

# Improvement of Liquid Absorption Properties of Nonwoven Polypropylene Substrates by Low Pressure Plasma Treatment with CH<sub>4</sub>-O<sub>2</sub> Mixture Gas

R. López, M. Pascual, D. García-Sanoguera<sup>1</sup>, L. Sánchez-Nacher<sup>1</sup>, and R. Balart<sup>1\*</sup>

*Textile Research Institute (AITEC), Plaza Emilio Sala 1, 03801, Alcoy, Alicante, Spain*

<sup>1</sup>*Materials Technology Institute (ITM), Polytechnic University of Valencia (UPV),*

*Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Alicante, Spain*

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**Abstract:** The use of nonwoven textile substrates for filtration and absorption purposes is generalized due to the high surface area they can provide. Many of these applications require good wetting properties to increase efficiency. In this work, low pressure plasma treatment with a CH<sub>4</sub>-O<sub>2</sub> mixture gas has been used to increase surface wettability and subsequent absorption properties on nonwoven polypropylene substrates. CH<sub>4</sub> plasma treatment leads to a plasma polymerization process which results in hydrophobic surface finishing, but in combination with O<sub>2</sub>, it is possible to form a functionalized plasmopolymerized layer thus improving wetting properties. Changes in wetting properties have been studied by contact angle measurements showing that optimum wetting properties are obtained with exposure times to plasma treatment of about 10 min, and no significant changes are obtained for longer exposure times. Absorption efficiency has been followed by determining three different parameters by the guidelines of the UNE-EN-ISO 9073-6 standard: wetting time, liquid absorption capacity (LAC) and liquid propagation rate or absorption speed. All these properties are remarkably improved as the exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma increases; this improvement is remarkably high for relatively short exposure times (5-10 min) and no significant changes are obtained for long exposure times so that, it is possible to conclude that previous plasma treatment with exposure times in the 5-10 min range is an efficient method to improve overall absorption properties of nonwoven polypropylene substrates.

**Keywords:** Cold plasma, Poly (propylene) (PP), Nonwoven, Liquid absorption, Organic gas

## Introduction

Textile substrates are highly used as membranes for filtration applications due to an excellent combination of surface-to-volume ratio, large porosity and variety of fibers for different purposes such as air filtration, wastewater purification, protein adsorption, blood filtration, liquid absorption, water transport, etc. [1-5]. There is a wide variety of textile structures and fibers which are capable to fulfill requirements for filter media and technical membranes. Many research works have focused on the use common fibers such as polypropylene, cotton, polyester, etc. as filter media due to their excellent absorption properties. In many cases, a previous surface pre-treatment is made to provide a particular functionality. So that, different surface treatments have been successfully employed on polyester, polypropylene, cotton, etc. textile substrates (woven and non-woven) to increase wettability, dyeability, adhesion behavior, absorption properties, etc. [6-14].

In the last years, a remarkable increase in research works on membranes based on the use of nanofiber webs has been detected [15-17]. This is due to the fact that these textile structures offer a surface-to-volume ratio higher to that obtained with nonwoven textiles with fibers in the micro-scale range. Different fibers have successfully used to obtain nanofibers' webs that can be deposited onto a textile substrate (fabric or non-woven) with optimum filtering

efficiency together with additional functionalities. Some interesting works have used chitosan as the base polymer for web formation thus giving additional antibacterial protection [18]. Polyacrylonitrile has been used as base polymer for the web formation and an additional chitosan coating can lead to improve overall efficiency as a wastewater membrane [17]. Nevertheless, the use of these nanostructures at industrial level is still low due to high costs.

Another interesting method to change wetting properties of textile fibers is by modifying surface properties to change wettability properties or to provide a particular functionality. In this field, some surface treatments have been used to change wetting properties of different textile substrates (fabrics, fibers, nonwovens, etc.) [19]. These treatments include physical and/or chemical modification [20]. Plasma techniques (cold, corona and atmospheric plasmas) have been widely used for textile treatments due to the excellent balance between improved properties and environmental efficiency [21-26]. Other interesting technique to provide a particular functionality on a textile substrate is surface coating in which different polymeric monomers can be deposited onto a textile substrate but in some cases, this procedure has the disadvantage that most of the functionality is lost after several washing cycles [27-30]. For this reason, important efforts to obtain durable modified surfaces are being done. In some cases it is possible to obtain good adhesion between the coated layer and the textile substrate by using photografting, plasma pretreatment or radiation induced processes [31-35]; these

\*Corresponding author: rbalart@mcm.upv.es

processes allow a mechanical interlock and chemical interaction which are responsible for durability of the coated layer. Plasma polymerization is an alternative method to previously described methods since it allows deposition of a thin functionalized layer characterized by high durability due to high interactions between the deposited layer and the textile substrate [36]. Other authors have obtained durable properties by combining surface plasma pre-treatment followed by crosslinking of a particular component [33]. In our previous works, we report the use of low pressure plasma with methane: oxygen mixture gas to promote formation of a thin organic layer on polypropylene films to achieve durable wetting properties [36,37].

In this work we have used low pressure glow discharge plasma with a CH<sub>4</sub>-O<sub>2</sub> (volume ratio 80:20) mixture gas to increase wetting properties of polypropylene nonwoven spunbond for absorption applications in which, durable effects of surface treatments are required for good efficiency. The effect of the exposure time to plasma has been followed by contact angle measurement; the effect of the plasma pre-treatment on absorption properties has been studied by determining different parameters following the guidelines of the UNE-EN-ISO 9073-6: absorption time, liquid absorption capacity (LAC) and absorption speed.

## Experimental

### Materials

As a base textile substrate, it was used a polypropylene nonwoven spunbond commercial grade PEGATEX<sup>®</sup> supplied by PEGAS NONWOVENS (PEGAS NONWOVENS s.r.o., Znojmo, Czech Republic) with a surface density of 16.5 g m<sup>-2</sup>.

### Plasma Modification of Polypropylene Substrates

The plasma treatment was carried out at low pressure conditions with a CH<sub>4</sub>-O<sub>2</sub> mixture gas (volume ratio 80:20). A radiofrequency RF generator (working at 13.56 MHz and a maximum working power of 150 W) mod CD 400 MC option PC (Europlasma, Oudenaarde, Belgium) was used. The plasma chamber is equipped with four Al shelves with a total volume of 64 L. The mixture gas flow rate was set to 100 cm<sup>3</sup> min<sup>-1</sup> and the working pressure was maintained in the 31-32 Pa range. The working power was set to 150 W for all plasma treatments. The exposure time to plasma treatment varied in the 0-30 min range.

### Characterization of Wetting Properties

Wettability changes were evaluated by contact angle measurements with a KSV CAM 200 goniometer (KSV Instruments, Helsinki, Finland). As contact angle changes with time in non-woven substrates, all contact angle values were determined after 30 s of liquid drop to obtain comparable results. Double distilled water and glycerol (Scharlab S.L., Barcelona, Spain) were used as test liquids.

## Microscopic Characterization

Surface morphology of non-woven polypropylene substrates was evaluated by scanning electron microscopy (SEM) with a FEI mod. Phenom (FEI, Oregon, USA). Prior to sample observation, samples were coated with an aurum-palladium alloy with a Sputter Coater EMITECH mod. SC7620 (Quorum Technologies Ltd. East Sussex, United Kingdom).

## Absorption Properties of Nonwovens

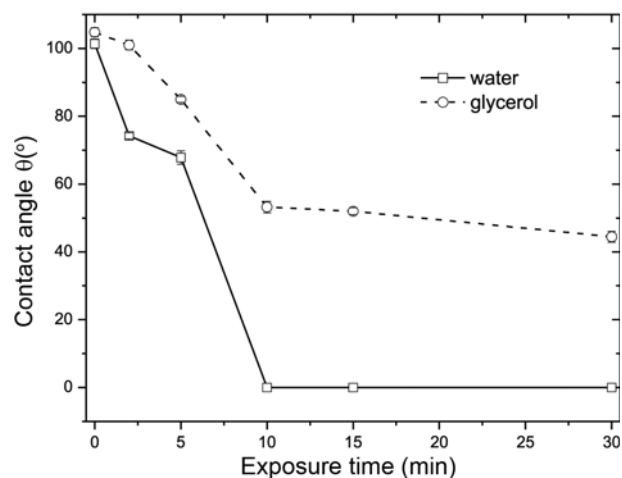
Characterization of absorption properties was carried out following the guidelines of the UNE-EN-ISO 9073-6 standard. This standard focuses on characterization of absorption efficiency of nonwoven textile substrates through determination of several properties: absorption time, liquid absorption capacity (LAC) and absorption speed. The absorption time is defined as the time taken for a sample to completely absorb the liquid used in the test. The liquid absorption capacity represents the quantity of liquid that a nonwoven can absorb, expressed as a percentage, under fixed conditions and time span. Finally, the absorption speed is a measure of the capillary capacity of a nonwoven, the speed at which a liquid can pass through.

## Results and Discussion

### Study of Wettability Changes Due to Plasma Treatment

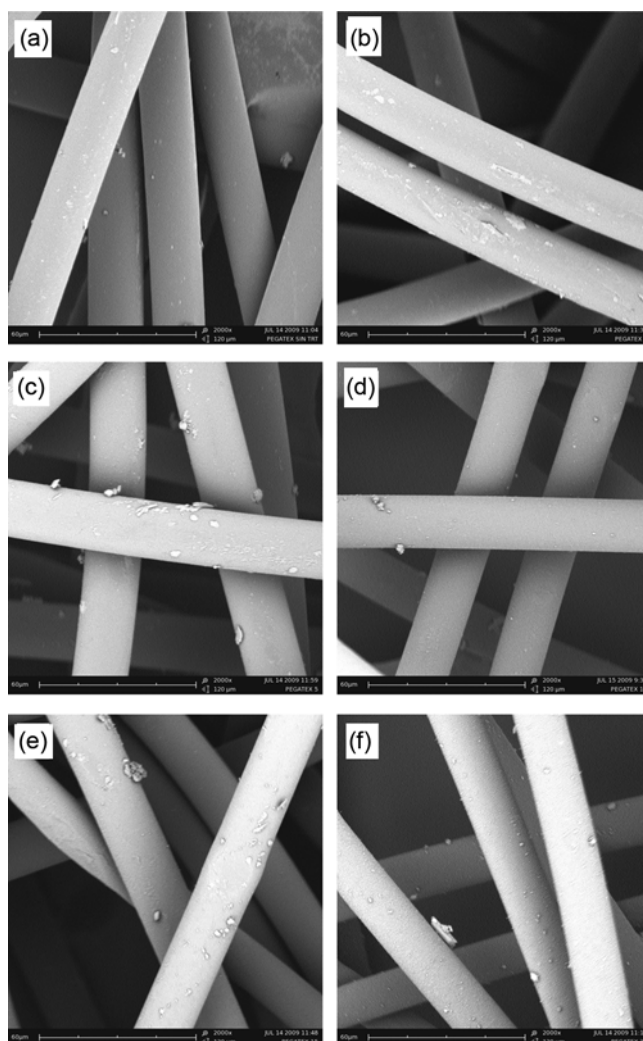
Changes in wetting properties of PP nonwoven substrates as a consequence of the plasma treatment, have been followed by contact angle measurements with different test liquids. As these substrates are nonwoven, the contact angle changes in time (dynamic contact angle), so that, in order to compare results, contact angle values have been taken after 30 s, when values tend to stabilize.

Figure 1 shows the evolution of the contact angle for water and glycerol as test liquids in terms of the exposure time to



**Figure 1.** Plot of the evolution of the contact angle of nonwoven PP substrates for different test liquids in terms of the exposure time to low pressure plasma with CH<sub>4</sub>-O<sub>2</sub> mixture gas.

plasma treatment. It is important to note that the initial contact angle for both test liquids is higher than  $100^\circ$  thus showing a marked hydrophobic behavior. Nevertheless, the plasma treatment produces a positive effect on general improvement of hydrophilic properties of nonwoven PP substrates. In the case of water as test liquid, the initial contact angle for untreated nonwoven PP is close to  $101.4^\circ$  and this value decreases as the exposure time to  $\text{CH}_4\text{-O}_2$  plasma increases. Furthermore, total absorption of the water drop is obtained for nonwoven PP substrates with a previous plasma treatment with exposure times higher than 10 min (contact angle =  $0^\circ$ ), thus showing a remarkable increase in hydrophilic behavior. Similar tendency can be observed for glycerol as test liquid but in this case, total wetting properties are not obtained. Despite this, we can observe a pronounced decrease in contact angle values from  $104.8^\circ$  (untreated nonwoven PP) up to values of about  $45\text{-}50^\circ$  for plasma exposure times higher than 10 min which is in total agreement with the results obtained with water as test liquid. As we have described in our previous works, plasma treatment with  $\text{CH}_4\text{-O}_2$  mixture gas on polypropylene substrates promotes formation of a thin functionalized organic layer in a similar process to plasma polymerization [38]; so that, changes in surface topography are expectable. In addition to this, it is important to consider that even in these plasma conditions (considered as cold plasma), excessive exposition can lead to overheating and this could have a negative effect on overall thermal stability as observed in jute fibres treated with Ar plasma. For this reason, we have carried out a SEM study of the nonwoven PP substrates with different exposure times to  $\text{CH}_4\text{-O}_2$  plasma treatment (Figure 2). In Figure 2(a), we can observe untreated PP microfibers with average diameter of 20 mm characterized by highly smooth surface. The effects of the plasma treatment with  $\text{CH}_4\text{-O}_2$  on surface topography are derived from a competition of two different processes: etching and plasma polymerization occurring at the same time and this can produce some changes in surface topography. Despite this, as our previous results have revealed, the overall effect is deposition of a thin functionalized organic layer which is responsible for wettability improvement and this indicates that plasma polymerization occurs in a greater extent than surface etching (material removal). So that, our previous results have revealed that slight changes in surface roughness are detected after the plasma treatment [38]. SEM photographs in Figure 2 are in agreement with our previous works as no significant changes in surface topography are detected by SEM analysis [37]. Figure 2(b) shows the SEM photograph for samples subjected to plasma treatment for an exposure time of 2 min. We can observe relatively high smooth surfaces on polypropylene individual microfibers thus indicating that a homogeneous thin-organic layer (from the plasma species) has been deposited onto polypropylene surface. Despite this, presence of small particles (with average size in the 1-5 mm range) derived from the etching-



**Figure 2.** SEM microphotographs of nonwoven PP substrates with different exposure times to  $\text{CH}_4\text{-O}_2$  plasma treatment (2000 $\times$ ); (a) untreated, (b) 2 min, (c) 5 min, (d) 10 min, (e) 15 min, and (f) 30 min.

plasma polymerization competition process can be detected. This situation is evident for all exposure times as observed in Figure 2(c) and 2(d) for exposure times to plasma treatment of 5 and 10 min respectively. For long exposure times (higher than 15 min) some degradation effects are evident: lots of adhered particles can be detected in Figure 2(e) and possible microfusion or softening (bulb formation) due to overheating. It is important to remark that this plasma is a cold type plasma but long exposure times can produce local overheating which is important for these polymers as they are highly sensitive to temperature changes. Finally, samples subjected to plasma treatment for an exposure time of 30 min (Figure 2(f)), show similar surface structure but slightly higher roughness can be observed thus indicating that etching mechanism occurs in a greater extent for long exposure times and some cracks are formed on the plasma

polymerized organic layer.

### Effect of the Plasma Treatment on Absorption Properties

The absorption time is representative for the tendency of a substrate to be impregnated by certain liquid so it is representative for its absorption properties. Table 1 shows measurements of the wetting times of nonwoven PP substrates as a function of the exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma treatment. The wetting time is directly related to contact angle measurements. We can clearly observe a decrease in wetting time values as the exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma increases.

The initial wetting time for untreated nonwoven PP is close to 49.4 s and this value is remarkably reduced for exposure times to plasma in the 2-10 min range, reaching values close to 29 s. For long exposure times, the wetting times remains almost constant with values close to 29-30 s, thus indicating that, exposure times to plasma treatment higher than 10 min do not lead to an increase in wetting properties. These values are in total accordance with those obtained with contact angle measurements.

Another parameter representative for the liquid absorption efficiency of a substrate is the liquid absorption capacity (LAC) which represents the weight percentage increase of a sample with controlled geometry and dimensions when submerged in a test liquid, in this case, distilled water. Table 2 shows LAC values for nonwoven PP substrates with different exposure times to plasma treatment. As we are working with

**Table 1.** Wetting times of nonwoven PP substrates with different exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma treatment obtained by the guidelines of the UNE-EN-ISO 9073-6 standard

Exposure time (min)	Wetting time (s)	Standard deviation	(%) variation
0	49.4	4.0	0.0
2	48.0	2.8	-2.8
5	45.0	2.4	-8.9
10	28.8	2.6	-41.7
15	29.2	2.9	-40.9
30	30.2	2.6	-38.9

**Table 2.** Liquid Absorption Capacity (LAC) of nonwoven PP substrates with different exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma treatment obtained by the guidelines of the UNE-EN-ISO 9073-6 standard

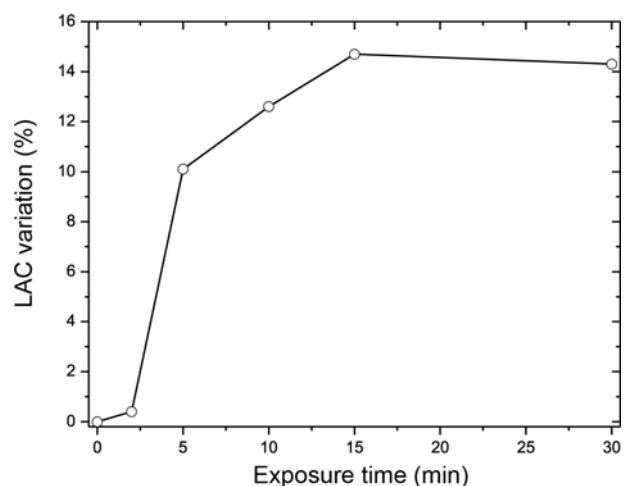
Exposure time (min)	Liquid absorption capacity, LAC (%)	Standard deviation
0	1155	140
2	1160	17
5	1271	90
10	1300	47
15	1324	98
30	1320	125

nonwoven textile substrates, the LAC values are high (more than 10 times higher) due to excellent absorption properties of these substrates. The initial LAC value of untreated nonwoven PP is around 1155 % and this value is increased up to 1300-1325 % for exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma in the 10-15 min range. Longer exposure times do not lead to a significant increase in LAC values and this totally agrees with previous results.

If we take as a reference the LAC value for the untreated material (1155 %), we can determine the percentage of LAC increase as shown in Figure 3. We observe a marked % increase in LAC values for relatively low exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma in the 2-10 min range (up to 13 %) and a tendency to stabilize for long exposure times as observed previously.

The absorption time and the liquid absorption capacity (LAC) are interesting to characterize nonwoven substrates for a certain time or in controlled absorption conditions. Sometimes it is interesting to obtain information on how the absorption process takes place. For this reason, we have also evaluated the absorption properties of nonwoven PP substrates by determining the liquid propagation rate or absorption speed. This is determined from the capillary height (or capillary propagation) for different times; with this information it is possible to know the liquid propagation rate in a considered substrate.

Table 3 shows numerical values for capillary height at different measurement times. As the measuring time increases, obviously the capillary height increases and this tendency is observed for all exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma treatment. But what it is more important for this data series is that capillary heights increase as the exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma increases which is representative for an improvement



**Figure 3.** Plot of the evolution of the LAC (liquid absorption capacity) % increase\* of nonwoven PP substrates in terms of the exposure time to low pressure plasma with CH<sub>4</sub>-O<sub>2</sub> mixture gas.\* It has been used as a reference for % calculation, the LAC value of the untreated nonwoven PP.

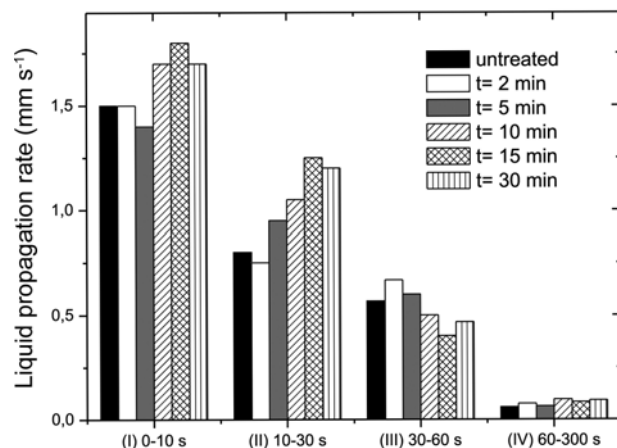
**Table 3.** Capillary heights (taken at different measuring times) of nonwoven PP substrates with different exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma treatment obtained by the guidelines of the UNE-EN-ISO 9073-6 standard

Exposure time (min)	Capillary height (mm)			
	t=10 s	t=30 s	t=60 s	t=300 s
0	15	31	48	62
2	15	30	50	68
5	14	33	51	66
10	17	38	53	76
15	18	43	55	75
30	17	41	55	77

on overall wetting properties of nonwoven PP substrate. If we consider a measuring time of 60 s, the initial capillary height for untreated nonwoven PP is 48 mm and this value is increased up to 55 mm for a plasma-treated PP substrate with an exposure time of 15 min. Similar tendency is observed for all the considered measuring times (10, 30 and 300 s). Nevertheless this tendency is more pronounced for higher measuring times (300 s) which leads to a capillary height difference of about 15 mm for the untreated and plasma-treated sample (with 30 min exposure time). Also, if we observe data for 300 s measuring time, we clearly see that a great increase in capillary height is obtained for relatively short exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma (5-10 min) and longer exposure times do not lead to a significant increase.

In addition to this information, it is possible to determine the liquid propagation rate as the ratio between the capillary height and the measuring time. We have calculated the average propagation rate for each range of time as the ratio between the capillary height difference and the measuring time difference. Figure 4 shows a comparative representation of the liquid propagation rate for the different measuring time ranges and the effect of the exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma treatment. If we consider the untreated nonwoven PP substrate, evolution of the liquid propagation rate is as follows: in the first measuring time range (I: 0 to 10 s), the propagation rate reaches the highest value (in this case, close to 1.5 mm s<sup>-1</sup>). In the second measuring time range (from 10 to 30 s), we observe a decrease on average propagation rate with values of about 0.75-0.80 mm s<sup>-1</sup>. In the third measuring time range (from 30 to 60 s), the propagation rate is around 0.6 mm s<sup>-1</sup> and this value is remarkably reduced up to values of about 0.06 mm s<sup>-1</sup> in the last measuring time range (from 60 to 300 s). This indicates that capillarity phenomena are quick at initial stages and slow down as the time passes.

We can also observe the effect of the previous exposure time to CH<sub>4</sub>-O<sub>2</sub> plasma if we compare the average propagation rates in terms of the exposure time to plasma. As it is clear from observation of Figure 4, at the initial stages of the



**Figure 4.** Comparative representation of the average liquid propagation rate or absorption speed on nonwoven PP substrates at different measuring time ranges for different exposure times to CH<sub>4</sub>-O<sub>2</sub> plasma treatment.

capillarity phenomena we can see an increase in the average propagation rate as the exposure time to plasma increases (range I and II) and this is representative for an improvement on wetting properties as wetting times can be reduced.

## Conclusion

The use of low pressure glow discharge plasma with CH<sub>4</sub>-O<sub>2</sub> (volume ratio 80:20) mixture gas is an efficient method to improve the overall wetting properties of polypropylene nonwoven substrates. Water contact angles are remarkably reduced from 101° (untreated PP substrate) to 0° (plasma-treated PP with an exposure time of about 10 min) and this is indicative of great improvement in wetting properties. Regarding liquid absorption characterization, it is important to remark that the water absorption time is reduced by 40% for plasma exposure times of about 10-15 min. On other hand, the liquid absorption capacity (LAC) is improved by 12-14% for intermediate exposure times to plasma (10-15 min). Concerning to absorption speed, the capillary height (for 300 s) changes from 62 mm (untreated PP substrate) up to 77 mm (plasma-treated PP substrate with exposure time of 10 min). So we can conclude that surface treatment with low pressure CH<sub>4</sub>-O<sub>2</sub> plasma is an efficient method to improve the overall liquid absorption properties of polypropylene nonwoven substrates and the optimum exposure time is close to 10 min since good balanced properties are obtained.

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