New Soft Microporous Green Materials from Natural Soybean Oil

Lyubov Lukyanova, Roberto Castangia, Sophie Franceschi-Messant, Emile Perez, Isabelle Rico-Lattes

Rheological Study: The rheological properties of the organogels were studied by an advanced rheological expansion system (Advanced Rheometer AR-1000, TA Instruments, USA). The gel was first dissolved in an oven at 100°C and then 1 mL of this solution was dropped between the two parallel plates of the AR-1000. The upper plate (cone diameter = 60 mm, cone angle 2°) was lowered to form a gap of 61 µm between the two plates. To obtain the storage modulus G’ (a measure of elasticity) of the gel, a dynamic temperature ramp test was carried out. The ramp rate was set at 1°C/min. The amplitude of the oscillation was controlled to obtain a strain of 1% and the frequency was set at 0.1 Hz. The T°gel was measured by following the variation of elastic modulus (G’) and viscous modulus (G”) with the temperature. The T°gel was determined when we observed an abrupt increase in the two moduli (gelation process). The accuracy of T°gel measurements was ±1°C.

Scanning Electron Microscopy (SEM) Analysis: The samples were allowed to dry naturally at room temperature, mounted on aluminum stubs (12 mm in diameter and covered with double-face carbon adhesive tape) and sputter coated with gold (10 mA, 1.5 kV for 30 s). Then the granulometry of the porogen particles, predesigned templates and organogel structure were examined using a scanning electron microscope (LEO 435VP, Zeiss, Germany) with an acceleration voltage of 7.5 kV.

Low Vacuum Scanning Electron Microscopy (LV-SEM) Analysis: The samples were allowed to dry naturally at room temperature and were mounted on aluminum stubs (35 mm in diameter and covered with double-face carbon adhesive tape). Then the surface and pore morphology of the materials were investigated using a low vacuum scanning electron microscope (JSM-6060 LV, JEOL, Japan) with an acceleration voltage of 30 kV at a pressure of 130Pa.

Image Analysis: The size distribution of the porogen particles and the pore size of the porous materials were quantified by image analysis (ImageJ software v.1.37, NIH, USA). The particle or pore size distribution and average particle or pore size were obtained by measuring about 600 to 1000 particles or pores in arbitrarily chosen areas of several micrographs (12 to 18).

Determination of Porogen Particle Residues: A known weight of porous material was dissolved in chloroform and the particle residues were extracted with distilled water. The saccharose residues were evaluated by thin-layer chromatography (specific revelation) and their concentration was less than the limit of detection (< 0.1 wt%). To evaluate sodium chloride residues, atomic emission spectroscopy (AES, X Inca Energy 250, Oxford Instruments) of Na ion was performed, and NaCl residues were less than 0.1 wt%. These experiments were performed in triplicate.

Effective Porosity Determination: The effective porosity of the organogel materials was measured by liquid displacement, with water as the displacement liquid. The porous organogel scaffold was immersed in distilled water for 7 days. After a rapid wipe of its surface with filter paper to remove the excess liquid, the water-saturated porous sample was weighed (m1). The dry weight of the sample (m2) was measured after freeze-drying. The density of nonporous organogel p was determined from the mass of organogel and the volume it occupied, and the porosity of the materials was calculated as P% = (V effective porosity/(V effective porosity + V organogel))x100, where V effective porosity = (m1-m2), V organogel = (m2/p). The weight was measured with an accuracy of 10^-4g. The experiments were performed in triplicate.

Draining Evaluation by Conductivity Measurements: A draining test was carried out using a SevenMulti (Mettler-Toledo GmbH, Switzerland) conductometer with an InLAB6720 conductivity cell. The porous materials were immersed in a solution of sodium chloride (0.15M) for 4-5 days at room temperature. After rapid wiping of the sample surface with filter paper, the sample was immersed in 100 mL of distilled water at 37°C±0.1°C, and conductivity was measured periodically until a constant value was reached. The results are means of three separate parallel experiments.
Figure S1  SEM micrograph of compact sugar template (3.5wt% of initial water content).