The utilisation of polydopamine interlayer to add silver nanoparticles (AgNPs) to PET fabrics

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ABSTRACT – REZUMAT

The utilisation of polydopamine interlayer to add silver nanoparticles (AgNPs) to PET fabrics

Silver nanoparticles are structures used in many areas such as antibacterial materials, increasing conductivity, wastewater treatment, etc. In recent years, demand for their use in textile products has been increasing. Starting from this point, this study aimed to produce silver nanoparticles on the surface of PET fabric to meet the different properties expected from silver. For this purpose, polydopamine coating was applied to PET fabric by in situ polymerization. Then, fabric structures containing polydopamine and silver nanoparticles were obtained with a reduction reaction of three different molarity silver nitrate salts. The after-washing condition of the fabric with the highest produced amount of silver nanoparticles was examined. Spectrophotometric colour measurement, FT-IR, SEM and EDX techniques were performed. The best results were obtained on the fabric where silver nitrate was applied at 50 mM. The amount of silver nanoparticles in this fabric was measured as 1.35% after washing.

Keywords: *polyethyleneterephthalate, polydopamine, silver, silver nitrate, nano-coating*

Utilizarea stratului intermediar de polidopamină pentru adăugarea de nanoparticule de argint (AgNP) la țesăturile PET

Nanoparticulele de argint sunt structuri utilizate în multe domenii, cum ar fi materialele antibacteriene, creșterea conductivității și tratarea apelor reziduale etc. În ultimii ani, cererea pentru utilizarea lor în produsele textile a crescut. Pornind de la acest punct, acest studiu a urmărit să producă nanoparticule de argint pe suprafața țesăturii PET pentru a satisface diferitele proprietăți așteptate de la argint. În acest scop, s-a aplicat un strat de polidopamină pe țesătura PET prin polimerizare in situ. Apoi, structurile țesăturilor care conțin polidopamină și nanoparticule de argint au fost obținute printr-o reacție de reducere a trei săruri de nitrat de argint cu molarități diferite. A fost examinată starea de după spălare a țesăturii cu cea mai mare cantitate de nanoparticule de argint produsă. Au fost efectuate tehnici de măsurare spectrofotometrică a culorii, FT-IR, SEM și EDX. Cele mai bune rezultate au fost obținute pe țesătura în care nitratul de argint a fost aplicat la 50 mM. Cantitatea de nanoparticule de argint din această țesătură a fost măsurată la 1,35% după spălare.

Cuvinte-cheie: *polietilentereftalat, polidopamină, argint, nitrat de argint, acoperire nano*

INTRODUCTION

Surface modifications have great importance in expanding the application areas of textile materials and in the production of technical textiles. Modifying surfaces enables control and improvement of surface properties and imparts new functions such as electrical conductivity, UV protection, antibacterial activity and super hydrophobic character. Many different approaches have been investigated in the literature to activate the surface of textile materials. Plasma treatments and chemical methods appear to be the most promising techniques for providing functional groups. However, these techniques damage the fabric surface and may negatively affect the mechanical properties of the fabrics. Apart from these, using an intermediate layer containing functional groups between the coating and the fabric is a new approach.

Dopamine, a substance with strong adhesion and high reactivity, can be used as an intermediate layer to provide fabrics with properties such as super hydrophobic, antibacterial, UV protection and dye adsorption on their surfaces. Adhesive polydopamine can be obtained by self-oxidative polymerization of dopamine under alkaline environmental conditions. Many surfaces can be functionalized by using this simple but versatile intermediate layer. The used surface modification technique here is both easy and solvent-free [1, 2]. Xu et al. tried to provide antibacterial activity by modifying the surface of cotton fabrics with dopamine. In their studies, the reduction of silver nitrate on the intermediate layer formed by the polymerization of dopamine was attempted. It has been observed that silver nanoparticles formed on the cotton fabric surface in this way provide significant antibacterial activity. In the study, dopamine molarity was used as 0.2 mol/l and silver nitrate solution as 0.29 mol/l. It was reported that there was no change in activity after 30 washings [3]. Xu et al. conducted similar studies on polyester fabric, where the application process is more difficult than cotton, by reducing the dopamine molarity by 50% (0.1 g/mol) without changing the silver nitrate molarity. Similarly, researchers reported significant antibacterial activity before and after washing [4]. Zhang et al. conducted studies to accelerate dopamine polymerization [5]. With the method recommended by these researchers, Ou et al. used polydopamine to make superhydrophobic cotton fabrics. The durability of nano-coated fabrics under different conditions was also examined by researchers [6]. Ding et al. tried to provide photocatalytic activity by using iron (III) chloride in addition to silver nitrate reduction on polydopamine-treated cotton fabric. The researchers, who achieved successful results in their studies, stated that they proposed an efficient method for cleaning dye-containing textile wastewater. They also emphasized that this would have a facilitating effect on the recovery of photocatalytic materials [7]. Miao et al. applied polydopamine and silver to a textile fabric whose contents they did not specify. It has been stated that the resulting surface can be used to effectively separate oil/oil mixtures of different polarities and oil/water mixtures. It has been emphasized that this surface can be efficient for dye removal and wastewater cleaning with the photocatalytic effect and that this effect can be significantly accelerated in the presence of sodium borohydride [8]. Sodium borohydride ($NaBH_4$) is a reducing agent used to produce nano-sized silver by reducing silver nitrate ($AgNO_3$) salt. It has been used for this purpose in many studies [9, 10]. However, these and similar materials are toxic and corrosive. The polydopamine technique is not dangerous to humans and the environment, unlike cross-linkers frequently used for surface functionalization, and is a green technology inspired by mussel shells. The functional groups of polydopamine have a high affinity for various functional molecules, giving it good adhesion properties. Therefore, the polydopamine film adheres well to the substrate. It can be estimated that it will have wide usage areas in the coming years due to its more harmless nature and it will be produced at lower costs.

As cotton was at the forefront of applying polydopamine to textile materials in previous years, there has been a focus on applying polydopamine to different textile materials in the last few years. Applications on fabrics such as acrylic [11], polypropylene [12] and wool [13] were reported. However, there are a limited number of studies on polyester fabrics, which are the most consumed among all fibres [14]. Silver nanoparticles are structures used in many areas such as antibacterial materials, increasing conductivity, wastewater treatment, etc. In recent years, demand for their use in textile products has been increasing. Starting from this point, this study aimed to produce silver nanoparticles on PET fabric to meet the different properties expected from silver. For this purpose, polydopamine coating was applied to PET fabric by in situ polymerization. Then, fabric structures containing polydopamine and silver nanoparticles were obtained with a reduction reaction of three different molarity silver nitrate salts. The after-washing condition of the fabric with the highest produced amount of silver nanoparticles was examined. Fourier Transform Infrared Spectroscopy (FT-IR), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) techniques were performed.

MATERIAL AND METHODS

In this study, PET fabric with a weight of 150 grams per square meter, was used. The fabric was supplied by Karesi Holding, a PET fabric manufacturer in Bursa/Turkey. The fabric was washed with acetone to remove impurities before use in the experiment. Polydopamine coatings were carried out on PET Fabric with oxidative self-polymerization. For polydopamine synthesis, dopamine hydrochloride (99 wt.%) obtained from Alfa Aesar and tris (hydroxymethyl)-methylamine (99 wt.%) obtained from Fisher Scientific were used. Dopamine hydrochloride was prepared as 10 mM by dissolving it in purified water. Aqueous dopamine solution was mixed homogeneously with tris (hydroxymethyl)-methylamine at 10 mM to keep the pH constant at 8.5 throughout the reaction. Tris buffer is an alkaline compound used to deprotonate dopamine solution. Then, the fabric was placed in the solution and polydopamine (dopamine-melanin) nanofilms on the fabric were formed by mixing at 50 rpm in the shaker of Ataç Laboratory machines, a textile machinery manufacturer in Turkey. The process was carried out for 24 hours in standard atmospheric conditions (20±2^{o}C, 65±2% relative humidity) according to EN ISO 139.

Polydopamine nanofilm-covered surfaces were formed by oxidative self-polymerization of dopamine on the fabric surface under constant alkaline conditions. The fabrics taken from the solution after 24 hours were rinsed with cold water and left to dry for 24 hours under standard atmospheric conditions. In this way, the first stage of the study was completed.

In the second stage, silver nitrate (99 wt.%) salt obtained from Sigma Aldrich was prepared in three different molarities (10 mM, 20 mM, 50 mM) for the synthesis of silver nanoparticles. Dried polydopamine-coated fabric surfaces were treated in these solutions for 6 hours. In this way, PET fabrics were coated with silver nanoparticles with the help of a polydopamine interlayer. Thus, the fabric structures consisting of polydopamine and silver nanoparticles were obtained by reduction reaction. These layered fabric surfaces were dried in an oven at 105^{o}C for 4 hours. To determine the durability of polydopamine (PDA) nano-coating and silver nanoparticles (AgNPs) on the fabric, washing according to EN ISO 105-C06 (A2S method) was applied to two of the dried samples (PET+PDA and PET+PDA+50 mM Ag).

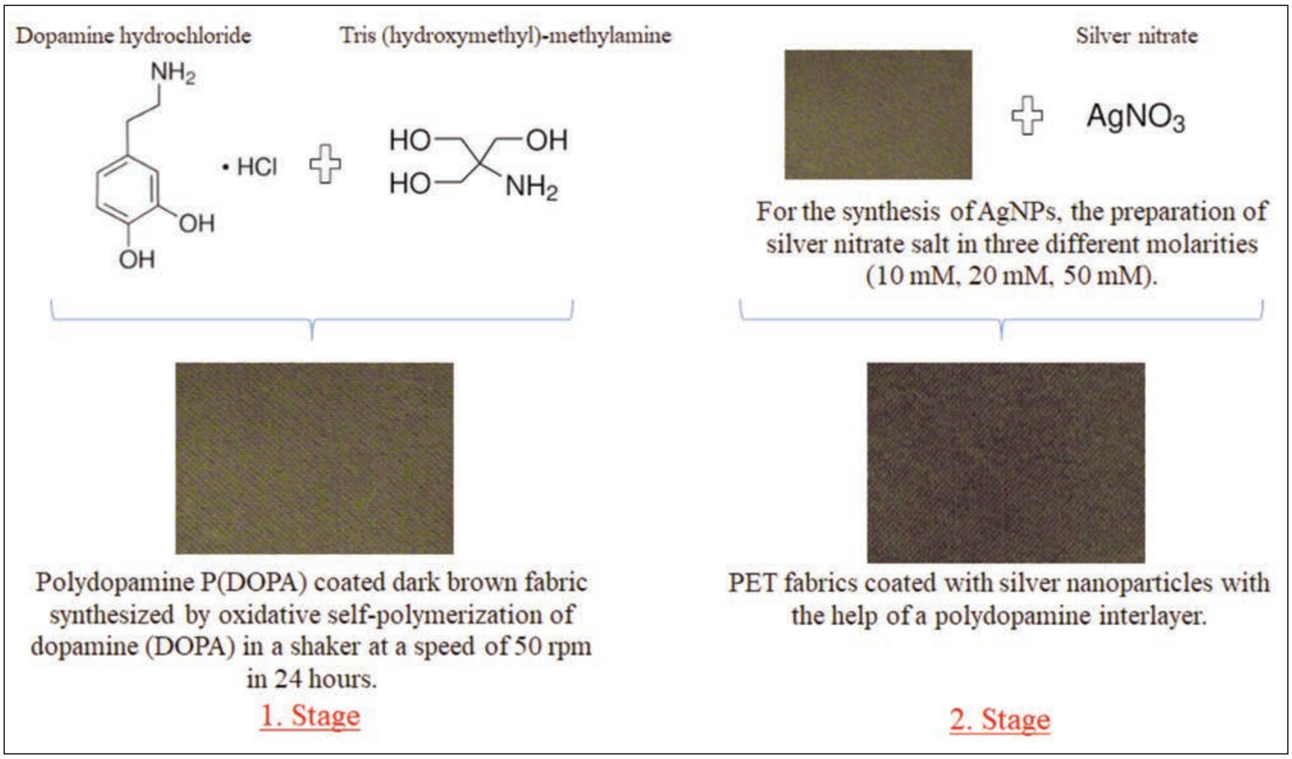

Fig. 1. Graphical abstract for all process steps

In this method, the fabric was washed with sodium perborate for 30 minutes at 40°C. In this way, the second stage of the study was completed. All process steps are summarized in figure 1.

After the applications were made, spectrophotometric colour measurements of the fabrics were measured using the Minolta CM 3600 D device and the lightness (L*), level of redness or greenness (a*) and level of yellowness or blueness values (b*) were examined. FT-IR (Jasco FT/IR-6700) spectrum, scanning electron microscope (FEI Quanta 650 Field Emission SEM) and energy dispersive spectrometer (EDX) were used to characterize the fabrics. The bonds in the sample structure, the status of the bonds and the functional groups were examined with FT-IR. The surface shapes of the samples were imaged at a magnification ratio of 10000 with a SEM device. The elemental analysis of the sample surface was performed with EDX.

RESULTS AND DISCUSSION

Colour (L*, a*, b*) values

Polydopamine (PDA) is a dark brown-black biopolymer, insoluble in organic solvents, synthesized by the oxidative self-polymerization of dopamine (DOPA). Additionally, the reduction of silver ions to silver nanoparticles is confirmed by the colour changing to dark yellow-beige [15]. For these reasons, spectrophotometric colour measurement results of fabrics were taken. Lightness (L*) values of fabrics are presented in figure 2.

According to figure 2, there was a significant decrease in the L* value of the PET fabric after polydopamine coating by 48.05% from 87.44 to 45.42, as expected. It was also similarly reported in previous studies that the colour of the samples changes towards dark brown-black with polydopamine coating [13–14, 16–18]. After washing, an increase in the L value was observed in the PDA-coated fabric. This shows that the washing process caused some removal of the coating from the fabric. This finding corroborates the results of Telli and Arabaci, who found that the washing process caused increases in the lightness values of PDA-coated fabrics [18]. It was determined that L* values decreased slightly with the production of silver nanoparticles on the fabric. The reduction was measured as 5.09% from 45.42 to 43.11 at 10 mM silver molarity, 6.52% from 45.42 to 42.46 at 20 mM silver molarity, and 11.74% from 45.42 to 40.09 at 50 mM silver molarity. A further decrease was observed with increasing applied molarity. The lowest L* value was measured on the fabric to which 50 mM silver nitrate was applied. Decreased L* value due to silver in this study confirms earlier findings of other researchers. Ilic et al. found that the silver nanoparticle ratio in the fabric caused insignificant colour changes at low levels, but there was a significant change in colour as the amount increased. Fabrics containing AgNPs were described as darker, redder and more yellow than the undyed control fabric [19]. After washing, the L* value of PET+PDA fabric increased by 13.18% from 45.42 to 51.41. Similarly, the L* value of PET+PDA+50mM Ag fabric increased by 13.17% from 40.09 to 45.37 after washing. After washing, a colour difference was seen that may indicate the presence of silver on the fabric when the L* values of PET+PDA fabric (51.41) and PET+PDA+50mM Ag fabric (45.37) were compared.

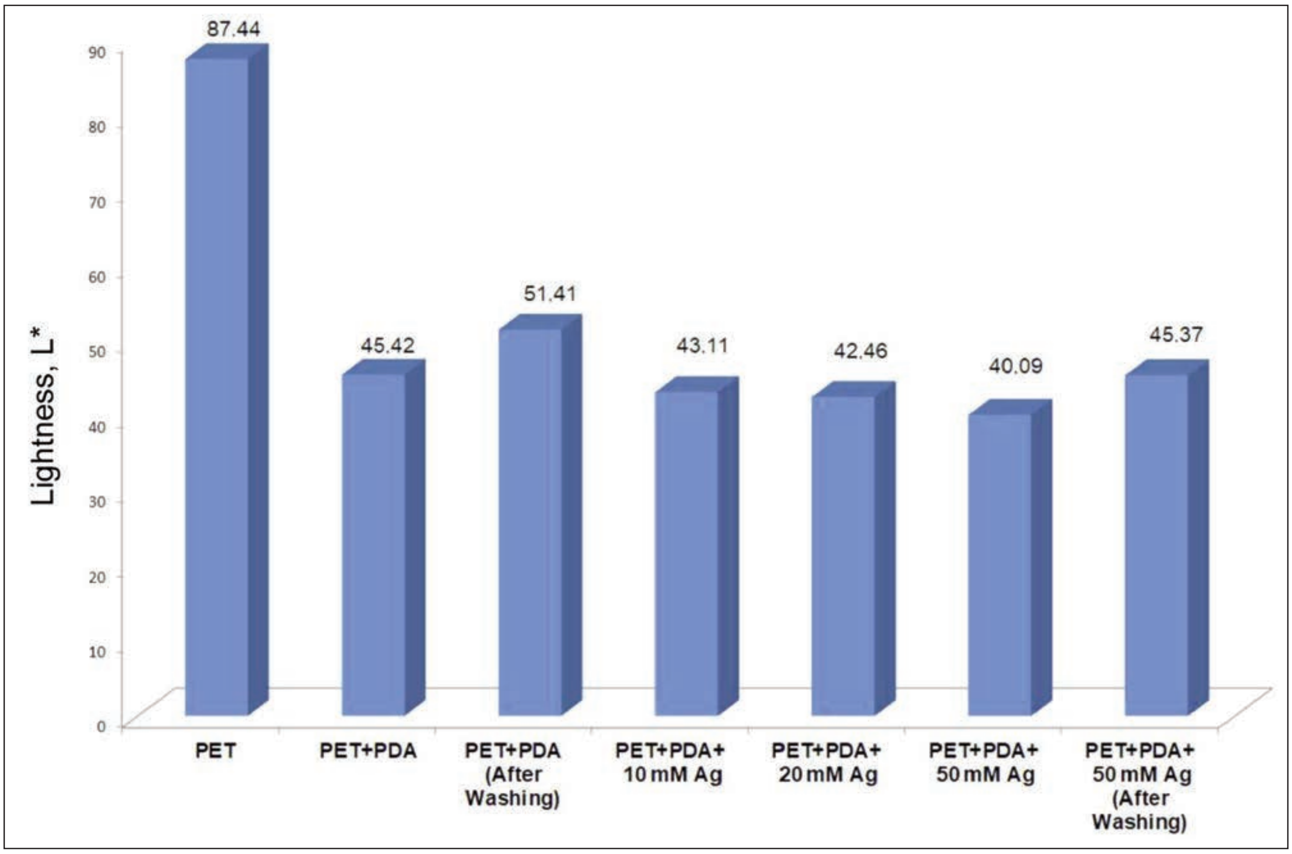

Fig. 2. Lightness (L*) values of fabrics

In previous studies, it was emphasized that the most significant change in colour with silver application was in the b* value [19]. Silver ions were used to provide antibacterial properties in a polyester and polyamide fabric by Yuranova et al. Researchers pointed out that Ag deposition on the fabric surface in the processes by RF-plasma and vacuum-UV techniques gave a more yellow colour depending on the deposition rate [15]. Figure 3 provides the b* values in this study.

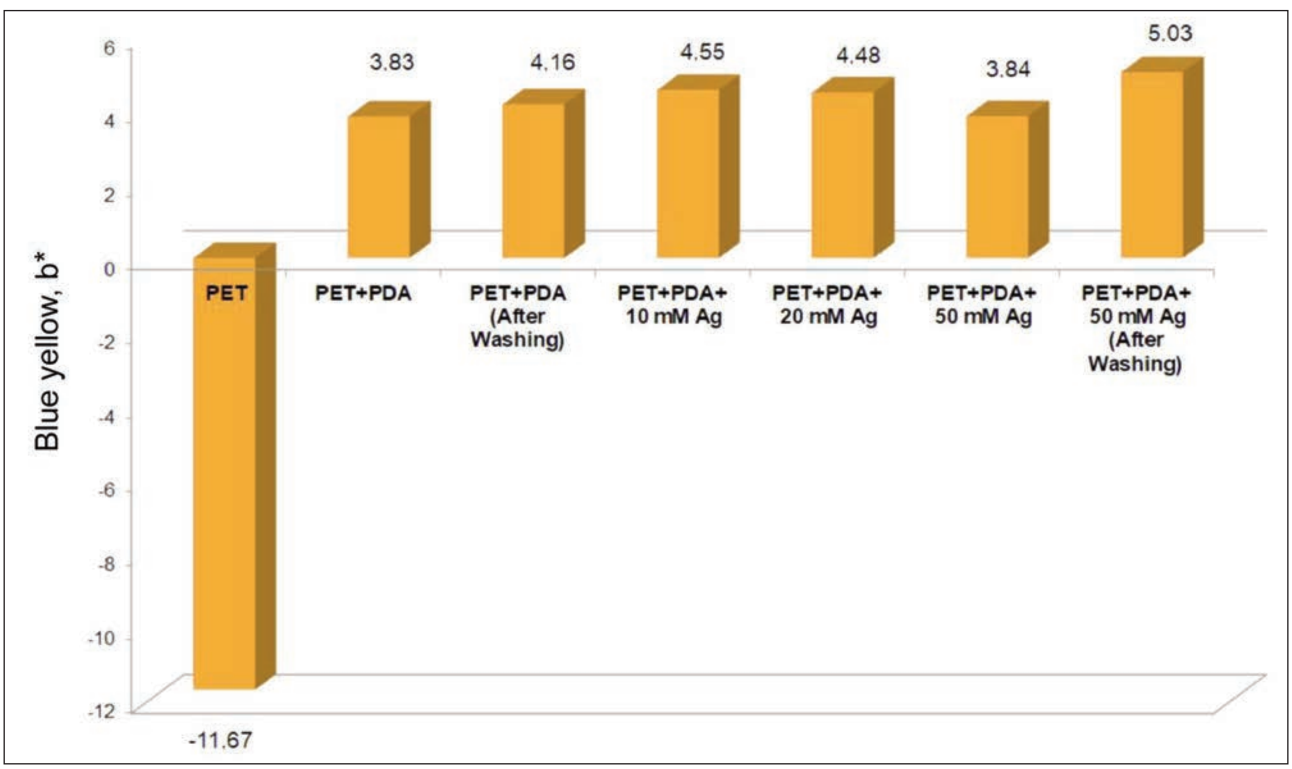

Fig. 3. Blue Yellow (b*) values of fabrics

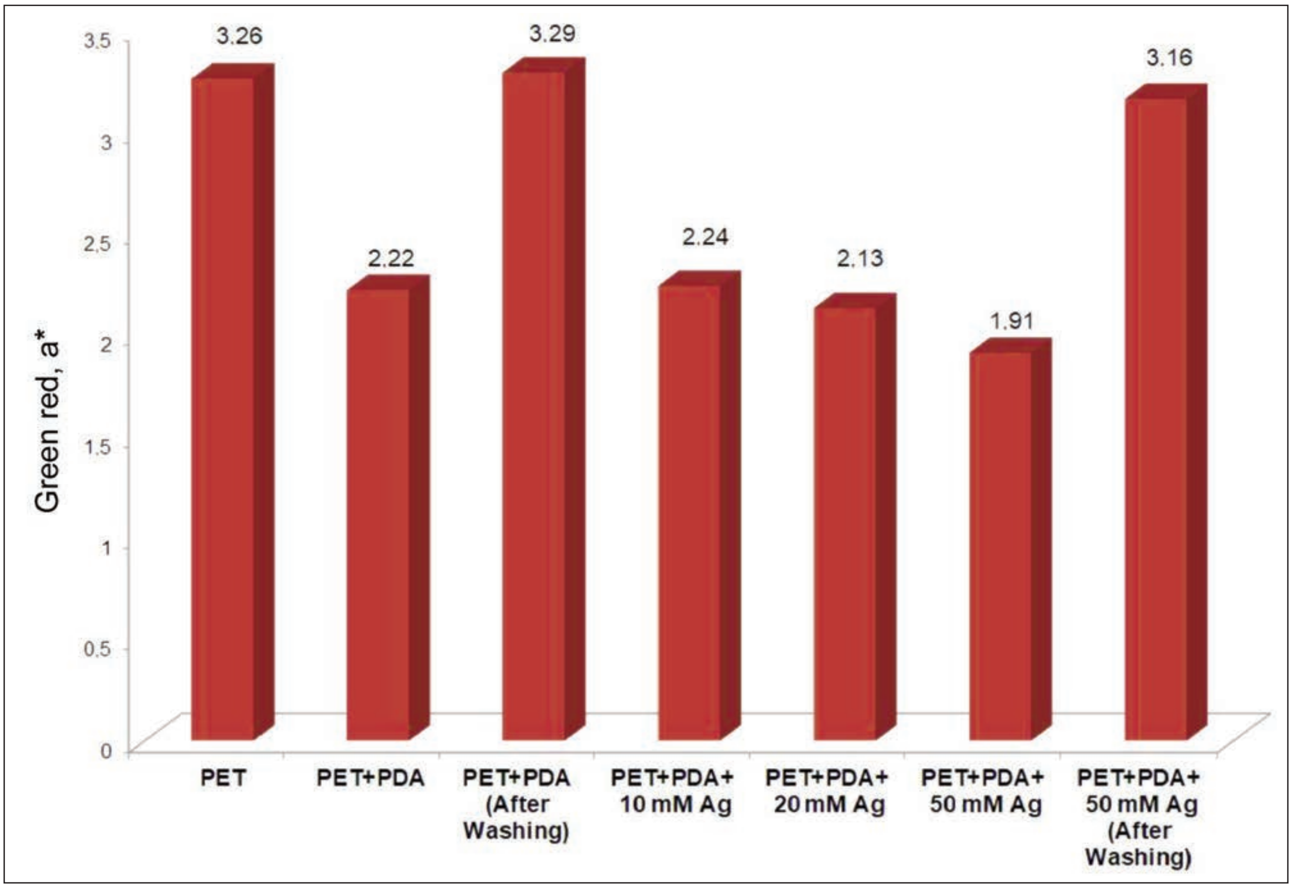

Fig. 4. Green Red (a*) values of fabrics

As the b value increases, yellowness increases and blueness decreases. Yellowness showed an increase clearly in coated fabrics compared to uncoated fabrics. Silver-applied fabrics were found to be more yellow than PDA-coated fabrics. However, the highest yellowness was obtained at the lowest molarity of 10 mM. As the molarity increased, yellowness decreased slightly. This is because the silver application was made on PDA-coated fabrics. Ilic et al. also stated that silver application caused a significant change in the dyed fabric colour like undyed fabric and that L, a* and b* values were directly affected by the dye colour or whether the silver application was made before or after dyeing [19]. After washing, a colour difference was seen that may indicate the presence of silver on the fabric when the b* values of PET+PDA fabric (4.16) and PET+PDA+50mM Ag fabric (5.03) were compared. The highest b* value in the study was measured on PET+PDA+50mM Ag fabric after washing. Figure 4 compares a* values in this study.

As a* value increases, redness increases and greenness decreases. PDA coating caused a slight decrease in a* value. Silver applications also caused a decrease in the a* value with increasing molarity. After washing, the a* values in both PET+PDA fabric and PET+PDA+50mM Ag fabric were close to the uncoated fabric.

FT-IR results

The framework of polyester fibres consists of ester bonds. When the FTIR spectrums of esters are examined, it is well known that they comply with "The Rule of Three peaks". PET fibres are also a type of polyester fibre. PET fibres have been reported in previous studies to have a series of strong peaks, including C=O stretch vibration in the ~1700 cm^{-1}, C-C-O Stretch vibration in the ~1200 cm^{-1}, and O-C-C stretch vibration in the ~1100 cm^{-1} [20]. The combined FT-IR results of the used fabrics in this study are presented in figure 5. In table 1, the obtained peaks and their intensities are shown in detail.

It can be seen from table 1 that the three-peak rule was valid in all tested four fabrics. These three peaks are highlighted in table 1. Additionally, due to weak C–H stretching and C–C bending vibrations of benzene rings, peaks were observed at 2967–2966 cm^{-1}, 870.7–871.67 cm^{-1} and 721.25 cm^{-1} in all fabrics.

When PET fabric and polydopamine-coated fabric were compared, it was seen that they gave similar results. Differently, the intensity values of the peaks seen in PET fabric showed a decrease in the polydopamine-coated fabric. In the presence of silver, it was seen that the intensity values of the broadband seen in the range of 3600–2100 cm^{-1} showed an increase. These values showed a decrease slightly after washing. In the polydopamine coating of PET fabric and the reduction of silver ions, bonds that indicate the presence of polydopamine and silver cannot

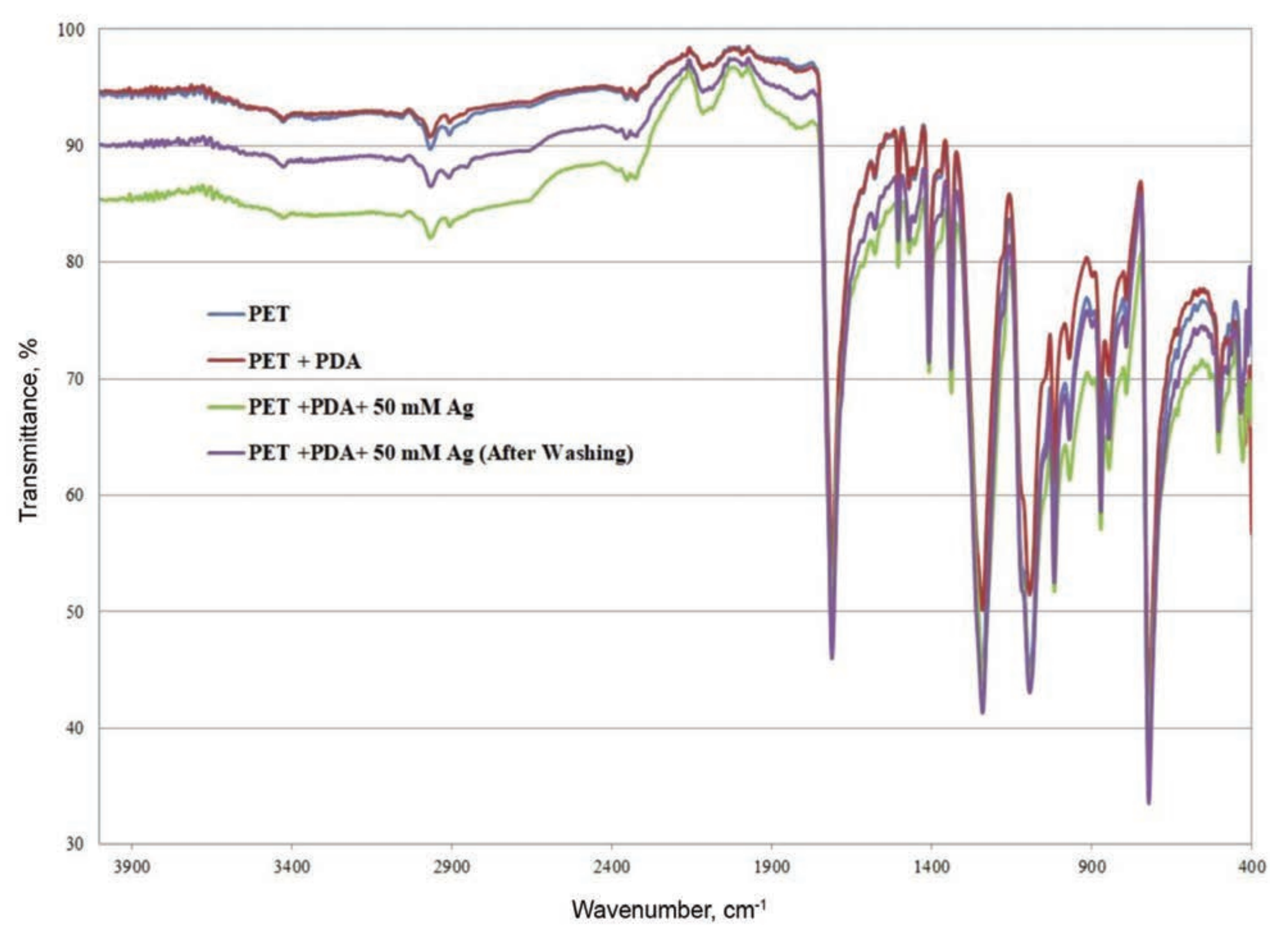

Fig. 4. The combined FT-IR results of the used fabrics

Table 1

THE OBTAINED PEAKS AND INTENSITIES FROM FT-IR RESULTS							
PET		**PET+PDA**		**PET+PDA+50mM Ag**		**PET+PDA+ 50mM Ag (After Washing)**	
Peak	Intensity	Peak	Intensity	Peak	Intensity	Peak	Intensity
400.16	31.87	400.16	61.37	400.16	42.21	401.12	23.60
		409.80	44.41	410.76	41.12	411.73	30.06
427.16	38.48	419.44	49.46	427.16	47.18	433.91	36.48
503.33	39.49	503.33	41.41	503.33	45.91	503.33	38.74
721.25	100.0	721.25	100.0	721.25	100.00	721.25	100.0
791.64	30.94	791.64	29.65	791.64	38.20	791.64	29.10
846.60	41.65	846.60	39.55	845.63	48.22	845.63	39.64
871.67	52.46	870.70	49.83	870.70	57.10	870.70	48.95
969.05	42.03	970.02	37.28	969.05	49.73	969.05	39.73
1016.3	62.70	1016.3	55.11	1016.3	67.11	1016.3	58.93
1093.4	**82.17**	**1093.4**	**74.62**	**1093.4**	**83.13**	**1093.4**	**77.13**
1241.0	**83.79**	**1241.0**	**77.56**	**1239.0**	**84.13**	**1241.0**	**80.94**
1338.4	32.25	1339.3	29.59	1338.4	38.09	1339.3	31.49
1407.8	30.89	1407.8	30.22	1407.8	35.43	1407.8	30.77
1470.4	15.18	1470.5	16.38	1470.5	21.69	1470.5	18.23
1504.2	16.75	1504.2	16.55	1504.2	23.23	1504.2	18.33
1712.5	**74.94**	**1711.5**	**69.64**	**1712.5**	**72.63**	**1711.5**	**71.06**
2967.0	11.19	2966.9	10.85	2966.9	20.05	2966.0	13.25

be distinguished in the FT-IR graph. For this reason, studies were continued with SEM and EDX analysis to detect the presence of silver nanoparticles on the fabric surface.

SEM and EDX analysis

SEM images of the fabrics taken at 10000x magnification were presented in figure 6. The presence of polydopamine nanocoating on the PET fabric is visible in SEM images. Nanospheres formed after the coating processes were observed in all fabric samples. In silver nanoparticle applications, extra particles accumulated in the regions where polydopamine nanospheres were located. Thanks to the reduction reaction, there were differentiated and denser regions in silver nanoparticle applications compared to PET + PDA fabric. However, no obvious differences can be detected between SEM images of different molarity applications and images after washing. EDX analyses were also performed to examine these differences.

EDX analysis is used together with SEM. Analysis of elements near the surface and their percentage amounts can be performed. The chemical composition results obtained as a result of EDX analysis were given collectively in table 2.

PET Polyester fibres are long-chain polymers that are the condensation product of ethylene glycol with

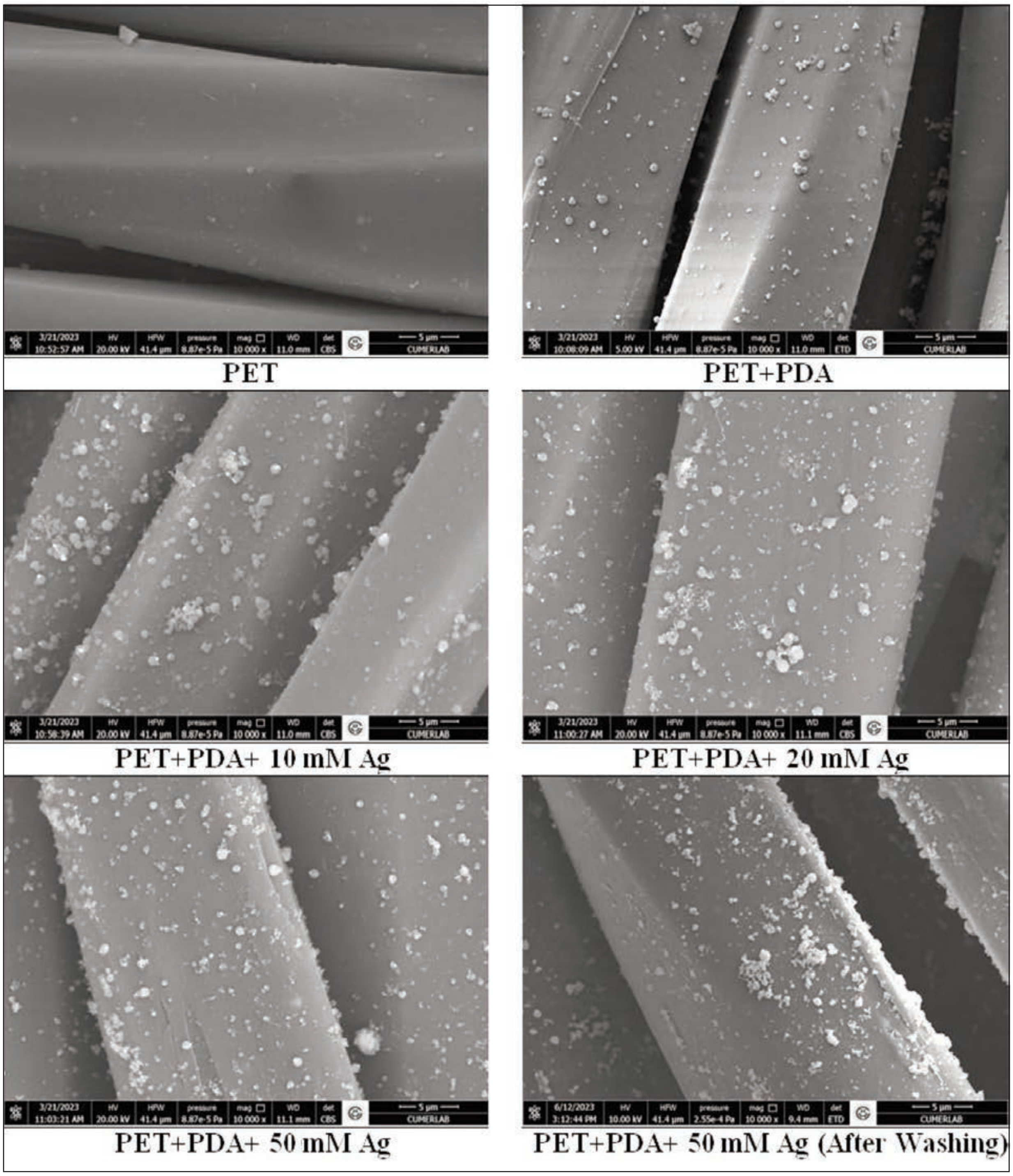

Fig. 6. SEM images of fabrics (10000x)

Table 2

ELEMENT WEIGHT (%) OF FABRICS				
Element weight %	**C (carbon)**	**O (oxygen)**	**N (nitrogen)**	**Ag (silver)**
PET	59.59	40.41	-	-
PET+PDA	54.79	39.72	5.49	-
PET+PDA (After Washing)	54.40	40.36	5.24	
PET+PDA+ 10mM Ag	55.16	39.02	5.50	0.33
PET+PDA+ 20mM Ag	53.98	39.38	5.58	1.06
PET+PDA+ 50mM Ag	53.84	39.02	5.43	1.71
PET+PDA+ 50mM Ag (After Washing)	54.71	38.76	5.18	1.35

terephthalic acid. In this chain, the ester group (-CO-O-) is repeated many times. The structure of PET consists of carbon and oxygen in addition to hydrogen. Differently, polydopamine has N molecules in its structure. Table 2 shows that polydopamine-coated fabric contains 5.49% nitrogen, unlike PET fabric. After the washing process, it was seen that this value decreased to 5.24%. The observed decrease in the N ratio seems to be similar to earlier findings. Ou et al. found that the nitrogen content of the polydopamine coating showed a decrease in all different durability tests, including washing, compared to the results before the test [21]. As expressed in the L* values from the colour results, it was seen that the PDA coating was removed to some quantity with the washing process.

When silver applications were evaluated, it was seen that there were the lowest silver nanoparticles in the PET+PDA+ 10 mM Ag fabric with 0.33%. Ahmed et al. achieved 99.33% antibacterial efficiency in PP melt-blown fabrics to which silver nanoparticles were applied with different techniques, with a silver content of 0.28% measured in EDX analysis [22]. From the data in table 2, it was seen that this silver ratio (0.33%) in the fabric, which can be considered successful, increases as the molarity of silver nitrate increases. The silver ratio increased to 1.06% at 20 mM Ag and 1.71% at 50 mM Ag. The highest silver content (1.71%) was reached at the highest molarity. 89% for S. aureus and 80% for E. coli antibacterial efficiency in cotton fabrics with a silver content of 1.58% measured in EDX analysis by Tania et al. were reported [23]. In previous studies on the application of silver nanoparticles to different fabrics with in situ formation, it was stated that the lowest efficiency was in polyester fabrics. It was stated that cotton fabrics were 8 times more efficient than polyester fabrics [24]. As table 2 shows, higher silver nanoparticle levels with the technique applied in the study were obtained in polyester fabric (1.71%) than earlier findings related to cotton fabrics (1.58%).

After the washing process, it was seen that this value decreased to 1.35%. According to previous studies, depending on the fabric structure, it can provide various functional properties to the fabric with the formation of at least 0.28% silver nanoparticles on the fabric [22]. In this study, a silver content of 1.35% was obtained even after washing in polyester fabric which was stated to be difficult to apply silver in previous studies. Furthermore, no pretreatment was applied to the polyester fabric in this study [24].

In addition, it was seen that the nitrogen content in PET+PDA+ 50 mM Ag fabric decreased after washing, as in the washing of PET+PDA fabric. It decreased from 5.43% to 5.18%. The decrease in nitrogen content of both fabrics after washing was proportionally similar. It was seen that both the polydopamine that enables the AgNPs to adhere to the PET fabric and some silver were removed from the fabric after washing.

CONCLUSIONS

In this study, PET fabrics were coated with silver nanoparticles with the help of a polydopamine interlayer. In the first stage, polydopamine-coated surfaces were formed by oxidative self-polymerization of dopamine on the fabric surface under constant alkaline conditions. In the second stage, silver nitrate salt was applied to the polydopamine-coated fabric in three different molarities (10 mM, 20 mM, and 50 mM) for the synthesis of silver nanoparticles. With the production of polydopamine coating and silver nanoparticles, fabric lightness values decreased. The lowest L* value was measured on the fabric to which 50 mM silver nitrate was applied. In the SEM images of the fabrics taken at 10000x magnification, the presence of polydopamine nano-coating on the PET fabric was seen. By EDX analysis, it was determined that the amount of silver nanoparticles in the fabric increased as the molarity of silver nitrate increased. The highest silver nanoparticle ratio was reached at 1.71% at 50 mM molarity. After the washing process, it was seen that this value decreased to 1.35%. Compared to the minimum ratios required to impart various functional properties to fabrics with silver nanoparticles in previous studies, the silver nanoparticle ratio obtained after washing in this study will enable polyester fabrics to gain functional properties such as antibacterial, electrical conductivity electromagnetic shielding etc.

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