Polyester fabric modification through fabrication of nano-copper compounds

Modificación de tejido de poliéster mediante la fabricación de compuestos de nano-cobre.

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SCIENTIFIC RESEARCH

How to cite this paper:
Jolaei M., Montazer M., Rashidi A., Bameni Moghadam M., Polyester fabric modification through fabrication of nano - copper compound, Iran. Innovaciencia 2020; 8 (1): 1-12. DOI:http://dx.doi.org/10.15649/2346075X.1005

Reception date:
Received: 30 May 2020
Accepted: 30 September 2020
Published: 1 December 2020

Keywords:
In situ synthesis, nano-plates copper, polyester fabric, cetyltrimethylammonium bromide (CTAB)

ABSTRACT

Introduction This research introduces a new method of polyester fabric surface modification in order to achieve distinctive features. **Materials and methods.** The copper sulfate, sodium hydroxide and cetyl trimethylammonium bromide (CTAB) were used to synthesize copper nanoparticles, and loaded on the polyester fabric surface. The optimal sample was considered by SEM-EDX, FT-IR and XRD devices. **Results** SEM images showed copper nanoparticles in shape of nano-plates with 150 to 600 nm in length and thickness of about 30 nm. The best results obtained on the modified fabric processed at boil for 120 min. According to the results of experiments CuO and Cu^0 were synthesized on the fabric.

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1. INTRODUCTION

At the moment, attention to the nanomaterials is very important due to the low production costs and novel properties in the nanoscale. The latest research indicates an increased anticancer effect of nanomaterials such as gold, silver, and copper nanoparticles [1].

The synthesis of copper nanoparticles has become very important for its inherent properties. One of the most important methods available for the synthesis of copper nanoparticles is the chemical reduction method [2] to [4]. The desire to use copper nanoparticles has increased due to new optical, catalytic, mechanical, electrical, magnetic and thermal conductivity properties compared to gold nanoparticles and silver nanoparticles [5].

Several methods have been proposed to improve the structure of the fabric, which can generally be classified into two categories of chemical and physical methods. The physical methods are: plasma, corona, laser, electron beam, ion and neutron and chemical methods are: surface grafting, sol gel, use of different materials and microcapsules [6].

Polyester fabric was modified by synthesizing copper nanoparticles using triethanolamine as polyester aminolysis and pH adjustment, copper sulfate as a metal salt, sodium hypochosphite as reducing agent, and polyvinyl pyrrolidone as stabilizer. The modified fabrics indicated high tensile strength and flame retardant properties [7].

Acetonitrile has been used for the first time as a protective agent for the preparation of copper nanoparticles. Copper nanoparticles usually have a cube-shaped form with diameters less than 100 nanometers [8]. A number of researchers worked on the synthesis of nano-sized copper through the colloidal solution and its application on cotton fabrics. Nano-colloid of copper by chemical reduction of copper salt using sodium borohydride as a reducing agent is used in the presence of trisodium citrate produced particles with the average particle size of 60 to 100 nm [9]. Also we investigated the usage of alkaline glucose for synthesis of copper nanoparticles on polyester fabric forming nano-copper with a thickness of 30-40 nm on the surface of the fabric [10].

Also, the modification of the polyester fabric with beta-cyclodextrin has been reported with citric acid, butane tetracarboxylic acid, dimethyl dihydroxyl ethylene urea, and two siloxane-based softeners as a crosslinking agent. They showed the higher durability of beta-cyclodextrin on the polyester after 10 washes by using cross-linking agents [10] to [13].

Further, the antibacterial activities of copper nanoparticles were studied and confirmed their usage as antibacterial agent [14] to [15].

In this study, the surface modification of polyester fabric was performed and three factors of weight changes, color changes and antibacterial properties were considered and the sample with the highest weight and least color change was selected as the optimal sample. In addition, the antibacterial property was reported excellent for all samples due to the complete elimination of bacteria.

Applicability, availability and simplicity of methods are some of the most important features of this study.

2. MATERIALS

Materials used in this research were: polyester fabrics with 165 g/m^2 from the local market (Iran) and copper sulfate ($CuSO_4{*}5H_2O$), sodium hydroxide (NaOH) and cetyltrimethylammonium bromide (CTAB) from Merck Co.(Germany).

3. INSTRUMENTS

The absorption rate of nano-copper was studied by a spectrometer called Varian, Cary100 UV-Vis-NIR. Reflection spectra of nano-copper on the fabrics were investigated by a Spectrophotometer Color-Eye 7000A (USA). Surface morphology of the fabric was observed by scanning electron microscope TESCAN VEGA 5130 mm SEM/EDX (Czech Republic). To determine chemical groups on the fabric, and FT-IR spectrometer (model Bomem-mb100) was (Canada) used. Also, the crystal structure of the fabric was investigated by an X-Ray diffraction system (model XRD PTS 300) from SEIFERT Co. (Germany).

4. EXPERIMENTS

The design of the experiments was based on the response surface methodology (RSM) and using the Central Composite Design method (CCD). According to the design, 25 samples of polyester fabric in the size of 10 * 20 cm^2 were prepared and then washed with a nonionic detergent (1 g/L) at 60 °C for 20 min. Finally, the samples were dried and cured. The experiments were carried out at the boiling point (Table 1).

Table 1. Different factors based on response surface methodology (RSM) analysis and weight and color changes

NO	Copper sulfate (w/v %)	NaOH (w/v %)	CTAB (w/v %)	Time (min)	W(%)	ΔE
1	1	0.29	1	77.42	-3.20	46.33
2	1	0.1	0.35	120	4.11	13.52
3	1	1	1	120	-2.88	19.70
4	0.1	1	0.01	45	-3.07	39.42
5	1	0.1	0.01	45	-2.94	21.45
6	0.1	1	0.01	120	-2.05	35.55
7	1	1	1	45	-2.16	29.81
8	0.1	0.1	1	45	2.99	18.85
9	0.41	0.1	1	120	-3.35	17.54
10	0.1	0.51	0.64	120	-1.00	6.27
11	1	1	1	45	-3.77	21.75
12	0.67	0.51	0.01	120	-1.90	9.81
13	0.55	1	0.5	81.94	-2.94	15.64
14	0.78	0.54	0.52	45	2.17	24.66
15	0.97	0.96	0.33	120	2.53	29.49
16	1	0.1	0.01	45	-3.76	8.10
17	0.1	1	0.01	120	-0.65	9.72
18	0.96	0.1	0.97	120	-4.11	9.28
19	0.1	0.1	0.01	88.92	-3.62	44.78
20	0.55	0.31	0.48	89.04	4.26	10.68
21	0.67	0.51	0.01	120	3.60	12.31
22	0.13	0.39	0.33	45	-1.79	20.21
23	0.1	1	1	75.69	-3.84	23.14
24	0.1	0.1	1	45	-3.34	18.85
25	1	1	0.01	75.54	2.59	13.54

In the first step, weight changes, color changes and antibacterial properties were calculated for different samples and the optimum specimen was determined.

In terms of the values obtained from color changes and weight changes using the Design-Expert software (DOE), related models and equations were obtained.

Figure (1) and (2) show the three-dimensional response level for weight variations and color variations.

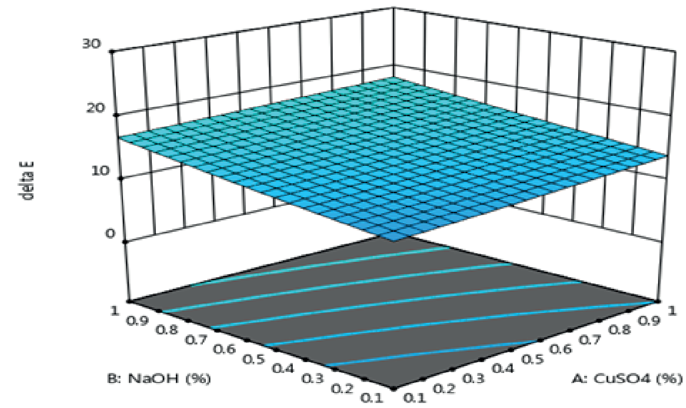
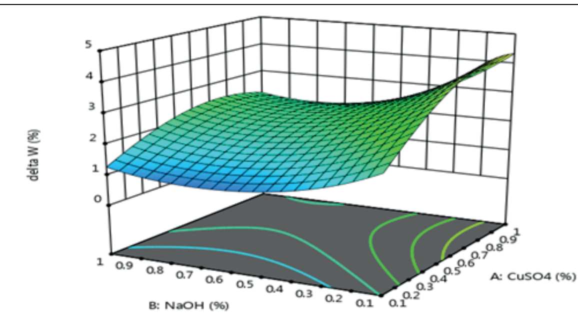


Fig1. *3D response surface for color variations*



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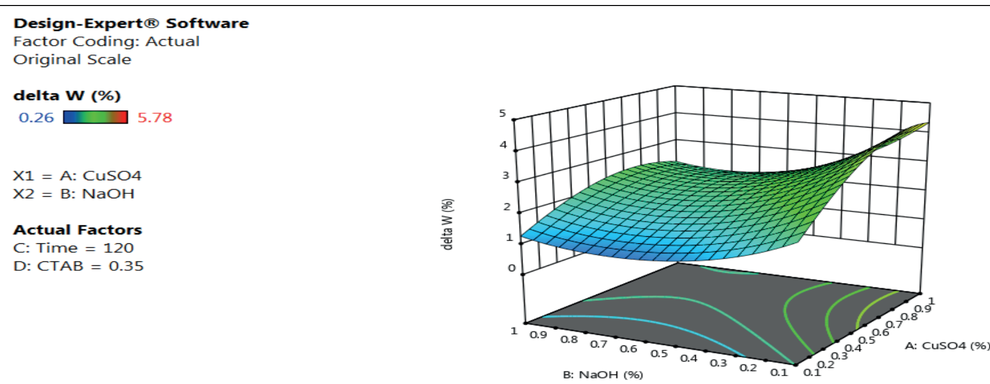

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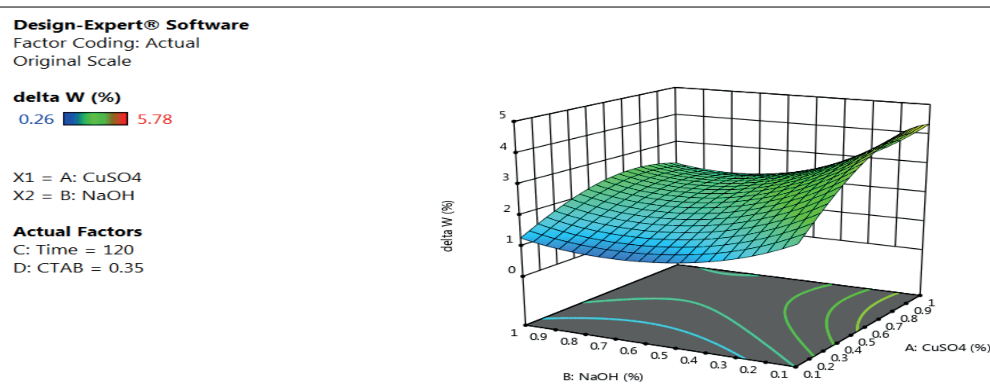

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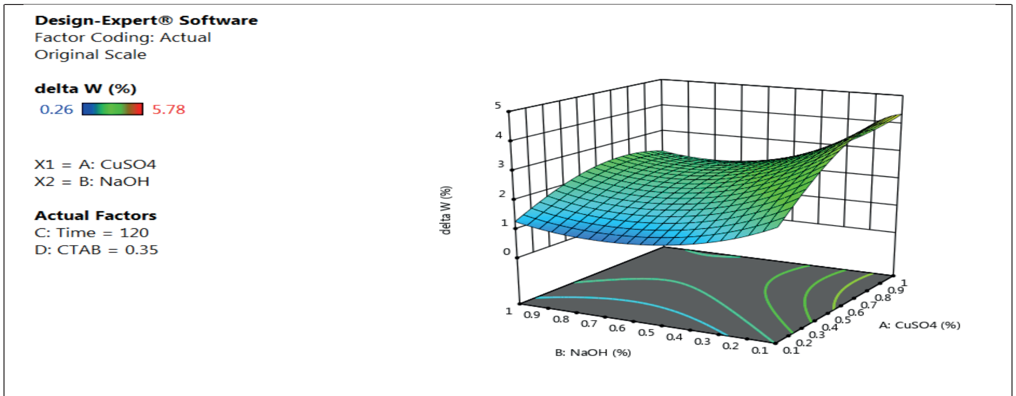

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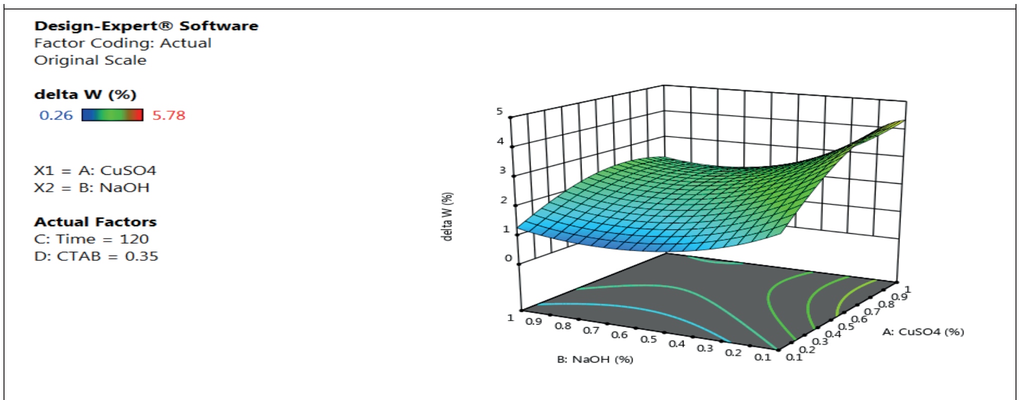

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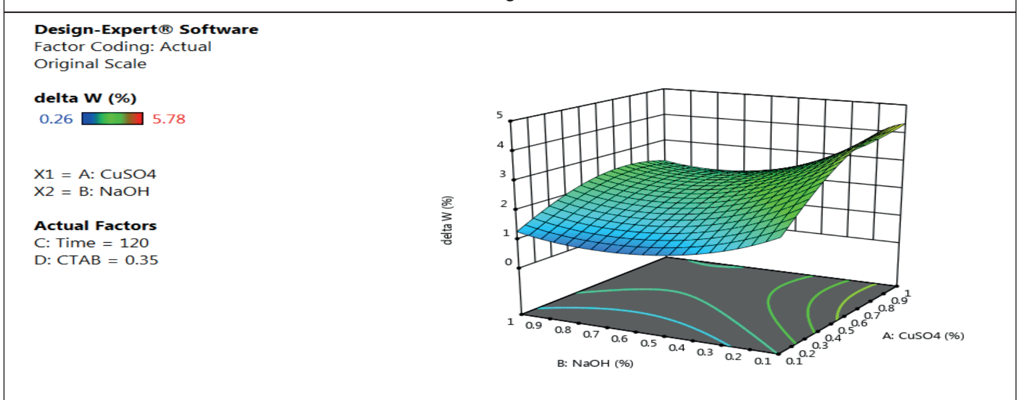

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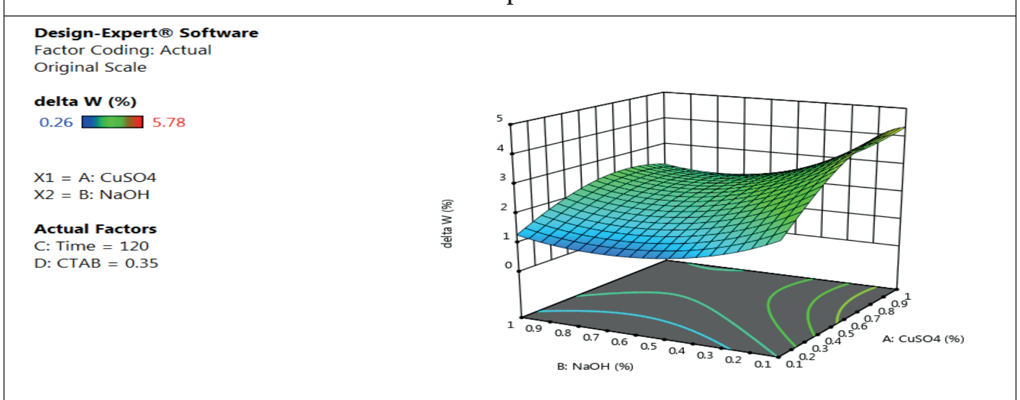


g

Fig2. 3D response surface for weight changes(fig a-g)

Figure (3) shows the 3D response surface for the desirability conditions. These conditions were adjusted based on the lowest amount of CTAB and the maximum operating time.

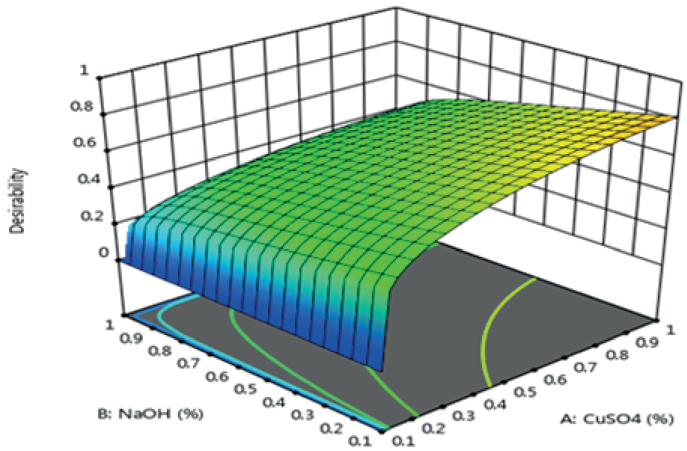


Fig3. Three-dimensional surface of response to achieve desirable conditions

According to the surface treatment performed on the fabric, a sample with more weight changes and less color changes in the surface of the fabric is selected as the optimum sample.

For statistical analysis of the operation, we used analysis of variance (ANOVA) based on weight and color variations. Color and weight variations are shown in Table (2) based on ANOVA analysis.

Table 2. ANOVA test for CTAB treated samples

	Sum of Squares	df	Mean Square	F	Sig.	
weight changes(ΔW)	Between Groups	64.906	24	2.704	2.121	0.013
	Within Groups	63.756	50	1.275		
	Total	128.662	74			
color changes(ΔE)	Between Groups	9134.647	24	380.61	1.896	0.028
	Within Groups	10035.189	50	200.704		
	Total	19169.836	74			

The obtained models based on the different copper sulfate and sodium hydroxide in the presence of CTAB are reported in equations (1) and (2).

$$\Delta E^{0.17}=2.11-0.59*CuSO_4-0.16*NaOH-0.004*Time-0.18*CTAB-0.029*CuSO_4*NaOH+0.003*CuSO_4*Time+0.52*CuSO_4*CTAB+0.002*NaOH*Time+0.10*NaOH*CTAB-0.002*Time*CTAB \quad (1)$$

$$\Delta W^{0.61}=1.21+1.12*CuSO_4-1.90*NaOH+0.028*Time-0.65*CTAB-0.48*CuSO_4*NaOH+0.013*CuSO_4*Time-0.089*CuSO_4*CTAB-0.006*NaOH*Time+0.58*NaOH*CTAB-0.0027*Time*CTAB-1.48*CuSO_4^2+1.87*NaOH^2-0.00019*Time^2+0.68*CTAB^2 \quad (2)$$

Finally, an optimum specimen is the sample treated with 1 % copper sulfate, 0.1 % sodium hydroxide, 0.35 % CTAB at boiling point for 120 min. At this stage, after determining the optimum sample. Subsequent experiments were performed to investigate the synthesis of nano-copper on the fabric.

To perform nano-copper synthesis studies, electron microscopy (SEM), x-ray diffraction (EDX), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) were used.

5. METHODS

5.1 Synthesis of nano-copper in solution

Possible reactions are (1-5) for the synthesis of nano-copper on a polyester fabric in an aqueous medium. CTAB can be reacted with copper sulfate and sodium hydroxide (1, 2).

According to reaction (3), due to the heat, copper hydroxide is first converted to copper oxide, and then through the formation of reducing agent in the environment through the reaction of alkali with the ester in polyester, to zero copper. This is shown in reaction (4).

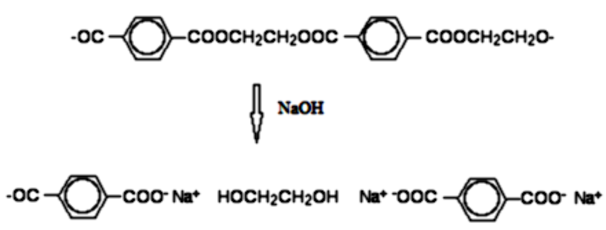
$$C_{19}H_{42}BrN + CuSO_4 \longrightarrow BrSO_4 + C_{19}H_{42}NCu \qquad (3)$$

$$C_{19}H_{42}NCu + 2NaOH \longrightarrow Cu\,(OH)_2 + C_{19}H_{42}Na_2N \qquad (4)$$

$$Cu\,(OH)_2 \xrightarrow{\text{HEATING}} CuO + H_2O \qquad (5)$$

$$CuO + \text{reducing groups} \xrightarrow{\text{HEATING}} Cu0 + H_2O \qquad (6)$$

Moreover, the polyester fabric in the presence of alkali is decomposed and turns into negative temple, where positive sodium ions can react with them according to reaction (5), and in the case of presence of positive copper replaces sodium ions.



(7)

Figure (4) shows nano-copper synthesis using copper sulfate, sodium hydroxide and CTAB in aqueous solution. Due to the simultaneous presence of the fabrics in the solution, these materials form bonds with active groups in the polyesters physically and chemically [11], [12].

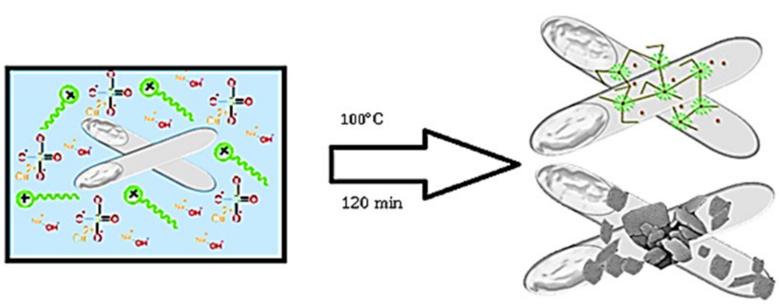


Fig4. Schematic of nano-copper synthesis on polyester fabric using CTAB

Therefore, based on chemical reaction (1-5), mainly using CTAB can cause different roles in the process of synthesis of nano-copper. It can act as controlling the particle size, reducing agent and preventing agglomeration and oxidation or bonding agents to the fiber and the fabric surface, depending on the conditions. This can carry out one or more of these tasks.

5.2 Surface reflection (K/S), ΔE

The amount of K/S was investigated at a wavelength of 600 nm and reported to be about 0.12. Also, the ΔE is reported at about 13.5. This shows very light color of the treated fabric that can be dyed in the following step.

5.3 Studying the chemical structure based on FTIR spectrum

In this experiment, the chemical structure of the raw polyester fabric and the nano-copper modified polyester fabric were examined. The results of changes in the size of the peaks or their displacement in FTIR spectroscopy show that it can be attributed to the effect of corrective actions on the chemical structure of the fabric. The FTIR spectra of samples on the fabric are shown in Figure (5).

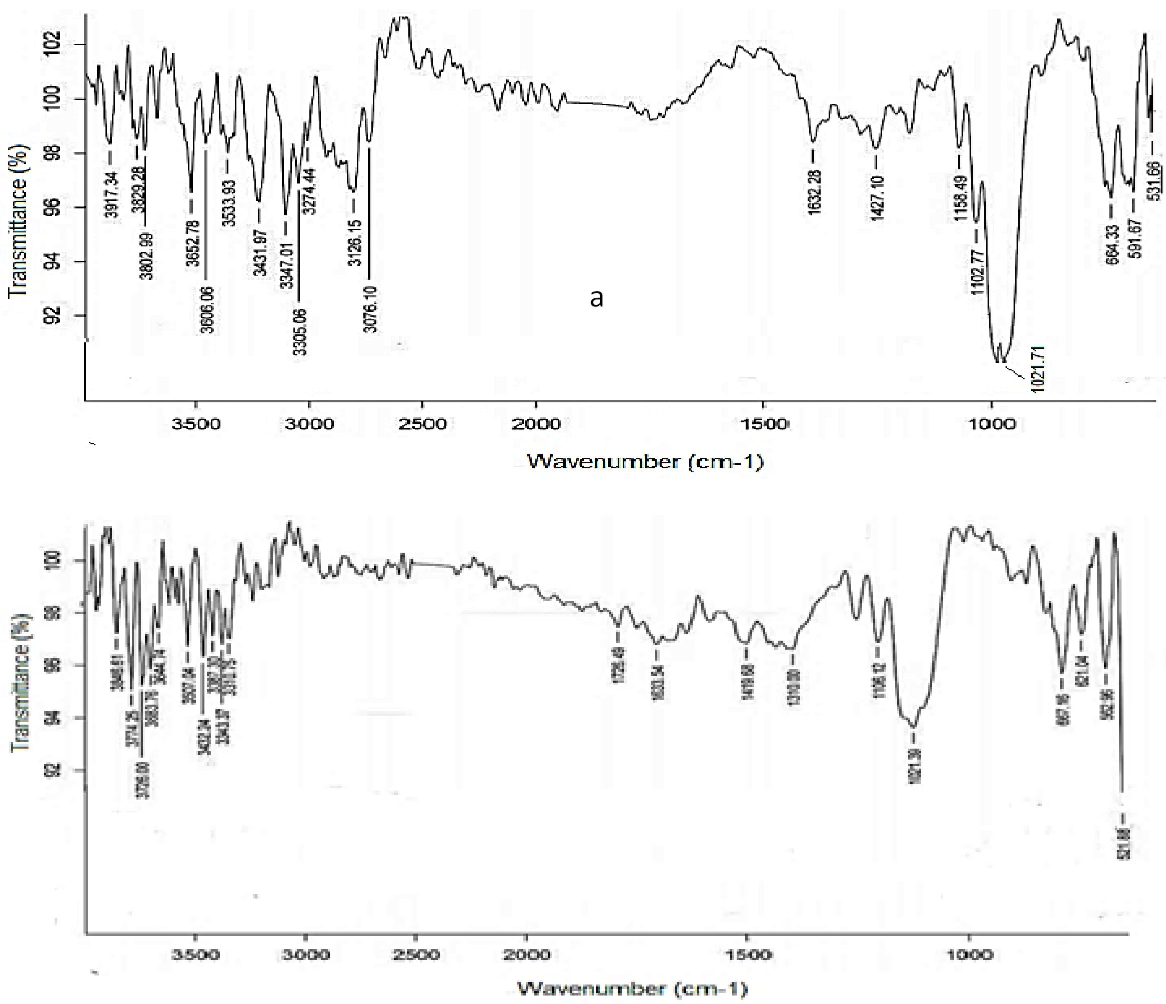


Fig5. FTIR spectrum related to a) raw and b) modified polyester fabrics with CTAB

These peaks or changes represent additional operations effect on the fabric chemical structure.

5.4 X-ray diffraction (XRD)

The crystal structure of the sample modified by XRD using electronic ray bombardment 20-40 KeV and wavelength (γ=0.154 nm) in the range of 2Theta from 10 to 100 degrees angle and at every step for 0.02 degrees has been scanned.

According to the standard JCPDS Copper: 04-0836, 2theta of nano-copper is specified in three values of 43.297, 50.433 and 74.420 that are mainly related to the Miller indices (hkl) for the peaks (111), (200) and (220).

The intensity and width of the peaks depend on the size of nanoparticles and their values [16]. The existences of prominent peaks related to nano-copper were shown near the standard range of the device. The results are reported in Figure (6).

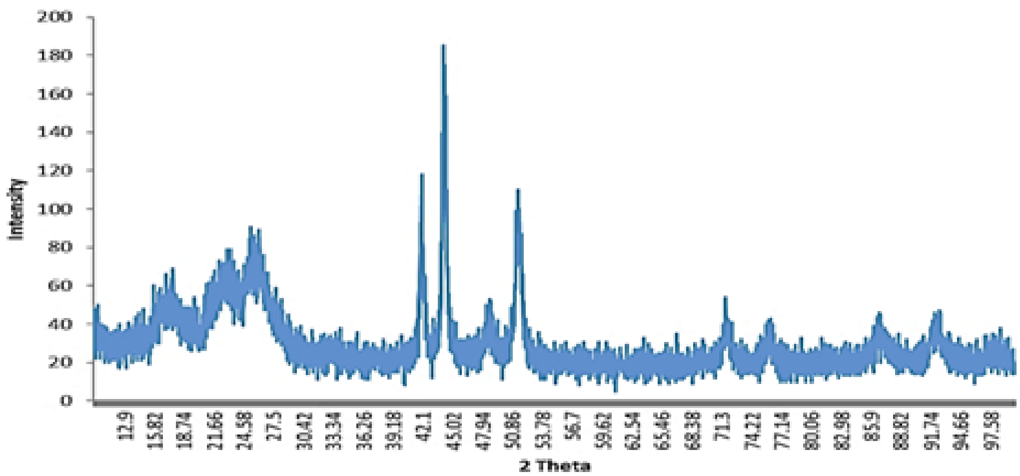


Fig6. X-ray diffraction related to the finishing sample using CTAB

5.5 Electron microscope analysis (SEM / EDX)

The nanoparticles on the surface of the fabric can be seen by using SEM images. Nanoparticle size can be measured using more magnification and their shape can be observed in different forms on the fabric [17].

In Figure (7), SEM image of the raw sample is shown where the fiber surface before treatment is smooth and uniform. Based on electron microscopic images, copper nanoparticles in Figure (8) related to the treated sample.

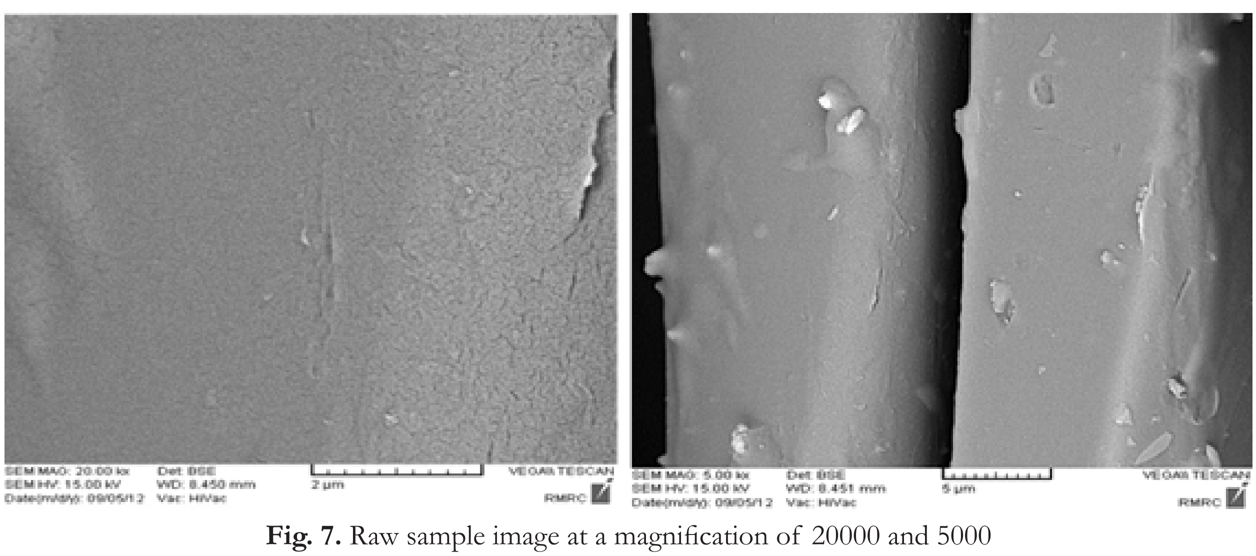


Fig. 7. Raw sample image at a magnification of 20000 and 5000

Figure (8) shows the synthesis of nano copper deposed on the fabric surface. The important point is the form of copper nanoparticles synthesized shaped in the form of sheets with a very small thickness about 30 nm.

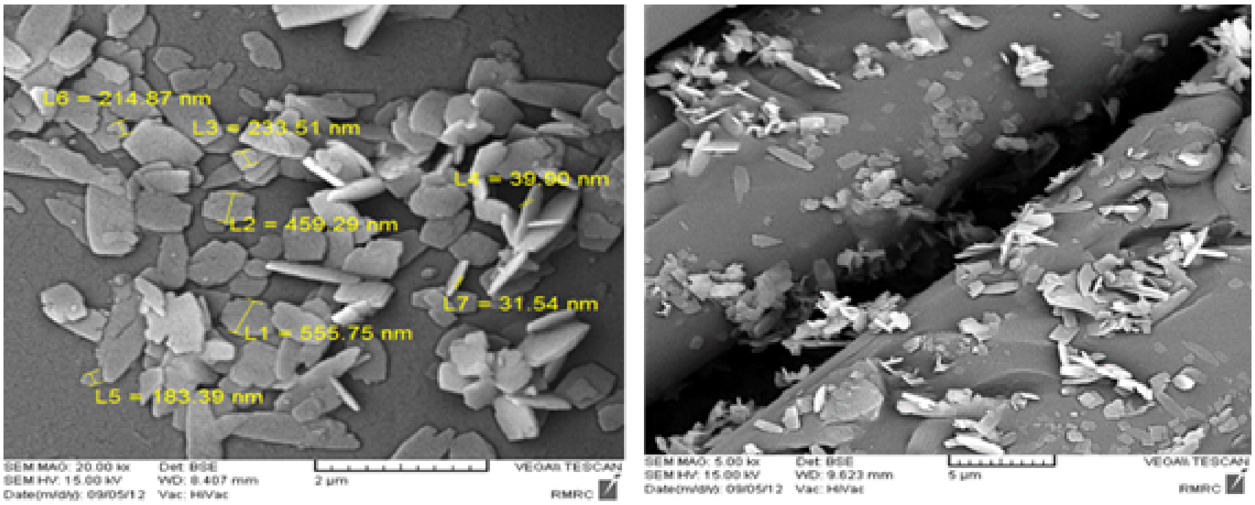


Fig. 8. Surface of the treated sample with copper sulphate and CTAB with a magnification of 20000 and 5000

The reason for the formation of nanoplates could be reaction of copper ions with CTAB and sodium hydroxide.

5.6 Analysis of the EDX patterns

The combination of copper nanoparticles was studied with X-rays and analyzed by EDX. This spectrum shows a number of sharp peaks that correspond to the various elements on the fabric surface [18], [19]. In Figure (9), the elements of copper, carbon and oxygen are shown based on the filter.

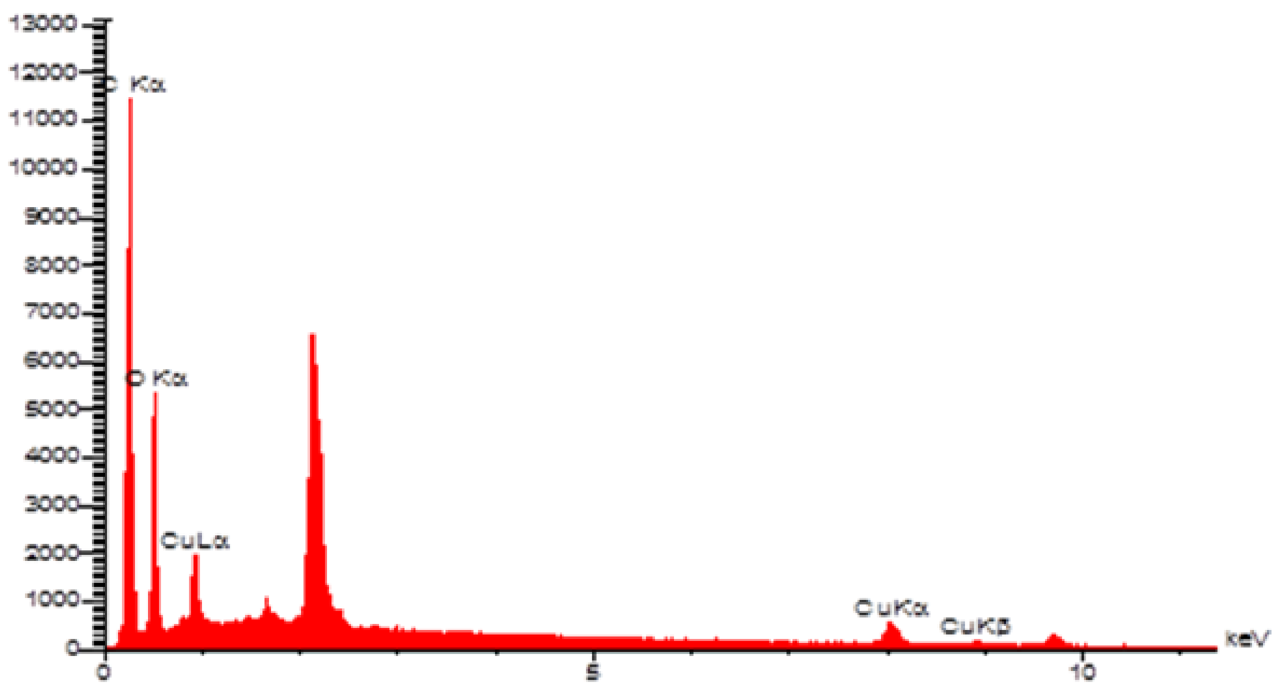


Fig9. EDX analysis of the treated sample with CTAB

According to EDX analysis, it can specify the existence of copper on the treated sample. Copper content on the surface of the fabric based on EDX analysis for the finished sample is 0.16 %.

6. CONCLUSION

This study shows the synthesis of nano-copper and surface modification of polyester fabric with using copper sulphate in the presence of sodium hydroxide and CTAB in one step. CTAB has a different role in the synthesis process of nano-copper on polyester fabric including controlling the nanoparticle size, reducing agent, preventing the oxidation acting as capping agent. Moreover, the placement of synthesized copper nanoparticles with different sheets with very low thickness occurred on polyester fabric. SEM images showed the nano-copper synthesis on polyester fabric as plates in length of 150 to 600 nm and thickness about 30 nm. Nanosheets in different shapes of roughly rectangle can be due to the molecular weight of the compounds created in this research.

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