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Improving the Adhesion Strength of Polypropylene Nonwoven Laminated Fabrics Using Low-Pressure Plasma

Abstract
In this study, the adhesion strength properties of laminated polypropylene nonwoven fabrics treated with low-temperature, low-pressure, radio frequency argon plasma were investigated. The change in wettability was determined by water contact angle measurements. In order to observe the effect of plasma treatment on the washing resistance of the laminated samples, washing treatment by means of 10 wash cycles was also carried out. After the peel-off test, the remaining adhesives on the peeled surfaces were examined by SEM images in order to see the effect of plasma treatment. Contact angles of highly hydrophobic polypropylene nonwoven samples decreased by increasing the plasma exposure time and discharge power. The peel bond strength of the plasma treated laminated fabrics improved by up to 150% compared to the untreated fabrics. SEM and AFM analyses also showed that the surface roughness increased due to the etching effect of the plasma treatment, leading to the improvement of the mechanical adhesion of the polypropylene nonwoven fabrics.

Key words: *plasma treatment, adhesion strength, polypropylene, nonwoven, laminated fabric.*

Introduction

Polypropylene is used in a wide range of technical and industrial applications. However, it has very low surface tension resulting in weak hydrophilic and adhesion properties [1]. Plasma technology can be used to improve the surface characteristics of polypropylene through surface modification [2 - 17].

Polypropylene laminated fabrics are used in the textile industry for their functional and technical properties instead of their aesthetic and appearance features. They are utilised mainly as automobile interior coverings, footwear interiors, edge folds and collars to support seaming in the apparel industry [18].

Plasma treatments were a breakthrough in textile processing technology being a clean and dry process that requires no water or organic solvent during processing. Plasma surface modification changes surface properties without affecting the bulk properties of materials, and less energy is consumed compared to conventional treatments [19].

Plasma induced surface modification is an effective way to produce functional textiles. Numerous studies indicated that plasma treatment improves the adhesion characteristics of surfaces. These studies stated that the etching effect of plasma gases caused nano- or micro-roughness, which contributes to the enhancement of adhesion on the surfaces [20 - 25]. Yeh et al. [26] stated that a higher amount of the total surface might be one of the main ways to improve the adhesion strength of laminated fabrics.

There are few studies in the literature studying the effect of plasma treatment on the peel-off behavior of laminated textile fabrics. It was found that the adhesion strength of ultra high molecular weight polyethylene (UHMWPE) laminated woven fabric increased by about 3 - 4 times compared with the untreated one when He/O_2/N_2 plasma treatment is applied [26]. In another study, the adhesion interface strength of PU and PVC coated polyester woven fabric was improved by CO_2 plasma treatment [27]. The adhesion improvement of polyamide laminated fabric through argon and CO_2 plasma treatment was also studied. It was observed that the adhesion properties of polyamide fabrics improved with plasma induced surface roughness compared to the untreated samples [28].

The main problems of laminated fabrics are low bonding strength between layers and resistance to washing. High adhesion strength is required in order to prevent delamination during the end-use of the laminated fabric. PP nonwoven fabrics are commonly used in laminated textiles. However, as the fibre surface is smooth, surface modification might be beneficial as a pretreatment before lamination to improve adhesion. Therefore in the current study, the adhesion properties of argon plasma treated polypropylene laminated nonwoven fabrics were investigated. Argon plasma treatment was selected to enhance the mechanical adhesion of laminated layers. There is very limited information in the literature on the improvement of the washing resistance of laminated fabrics through plasma modification. The washing resistance of these products also needs to be investigated because polypropylene laminated fabrics are also used for apparel purposes besides their use in automotive interiors, as backing materials in home textiles and carpeting etc. Therefore this study also investigated the effect of plasma treatment on the washing resistance of these products. The peel bond strength of the plasma treated laminated samples was observed after 10 wash cycles.

The effects of the plasma treatment on the fabric surfaces were determined by measurements on static contact angle of water. The wettability behaviour of a material can be determined by the contact angle values. AFM and SEM analyses were carried out to characterise the change in surface modification and roughness. After the peel-off test, the remaining amount of adhesives on the peeled surfaces was compared by SEM analysis.

Materials and method

Point-bonded polypropylene spunbonded nonwoven fabrics with a weight per unit area of 70 g/m^2 and thickness of 0.46 mm were supplied by Mogul Tekstil San. Tic. A.S. (Turkey). Nonwoven samples with a length of 12 cm and width of 10 cm were washed with ethanol, rinsed with distilled

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water twice, dried at 40 °C in an oven for half an hour and kept in a desiccator. Pure argon (purity higher than 99.99%) was used as processing gas. Fixamin PUK (polyurethane based adhesive), Mirox AS 1 (defoamer), and Mirox VD P5 (thickener) were used for preparation of the adhesive solution, which was then used for lamination. Polyurethane-based adhesives are commonly used in laminated textiles and have high resistance to washing. Chemicals were supplied by Bozzetto Kimya San. ve Tic. A.S. (Turkey).

Plasma processes

A Diener PICO RF plasma device (Diener Electronics, Germany) was utilised in this study. It has low temperature, low pressure, glow discharge plasma generation. Only one side of the double-layered laminated fabrics was exposed to various plasma treatments, as seen in ***Table 1***. Pure argon (purity higher than 99.99%) was used as the processing gas. Plasma conditions were determined based on the experimental design and statistical significance analysis of water contact angle data using Minitab statistical software.

Table 1. *Plasma treatment conditions.*

Fabric	Plasma pressure, Pa	Plasma gas	Radio frequency, MHz	Plasma power, Watt	Plasma treatment time, min
Polypropylene spunbond nonwoven fabric, 70 g/m^2	30	Argon	13.56	40	3
					5
					10
				60	3
					5
					10
				80	3
					5
					10

Water contact angle

Static contact angles of water on untreated and plasma treated samples were measured using a contact angle meter with a goniometer (KSV CAM200, Diener Electronics) at room temperature. Three measurements were made on five different samples and the average contact angle values were recorded for untreated and plasma treated samples. Droplets of distilled water with a constant volume of 5 µl were dispensed using a microsyringe. Measurements were taken at one-second time intervals for the untreated hydrophobic nonwoven surface, because water droplets remained stable on the surface. Conversely measurements were taken at 17 ms time intervals for the plasma treated nonwoven surface, because water droplets were absorbed immediately by the plasma treated nonwoven surfaces. Water contact angle values were taken at different time intervals: from 0 to 0.05 s.

Preparation of laminated PP nonwoven fabrics

For preparing the adhesive solution, 100 g of Fixamin PUK adhesive was stirred by a mechanical mixer, and 0.5 g of Mirox AS1 was added to the solution and stirred for about 5 minutes. 0.2 g of Mirox VDP5 was added to the solution at 5 minute intervals until the appropriate viscosity, around 10000 - 15000 mPa·s, was obtained similar to industrial applications. Viscosity measurements were performed using a Brookfield DV-II type rotational viscometer at 20 °C. The laminated samples were dried and cured at 120 °C for 7 minutes. The adhesive solution becomes active at a curing temperature of about 120 °C.

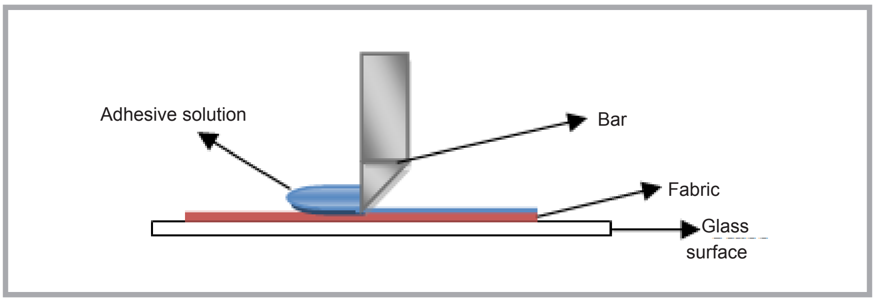

Figure 1. *Schematic of adhesive solution application.*

Two layers of polypropylene nonwoven fabrics were laminated. Adhesive solution application is shown in ***Figure 1***. PU based adhesive solution was coated on the plasma treated side of the nonwoven fabric with a bar adjusted so that the coating thickness was 150 microns, then a second layer of untreated fabric was immediately adhered the adhesive coated base fabric under constant pressure. The samples prepared were dried and cured in an oven at 120 ºC for 7 minutes. The same procedure was also applied to nonwoven fabric samples which were not exposed to plasma treatment in order to compare their adhesion properties.

The samples prepared were coded based on the plasma conditions (***Table 2***). The plasma discharge power was represented by "W" and the plasma exposure time by "M". For example, "40W-5M" means that the sample was treated by 40 Watt plasma discharge power for a five-minutes plasma discharge time.

Table 2. *Actual units and codes of the plasma treatment parameters.*

Plasma parameters	Actual units	Codes
Discharge power	Watt	W
Exposure time	Minute	M

Peel bond strength test

The adhesion strengths of untreated and plasma treated laminated fabrics were determined by a peel bond strength test. The peel bond strength of laminated samples was measured according to the ASTM D 2774 test standard with a James H. Heal - Titan^2 Universal test device [29]. The testing mechanism is illustrated in ***Figure 2*** (see page 106). Test specimens of 25 mm × 100 mm were prepared from the laminated fabrics. Adhesive was only applied on 50 mm of the sample length. The remaining uncoated 50 mm fabric layers were attached to the jaws from both sides at a distance of 25 mm. The peeling speed was selected as 10 mm/min due to the high adhesion strength of the adhesive, shown by previous studies [26, 27], while it was set at 300 mm/min in the test standard. The peeling strength was measured in N/25 mm. Three different measurements were performed and average values calculated. The peel bond strength of the laminated fabrics after 10 wash cycles was also measured for three different samples. The TS 5720 EN ISO 6330 test method was applied to study durability against washing [30].

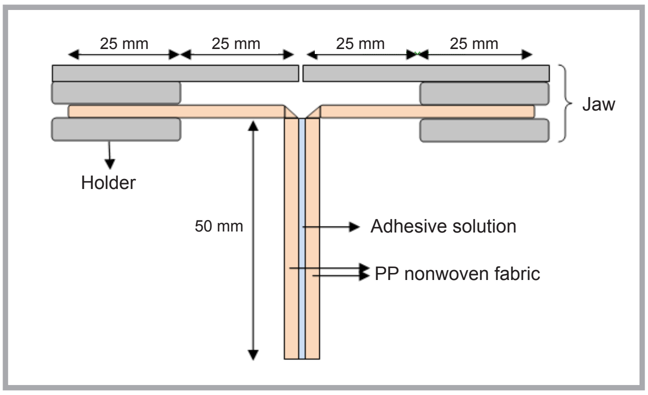

Figure 2. Laminated fabric sample and peel bond strength test scheme.

The bonding interface of double-layered nonwoven laminated fabric is shown in ***Figure 3***. Plasma treated and untreated sides of the peeled nonwoven laminated fabric can be seen.

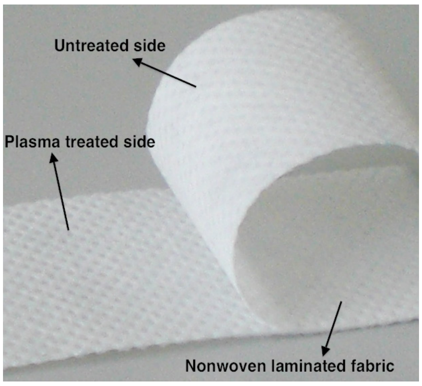

Figure 3. *Photograph of bonding interface of two layered nonwoven laminated.*

Atomic force microscopy (AFM) analysis

The surface morphology of untreated and argon plasma treated polypropylene nonwoven samples were scanned with an AFM device (Shimadzu/SPM-95000J3). A triangular-pyramidal silicon nitride tip was used as a probe, and an area of 2 × 2 μm^2 was scanned in the dynamic mode (tapping mode). The root mean square roughness of the surface (R_{rms}) was calculated from the AFM profiles.

Scanning electron microscope (SEM) analysis

A JEOL JSM 6390LV scanning electron microscope was utilised to observe the change in surface characteristics of polypropylene fibres of the nonwoven fabric after plasma treatment. Samples were coated with gold prior to imaging. Remaining adhesives on the peeled surfaces of each laminated fabric after the peel test were analysed using a LEO 1550VP Field Emission scanning electron microscope. Samples were coated with carbon before analysis.

Results and discussion

Contact angle and wettability

The wettability behaviour of a material can be determined from the contact angle values. A decrease in the contact angle indicates an increase in wettability [1, 13]. Materials that have over a 90 degree contact angle are accepted as having a hydrophobic structure and those that have a less than 90 degree contact angle are accepted as having a hydrophilic structure [31].

The mean water contact angle of the plasma treated samples is shown in ***Figure 4*** for droplet absorption times of 0 and 0.05 sec. The average static water contact angle of untreated polypropylene nonwoven fabric was 127º, showing a highly hydrophobic structure. This showed that cleaning the untreated sample in ethanol before the contact angle measurement did not improve the surface wetting behaviour, which may be due to the fact that the inter-fibre capillary spaces may be blocked by remaining contamination on the fibres [13].

The plasma treatments dramatically decreased the water contact angle and wetting time. Moreover the plasma treated fabrics absorbed water droplets within 0.3 seconds. It was seen that the contact angle decreased by increasing the plasma power and plasma exposure time. The highest decrease in the water contact angle was observed at 10 minutes of plasma exposure time, as can seen from ***Figure 4***. Within 0.05 seconds, the contact angles of the fabrics decreased by about 25°. Moreover the contact angle could not be taken from the 80W-10M plasma treated fabric because the water droplet was completely absorbed by the treated surface. The results showed that as the treatment time was increased from 3 to 10 minutes, the wettability also increased.

Peel bond strength

Adhesion strength was measured by way of the peel bond strength of the laminated fabrics. The peel bond strength of the untreated and plasma treated PP nonwoven laminated samples is given in ***Figure 5***. The adhesion bond strength of plasma treated laminated samples showed an

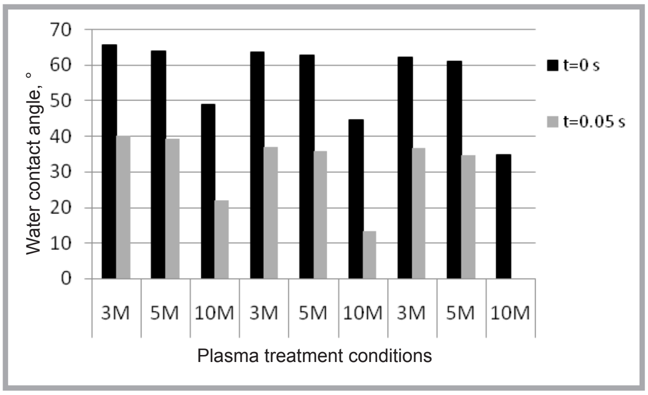

Figure 4. *Mean water contact angle results for PP nonwoven fabric treated under various plasma conditions.*

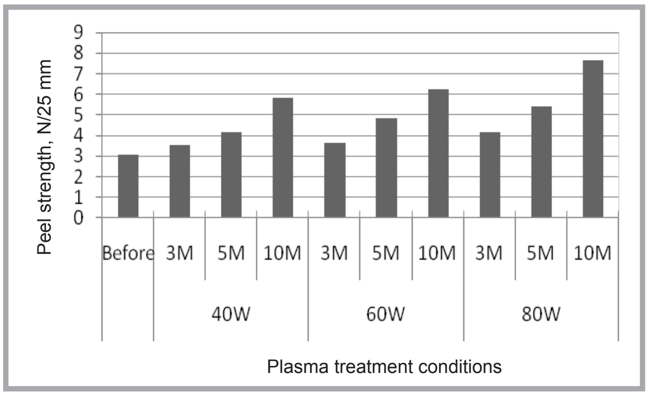

Figure 5. *Peel bond strength results for PP nonwoven laminated fabrics.*

Table 3. *Peel bond strength comparison of laminated fabrics before and after 10 washing cycles.*

Plasma treatment conditions	Peel bond strength, N/25 mm	Peel bond strength, (N/25 mm) /10 wc
Untreated	3.09	2.75
40W-3M	3.54	3.34
60W-3M	3.64	3.47
80W-3M	4.16	3.98
40W-5M	4.15	4.03
60W-5M	4.84	4.57
80W-5M	5.40	5.02
40W-10M	5.82	5.42
60W-10M	6.24	5.98
80W-10M	7.66	7.06

increase of about 150% compared to the untreated laminated samples.

As can be seen from ***Figure 5***, increasing the plasma power and exposure time enhanced the peel bond strength. As the plasma treatment time was increased from 3 min to 10 min, the peel bond strength increased by 64%, 71% and 84% at a plasma power of 40, 60 and 80 W, respectively. The highest peel bond strength values were obtained at a plasma power of 80 W and plasma time of 10 min. The results showed that the plasma treatment time was more effective in improving the adhesion than the plasma power. The results are in agreement with recent studies showing that the degree of plasma modification depended on the plasma exposure time and discharge power [26, 32].

A comparison of results of the peel bond strength of the laminated fabrics before and after 10 washing cycles is given in ***Table 3***. After 10 WC (wash cycles), untreated laminated fabric had an 11% decrease in the peel strength, while plasma treated laminated fabric showed a lower peel strength decrease ranging from 2.9 to 7.8%. This showed that the washing resistance of laminated textiles was not significantly improved after argon plasma treatment. Moreover the washing treatment of untreated laminated samples did not result in a significant decrease in peel bond strength as well, which may be due to the high adhesion strength obtained by the PU-based adhesives.

Table 4. *Roughness values (R_{rms}) of the untreated and plasma-treated PP nonwoven fabrics.*

Sample	R_{rms}, nm
Untreated	4.977
80W-5M	14.828
40W-10M	22.416
80W-10M	36.816

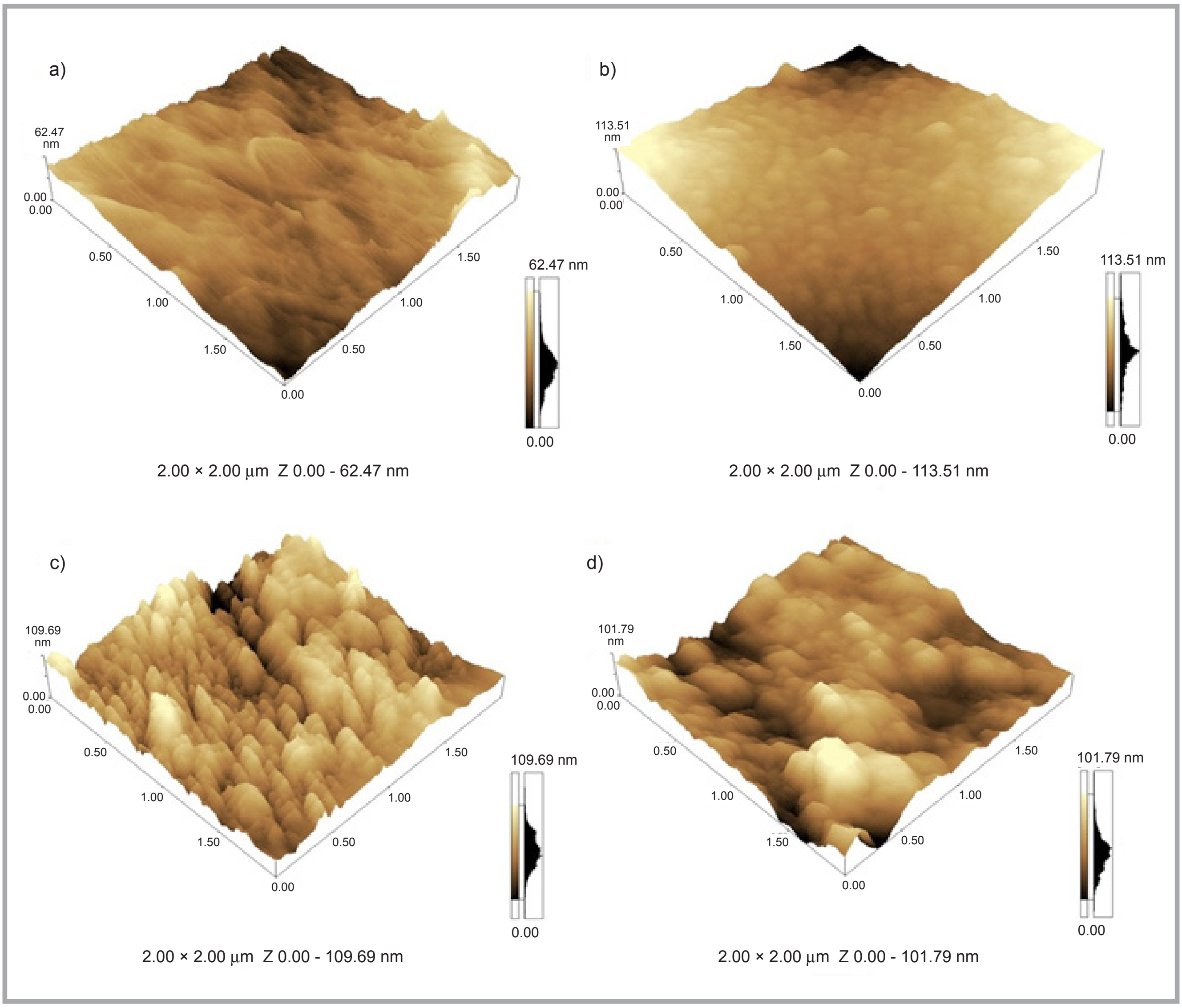

Figure 6. *The AFM images of Ar plasma treated PP nonwoven fabrics; a) untreated, b) 40W-10M treated, c) 80W-5M treated, d) 80W-10M treated.*

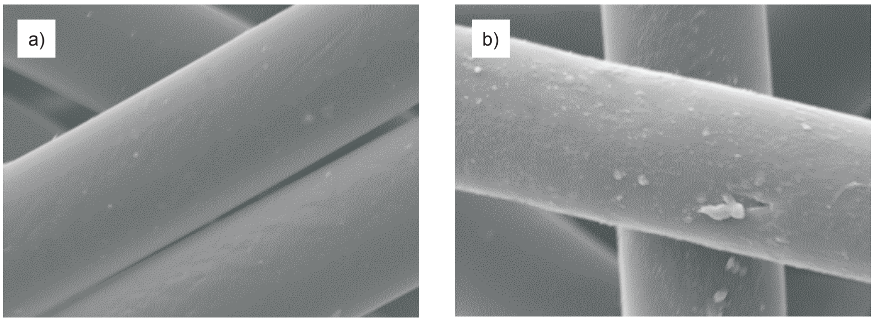

Figure 7. *SEM images of PP fibres, untreated and treated at 80W-10M Ar: a) untreated (×5060), b) treated (×5490).*

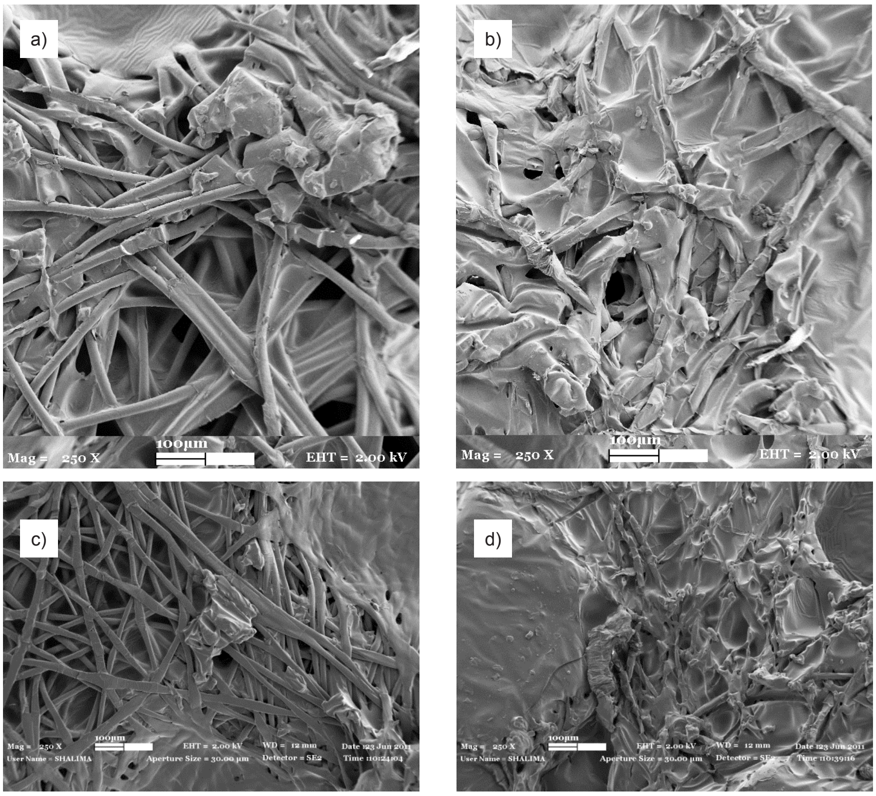

Figure 8. *SEM images of remained adhesives on the surface (x250): (a) untreated, (b) 40W-10M Ar plasma treated side (c) untreated, (d) 80W-3M Ar plasma treated side.*

The peel bond strength of the untreated laminated sample increased from 3.09 to 7.66 N/25 mm after plasma treatment at a plasma power of 80 W and treatment time of 10 min. After 10 wash cycles the peel bond strength decreased from 7.66 to 7.06 N/25 mm, showing a decrease in the peel strength of only 7.8%, while the decrease for the untreated sample was 11%. Overall, argon plasma treatment improved the peel bond strength of laminated samples compared to untreated samples with increasing plasma power and treatment times.

AFM (Atomic force microscopy) analysis

The surface modifications of untreated and various argon plasma treated PP nonwoven fabrics were observed using AFM images, the results of which are given in ***Figure 6***. As can be seen from ***Figure 6***, argon plasma treatment resulted in a roughening effect on the PP surface.

Table 4 shows the surface roughness values of untreated and plasma treated samples. The untreated fabric surface had a 4.977 nm roughness value, while the surfaces of the plasma treated sample had higher roughness, as indicated in ***Table 4***. The root mean square roughness of the surfaces increased remarkably after plasma treatment. It was observed that increasing the plasma exposure time and discharge power led to an increase in surface roughness due to the etching effect. The increased adhesion strength of laminated fabrics may be attributed to the increase in surface roughness due to argon plasma treatment.

SEM (Scanning electron microscope) analysis

Figure 7 shows SEM images of untreated and plasma treated polypropylene nonwoven samples. Morphological changes on the fibre surface after argon plasma treatment can be observed. The untreated PP fibre surface has smooth-like properties, while plasma treated PP fibres show rougher surfaces. Plasma induced roughening was observed on the polypropylene fibre surface, as also shown in previous studies [1, 33, 34].

It is clear that argon plasma treatment etched the fibre surfaces. Morphological alteration of the fibre surface might lead to improved mechanical adhesion due to the roughening effect.

After the peeling test, the remaining adhesive on the separated lamination layers can be seen in the SEM micrographs in ***Figure 8***. There was more remaining adhesive on the plasma treated surfaces than that of untreated surfaces, as also revealed in previous studies [26, 28]. PU adhesive is mainly attached to the surface of plasma treated PP filling the spaces between fibres.

Conclusions

The adhesion strength of argon plasma treated polypropylene nonwoven laminated fabric was investigated in this study. It was seen that the contact angle decreased by increasing the plasma power and plasma exposure time, indicating that the wettability increased through plasma treatment. The peel bond strength was measured for various plasma conditions, i.e., exposure time and plasma power, in order to evaluate the effect of plasma treatment on adhesion between the laminated fabric layers. The results showed that the plasma treatment time was more effective in improving the adhesion than the plasma power. The adhesion bond strength of plasma treated laminated samples showed an increase of about 150% compared to the untreated laminated samples. On the other hand, the washing resistance of laminated fabrics was not significantly improved by plasma treatment, which was explained by the high adhesive strength of PU-based adhesive, providing a minor decrease in adhesive strength after 10 wash cycles.

SEM and AFM images indicate that surface roughness increases with plasma treatment, and etching on the fibre surface was an important factor for the improvement of adhesion strength.

This study will be extended using reactive plasma gases such as oxygen and also using acrylic based adhesives, which are less resistant to washing.

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