

Modification of PP Fabric with Polyols by the Plasma Composite Technique

Wen-Xiao Ma^{1*}, Bing-Nan Hu¹, Yun Bai¹

¹ Hohhot City, Inner Mongolia 010080, China

* Corresponding author. E-mail: mawenxiao1978@hotmail.com

Abstract

In order to endow polypropylene (PP) fabric with hydrophilic and antistatic properties, PP was grafted with polyols (sorbitol, maltitol and polyethylene glycol (PEG)) by the oxygen plasma treatment and subsequent bridging process, where ethylene glycol diglycidyl ether (EGDE) and trimethylolpropanetriol (2-methyl-1-aziridinepropionate) (TTMA) were used as crosslinkers. The highest grafting rate was 7.48%. The chemical structure changes were analysed by Fourier transform infrared spectroscopy (FTIR). A scanning electron microscope (SEM) was used to observe the changes in polypropylene fiber surface morphology. The hydrophilicity was characterized by testing loose fiber suspension and moisture retention. The modified PP was more easily immersed in water and the desorption behaviour continued for 2h without equilibrium. The half-life of the grafted PP was reduced to 7s. The results showed that the modified PP with polyols possessed hydrophilicity and antistatic properties.

Keywords

PP fabric, polyols, crosslinker, antistatic property, hydrophilicity.

1. Introduction

PP fiber is a semi-crystalline thermoplastic polymer, widely applied in decoration cloth, packing fabric, non-woven fabrics, workwear and medical care dressing due to its excellent mechanical properties and good chemical stability. But PP fiber has no polar group on its surface, which makes it difficult to be dyed on the one hand, and static electricity easy to generate on the other hand [1]. Thus, it is very necessary to pursue the hydrophilic modification of PP fiber. Universal hydrophilic modification of PP involves surface coating [2, 3], wet chemical [4-6] and physical radiation containing plasma discharge [7-10]. It is reported that low temperature plasma treatment can positively produce large amounts of the polar group on the PP surface [11, 12]. However, the surface properties improved by plasma usually weakens with the lay-up time, known as the ageing effect of plasma treatment, because the polar groups produced will decay [13]. To attain lasting hydrophilicity, the grafting polymerisation of hydrophilic monomer is developed after plasma treatment [14-17]. However, in this method it is difficult to control the grafting rate [18, 19].

Actually, when many polar groups are produced on the surface of PP fiber after plasma treatment, the fibers can attach hydrophilic agents, aided by crosslinkers.

Bratskaya et al. described the coating of chitosan onto O₂-plasma treated PP surfaces through epichlorohydrin as the crosslinker [20]. Tsou et al. [21] reported quaternary methyl diallyl ammonium salt grafting onto O₂ plasma treated PP nonwovens; the finished PP fabric indicated favourable antibacterial and hydrophilic properties. Wu et al. [22] introduced a modification of polypropylene treated by air plasma and feather keratin graft, the results of which showed that the surface hydrophilicity and printing property were greatly improved. Ma et al. [23] achieved three hydrophilic biomolecule grafting onto PP fabric using NH₃ and N₂ low temperature plasma pre-treatment.

As is known, polyols possess good hydrophilicity due to its multi-hydroxy groups, including sorbitol, maltitol and polyethylene glycol. Sorbitol is an important food humectant and has been gradually applied in pharmacology, cosmetics, and other fields [24-26]. Maltitol, a disaccharide consisting of a glucose unit linked to a sorbitol one via an α -1,4 bond, is obtained from maltose by hydrogenation. It is mostly used in food, toothpastes, and tablets owing to its high affinity for water, low calorie sweetener and effective acaricidal activity [27, 28]. PEG is a polymer from ethylene glycol, which exhibits excellent moisture retention, adhesive, and antistatic

properties in many fields [29-31]. Once polyols are linked to the surface of the plasma-treated PP fabric, permanent hydrophilicity can be obtained.

For polyol grafting, a crosslinker which can react with the hydroxyl group is necessary. N-methylol reagents (DMDHEU), polycarboxylic acids (BTCA), acrylamide (FAP or MBA), blocked-diisocyanate (HDACS), and divinyl sulfonyl sulfate are effective reagents and have been well demonstrated in cotton or cellulose fiber crosslinking [32-38]. However, formaldehyde will be released when DMDHEU-treated fabric is subjected to multiple laundering cycles, and the reaction of other crosslinkers needs high temperature. Considering saving energy, low temperature crosslinking is the most desirable. Epoxy and aziridine crosslinkers were reported to react at low temperatures [39, 40], due to the three-membered heterocyclic ring at the less substituted carbon atom, which is susceptible to attack by a range of nucleophiles (e.g., amines, alcohols, acids) [41, 42]. Kurmaev et al. [43] used X-ray fluorescence measurements to show that the epoxy group of EGDE as a crosslinker reacted with the hydroxyl group of chitosan. Xiao-Qiong Li and Ren-Cheng Tang [44] analysed the FTIR spectra of crosslinked chitosan fiber with an SaC-100 crosslinker, implying that the hydroxyl groups in chitosan react with the aziridine.

Based on the above investigation, this study aimed to graft polyols (sorbitol, maltitol, polyethylene glycol) onto the surface of PP to develop a hydrophilic and antistatic fabric. To provide a grafting site for PP, an O_2 plasma pretreatment was firstly designed to produce $-COOH$ or $-OH$. Then, the attachment of polyols on the surface of the fiber was performed using EGDE and TTMA, respectively. Finally, the wettability, moisture retention, antistatic and coloring performance of modified PP fabric were determined and analysed.

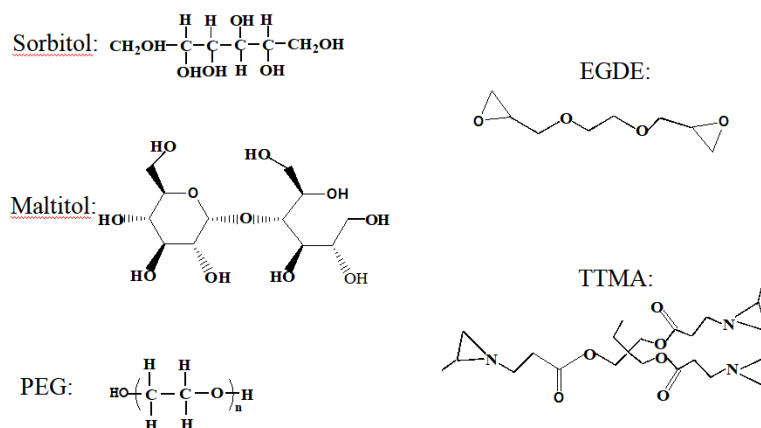


Fig. 1. Structure diagram of polyols (sorbitol, maltitol, PEG) and crosslinkers (EGDE, TTMA)

2. Experiments and test method

2.1. Materials and reagents

The PP woven fabric (warp/weft: 182/96, 364 g/m²) used in this study was supplied by Anping Country Yongding Filter Cloth Weaving Co., Ltd (China). The samples (4cm×8cm) were cleaned with acetone and distilled water in an ultrasonic cleaning machine before the experiment to remove impurities. Three kinds of polyols (sorbitol, maltitol and PEG-800) were purchased from Tianjin Fengchuan Reagent Technology Co., Ltd (China). EGDE and TTMA crosslinkers were purchased from Chengdu Aikeda Chemical Reagent Co., Ltd (China). **Figure 1** shows the structure of the polyols and crosslinkers used in this experiment. The reactive dyes (reactive yellow KE-4R, reactive scarlet B-3G, and reactive deep blue K-R) were supplied by Tianjin Chemical Reagent Co., Ltd (China).

2.2. Treatment methods

The whole experiment was divided into two parts: plasma pretreatment of PP fibers and polyols post-grafting. **Figure 2** is a flow chart of the total processing.

2.2.1. Plasma treatment

Plasma technology is a new modification technology for fiber and textile which can improve the hygroscopicity of

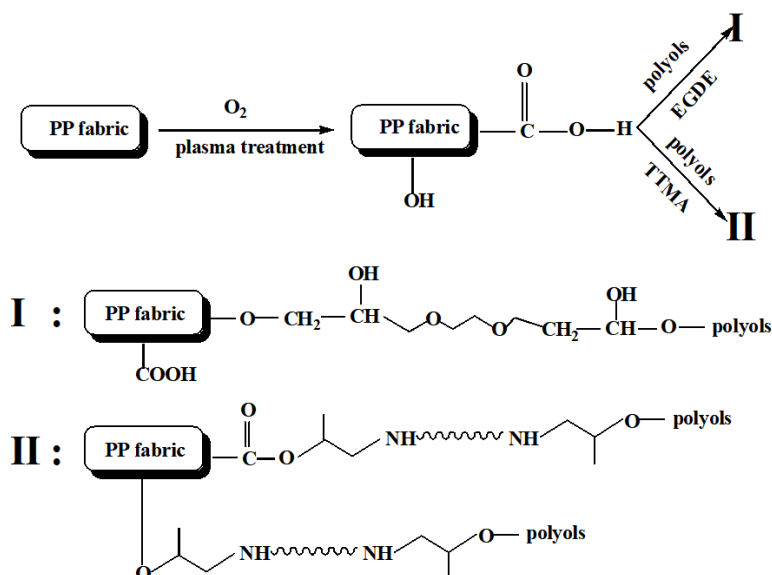


Fig. 2. Flow chart of plasma treatment and polyol modification

fiber and the dyeing property of textile. Depending on the type of dopant used, two main classes of treatments can be distinguished: grafting and coating. This paper mainly adopted the grafting way to activate and etch the sample surface [45].

PP fabric (4cm×8cm) was treated on glow-discharge plasma apparatus (Omega DT-03), which was supplied by Suzhou Optics Plasma Co., Ltd (China). Oxygen was used in the plasma pretreatment. The electrode distance was 4.5cm. The entry flow (200sccm, 250sccm, 300sccm), discharge power (50W, 100W, 150W, 200W, 250W, 300W), and treatment time (90s, 120s, 150s, 180s, 210s, 240s) were set as variables for the experiment.

2.2.2. Polyols immobilisation onto pretreated PP fabric

Three 5wt% polyol solutions (sorbitol, maltitol, PEG-800) were individually prepared by dissolving the crosslinker in distilled water (100mL). The pH value was adjusted to 7-8 for the EGDE solution, and the pH value was 6-7 for the TTMA solution. Then the plasma-treated PP fabric was dipped into the polyol solution for a grafting effect. It was a single factor variable experiment, whose variables were the amount of crosslinkers (2%, 4%, 6%, 8%, 10%) (o.w.f), temperature (AMB, 50°C, 80°C), and time (8h, 12h, 16h, 20h, 24h). After the experiment, the samples were heated

at 120°C for 3min, then rinsed with water, and finally dried again.

2.2.3. Fourier transform infrared spectroscopy analysis

The surface chemical composition of the samples was determined using a Fourier transform infrared spectrometer (IR affinity, Shimadzu, Japan). The KBr tablet method was used for sample preparation. The samples were cut up and ground in a mortar with KBr, then placed in a press for tablet pressing. The samples were then put onto the spectrometer for testing. The scanning range was 500 ~ 4000cm⁻¹, the number of scans 200, and the resolution was 4cm⁻¹.

2.2.4. Coloring analysis

Reactive dyes (reactive yellow KE-4R, reactive scarlet B-3G, reactive deep blue K-R) were used to dye the PP fabrics at a

bath ratio of 1:50. The PP fabric was dyed at 60°C for 30 minutes, and NaCl and Na₂CO₃ were added. After that, the PP fabric was dyed at 90°C for 30 minutes, then washed and dried. The dye-uptake was calculated by *Equation 1*, where A₁, A₂ are the absorbances of the dye solution before and after dyeing, N₁, N₂ are the dilution factors of the dye solution before and after dyeing. The absorbance was tested with an ultraviolet spectrophotometer of UV-1750 (Shimadzu, Japan). The colour depth was evaluated according to the K/S value, and the value was tested with a computerised colour matching instrument (CM-3600A, MINOLTA, Japan).

$$\text{Dye}_{\text{uptake}} = \left(1 - \frac{A_2 N_2}{A_1 N_1}\right) \times 100\% \quad (1)$$

2.2.5. Grafting rate analysis

The grafting rate was used to analyse the immobilisation results of the polyols. *Equation 2* is the calculation formula of the grafting rate, where G is the grafting rate; M₀ is the weight of PP after plasma treatment, and M₁ is the weight of PP after graft modification.

$$G = \frac{M_1 - M_0}{M_0} \times 100\% \quad (2)$$

2.2.6. Scanning electron microscope analysis

An S-3400N scanning electron microscope was used to observe the surface morphology changes of the original sample, the sample after plasma treatment, and the sample after modification. Before the SEM observation, the samples were coated with a carbon film, and then with a gold film (5nm) by ion sputtering. The voltage was 20kV, the working distance 11.8 mm, and the magnification was 2000x.

2.2.7. Loose fiber suspension analysis

Loose fiber was suspended in H₂O to test the hydrophilicity of PP fabrics. First of all, the test sample is separated into fibers and marked. After that, the fiber was put in a beaker with 100mL of water for 3 min, and then the fiber settling velocity was observed and recorded. To avoid contingency, each group was tested three times.

2.2.8. Moisture retention measurement

The level of moisture release of PP fiber was used to characterise its moisture retention. The samples were firstly completely dried, and then they were placed in a humidity chamber under normal conditions (25 °C, 65% RH) and high humidity conditions (30 °C, 95% RH) successively for 80min. Lastly, the samples treated with high humidity were taken out and put into normal conditions (25 °C, 65% RH) again for 160 min. The sample was weighed for a random period of time. Moisture regain is defined by *Equation 3*, in which W_m is the weight (g) of the moisturised sample, and W_d is the weight (g) of the sample dried in an oven (100°C, 2 h):

$$\text{Moisture}_{\text{regain}}(\%) = \frac{W_m - W_d}{W_d} \times 100 \quad (3)$$

2.2.9. Antistatic performance measurement

The antistatic performance of the modified PP was analysed according to the half-life value. The samples (6cm×8cm) were treated by a fabric static tester (YG (L) 342D, Shandong Textile Research Institute (China)). The test conditions were 20 °C, and the relative humidity was 35%. The sample was placed in a high voltage electrostatic field when the charge was stable, and then the voltage was naturally attenuated through a grounding metal station. When the voltage value had decayed to the half, the corresponding time was recorded.

3. Results and discussion

3.1. PP fabric after plasma treatment

The more O-containing groups produced on the PP surface after O₂ plasma pre-treatment [23, 46], the more favorable polyol post-grafting is. Hence, it is necessary to optimise the plasma treatment of PP fiber.

3.1.1. FTIR spectra of PP fabric after plasma treatment

The original PP lacks polar functional groups, but the chemical composition of its surface will change after plasma treatment. *Figure 3* shows the infrared spectra of PP fiber before and after O₂ plasma treatment. Obviously, the characteristic peak of -OH and -C=O groups at 3340 cm⁻¹ and 1716 cm⁻¹ appeared successively in the case of plasma (O₂)-PP. This shows that the PP surface is oxidised after oxygen plasma treatment. This is consistent with the emergence of new groups after the oxygen plasma treatment of paper [47]. Therefore, the amount of -COOH and -OH can be used to evaluate the effect of PP fiber treatment with O₂ plasma.

Table 1 shows absorption values of the characteristic peak in single variable plasma experiment. It can be seen that the absorption value of hydroxyl and

Factors	1	2	3	4	5	6	7	8	9	10	11	12	13
Power (W)	50	100	150	200	250	300	250	250	250	250	250	250	250
Time (s)	90	90	90	90	90	90	120	150	180	210	240	180	180
Flow (sccm)	300	300	300	300	300	300	300	300	300	300	300	250	200
O-H (3340 cm ⁻¹)	0.10	0.10	0.10	0.11	0.20	0.14	0.26	0.32	0.42	0.28	0.22	0.23	0.15
C=O (1716 cm ⁻¹)	0.13	0.13	0.13	0.18	0.28	0.22	0.37	0.41	0.50	0.39	0.24	0.31	0.19
Dye uptake (%)	12	15	18	25	30	28	32	33	37	29	27	26	21
K/S value	0.24	0.37	0.51	0.68	0.65	0.72	0.84	0.89	1.03	0.88	0.77	0.63	0.49

Table 1. Absorption intensity of the characteristic peak and colouring performance of PP fabric after plasma treatment

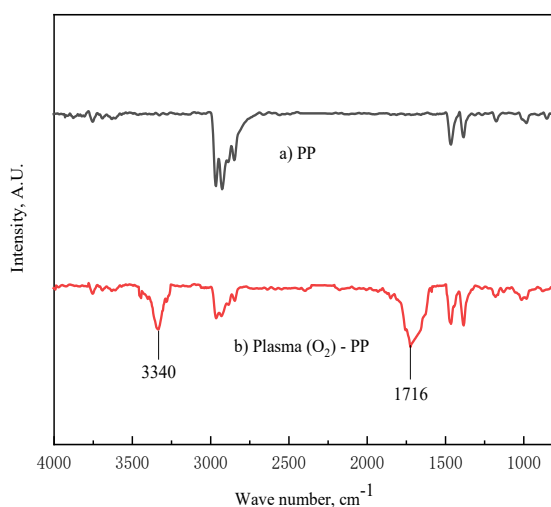


Fig. 3. Infrared spectra of PP before and after O₂ plasma

carbonyl groups reaches the maximum with the treatment condition of 250W, 180s and 300sccm. The value does not rise with an increase in power to 300W or discharge time to 240s. It may be that the newly formed functional groups were further disrupted under high-power processing for a long time. Therefore, 250W, 180s and 300sccm are considered as the optimal process of O₂ plasma treatment. The PP obtained by the optimal plasma treatment were used to graft polyols.

3.1.2. Coloring performance of PP fabric after plasma treatment

The effect of O₂ plasma treatment on PP fiber can also be evaluated by a colouring experiment with reactive dyes because the reactive groups of dyes react with the O-containing groups (-OH or -COOH) of plasma (O₂)-PP fibers in the form of

covalent bonds. **Figure 4** shows photos of the original sample and PP fabric dyed with three dyes after O₂ plasma optimal treatment. Evidently, the effect of three dyes on plasma treated fabric is much better than that of untreated fabric. This indicated that PP fabric treated with O₂ plasma has a good dye permeability and bonding force. The colouring performance of reactive yellow dye is the best. Thus, reactive yellow dye is used to dye the PP after plasma treatment. The colouring performance of plasma treated PP was analysed by testing its K/S value and dye uptake, the results of which are shown in **Table 1**. It can be seen that the dyeing results were the best when the plasma treatment conditions were 250W, 180S and 300sccm. This is consistent with the conclusion of IR characteristic peaks, indicating that the more the amount of -COOH and -OH on the surface of PP, the better the colouration effect of PP will be.

3.2. Polyol grafting onto plasma (O₂)-PP fabric

The single factor variable experiment was used to analyse optimal polyol grafting onto plasma (O₂)-PP fabric. **Figure 5** shows the polyol grafting rate after different processing.

3.2.1. Influence of temperature on polyol grafting rate

To investigate the grafting effect at low temperature and to prevent the self-polymerisation of polyols at high temperature, 80°C was set as the upper limit of grafting. The effect of the reaction temperature on the grafting rate was investigated under the condition of 4% crosslinker for 12h. Clearly, the grafting rate increased with the increase of temperature (**Figure 5a**), which is consistent with the result of other papers [42, 44]. It indicates that high temperature is conducive to the reaction of hydroxyl with epoxy and aziridine groups. The grafting rate of TTMA at 80°C was similar to that at 50°C, therefore 50°C was selected as the reaction temperature for TTMA. The grafting rate of EGDE increased with the increase of temperature, hence 80°C was selected as the reaction temperature for EGDE.

3.2.2. Influence of reaction time on polyol grafting rate

Under conditions of 80°C, 4% EGDE crosslinker usage, and 50°C, 4% crosslinker usage, the influence of time

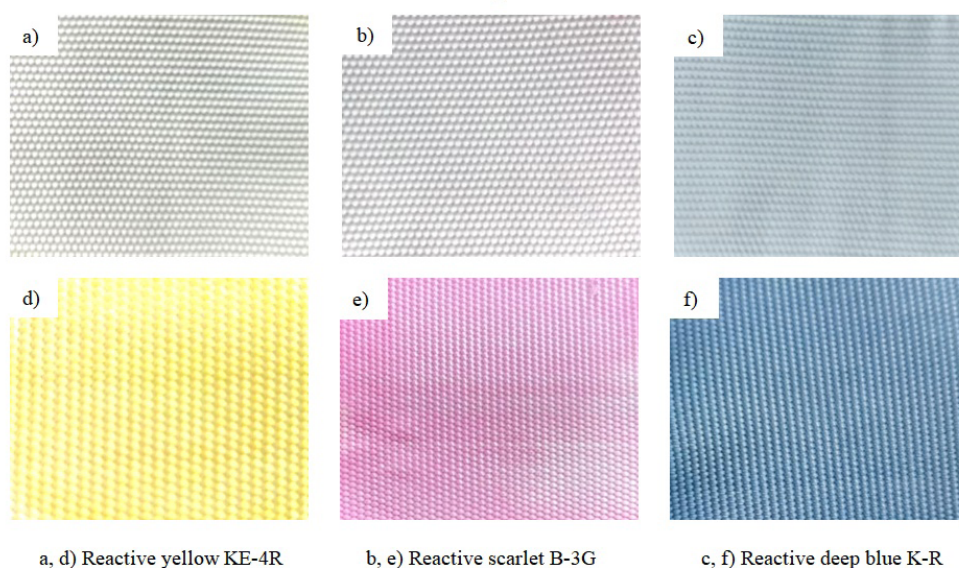


Fig. 4. Colouring photos of PP fabric before and after O_2 plasma treatment: a-c) original PP fabric, d-f) plasma (O_2)-PP fabric

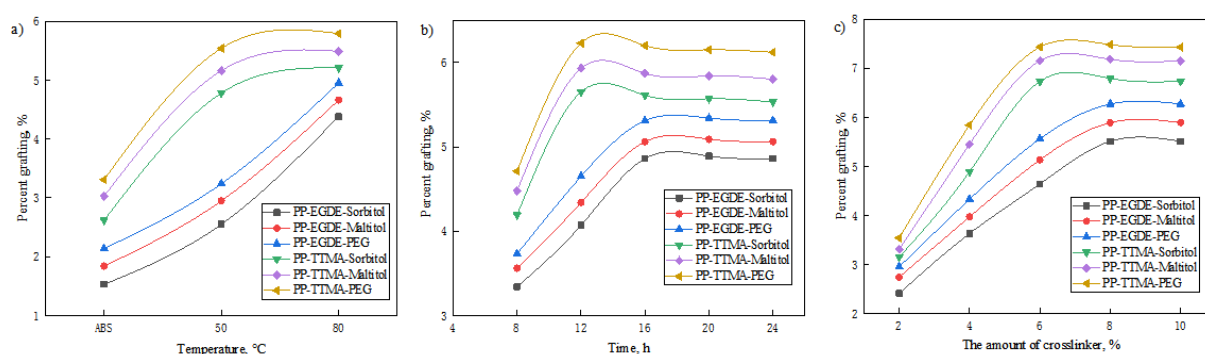


Fig. 5. Influence of different factor levels on the grafting rate: a) the influence of temperature on the grafting rate (12h, 4% crosslinker), b) the influence of time on the grafting rate (EGDE: 80°C, 4% crosslinker. TTMA: 50°C, 4% crosslinker.), and c) the influence of the amount of crosslinker (EGDE: 80°C, 16h. TTMA: 50°C, 12h.)

on the grafting rate is investigated. The corresponding result is presented in **Figure 5b**. The grafting rate of EGDE increased with the increase of reaction time, but did not increase after 16h. TTMA reached the maximum grafting rate when the reaction time was 12h, and then the grafting rate decreased, which was because TTMA self-polymerised with the increase of the reaction time. Therefore, 16h and 12h were selected as the reaction time for EGDE and TTMA. The less equilibrium time for TTMA may be due to the reaction of -COOH on the plasma (O_2)-PP surface with aziridine crosslinker being much faster than that of -OH on the plasma (O_2)-PP surface with the EGDE crosslinker at low temperature.

3.2.3. Influence of crosslinker dosage on polyols grafting rate

Polyols grafting experiments were carried out with various amounts of the crosslinker under conditions of 16h at 80°C for EGDE, and 12h at 50°C for TTMA. **Figure 5c** shows the results of the crosslinker amount with respect to the grafting rate. It can be seen that the grafting rate presents an increasing trend with the increase of the amount of crosslinker until 8% EGDE and 6% TTMA, respectively. Then, with the increase of the amount of crosslinker, the grafting rate did not change much. Therefore, a crosslinker dosage of 8% EGDE and 6% TTMA were separately selected. Comparatively, the polyol (no matter if it is sorbitol, maltitol, or

PEG) grafting rate using EGDE as the crosslinker is less than for TTMA. This is because TTMA is more active and easier to react with carboxyl and hydroxyl groups. In addition, it was found that the grafting rate of three polyols followed the same order of PEG>maltitol>sorbitol, no matter whether it was EGDE crosslinker or TTMA crosslinker. This sequence may be caused by the different molecular weight of polyols, as the larger the molecular weight, the higher the grafting rate.

3.3. SEM analysis

The surface morphology of PP fabric will change after plasma treatment and grafting modification; thus, the surface

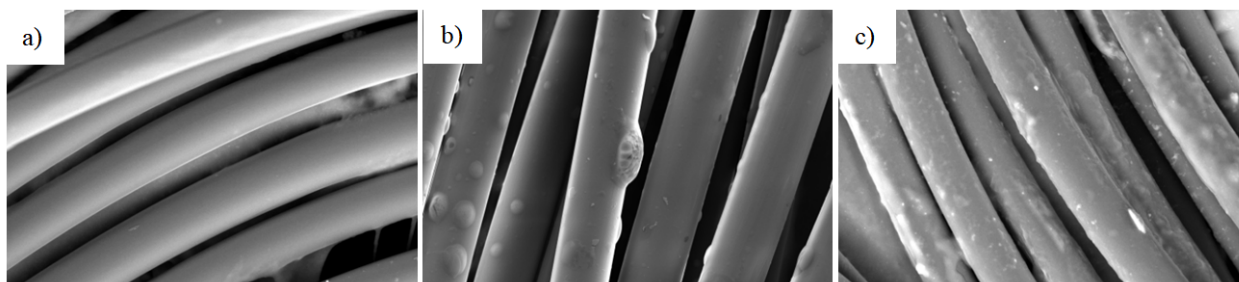


Fig. 6. SEM images of the three finishing stages of PP fabric: a) untreated PP, b) PP treated by oxygen plasma, and c) PP grafted with PEG

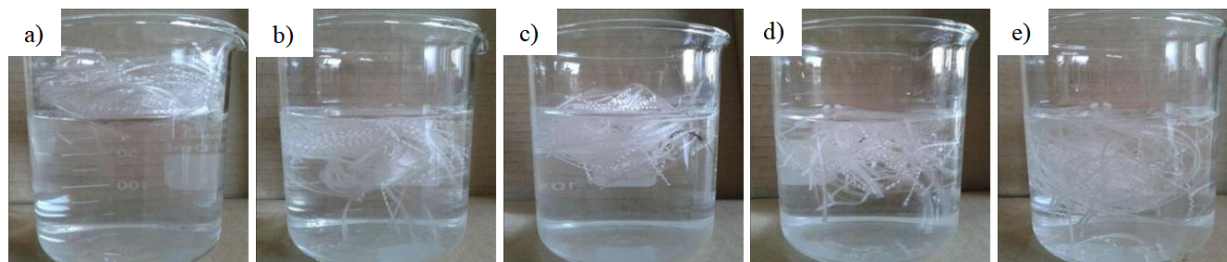


Fig. 7. Loose fiber suspension picture with different finishing methods: a) untreated PP, b) PP treated by oxygen plasma, c) PP grafted with sorbitol, d) PP grafted with maltitol, and e) PP grafted with PEG.

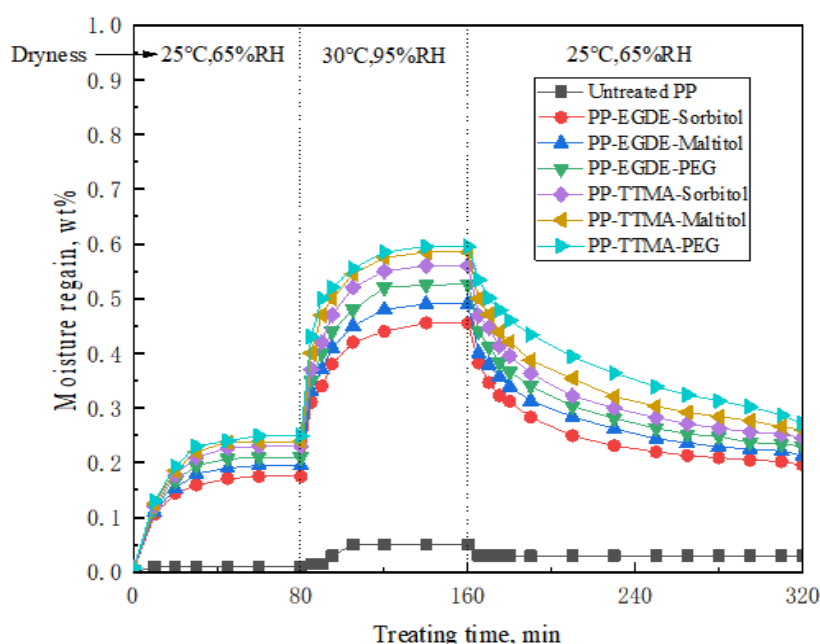


Fig. 8. Changes in moisture absorption and desorption of untreated PP and grafted PP

morphology of PP fabric was observed in three stages by scanning electron microscope. **Figure 6** shows SEM images of the three finishing stages of PP fabric. It is clearly seen that every single fiber of untreated PP has a smooth surface. However, there are bubbles on the surface of PP after plasma treatment, reason for which is that PP fabric is activated to produce new groups after

oxygen plasma treatment. Some of the grooves are attributed to plasma etching. Compared to each other, the surface of PEG-PP fiber is much rougher, and seems to be covered with a membrane. This indicates that more PEG was grafted onto the PP surface by the crosslinker. In another paper, the same changes are reported after plasma treatment and graft modification [48].

3.4. Hydrophilicity of PP fiber

The hydrophilicity change of the PP fibers was investigated by a water immersion experiment of loose fibers. **Figure 7** shows the loose fiber suspensions prepared with different finishing methods. It was found that the original PP fiber was still not immersed in water even after a long period of storage, while the plasma (O_2)-PP and Polyols-P (O_2)-PP fibers could completely immerse in water. That suggests PP hydrophilicity is greatly improved after oxygen plasma treatment and subsequent polyol grafting. This is attributed to numerous O-containing groups produced by the O_2 plasma treatment of the fibers and the large number of hydroxyl groups in the grafted polyols. It can also be seen that the immersion state of PEG-PP fiber is better than for the other two, which is due to its higher grafting rate.

3.5. Moisture retention of PP fabric

Moisture retention of fibers determines comfort in application. **Figure 8** shows the changes in moisture absorption and desorption of untreated PP and polyols-grafted PP. Except for untreated PP,

Samples	Electrostatic pressure peak value [V]	Half-life period [s]
Untreated	1102	>99
PP-EGDE-Sorbitol	507	25
PP-EGDE-Maltitol	485	22
PP-EGDE-PEG	463	17
PP-TTMA-Sorbitol	405	14
PP-TTMA-Maltitol	389	13
PP-TTMA-PEG	342	9

Table 2. Electrostatic pressure peak value and half-life period of PP fabric with different finishing methods

hygroscopic equilibrium was reached after 60 min at (25 °C, 65% RH) or (30°C, 95% RH), while the desorption behavior went on for close to 2h, especially for PEG-PP fiber. It is quite obvious that the velocity of desorption is much slower than that of the hygroscopic behaviour [49]. Furthermore, the moisture retention of these PP samples maintained the same order as for the water immerse ability, namely PEG>maltitol>sorbitol. This result indicate that polyols-PP possesses different degrees of moisture retention.

3.6. Antistatic performance of PP fabric

The antistatic property of PP fabric was tested by the half-life method. **Table 2** shows the electrostatic pressure peak value and half-life period of the original PP and polyols-PP fabric.

The half-life period can represent the time of charge loss; hence, the shorter the half-life, the better the antistatic performance [50]. The untreated PP fabric is not antistatic according to the data. After polyol grafting, the electrostatic voltage and half-life of PP decreased sharply, indicating the finished fabric possessed antistatic ability, among which PEG-PP fabric has the best. Besides this, the polyols-PP fabric obtained with a TTMA crosslinker had stronger antistatic ability than that with EGDE crosslinker, as a result of the higher grafting ratio.

4. Conclusions

Polypropylene fabric was modified with plasma pretreatment and polyol post-grafting. The modified PP fabric has hydrophilic, colouring and antistatic properties. Some conclusions can be drawn based on the results:

1. The optimal process (250W, 180s and 300sccm) of O₂ plasma treatment on PP fabric was obtained by evaluation of the IR characteristic peak and dyeing effect of experimental samples.
2. With EGDE or TTMA crosslinking, three polyols could be successfully grafted onto plasma (O₂)-treated PP fiber, but the effect using TTMA as a crosslinker was better than for EGDE. The grafting ratio order of PEG>maltitol>sorbitol was maintained, regardless which crosslinker was used. The optimal grafting process, 16h at 80°C for 8% EGDE and 12h at 50°C for 6% TTMA were obtained respectively.
3. After polyol grafting, the PP fabric possessed distinct water immersion, moisture retention and antistatic ability.

The above shows that the experiment of grafting polyols onto PP fabric after oxygen plasma treatment is feasible, which can improve the hydrophilic and antistatic properties of PP fabric.

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