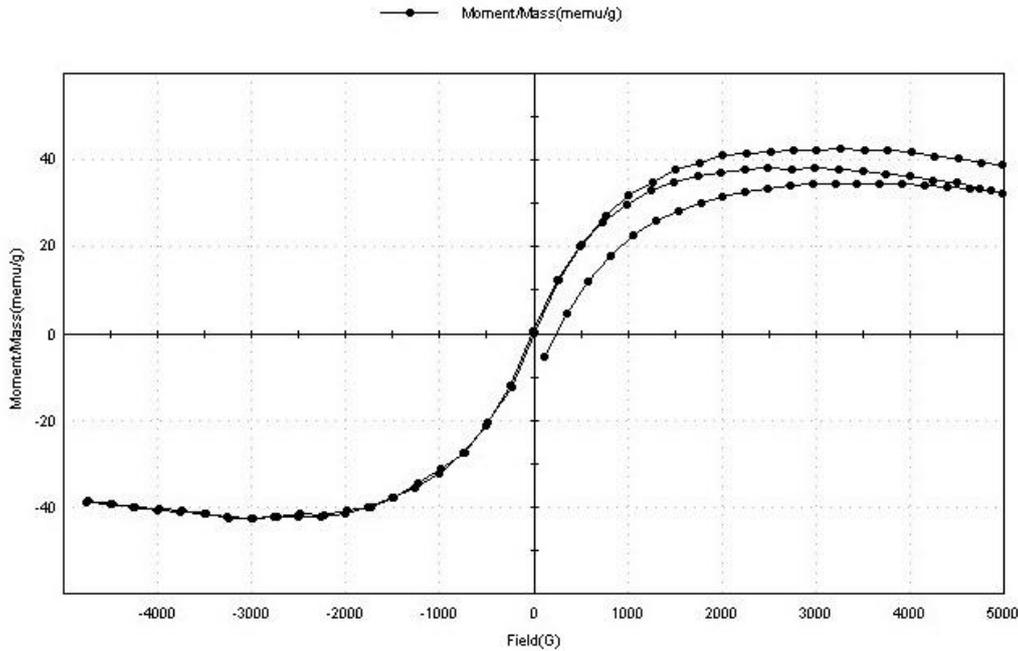


Fig. 15. Saturation magnetization graph for the prepared sample in optimum conditions (from 5000 to -5000 G)

Table 3. The output of VSM for the sample fabricated with the Ag/kaolin/silica nanocomposite in optimum conditions

Coercivity (Hci)	240.169 G
Initial Slope	35.218E-6 emu/(g G)
Magnetization (Ms)	42.195E-3 emu/g
Mass	40.349E-3 g
Retentivity (Mr)	2.1826E-3 emu/g

Table 4. Optimum formulation for making the saturated magnetic polyester fabrics

Solutions	UV*	silitin n 85*	silver*(0.4N)
	44.76 min	4.18 gr	43ml

*Evaluation of electromagnetic properties of the coated polyester fabrics*

An electromagnetic field is defined as a physical field produced by electrically charged objects. Once, an electromagnetic field is moved onto a polyester fabric coated with a three-component nanocomposite, there may occur three phenomena including reduction of absorbance, reduction resulting in reflection, and reduction due to internal transmission of electromagnetic waves. Electromagnetic Reflection, EMR, is a term used to characterize the electromagnetic waves reflected in the interface of two materials (surface reflection) or internal materials (volume reflection), while transmission means to transfer electromagnetic waves through a material (EMT). Absorbance represents conversion of irradiation energy into energies, mainly thermal energy, that are released and result in interaction with a material. The electromagnetic diagrams of the samples fabricated

based on experiments 4, 9, 12 and 19 were recorded in the range of 5000 - 8200 Hz, as presented in Figures 16 and 17.

Figure 16 indicates that the blank polyester fabric had the lowest electromagnetic reflection, but it increased with an increment in the amount of the Ag/kaolin/silica nanocomposite. From EMT and EMR diagrams related to the blank and the treated polyester fabrics at 5000-8000 Hz, it can be understood that the nanoparticles deposited on the fabrics underwent regular variations in the transmission range of 28-90 db. UV irradiation on the cationic Ag in the colloid dispersion as well as on the coated polyester fabrics caused the transmission rate to decrease due to addition of Ag nanoparticles to kaolinite layers. The reflection curves for the fabrics treated with the three-component nanocomposite show six sharp peaks at 5200, 5542, 6404, 6772, 7631 and 7800 MHz. Enhancements in the peak intensity could be due to resonance and matrix deformations and Ag features. When electromagnetic waves hit on the fabrics coated with nanocomposites, the waves interacted with kaolinite and were reflected. The internal waves were not reflected parallel to the external waves, and this reduced the wave intensity. As a result, EM absorbance by the nanoparticles was increased. Some researchers have stated that most of the absorbed energy is released as heat [19]. Our study indicates that, after irradiation of EM waves on coated polyester fabrics, Ag particles show bi- and multi-polar surface Plasmon resonance behaviors. Creation of these types of resonance can cause emission of electrons to the adjacent conductive

material and, in turn, cause an increase in the EMR of the nanocomposites deposited on fabrics. In

addition, interaction rate depends on the concentration, size and distribution of nanoparticles

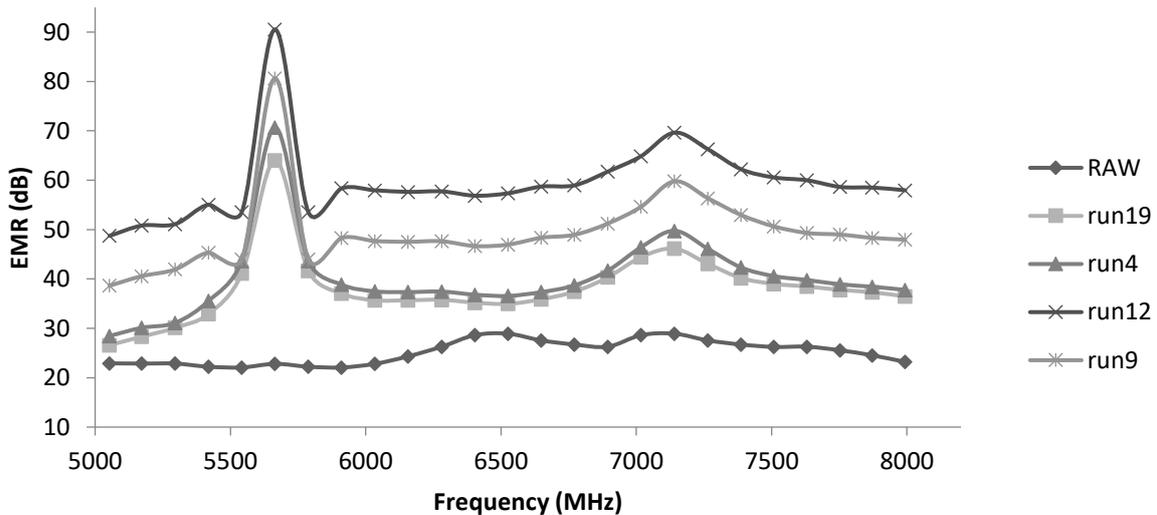


Fig. 16. The reflection graph of electromagnetic waves on the polyester coated with the Ag/Kaolin/silica nanocomposite at 5000-8200 Hz

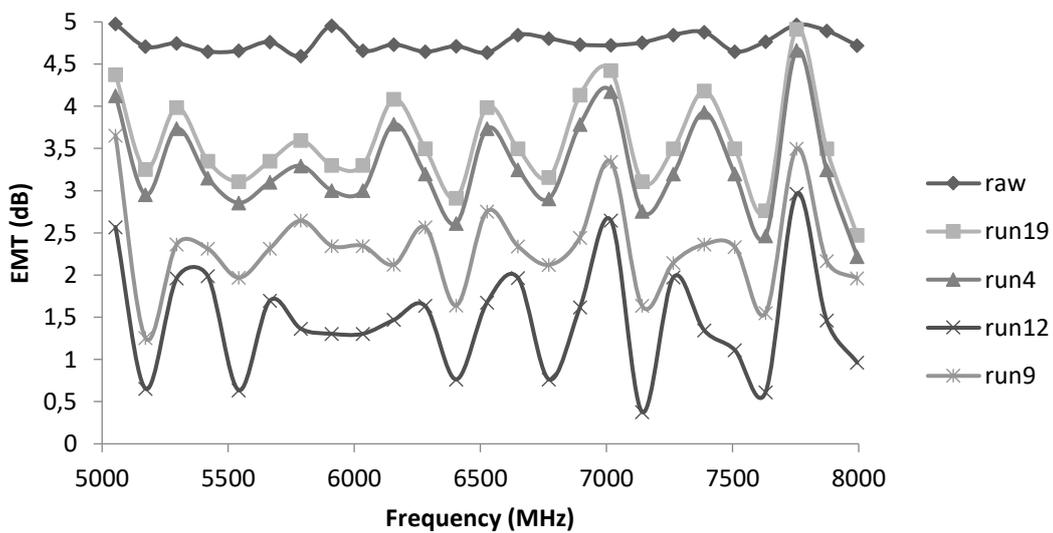


Fig.17. The transmission graph of electromagnetic waves on the polyester coated with the Ag/Kaolin/silica nanocomposite at 5000-8200 Hz

### CONCLUSION

In this research, Silica/Kaolin/Silver nanocomposite was synthesized and applied as a coating on polyester fabrics. The nanocomposite was made by the UV irradiation method using the formulation suggested in the literature. The existence of the nanocomposite deposited on the polyester fabrics was confirmed by EDX and FESEM. An FTIR analysis indicated that the nanocomposite was fixed on the polyester fibers. Thus, the proposed method can be used for fixation of the Silica/Kaolin/Silver nanocomposite on polyester fabrics. The electromagnetic transmission analysis of the treated fabrics showed that they block

electromagnetic waves; while blank fabrics let the waves pass through. This is obvious in the case of fabric treated according to experiment run 12. An electromagnetic reflection analysis also proved that treated polyester fabrics reflect electromagnetic waves more than untreated polyesters. After the experiments were designed and the saturated magnetic properties of the samples were modeled using DX software, it was confirmed that all the treated samples had saturated magnetic features in terms of the studied factors. VSM tests showed that the fabricated samples have  $42.195E-3$  emu/g saturated magnetic features under optimum conditions.

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