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# Facile synthesis of highly ordered through-micro-porous polyethylene microfiltration membrane via micro-casting

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ABSTRACT

A polyethylene (PE) microfiltration membrane with highly ordered and identical straight through-pore microstructure was facilely synthesized via micro-casting. Initially, through embedding the micropillars of silicon micropillar template into a polydimethylsiloxane (PDMS) layer under a press force, we built a micropillar mold cavity (MPMC) and infiltrated PE melt into the cavity by submerging the MPMC into the melt to perforate the PE thin film. Then after a release of the PDMS layer, the solidified PE membrane was peeled off from the template. We synthesized membranes with different thicknesses from tailored MPMCs constructed under various press forces, and achieved a perfect peeling off without damage belonging to the thickness of 11 ± 0.3 μm due to its small peeling force. Water filtration experiment demonstrated membrane's functional performance on separation.



## 1. Introduction

For water purification, compared with inorganic microfiltration (MF) membranes [1], polymeric membranes [2,3] are more popular due to their cost-effectiveness and tunable properties [4,5]. However, the conventional polymeric MF membranes synthesized from phase separation [6], electrospinning [7,8] and stretching [9] have inherent defects like tortuous, overlapped and irregular pores, rough surface and great thickness, compromising their separation accuracy and throughput. The track-etched MF membranes [10] possess ideal straight through-pores but the inborn irregular distribution of pores and low porosity impede their applications.

Herein, we described a facile method to synthesize polymeric MF membranes with highly ordered and uniform straight through-pores (pore size: 2 μm, pitch: 4 μm) via micro-casting. Low-cost thermoplastic polyethylene (PE) was employed as the membrane material due to its excellent mechanical properties and chemical stability. Firstly, a micropillar mold cavity (MPMC) was constructed by embedding the micropillar tips of a silicon micropillar (SMP) template into a polydimethylsiloxane (PDMS) layer under a press force. Afterwards, by submerging the MPMC in PE melt, PE melt was infiltrated into the cavity. Finally, the solidified membrane was peeled off from the template after a release of the PDMS layer. We synthesized membranes with different thicknesses from tailored MPMCs built by various press forces and studied their peeling effect difference. Water filtration experiment was performed to evaluate the obtained membrane.

## 2. Experimental

### 2.1. Synthesis of membrane

Initially, we prepared a SMP template (micropillar diameter: 2 μm, height: 12 μm, pitch: 4 μm and square pattern area: 2.5 cm × 2.5 cm) by conventional photolithography and micromachining (Fig. S1).

In a hot-press machine (WENHUA CHIPTEK, CHINA), for forming a MPMC (Fig. 1a), a press force precisely applied by the piston pushed a fused silica plate (3.5 cm × 3.5 cm) onto a SMP template, to embed the micropillar tips into the soft PDMS layer (thickness: 30 μm, cured at 70 °C for 30 s) on the fused silica plate. Then we further cured the PDMS layer (110 °C for 3 min) to promote the MPMC's firmness.

The PE melt (The added solid PE was heated at 145 °C by the heating plate and melted.) gradually submerged the MPMC (Fig. 1b). For removing the air in the melt and the MPMC and adequately infiltrating the melt into the MPMC, we vacuumized the chamber and keep the vacuum state for 20 min. Then following the entire MPMC was exposed by discharging the melt (Fig. 1c), we ceased heating to cool it naturally. During cooling, the pressure applied on the sample was maintained. When cooled to room temperature, the applied pressure was removed. We took out the sample and released the PDMS layer (Fig. 1d). Then the membrane

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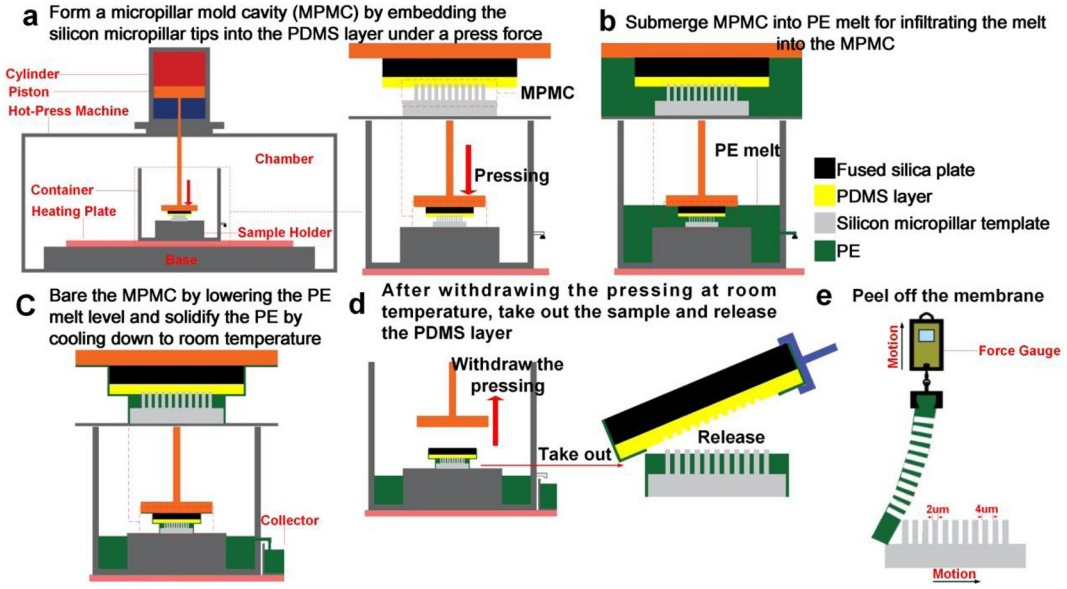


**Fig. 1.** Schematic illustration of the membrane synthesis.

thickness was measured by a film thickness tester (F20, Filmetrics Inc., USA). Finally, the PE membrane was grabbed by a clamp attached to a force gauge and peeled off (Fig. 1e). To ensure a constant peeling angle, we moved the force gauge and the SMP template along vertical and horizontal direction respectively by two linear motors at a uniform velocity (0.5 mm/s). We applied press forces (5.1 kgf, 6.4 kgf and 7.8 kgf) to tailor the MPMC's spacing and repeated the process to synthesize the membranes with thickness of 11 ± 0.3 μm, 10 ± 0.3 μm and 9 ± 0.3 μm respectively. The membrane morphologies were analyzed by a scanning electron microscope (HATACHI SU8010). The membrane porosity was estimated by the typical density method:

$$Porosity = [1 - (\rho_a/\rho_0)] \times 100\%$$

where $\rho_a$ is the membrane's apparent density, and $\rho_0$ is the PE density.

### 2.2. Evaluation of filtration performance

In various industrial fields, a large number of tiny matters ranging from 3 μm to 10 μm in solutions are required to be separated from water. Therefore, for comprehensively evaluating the membrane capability on filtering the matters bigger than 3 μm, we employed the polystyrene (PS) particle solution (0.15 mg/L, average particle size: 3 μm) as surrogate to perform an integrity test. The membranes were tested under various pressures in a homemade filtration device and at each pressure cycle-test was carried out: each cycle consisted of pure water (200 mL) flux test, PS particle solution (200 mL) filtration (flux and retention ratio test) and back flushing by pure water (300 mL) to clean the membrane. Ten samples were tested under the same condition for averaging the experimental results.

## 3. Results and discussion

### 3.1. Synthesis of membranes

#### 3.1.1. Formation of perforation

The SMP template is shown in Fig. 2a. Fig. 2b exhibits that the micropillars perforate the PE film. The embedment of the micropillar tips inside the PDMS layer leads to a direct formation of perforation as the PE melt fills in the MPMC. The temperature of PE melt does not influence the PDMS layer, fused silica plate and SMP template, since they can endure much higher temperature. After

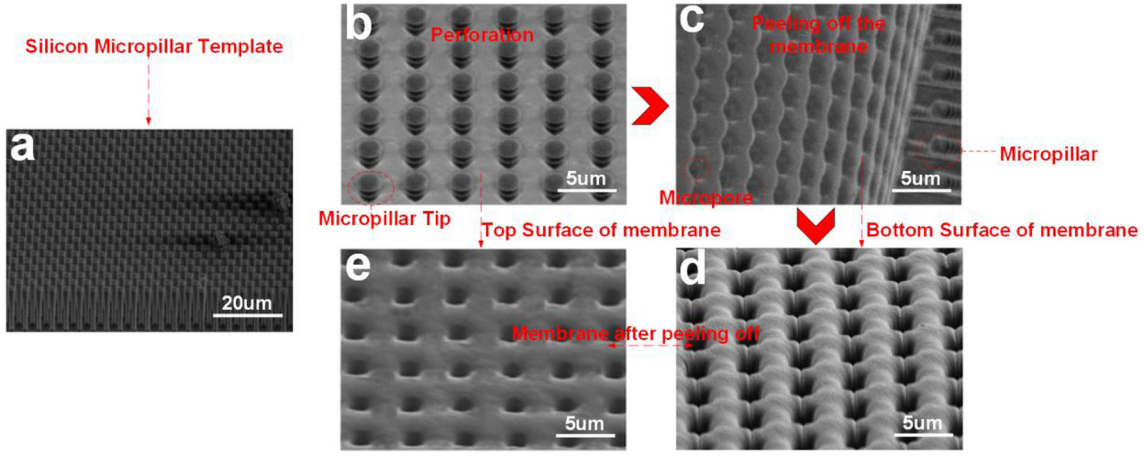


**Fig. 2.** SEM images of (a) silicon micropillar (SMP) template, a typical result of (b) perforate the PE film, (c) the membrane during peeling, (d) and (e) the membrane after peeling off.