



## Aminolysis of polyethylene terephthalate fabric by a method involving the gradual concentration of dilute ethylenediamine



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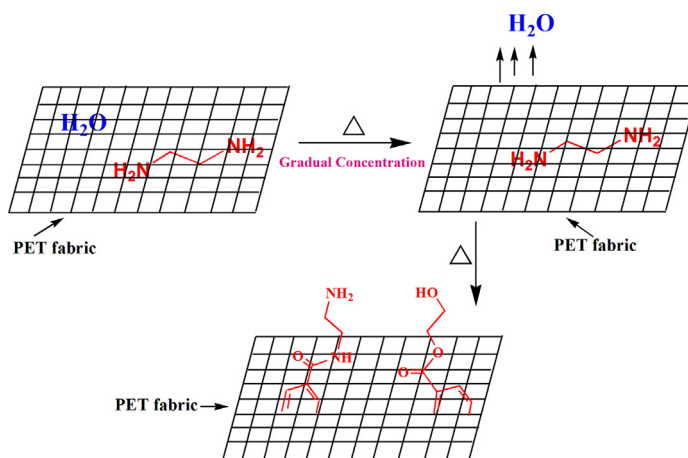
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### HIGHLIGHTS

- A hydrophilic PET fabric was prepared by a facile aminolysis method about the gradual concentration of ethylenediamine.
- Dilute ethylenediamine gradually became concentrated enough to aminate the PET fabric during the curing process.
- The reactive  $\text{-NH}_2$  groups were introduced on PET fibers by dilute ethylenediamine gradually concentrating aminolysis method.
- The hydrophilicity of aminated PET fabric was improved greatly.
- The mechanical properties of aminated PET fabric were kept well.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Aminolysis of polyethylene terephthalate (PET) fibers can endow fibers high hydrophilicity and reactive  $\text{-NH}_2$  groups; however, the ethylenediamine solution aminolysis method severely damages the PET fibers. In this paper, a facile aminolysis method involving the gradual concentration of dilute ethylenediamine was developed to aminate PET fibers. The results showed that the hydrophilicity of the PET fabric substantially improved and that reactive  $\text{-NH}_2$  groups were introduced onto the PET fibers. The water absorption time and contact angle of the treated PET fabrics decreased to 15 s and  $0^\circ$ , respectively. The liquid wicking height of the treated PET fabric increased from 0.4 cm (original PET fabric) to 11.0 cm. Fourier transform infrared spectra demonstrated that  $\text{-NH}_2$  groups were successfully introduced onto the PET fibers. Scanning electron microscopy showed almost no difference between the surfaces of the original and treated PET fibers. X-ray diffraction analysis indicated that the main structure of the PET fibers was well maintained. The aminated PET fabrics retained the weight loss, breaking strength and

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elongation, crease recovery angle, stiffness and whiteness of the original PET fabrics. The aminolysis of PET fibers via the gradual concentration of dilute ethylenediamine is a promising method for the efficient, rapid, and eco-friendly modification of PET fibers.

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## 1. Introduction

Polyethylene terephthalate (PET) is the most popular synthetic fiber in the textile industry [1–3] because of its many excellent properties [4], which include good wash and wear, excellent anti-wrinkle performance, and outstanding mechanical properties. However, PET fabric exhibits poor hydrophilicity and PET macromolecules contain no reactive groups. Its low moisture regain (0.42%) [5] and hydrophobicity cause a series of adverse effects with respect to consumer use. PET fabrics cannot be worn next to the skin because of their tendency to accumulate static electricity and their poor wet absorption property. Thus, surface hydrophilic modification of PET fabric is critical for its wider application in close-fitting clothing and biomedicine.

In recent years, numerous technologies (alkali treatment, plasma treatment, aminolysis, and surface grafting) have been applied to improve the hydrophilicity of PET fabrics. Among these methods, the hydrophilic modification of PET fabric by sodium hydroxide [6,7] has long been used. The alkali treatment method hydrolyzes the ester bonds of the PET to produce hydroxyl and carboxyl groups, thereby increasing the wettability of PET fibers. The mechanical properties may be adversely affected if the processing parameters are not well controlled [8]. Plasma treatments have been widely studied as methods to modify the surface properties of PET fabrics, especially their wettability. The gases used for plasma treatments include  $\text{NH}_3$  and  $\text{O}_2$  [9],  $\text{H}_2\text{O}$  vapor [10], He and Ar [11],  $\text{O}_2$  and  $\text{CF}_4$  [12],  $\text{SF}_6$  [13], and air [14]. These plasma treatments can make the PET fabric surface hydrophilic by introducing highly reactive species (radicals, ions, electrons, excited molecules, photons, etc.). However, the use of plasma treatments in large-scale production is difficult.

Many surface-grafting techniques have been developed to improve the hydrophilicity of PET fabrics, including the grafting of nanocrystalline cellulose [15], chitosan [16], silk fibroin [17], wool keratin [18], and silk sericin [19]. These grafting methods can improve the hydrophilicity of PET fabrics to some extent. However, the contact angle of such treated PET fabrics cannot reach  $0^\circ$ , and the increase in stiffness because of a layer of grafting materials leads to a bad hand-feel.

The aminolysis of PET fibers with various diamines [20,21] can introduce amino groups onto PET macromolecules. This approach is effective because  $-\text{NH}_2$  groups are hydrophilic; thus, aminated PET fabric exhibits excellent hydrophilicity. The  $-\text{NH}_2$  groups are also reactive; thus, other functional compounds can be grafted onto the PET fabric to endow the PET macromolecules with various functions. However, in the reported papers, when PET fibers or films were treated in a diamine solution, their weight and tensile strength decreased substantially. Thus, the development of a new method to aminate PET fabric while maintaining its tensile strength and weight is very indispensable.

In this work, a rapid and efficient method was developed to improve the hydrophilicity of PET fabrics. The PET fabric was aminated via the gradual concentration of dilute ethylenediamine. The aminated PET fabric exhibited good hydrophilicity and bore reactive  $-\text{NH}_2$  groups, and its mechanical properties and weight were well maintained.

## 2. Experimental

### 2.1. Materials

Polyethylene terephthalate (PET) fabrics (318/10 cm  $\times$  284/10 cm, 145 g/m<sup>2</sup>) were purchased from Fengqiang Textile Company, China. Sodium dodecyl benzene sulfonate (SDBS), sulfuric acid and ethylenediamine (AR) were purchased from Chengdu Kelong Chemical Reagent Factory.

### 2.2. Hydrophilic modification of PET fabric

PET fabrics were cut into 30 cm  $\times$  20 cm pieces, cleaned with distilled water, and then dried in a drying oven at 100 °C. The fabric samples were then immersed into ethylenediamine solutions at different concentrations ranging from 0 to 200 g/L at ambient temperature for 5 min with moderate agitation. The bath ratio was 1:30. These PET fabric samples were then cured in a drying oven at different temperatures ranging from 50 °C to 90 °C for 30 min. After being cured, these PET fabric samples were washed twice with distilled water and then dried at 100 °C. The hydrophilicity of the treated PET fabric samples was subsequently analyzed, and the samples were subjected to various characterizations.

### 2.3. Weight-loss rate of treated PET fabric samples

The weight-loss rate (WLR) of treated PET fabric samples was calculated according to Eq. (1):

$$\text{WLR} = \frac{m_0 - m_1}{m_0} \times 100\%, \quad (1)$$

where  $m_0$  is the weight of the original PET fabric (g) and  $m_1$  is the weight of the treated PET fabric (g).

### 2.4. Amino value of treated PET fabric

The amount of amino groups on the aminated PET fabrics was quantitatively evaluated via the sodium dodecyl benzene sulfonate (SDBS) adsorption process. The pristine and treated PET fabrics were immersed in a solution containing 1 g/L SDBS and 0.5 mol/L  $\text{H}_2\text{SO}_4$  for 1 h at 40 °C. The bath ratio was 1:50. The wavelength of maximum absorption was 224 nm, as measured using a TU-1810 UV-vis spectrophotometer (Beijing Purkingje General Instrument Co., Ltd.). The absorption (Abs) of residual SDBS solution was measured after the SDBS adsorption process to evaluate the amine value. The amount of amino groups on 1 g aminated PET fabric was calculated according to Eq. (2) [22]:

$$\text{AMV} = \frac{A_0 - A_1}{A_0} \times \frac{CV}{Mm_1} - \frac{A_0 - A_2}{A_0} \times \frac{CV}{Mm_2}, \quad (2)$$

where AMV (mol/g) is the amino value,  $A_0$  is the Abs of SDBS solution before the adsorption process,  $A_1$  is the Abs of residual SDBS solution after the adsorption of aminated PET fabric,  $A_2$  is the Abs of residual SDBS solution after the adsorption of the original PET fabric,  $C$  is the concentration of SDBS solution used in the adsorption process,  $V$  is the volume of SDBS solution used in the adsorption

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