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Application of Microwave Plasma Pre-treatment in Enhancement of the Desizing Efficiency of WSP-Sized Woven PET

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This paper aims to replace the traditional alkali desizing of PET fabrics with the combination of microwave plasma treatment and water desizing. To this end, the microwave plasma pre-treatment was applied to enahnce the desizing efficiency of PET fabrics in pure water. With a 2.45GHz plasma source and a power of 450W, the 80s-long treatment was found to significantly increase the desizing rate of WSP in water due to the etching effect of microwave plasma. The SEM observation disclosed the etching effect of WSP on PET fabrics induced by plasma, and verified the complete removal of WSP on PET fabrics through plasma pre-treatment and water desizing. The test on the water contact angle revealed the contribution of plasma treatment to the improved wetting property of the sized PET fabrics. This study opens up a new way for desizing of PET fabrics that reduces wastewater discharge and the consumption of chemicals, energy and time.

1. Introduction

Polyethylene terephthalate (PET) fabrics are one of the most popular textile materials. The production of woven PET fabrics hinges on the sizing of the warp threads. In recent years, the traditional sizing agent polyvinyl alcohol (PVA) is gradually being replaced by some novel and eco-friendly sizing agents. For example, the biodegradable and water-soluble polyester (WSP) sizing agent has been widely applied in the manufacturing of tight PET fabrics (Jin et al., 2010; Zhu et al., 2009). The WSP size contains hydrophilic groups (e.g. carboxyl group and sulfonic group), and exhibits excellent sizing effect on the warp threads of PET fabrics. However, the strong adhesion forces between WSP and PET fibres brings numerous difficulties to the desizing process, as the residual sizing agents can exert a negative impact on the subsequent processes (e.g. dyeing and printing), and the wearability of end products. Traditionally, an alkali desizing process was employed to remove all WSP sizing agents. The problem is the traditional measure consumes large amounts of chemicals, energy and water. This calls for plasma pre-treatment before the desizing process (Zille et al., 2015). Previous studies have shown that plasma pre-treatment works well in removing PVA and polyacrylic acid sizing agents from cotton and polyester fabrics (Cai et al., 2003; Cai et al., 2002; Bae et al., 2006; Li et al., 2013). Nevertheless, most of the scholars merely tackled the effect of temperature and atmospheric-pressure plasma treatment on the desizing of modified starch, PVA and polyacrylate (Peng et al., 2009; Peng et al., 2010; Peng et al, 2010; Li et al., 2012; Li and Qiu, 2012; Cui et al., 2011), while only a few reported on the influence of plasma pre-treatment on the desizing of WSP.
Plasma treatment, which leads to the generation of active ions and high energy electrons, can modify fibre surfaces by means of etching, implantation reaction and polymerization. The common plasma generation approaches include DC discharge, AC discharge, laser discharge, RF discharge and microwave discharge. Among them, microwave discharge is the most promising way to process textile materials, because it can generate stable and high-density plasma at near-ambient temperature and produce less ozone and noise pollution than other methods. Focusing on the PET fabric with warp threads coated by WSP, this paper treats the fabric with microwave plasma, aiming to improve the desizing efficiency of WSP in water; then, the author

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discussed the effects of plasma treatment time on the desizing rate of WSP in water as well as the tensile strength, surface morphology and wetting property of PET fabric. The combination of microwave plasma treatment and water desizing was hoped to replace the traditional alkali desizing with the aim to reduce wastewater discharge and the consumption of chemicals, energy and time.

2. Experiment

2.1 Chemicals

The PET plain fabrics were purchased from Jiangsu Shenghong Chemical Fibre Co. Ltd., China. The specifications are as follows: warp count, 75D/36F; warp density, 1,200 threads/10cm; weft count, 100D/36F; weft density, 1,300 threads/10cm. The dry weight of the fabric sized with WSP was 0.8g/cm^2. The NaOH was purchased from Shanghai Aibi Chemistry Preparation Co. Ltd., China. The glacial acetic acid was bought from Nanjing Chemical Reagent Co. Ltd., China. The penetrating agent JFC was provided by Jiangsu Haian Petroleum Chemical Factory, China. The deionized water was used for all experiments.

2.2 Microwave plasma pre-treatment

The PET fabrics were firstly treated by microwave plasma. Figure 1(a) details the microwave plasma treatment process. Considering the cost and time of potential industrialization, the plasma source was set with a stable frequency of 2.45GHz, and the power was set to 450W with the air as the working gas. Before the plasma treatment, the chamber was depressurized to 100Pa, and purged with a small amount of working gas for three times. After the purging, the intake valve was adjusted to create a stable and desirable pressure. Then, the fabrics were processed at the set power for the pre-set period of time. The microwave plasma apparatus YZ-150 from Anhui Unileading Materials Technologies Co. Ltd., China is shown in Figure 1(b).

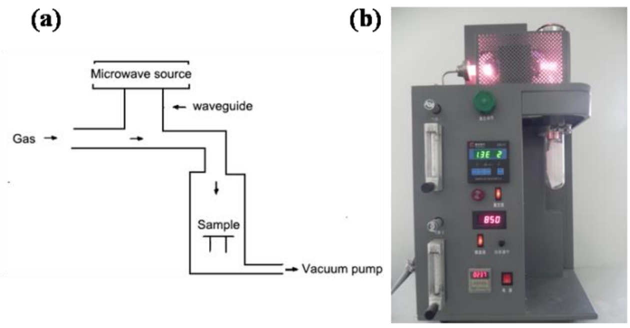


Figure 1: The microwave plasma treatment (a) and the microwave plasma apparatus (b).

2.3 Desizing

Desizing in water: The 30cm-long, 5cm-wide fabrics with/without microwave plasma pre-treatment were put in water at 80oC for 6min-long desizing. The liquor ratio (i.e. the liquor volume to fabric weight) was 100:1. The temperature of the water bath was controlled by a SHA-C constant temperature oscillator (Hui Electronics Co. Ltd., China).

Desizing in alkali: The fabrics were desized in NaOH (5g/L) solution with the liquor ratio of 100:1 at 40oC for 6min.

2.4 Measurements

(1) Weight loss

The samples were weighted just before and right after microwave plasma pre-treatment. During the pre-treatment, the weight loss mainly occurred due to the etching effect. The weight difference between treated and untreated fabrics was taken as the weight loss.

To measure the weight of the desized fabrics, the treated and untreated samples were placed in a standard environment (temperature: 20±5oC; relative humidity: 60%) for 24h, and then weighted. The desizing rate or weight loss was calculated based on the weight difference between treated and untreated samples. Each test was repeated 5 times to obtain the average weight loss.

(2) Tensile strength

The tensile strength of PET fabrics was measured by a YG(B) 02617D-250 electronic fabric strength testing machine (Wuhan Guoliang instrument Co. Ltd., China). Three parallel tests were performed to obtain the average tensile strength.

(3) Wetting property

The samples were fixed in holders and placed on the bench of a JC2000D2 contact angle meter (Shanghai Digital Technology Co. Ltd., China). Then, 30µL of distilled water was put on the top of the samples. The dynamic wetting process was recorded, and the dynamic water contact angle was measured second by second.

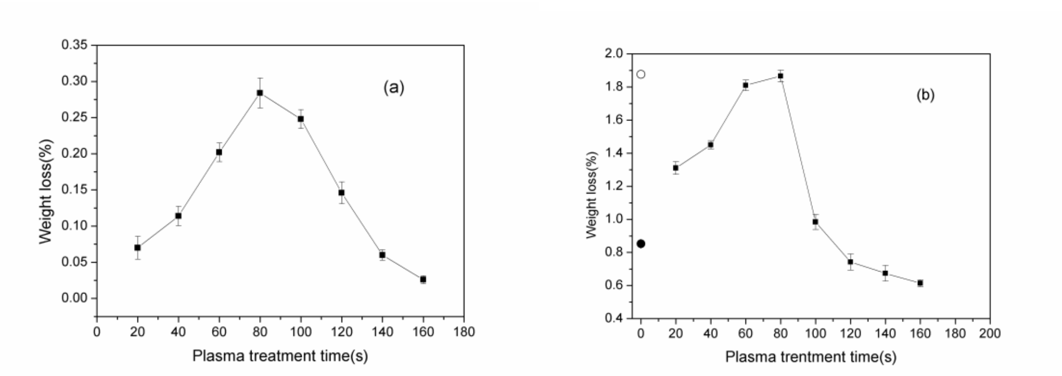
(4) Surface morphology

The original fabrics and sample pre-treated for 80s were chosen for the surface observation by a field emission scanning electron microscope (SEM) (FEI, US). The images were taken at an acceleration voltage of 10.0kV and a magnification of 250 to 5,000 times. Prior to SEM observation, all the samples were coated with gold in a vacuum environment.

3. Results and Discussion

3.1 Weight loss

As shown in Figure 2(a), the weight loss of pre-treated fabrics increased linearly as the treatment time increased up to 80s. However, further increase in time led to a decline in the weight loss. During plasma pre-treatment, the fabrics were under the joint action of etching and implantation reaction. At the beginning (<80s), the etching effect had the upper hand. In this stage, it was easy for WSP molecules in the amorphous region to react with the air plasma, and produce small molecules that could be pumped away. With the decrease in the amorphous content, the extent of crystallinity was automatically increased. Then, the weight loss reached the saturation point under the high order and high bond energy in the crystalline (Matthews et al., 2004; Lazea et al., 2005; Matthews et al., 2005). Owing to the implantation of oxygen- and nitrogen- functional groups on the etched surface, the implantation reaction surpassed the etching effect, resulting in the decrease of weight loss.



(a) microwave plasma treatment only *(b) water desizing after microwave plasma pre-treatment, pure water desizing (●) and alkali desizing (○).*

Figure 2: Weight loss of the fabrics subjected to different treatments

In this study, the sizing rate of warp threads was 2%. The traditional one-step alkali desizing worked well and removed almost all the sizing agents. As shown in Figure 2(b), the weight loss of the alkali desized fabrics reached 1.85%, indicating that 95% of the sizing agents were removed. For pure water desized fabrics, the weight loss only stood at 0.86%. For the fabrics subjected to plasma pre-treatment and water desizing, the weight loss increased with the time of plasma treatment up to 80s. At the treatment time of 80s, the plasma pre-treated sample had a weight loss (1.9%) almost comparable to that (1.85%) of alkali desized sample. This means the etching effect of plasma pre-treatment can markedly enhance the removal of sizing agents, and the alkali desizing can be replaced by the combination of plasma treatment and water desizing. However, it should be noted that the weight loss in water desized sample declined with any further increase in the time of plasma pre-treatment. The trend agrees well with that in Figure 2(a). It might be attributed to the complex chemical reactions between the reactive ions and electrons in air plasma induced by the implantation reaction.

3.2 Surface morphology

Figure 3(a) illustrates the surface morphology of sized fabrics. With the accumulation of particles, the fibres were covered uniformly and smoothly by sizing agents. After plasma pre-treatment, the coatings on the fibre surface and between fibres carried etching marks and loopholes (Figures 3 (b) and (c)), and clear micro-voids were observed on the sizing agents attached to the fibre surface (at the arrowhead in Figure 3(d)). The morphological changes can be explained by the following reasons: the etching effect on sizing agents; the decomposition of sizing agents; the removal of pieces of small degraded sizing agents from the surface. Interestingly, the fibre itself was not influenced by plasma etching due to the protection effect of sizing agents. After plasma pre-treatment and water desizing, the fibre became smooth and clean on the surface (Figure 3(e)), a sign of the complete removal of the sizing agents covering fibres.

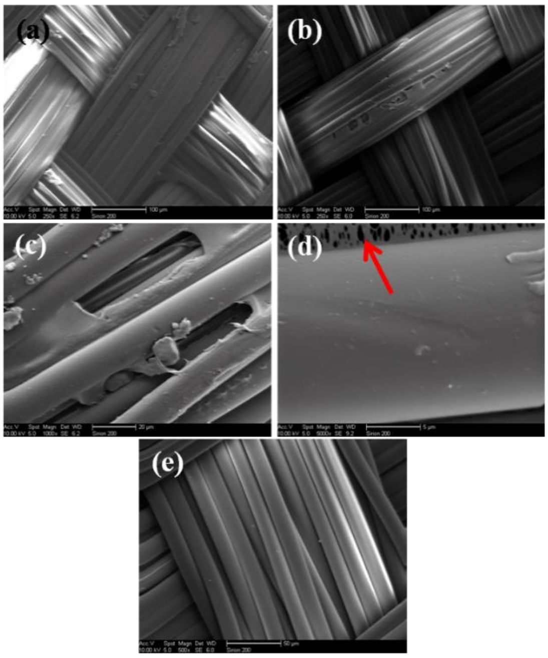


Figure 3: SEM images of the fabrics after different treatments: (a) original, alkali-desized fabrics; (b), (c) and (d) fabrics treated by microwave plasma; (e) fabrics treated by microwave plasma and desized by water

3.3 Tensile strength

The tensile strength of the fabrics subjected to combined plasma treatment and water desizing is shown in Table 1. Compared with the alkali-desized fabrics (tensile strength: 687N), the samples treated by microwave plasma and desized by water showed almost no change in tensile strength. Hence, the new desizing approach does not affect the mechanical performance or wearability of the fabric.

Table 1: Tensile strength of the fabrics after microwave plasma treatment for different durations and water desizing

Treat duration (s)	*60*	*80*	*100*	*120*	*140*	*160*	*200*	*300*	*400*	*500*	*600*
Tensile strength (N)	687	679	692	690	691	697	691	687	682	678	677

3.4 Wetting property

A water contact angle test was carried out to evaluate the surface wettability of the fabrics undergoing different treatments. The dynamic contact angle was captured for every second in a high speed video system (Figure 4).

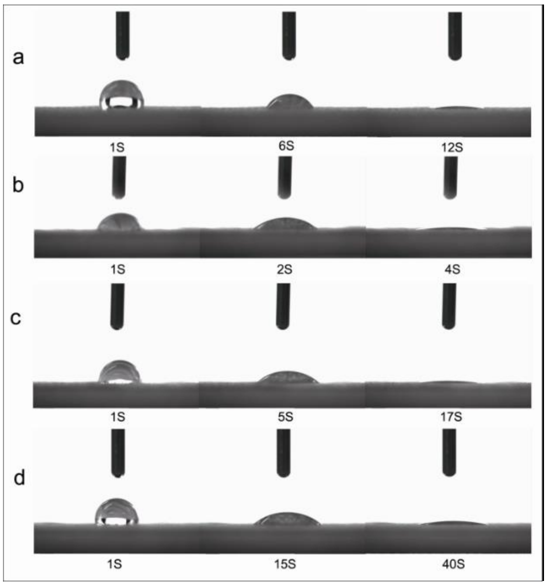


Figure 4: Water contact angle of the fabrics undergoing different treatments: (a) original fabric; (b) microwave plasma treatment for 80s; (c) microwave plasma treatment for 80s+water desizing; (d) alkali desizing

According to Figure 4(a), the original, alkali-sized fabrics exhibited good hydrophilicity, and its complete wetting time was about 13s. The results reflect the presence of hydrophilic groups (e.g. carboxyl group and sulfonic group) in the WSP. However, the wetting time shortened greatly after the fabrics received the microwave plasma treatment, and the complete wetting time fell to only 4s (Figure 4(b)), revealing a huge improvement of surface hydrophilicity caused by the etching effect of plasma treatment. The enhanced hydrophilicity can increase the water solubility and dispersion ability of sizing agents, and accelerate the removal of sizing agents.

After plasma treatment and water desizing, the fabrics consumed a longer time (17s) to reach complete wetting (Figure 3(c)) than those only treated by plasma. This is because hydrophilic slurry on the fabric surface had been removed. In this case, the desized fabric displayed the hydrophobic property of polyester fibres. The complete wetting time of the alkali-desized fabric was 40s (Figure 4(d)). The comparison between Figures 4(c) and 4(d) shows the hygroscopicity enhancement of the fabrics by plasma treatment. This might be related to the slightly increase in oxygen of the plasma-treated and desized fibres, despite the absence of the fibre etching in Figure 3(e).

4. Conclusions

This paper explores the effect of microwave plasma pre-treatment on PET fabric sized with WSP. The complete removal of WSP on PET fabrics was verified by SEM observation, and etching effect of plasma on the sizing agents on the surface of PET fabric was revealed by the SEM observation and wetting time test. It is concluded that fabrics, after 180s of plasma treatment and 6min of 80oC water desizing, had almost the same weight loss and tensile strength with the alkali-desized fabrics. This research demonstrates that the traditional alkali desizing can be replaced by the combination of microwave plasma treatment and water desizing.

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