

## Study on Hole Filling of Porous PU/PEG Membrane and Its Mechanical Properties

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**Abstract:** This study attempted to introduce porous technology into functional textile's formation, and developed a new way to improve working materials content in phase change material. Porous polyurethane (PU)/ polyethylene glycol (PEG) membrane was prepared and selected as a supporting material. Considered to be convenient for filling, anhydrous ethanol was used to decrease PEG viscosity according to the rule of similarity. The solution of PEG and anhydrous ethanol were filled into porous PU/PEG membrane by vacuuming and then PU dry-film technology was used for the sealing of the membrane hole. The effects of PEG mass percentage in ethanol solution on filling rate and the filling rate on the membrane morphology and mechanical properties were studied. The results show that, the filling rate increased with the increase of PEG content in the blending solution. As the filling rate increased, the elongation at break decreased obviously, while the initial modulus increased largely. After sealing the filled membrane using PU dry-film technology, the initial modulus of the membrane decreased, while the elongation at break increased. In addition, DSC analysis indicates that the sealed membrane and pure PEG show similar thermal properties, while the compound membrane can retain its solid shape during phase change.

**Keywords:** Porous phase change membrane, polyurethane, polyethylene glycol, filling, mechanical properties.

### 1. Introduction

Technology development and the improvement of people's lifestyle brought forward higher demands to textile comfort performance. Self-adaptive textiles with heat storage and preservation function thus came into being and then the representative materials are phase change materials (PCMs) and corresponding techniques [1-4]. Among all the studies, new compound solid-solid PCMs are paid more and more attention [5-10]. Porous materials have light mass and big porosity. The combination of porous material and phase change material (PCM) is one method to obtain compound solid-solid PCMs. Porous composite phase change materials have been used as heat preservation materials in buildings. And relative study has been done widely at home and abroad [11-16]. The control of pore structure and PCM content are two key problems in porous composite PCM study. PCM content decides heat storage density and temperature adjusting ability of the composite material [17]. Pore structures and characters not only effect PCM content but also heat insulation [18]. For the present study, there are still problems such as low latent heat during phase change and separation of work material and supporting material [19]. And polyurethane membrane as porous skeleton in phase change composite material

is seldom reported.

In this study, porous polyurethane (PU)/polyethylene glycol (PEG) membrane was prepared and selected as supporting material and PEG was selected as filling component. For pure PEG has high viscosity hole of the PU/PEG membrane was shaped as a beehive, it was hard to fill PEG into the membrane fully if only rely on soaking and wicking. To improve the filling effect, PEG and anhydrous ethanol was blended in a certain proportion to reduce PEG viscosity based on the law of similar mutual solubility. Porous PU membrane was soaked in the blended solution and the filling of the membrane with PEG was fulfilled under a certain vacuum degree. And PU dry film was used for the sealing of the filled membrane.

### 2. Experimental section

#### 2.1 Materials and reagents

PEG2000, N, N-Dimethylformamide (DMF) and anhydrous ethanol were of chemical grade and supplied by Shanghai Sinopharm Chemical Reagent Co. Inc. Liquid PU (solid content 30wt%) was of chemical grade and purchased from Kunshan Gaoding Chemicals Industry.

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## 2.2 Preparation of porous PU/PEG membrane

The homogenous mixture solution was obtained by dissolving PU and melted PEG in DMF by vigorous stirring. When the mixture solution was cooled down to room temperature, 28.6wt%  $(\text{NH}_4)_2\text{CO}_3$  superfine powder was dispersed in DMF and the emulsion was added into the mixture and stirred vigorously for 1h to form a homogenous solution. After degassing, the resulting solution was cast onto a plane glass model using a glass bar. The cast film was placed at ambient temperature for 2min, and then immersed into water at room temperature for 3 min. And then the model was taken out of water and dried in air for about 5h. Finally the membrane was removed from the model and placed in climatic chamber at temperature  $50^\circ\text{C}$  and 70~80% R.H. for at least three hours. After steam treatment, the films were stored in a ventilated environment for at least one week before measurements.

## 2.3 Filling and sealing of the membrane

Melt PEG and anhydrous ethanol were mixed up in different mass ratio till a white transparent solution obtained. Then the porous membrane was vacuum-impregnated (vacuum degree 0.1MPa) with the blended solution. The membrane was taken out when no bubble appeared in the solution and was dried until anhydrous ethanol volatilized entirely.

14~19wt% PU (30wt%PU diluted with solvent DMF) was poured into a self-made glass plate and scrapped to film formation with a glass bar. The cast film was dried at  $25\sim60^\circ\text{C}$  for 1~10min and then covered with the filled membrane (loosen bottom down). Then the combined membrane was flattened and treated at  $20\sim30^\circ\text{C}$  for 5~30min under 60~80% relative humidity. After becoming stable, the film was peeled off and stored in sealed container for measurement.

## 2.4 Characterization

### 2.4.1 Morphology observation

Pore morphology of PU/PEG membrane and the surface of the sealed membrane were investigated using a scanning electron microscope (SEM) (HITACHI, model X-450).

### 2.4.2 Void fraction measurement of the membrane

Void fraction of the porous PU membrane was calculated according to Eq.1:

$$V = (V_1 - V_0) / V_1 \times 100(\%) \quad (1)$$

where  $V$  is void fraction of the membrane,%;  $V_0$  is entity volume of the membrane (Assuming the membrane has no pore.);  $V_1$  is real volume of the membrane.

### 2.4.3 Viscosity measurement of blended PEG and anhydrous ethanol

PEG and anhydrous ethanol were blended evenly by different mass ratio. The viscosity of the mixture was measured using NDJ-79 rotational viscometer.

### 2.4.4 Filling rate of PEG into porous PU/PEG membrane

Filling rate was calculated according to Eq.2:

$$W\% = \frac{W_1 - W_0}{W_1} \times 100\% \quad (2)$$

where  $W$  is the filling rate of PEG into PU/PEG membrane;  $W_1$  is the dry weight of the filled membrane;  $W_0$  is the dry weight of the membrane before filling.

### 2.4.5 Mechanical property measurement

Mechanical performance test of the membrane was carried out using Instron5566 apparatus with 50mm gauge at a crosshead speed of 100mm/min, using rectangular (100mm×50mm) type tensile bars as testing samples.

### 2.4.6 DSC Measurement

Differential scanning calorimetry (DSC) was carried out using a DSC 204 F1 (NETZSCH). Samples (5~10 mg) were sealed in aluminum pans and measurements were performed in nitrogen atmosphere (60ml/min) at a heating rate of  $10^\circ\text{C}/\text{min}$  in a temperature range of 0 to  $100^\circ\text{C}$ . In the first scanning, the sample was kept at  $0^\circ\text{C}$  for 10 min and heated to  $100^\circ\text{C}$ , then kept at this temperature for 10 min. In the second scanning, the sample was cooled down to  $0^\circ\text{C}$  and the second scan was measurements were recorded.