Polydopamine nanocoating to use surface functionalization of polypropylene fabrics with a closed structure

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ABSTRACT – REZUMAT

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In this study, an experimental investigation was conducted to explore polydopamine (PDA) nano-coating to use surface functionalization of polypropylene (PP) fabrics with a closed structure. Nanocoatings were made on polypropylene nonwoven fabric with the oxygen-induced polymerization method of dopamine (DA) at six different times. Uncoated control samples, coated samples and washed samples after coating were compared. A change in the solution towards a dark brown-black colour was observed during the experiment as the reaction time increased. Based on this finding, spectrophotometric measurements of samples were made. The lowest lightness and highest colour strength values were observed in fabric coated for 32 hours. In before and after washing SEM images, the formed nano-coating could be easily seen on this fabric (32 h). The nitrogen ratio indicating the presence of polydopamine was detected as 17.05%. After washing, it was observed that this high percentage decreased up to 1.35% but the nitrogen element was distributed homogeneously on the fabric surfaces from EDX mapping. FTIR analysis results confirmed that the PDA coating formed a bond with the polypropylene fabric and that these bonds continued after washing. Differently from uncoated fabric, extra bands and signals were observed in two different regions on the FTIR graph due to polydopamine. The results presented here may facilitate improvements in the surface activation of PP fabrics, which have a more closed structure for functionalization.

Keywords: *polypropylene, polydopamine, nonwoven, nano-coating, oxygen-induced polymerization*

Acoperirea cu nanoparticule de polidopamină pentru funcționalizarea suprafeței materialelor textile din polipropilenă cu structură închisă

În acest studiu, a fost efectuată o investigație experimentală pentru a explora acoperirea cu nanoparticule de polidopamină (PDA) pentru funcționalizarea suprafeței materialelor textile din polipropilenă (PP) cu structură închisă. Acoperirile cu nanoparticule au fost realizate pe nețesute din polipropilenă folosind metoda de polimerizare indusă de oxigen a dopaminei (DA) în șase timpi diferiți. Au fost comparate probele de control neacoperite, probele acoperite și probele spălate după acoperire. O schimbare a soluției către culoarea maro închis-negru a fost observată în timpul experimentului pe măsură ce timpul de reacție a crescut. Pe baza acestei constatări, au fost efectuate măsurători spectrofotometrice ale probelor. Cele mai scăzute valori de luminozitate și cele mai mari valori de rezistență a culorii au fost observate la materialul acoperit timp de 32 de ore. În imaginile SEM înainte și după spălare, învelișul nano format poate fi observat cu ușurință pe acest suport textil (32 de ore). Raportul de azot care indică prezența polidopaminei a fost detectat la 17,05%. După spălare, s-a observat că acest procent ridicat a scăzut până la 1,35% dar elementul azot a fost distribuit omogen pe suprafața materialului textil din cartografierea EDX. Rezultatele analizei FTIR au confirmat că învelișul PDA a format o legătură cu materialul textil din polipropilenă și că aceste legături au continuat după spălare. Spre deosebire de materialul textil neacoperit, s-au observat benzi și semnale suplimentare în două regiuni diferite pe graficul FTIR datorită polidopaminei. Rezultatele prezentate aici pot facilita îmbunătățiri în activarea suprafeței materialelor textile PP, care au o structură mai închisă pentru funcționalizare.

Cuvinte-cheie*: polipropilenă, polidopamină, nețesut, acoperire cu nanoparticule, polimerizare indusă de oxigen*

INTRODUCTION

Various functional properties can be imparted to textile materials by coating. In recent years, microcapsule applications have come to the fore to add lasting pleasant odours to fabrics. In these applications, durability against washing is still an unresolved research issue [1]. Nano-sized textile coatings have greater potential for the textile industry. For instance, it has been reported that better results were obtained in samples processed with nano-emulsion silicone softeners compared with micro and macro-emulsion silicone [2]. It was stated that the surface free energies and various structural properties of fabrics at nano-sized coatings changed compared to conventional size [3]. In a study where nano-sized zinc oxide was used to increase resistance to UV rays, a more effective UV radiation blockade was achieved compared to conventional size [4]. Especially with nanocomposite applications, various functional properties such as electromagnetic protection can be gained from fabrics [5]. In these applications, cross-linkers are generally used and the majority of these

binders are harmful to the environment and workers [6]. There is a need for nanostructures and surface modification in closed-structure fabrics such as PES and PP, which are not moisture-absorbent. One of the outstanding applications for this purpose is the plasma technique. There are different types of this technique. It has been reported that it contributes to the fabric in terms of wrinkle resistance, drape ability and water repellency. These applications are currently expensive and negatively affect the mechanical properties of the fabric [7]. Surface activation has been studied over the last few decades because the majority of textile polymers have a closed chemical structure. Additional activation steps are required for functionalization.

Polypropylene (PP) fibres are preferred in many sectors due to their low density and cheap costs. PP has low surface energy due to its hydrocarbon chains, which results in poor wettability properties. Low adhesion properties create a disadvantage in dyeing, coating and use together with different materials. It restricts their use. For this reason, various surface treatments must be applied to these fibres to increase their surface energy [8]. Recently, research has been conducted on the preparation of polydopamine-coated complicated surfaces by polymerization of dopamine. Polydopamine (PDA) is a biopolymer synthesized by the oxidative self-polymerization of dopamine. It is estimated that surfaces functionalized with a PDA layer will be widely used in the coming years. The PDA layer can be used as an intermediate structure to anchor functional molecules on the surface through chemical bonds or other physical bonds [9–10]. There are a limited number of studies in the literature on the use of polydopamine in the textile industry [10–22]. It was reported in the literature that the oxidative polymerization of dopamine occurred mainly on the surface and in the amorphous regions of the textile fibres [23]. Previous studies have primarily concentrated on providing functional properties (hydrophobic character, self-cleaning, flame retardant, electrical conductivity) to cotton fabrics. In these studies, various nanoparticles such as silver and titanium dioxide were tried to be anchored to fabrics by applying polydopamine as an intermediate structure [12–13, 17, 19–21]. Liu et al. (2019) applied dopamine to the fabrics coated with Ag film. It was said that the antibacterial activity of the fabric and the laundering durability was enhanced. The developed fabrics showed more resistance against sodium sulphide corrosion with dopamine application [14]. Miao et al. (2022) developed a textile product by chemically depositing Ag particles on the textile surface using polydopamine as the binding layer. Researchers pointed out that this product can be used in the separation of oil/water mixtures and decomposition of the organic dyes under UV light [15]. Li et al. (2022) produced a superhydrophobic and conductive cotton fabric even after 18 cycles of accelerated washing through the PDA-assisted deposition of photocatalyst Ag/CdS [24]. Wang et al. (2023) produced a superhydrophobic coating providing functional properties such as self-cleaning, oil-water separation, oil sorption and flame retardancy. This nano-coating was achieved with polydopamine-boehmite modification. The boehmite particles were adhered to stainless steel mesh through polydopamine (PDA). Furthermore, cetylamine (CTA) with low surface energy and amino group was grafted onto PDA [18].

Literature reviews have indicated that there were no studies on the detailed performance of PDA nano-coating on polypropylene nonwoven fabrics. The objectives of this paper are to determine whether PDA nano-coating could be used for surface functionalization of polypropylene fabrics with a closed structure. For this aim, dopamine hydrochloride was preferred as the monomer. The oxygen-induced polymerization method of dopamine (DA) was used. The behaviour of nano-coating PP fabrics before and after washing was examined.

MATERIAL AND METHODS

In this study, polypropylene spunbond nonwoven fabrics with a weight of 35 grams per square meter, obtained from Teknomelt nonwoven company, were used. The fabric was washed with acetone to remove impurities before use in the experiment. Chemicals were obtained from Sigma-Aldrich Chemie GmbH in Germany. Dopamine hydrochloride (99 wt.%) and tris(hydroxymethyl)aminomethane (99 wt.%) were used without an additional purification step.

At first in the synthesis of polydopamine, 2 mg/ml dopamine hydrochloride as a monomer was dissolved in water. After this step, 1.2 mg/ml Tris(hydroxymethyl)aminomethane was added to the solution to stabilize of pH and mixed quickly. During this process, the solution, which was initially colourless and transparent, turned pale yellow with the oxidation of catechol to benzoquinone. After a homogeneous mixture was achieved, the fabric was left in the solution. Fabric in solution was starting to put to a continuous movement in the horizontal direction using a shaker. In this way, dopamine-melanin aggregates on fabric surfaces started to form under alkaline and stable environmental conditions as reported in the literature [10, 14, 16]. The self-polymerization reaction began to occur in the presence of atmospheric oxygen. The oxidative self-polymerization of dopamine was made six different times as powers of two (2^0 = 1, 2^1 = 2, 2^2 = 4, 2^3 = 8, 2^4 = 16, 2^5 = 32 hours). As the reaction time increased, a gradual change in the solution colour towards dark brown-black was observed. PDA's natural colour becomes more evident as the layer thickness of the PDA increases successfully and the aggregates are observed more. Depending on the reaction time, this shows itself as a colour change [25–27]. The obtained coated samples were rinsed with cold water. Following this, samples were dried flat under standard atmospheric conditions. To determine the durability of the nano-coating, washing was carried out using sodium perborate for 30 minutes at 40 °C in the GyroWash Colour Fastness

Tester of James Heal according to EN ISO 105-C06. The A2S method was used for the experimental conditions.

Since the colour of the solution darkens with polymerization, spectrophotometric colour measurements (Minolta CM 3600 D Spectrophotometer and RealColor® software) were made on all samples. CIELab values of the fabrics were determined under standard D65 light. Among these values, lightness (L*) values were examined because it was thought that polymerization would be most successful as the darkness was increasing. In addition, colour strength values (K/S) were calculated using the percentage reflectance (%R) values at the 400 nm wavelength, where the maximum absorption of the colour in the dyes is found. The results obtained from uncoated control samples, coated samples and washed samples after coating (AW) were compared. Scanning electron microscopy (FEI Quanta 650 Field Emission SEM), energy dispersive spectrometer (EDX and mapping), FT-IR (Jasco FT/IR-6700) spectrum with ATR technique and static contact angle measurements for the surface wettability (Theta lite Contact Angle Measurement System) were used to examine and characterize the structure of the samples that were successful after colour measurements. Figure 1 shows the used methods as a schematic.

RESULTS AND DISCUSSION

Lightness (L*) results

The decrease in L* values indicates increasing darkness. Lightness (L*) values obtained at six different times are given in figure 2.

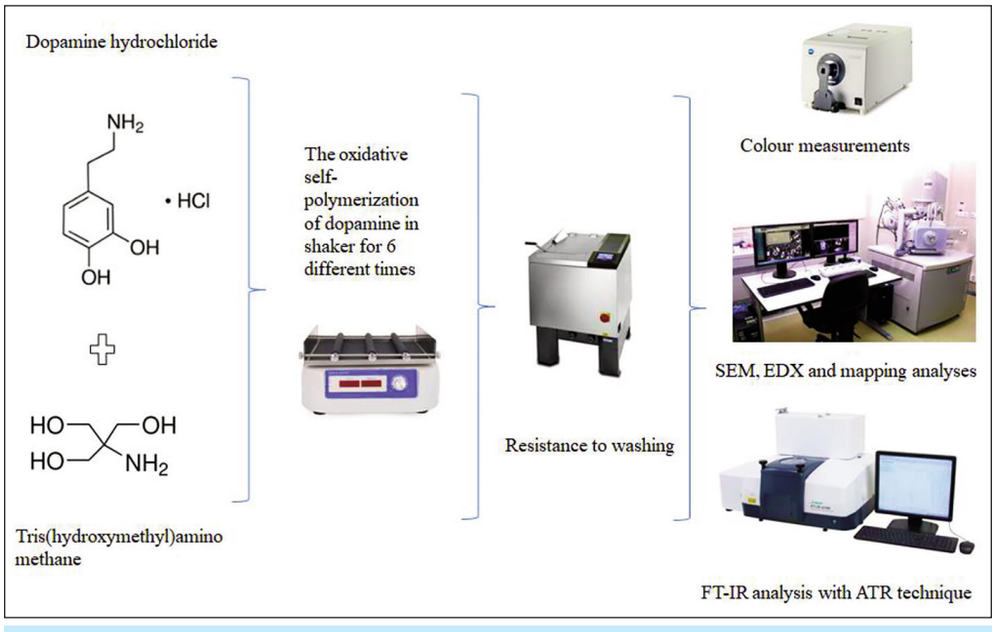

Fig. 1. The schematic representation of used methods

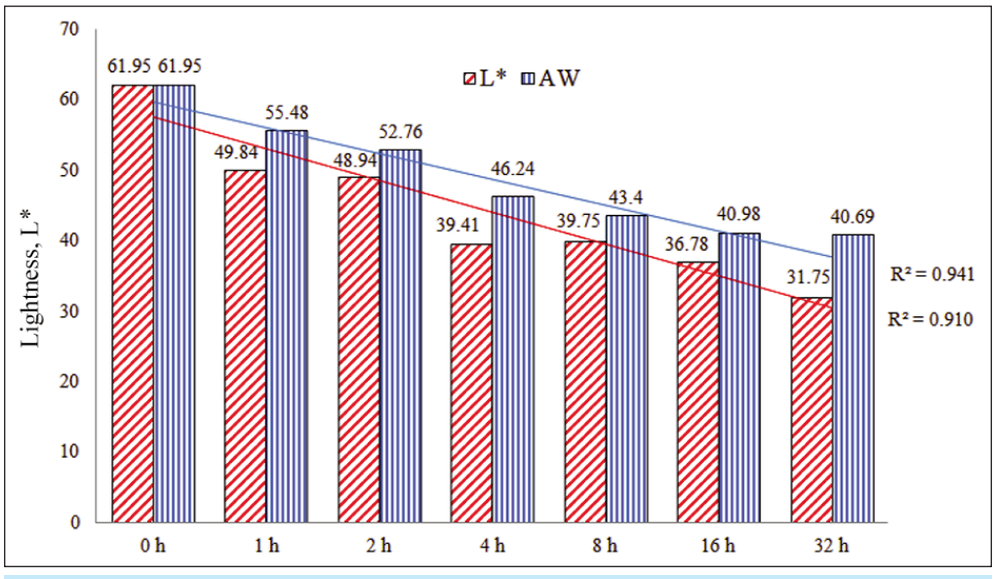

Fig. 2. Lightness (L*) values and changes after washing (AW)

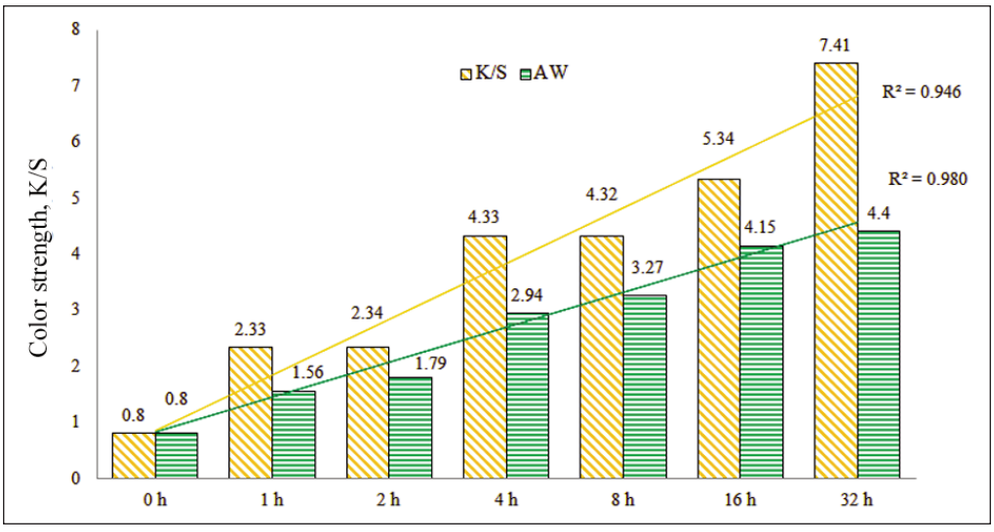

Fig. 3. Colour strength (K/S) values and changes after washing (AW)

A decrease in L* values was observed as the coating time increased, except for 8 hours. The lowest L* (L* = 31.75) were obtained in the coating applied for 32 hours. Similarly, after washing, the lowest L* (L* = 40.69) were measured in the coating applied for 32 hours. When the trend line of the coated fabrics was examined, high regression coefficients were observed for both lightness (R^2 = 0.910). After washing, significant and higher regression coefficients were seen in L* (R^2 = 0.941).

The colour strength (K/S) results

Fabrics with high cover factors have high K/S values. In this situation, it can be said that the increase in K/S values shows the success of coating. Colour strength (K/S) values obtained at six different times were presented in figure 3.

An increase in K/S values was observed as the coating time increased, except coated for 8 hours (figure 3). The highest K/S value (K/S = 7.41) was obtained in the coating applied for 32 hours. Similarly, after washing, the highest K/S value (K/S = 4.4) was measured in the coating applied for 32 hours. When the trend line of the coated fabrics was examined, high regression coefficients were observed for colour strength (R^2 = 0.946). After washing, significant and higher regression coefficients were seen in K/S (R^2 = 0.980).

Based on the results presented in figure 2 and figure 3, L* values and K/S values showed compatible results and it can be estimated that the most successful coating occurs in 32 hours which has the lowest L* and highest K/S. It was determined that washing removes the coatings to some extent at all application times. In washed samples, the darkest appearance, lowest lightness and highest colour strength values were measured in 32 hours. Before and after washing images of the coated (32 h) polypropylene fabric are shown in figure 4. It was seen that the coating applied for 32 hours was homogeneous and very close to black. But there was a significant colour fading with washing. For this reason, the study focused on 32 hours coated fabrics in subsequent processes.

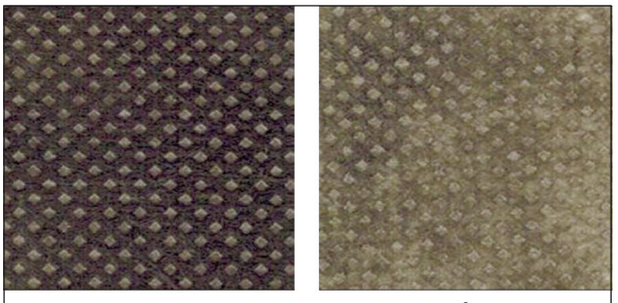

Fig. 4. Before and after washing images of the coated (32h) polypropylene fabric:
a – coated (32 h); *b* – washed

Presence of coatings in SEM images

To examine the presence of polydopamine nanocoating on the fabrics after the coating process and washing, images were taken at 10000x magnification on the SEM device. Compared to the uncoated control fabric in figure 5, the nanocoatings formed after the coating process can be easily seen on fabrics. It was seen that the polydopamine nanospheres remained on fabric surfaces after washing although its density decreased slightly.

Atomic percentages of elements in the EDX and Mapping

EDX analysis is used with SEM. Elements and percentage amounts on the surface can be analysed and mapped. The chemical composition results obtained from EDX analysis are given in table 1.

Polypropylene consists of methyl groups. For this reason, it contains only carbon and hydrogen elements. Since hydrogen could not be detected in the EDX analysis, it could be seen from table 1 that the structure of PP only consists of carbon. PP does not

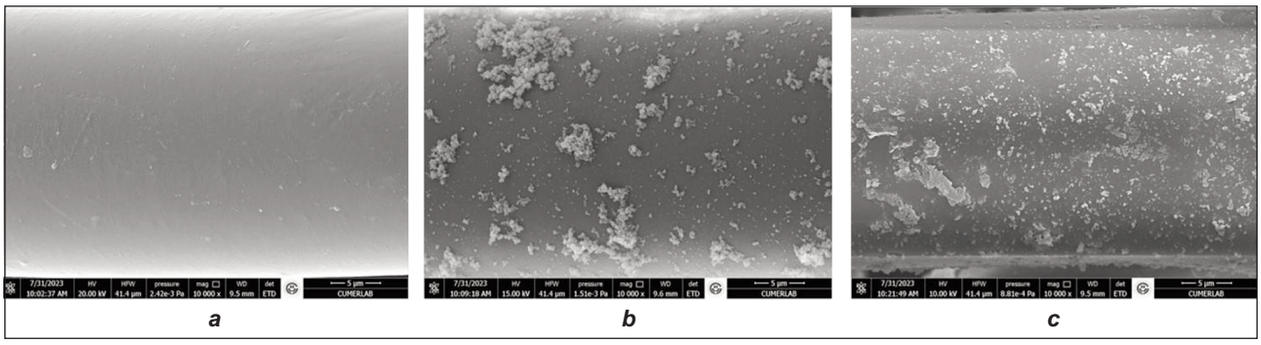

Fig. 5. SEM images of fabrics (10000x): *a* – uncoated control samples; *b* – coated samples (32 h); *c* – washed samples

contain a nitrogen element in its structure. In PP+PDA, 17.05% nitrogen content was detected thanks to PDA. After washing, it was observed that this high percentage decreased up to 1.35%. This situation is compatible with the colour change seen in the PP fabric after washing in figure 4. PDA continued its presence in the fabric although the rate decreased.

Table 1

CHEMICAL COMPOSITIONS IN FABRICS		
Atomic %	**C (carbon)**	**N (nitrogen)**
PP	100*	-
PP+PDA	82.95	17.05
PP+PDA (AW)**	98.65	1.35

Note: *Hydrogen cannot be detected in EDX analysis; **AW: After Washing.

Additionally, before and after washing mapping of the coated PP fabrics was given in figure 6. In this figure, yellow colour for N and green colour for C were represented. According to figure 6, it was seen that the nitrogen element is distributed homogeneously on the surface of the PDA-coated polypropylene fabric. A similar appearance was obtained after washing. A homogeneous distribution continued after washing. The results confirmed that PDA coatings were successfully formed using the in-situ polymerization technique on PP fabric and remained valid after washing.

FT-IR results

Each type of bond absorbs energy of a specific frequency. In this way, bond types in an element can be determined using the FTIR spectrum. In figure 7, FTIR results of PP, PP+PDA and PP+PDA (AW) fabrics were presented as unified in a single graphic.

In PP, CH3 stretching peaks at wavelengths of 2950.55 (asymmetric) and 2867.63 (symmetric) cm^{-1} and CH2 stretching peaks at wavelengths of 2916.81 (asymmetric) and 2837.74 (symmetric) cm^{-1} were observed. These peaks are the distinctive peaks in the FTIR graph of each polypropylene fabric. Four C-H stretching peaks were measured between wavelengths of 3000-2850 cm^{-1}. Since both methyl and methylene groups have two peaks in this range, the presence of four peaks means that both were present [28–29]. Additionally, two CH3 bending peaks were observed at wavelengths 1454.06 (asymmetric) and

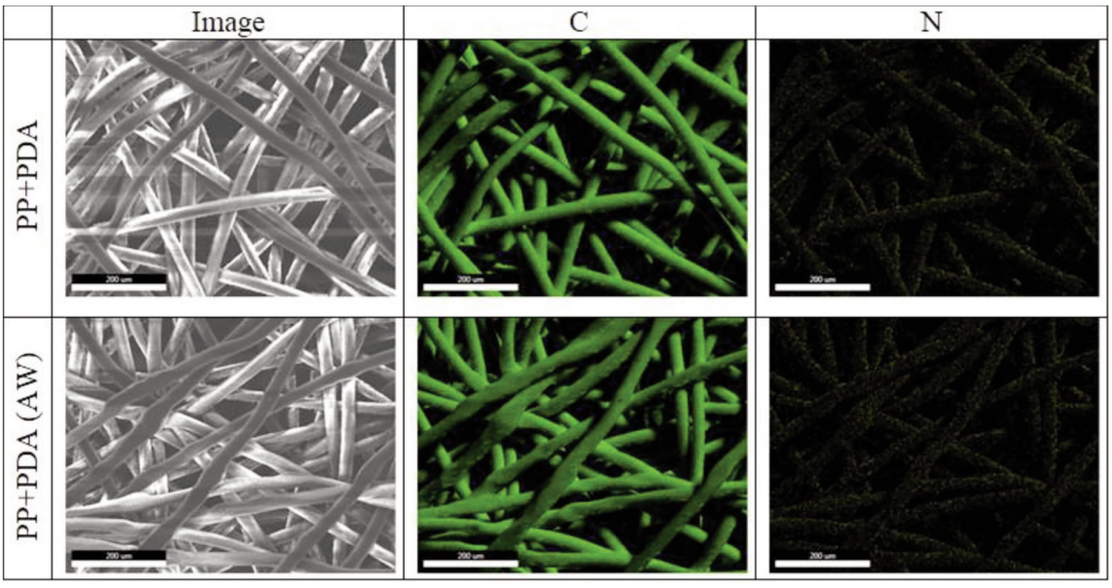

Fig. 6. Before and after washing mapping of the coated (32 h) PP fabric

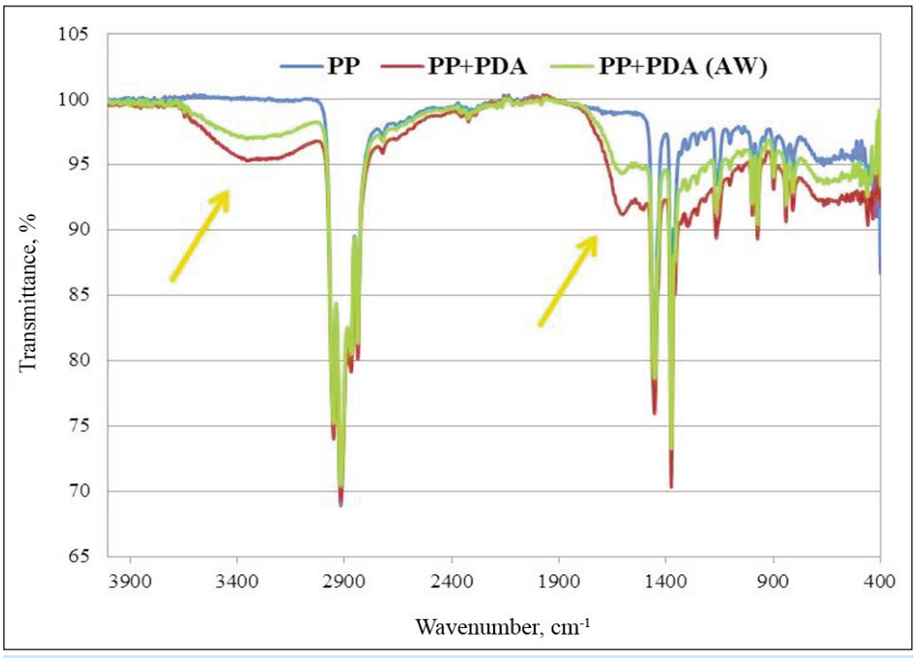

Fig. 7. FTIR results of PP, PP+PDA and PP+PDA (AW) fabrics

1375 (symmetric) cm^{-1} [29–31]. There were peaks with similar transmittance values at the same points in PP+PDA and PP+PDA (AW). However, differently from PP, the most striking result to emerge from the FTIR data is, that extra broad signals were observed in the two regions indicated by the arrows in figure 7 due to the presence of polydopamine.

The broad signals seen between the high wavelengths of 3600–3200 cm^{-1} originate from the catechol groups in the polydopamine structure. The presence of a broad signal at higher wavelengths indicates the presence of hydrogen bonds. If there is a very broad signal to the left of 3000 cm^{-1}, this is due to OH bonds. OH gives a broad signal due to its bond with hydrogen. Furthermore, symmetric N-H bond stretching (secondary amine) may occur at wavelengths of 3300±10. In figure 7, significant signals were detected at 3354.57 cm^{-1} in PP+PDA and 3355.53 cm^{-1} in PP+PDA (AW). Previous studies also emphasized that the signals in this region belong to (N-H) and (-OH) stretch vibrations in the dopamine structure [10, 32].

The other characteristic strong band was seen in the double bond region between wavelengths of 1650-1500 cm^{-1}. Signals were detected at 1602.56 cm^{-1} in PP+PDA and 1601.59 cm^{-1} in PP+PDA (AW). It was stated that these signals arise from the bending vibrations of the C=O double bond (COOH) of the carboxylic function, the aromatic C=C ring in the dopamine-quinone structure, and the C=N bond [10, 32]. When PP+PDA and PP+PDA (AW) were compared, the effect of washing was seen in the SEM images and EDX analysis results.

Transmittance values of specific peaks were found to be higher in PP+PDA (AW) fabric due to the slight decrease in nano-coating after washing. FTIR analysis results in figure 7 confirmed that the PDA coating formed bonds with the polypropylene fabric and these bonds continued after washing.

Surface wettability

The uncoated polypropylene (PP) fabric exhibited a high contact angle of 116 degrees, indicating a significant degree of inherent hydrophobicity. After coating the fabric with polydopamine (PDA), the contact angle was slightly reduced to 114 degrees. This minor decrease, however, fell within the margin of experimental error, suggesting that the PDA coating did not significantly alter the hydrophobic nature of the PP fabric. Thus, while the PDA coating modified the surface properties of the PP fabric, it did not drastically alter its overall hydrophobic nature. The proximity of the contact angles indicated that the PDA layer while affecting the surface chemistry did not significantly enhance the hydrophobicity of the already hydrophobic PP fabric (figure 8).

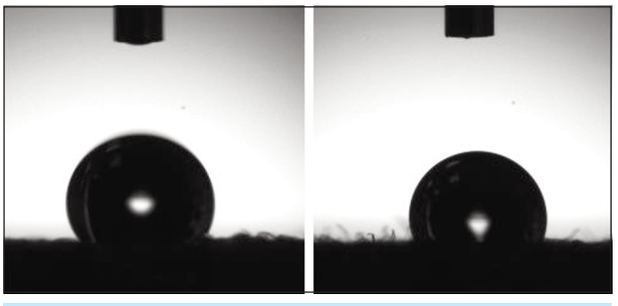

Fig. 8. Water contact angle results of PP and PP+PDA fabrics

CONCLUSIONS

In this study, polydopamine coatings six different times were made on polypropylene spunbond nonwoven fabric with the oxygen-induced polymerization method of dopamine (DA). This paper set out to compare the properties of uncoated control samples, coated samples and washed samples after polydopamine nano-coating. According to colour measurement results, the lowest lightness and highest colour strength values were observed in polypropylene fabrics coated for 32 hours. In SEM images, the formed nano coating after the coating process could be easily seen on the fabric. Although its density decreased slightly after washing, it was determined

that polydopamine nanospheres remained on fabric surfaces. According to SEM EDX analysis results, the nitrogen content in PDA-coated PP fabrics was determined as 17.05%. After washing, it was observed that this high percentage decreased to 1.35%. This finding has shown that PDA continued its presence in the fabric although the rate decreased after washing. Furthermore, this study has shown that the nitrogen element was distributed homogeneously on the fabric surfaces before and after washing with EDX mapping of the coated fabrics. The other major finding was that FTIR analysis results confirmed that the PDA coating formed a bond with the polypropylene fabric and that these bonds continued after washing. Since there is no nitrogen in the polypropylene fabric structure, the presence of polydopamine could be detected. Extra bands and signals were observed in two different regions on the FTIR graph due to polydopamine. These were the broad signals in the high wavelengths of 3600–3200 cm^{-1} and peaks in the wavelengths of 1650–1500 cm^{-1} in the double bond region. Water contact angle results showed that the PDA coating did not significantly alter the hydrophobic nature of the PP fabric.

The SEM, EDX, Mapping and FTIR results of this research support the idea that the polydopamine (PDA) nanocoating intermediate layer, which enables the functionalization of fabrics to be carried out easier, better and more homogeneously with fewer chemicals, will be successful in PP fabrics. The evidence from this study suggests that PDA nano coating can be considered a sustainable alternative for surface activation of PP fabrics.

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