

# Preparation of hydrophilic/hydrophobic porous materials

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## Abstract

A novel porous material was designed and prepared in this work. A hydrophobic open-celled porous polystyrene (PS) was first synthesized via a concentrated emulsion polymerization of water in styrene. Subsequently the porous polystyrene was saturated with an aqueous solution of acrylamide (AM) and an initiator, which was subjected to another polymerization and the resulted polyacrylamide (PAM) penetrated in the cells and intercellular pores of the PS matrix. The PAM would change its volume according to the environmental humidity and thus adjusted the permeation of the material. The morphology, pore size distributions, water absorption, and vapor permeation of the materials were investigated.

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**Keywords:** Concentrated emulsion; Porous material; Polystyrene; Polyacrylamide; Permeation

## 1. Introduction

In recent years, much attention has been focused on porous polymer materials with interconnected porosity, due to their wide range of possible applications, such as biomedical and pharmaceutical fields [1–4], chromatographic medias [5], catalysis of chemical and biochemical reactions [6], and electronic devices [7]. In order to produce open-celled porous polymeric materials, numerous techniques have been developed, including chemically and/or thermally induced phase separation [8,9], fiber bonding [10], solvent evaporation [11], gas foaming [12, 13] and so on.

However, in certain applications such as drug release, a special behavior of the porous materials was required, that is, a higher permeation was exhibited in some kind of situation and a lower permeation in the inverse situation. In other words, the material could adjust its permeation according to the environment. Numerous research works were carried out for preparing various functional materials. Ito et al. synthesized several stimuli-sensitive porous polymers, in which the permeation rate

of liquid was altered responding to the change in temperature [14], pH [15], oxido-reduction [16], and photo field [17]. Choi et al. grafted *N*-isopropylacrylamide (NIPAM) onto a porous membrane, and the obtained membrane was able to absorb hydrophobic solutes above the lower critical solution temperature (LCST) and hydrophilic solutes below the LCST [18].

In this work, a novel material was designed and prepared. A hydrophobic open-celled porous material was first prepared, which was impregnated in hydrophilic monomers, and the latter was allowed to polymerize in the cells and intercellular pores penetrating in the open-celled matrix. However, because of the volume contraction during the polymerization, some degree of connectivity of the intercellular pores remained in the matrix. The basic procedure was summarized in Fig. 1. With such a structure, in conditions of high humidity, the hydrophilic polymer would absorb water resulting in block of the pore. On the other hand, at low humidity, the hydrophilic polymer would lose water leaving the cells open. As a sample work, polystyrene (PS) and polyacrylamide (PAM) were selected as the hydrophobic and hydrophilic polymers, respectively. The morphology, pore size distributions, water absorption, and vapor permeation of the materials were investigated.

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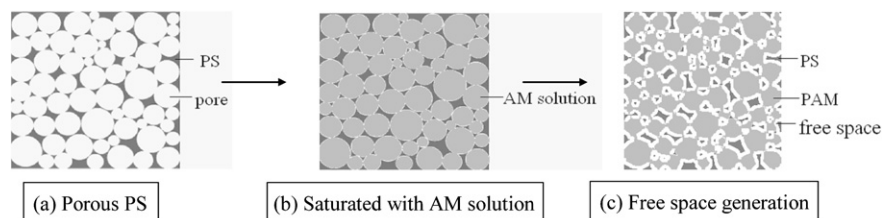


Fig. 1. Schematic representation of the general process in synthesizing PS/PAM composite.

## 2. Materials and methods

### 2.1. Materials

Styrene (St, AR), divinyl benzene (DVB, AR), Span80, acrylamide (AM, AR) and the water-soluble initiator potassium peroxydisulfate ( $K_2S_2O_8$ , AR) were purchased from Beijing Chemical Manufacture, China. The initiator of azobisisobutyronitrile (AIBN, CP) was provided by Shanghai Chemical Manufacture, China, and was purified by recrystallization from methanol. Poly(ethylene glycol) dimethacrylate (PEGDMA) was provided by Aldrich Chemical Company. The water used was distilled and deionized.

### 2.2. Syntheses

#### 2.2.1. Preparation of porous PS

An organic phase was placed in a flask composed of styrene monomer, Span80 (12 wt% of St, HLB = 4.3) as an emulsifier, divinyl benzene (DVB, 25 wt% of St) as a crosslinker, and azobisisobutyronitrile (AIBN, 2 wt% of St) as an initiator. With vigorous stirring, deionized water was introduced drop-wise into the organic phase as the dispersed phase until the volume fraction of the latter reached a value larger than 74%. A concentrated emulsion of water in styrene was thus formed, which was subjected to polymerization in a water bath at 50 °C for 20 h. The water was removed from the obtained porous PS via drying in a vacuum oven at 50 °C for 2 days until a constant weight was achieved.

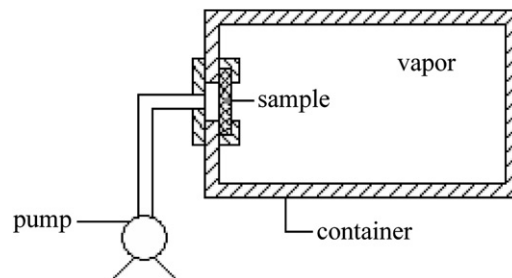
#### 2.2.2. Polymerization of acrylamide in porous PS

The obtained porous PS was immersed in a solution of acrylamide, potassium peroxydisulfate (2.5 wt% of AM) as an initiator, and poly(ethylene glycol) dimethacrylate (40 wt% of AM) as a crosslinker in deionized water at room temperature. A vacuum pressure cycle was used to help the solution permeate into the porous PS. The saturated system was heated in a test tube at 50 °C for 10 h to complete the polymerization of AM.

### 2.3. Characterization

#### 2.3.1. Scanning electron microscopy (SEM)

Structural morphologies of PS/PAM composites were studied with a JSM-6360LV SEM. The samples were mounted on aluminum studs using adhesive graphite tape and sputter coated with approximately 10 nm of gold before analysis.



Scheme 1. Equipment of vapor permeation experiment for PS/PAM composites.

#### 2.3.2. Mercury intrusion porosimetry measurement (MIP)

Pore size distributions were recorded by MIP using an Auto-pore II 9220 porosimeter. Samples were subjected to a pressure cycle starting from approximately 0.57 to 60,000 psia in predefined steps to give pore size/pore volume information. Two pressure ranges were applied: low-pressure range (0.5–25 psi) was used to measure pore size >6 μm and high-pressure range (25–60,000 psi) was used to measure pore size <6 μm.

#### 2.3.3. Water absorption

The PS/PAM composites were immersed into water at room temperature. The water absorption was calculated as follows [19,20]:

$$WG = [(m_{\text{wet}} - m_{\text{dry}}) / m_{\text{dry}}] \times 100\%,$$

where  $m_{\text{dry}}$  is the mass of the dry PS/PAM specimen, and  $m_{\text{wet}}$  is the mass of the PS/PAM with imbibed water.

#### 2.3.4. Vapor permeation

Saturated vapor was allowed to permeate the porous composite and the pressure difference between the inlet and outlet was recorded as a function of time, as illustrated in Scheme 1.

## 3. Results and discussion

### 3.1. Scanning electron microscopy (SEM)

The most compact packing volume fraction of monodispersed spherical drops in a concentrated emulsion is 0.74. Above this value, the drops are either deformed or polydispersed [21–24]. Since the wall separating the dispersed drops contains also microdrops, the porous material obtained from a concentrated emulsion polymerization possesses an open-celled structure.

The SEM micrographs of the porous PS from precursor concentrated emulsions with various volume fractions of the

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