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Controlled Nanoporosity of Organic Aerogels by Dispersing Clay Platelets

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Experimental Section

Materials: Laponite RD (gel grade) and RDS (sol grade) were obtained from Rockwood Clay Additives Inc., UK (www.laponite.com). Both materials consist of disc-shaped particles with a thickness of 0.92 nm and a diameter of 25 nm (from Laponite brochure in Southern Clay Products, Inc., www.scprod.com/pdfs/LaponiteBrochureE.pdf). This high purity grade material had an empirical formula of $Na^{0.7+}[(Si_8Mg_{5.5}Li_{0.3})O_{20}(OH)_4]^{0.7-}$. Laponite RD was a synthetic layered silicate and can produce highly thixotropic gels at the concentrations of 2 wt% or greater in water. Whereas laponite RDS was sol grade and remains free flowing up to the 10 wt% concentration in water. RF hydrogels were synthesized by the polycondensation of resorcinol and formaldehyde, using sodium carbonate as the base catalyst, and distilled water as the diluent. The R/F molar ratio was 0.5 and normally 1 g of RF solid dissolved in 10 ml of distilled water (target density of 0.1 g cm^{-3}) with varying wt% of catalyst concentration. Resorcinol, 37 wt% formaldehyde solution and sodium carbonate were purchased from Sigma Aldrich co. and used as received. Open-celled melamine foam used as support was kindly provided by Foam Hightech Co. (Korea).

Sample preparation: At first, clay dispersions were prepared at a concentration of 1~3 wt% in water by adding clay gradually with vigorous stirring for about 10 min. At the same time, resorcinol was dissolved in formaldehyde solution. After mixing two liquids, the solution was stirred for 30 min and poured into 20 ml glass vials (inner diameter of 25 mm), which were then sealed and cured at 70 °C oven for 1 day. To fabricate crack-free slab samples (20 x 20 x 1.5 cm) for the purpose of measuring thermal conductivity, we utilized open-celled melamine foam as supporting template for hydrogel. We tested several open-celled foams and found that melamine foam produced by BASF was best suited to our purpose. The picture of melamine foam and its microstructure using optical microscopy was shown in Fig. 4S in Supporting Information. To our knowledge, melamine foam showed the lowest density (~ 0.006 g cm^{-3}) of all existing polymer foams, soaked aqueous sol completely and exhibited no swelling during soaking. RF sol mixture was poured into teflon square mold and melamine foam was immersed into RF sol. The cured hydrogels removed from the teflon mold were placed in methanol for at least 3 days with replacement of fresh methanol each day in order to substitute methanol in the RF gel for water. The synthesized RF hydrogels were then dried with super critical carbon dioxide by following the standard procedure. (reference 3)

Analysis: Specific surface area and pore size distribution of aerogel composites were analyzed by BET and BJH nitrogen gas absorption and desorption method (Tristar II 3020, Micromeritics, USA) after degassing at 95 °C for 24 h. Pore morphologies were measured using a ultrahigh resolution scanning electron microscope (UHR-SEM, S-5500). SEM observations were conducted at an accelerating voltage of 5 kV with Os coating. The microstructure of the aerogels was studied using the transmission electron microscope (FE-TEM) at a voltage of 200 kV. The thermal conductivities at a temperature of 20 °C for aerogel foam composites were determined in accordance with the procedure given in ASTM C518 utilizing a custom heat flow meter (HFM 437, Netzsch Inc., Germany).

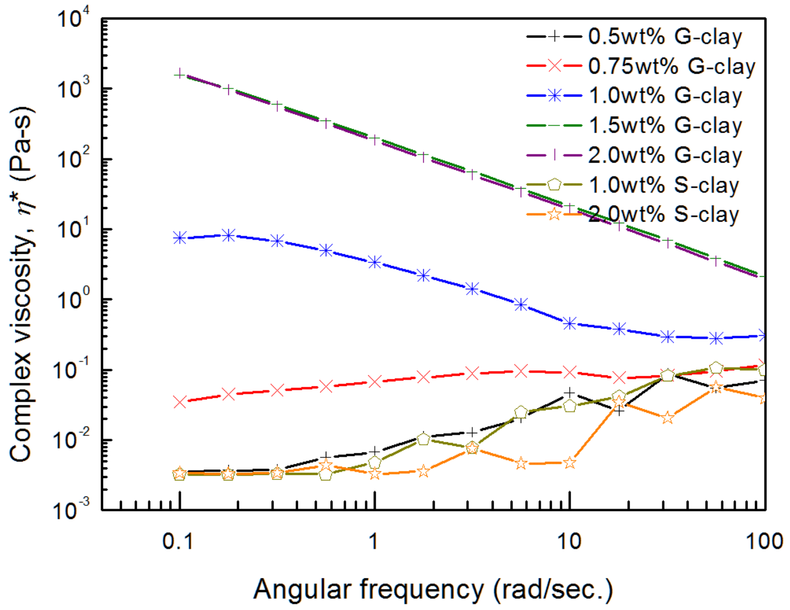

Fig. S1. Complex viscosity for S-clay and G-clay solutions as a function of angular frequency at various clay concentrations

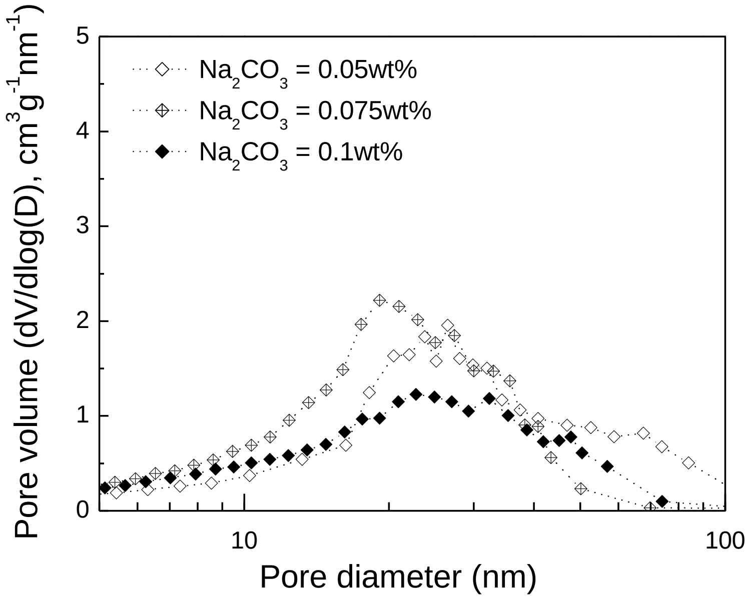

Fig. S2. Particle size distribution for RF aerogels with different amount of sodium carbonate (Na2CO3) as catalyst.

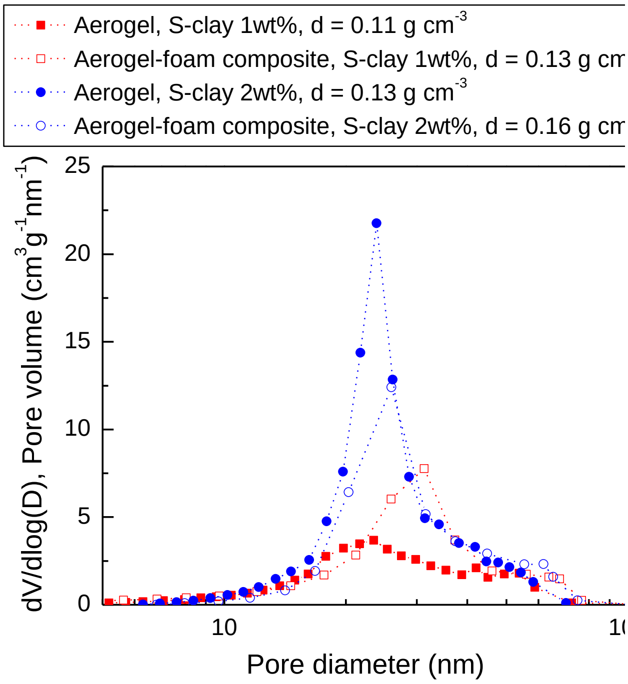

Fig. S3 The pore size distribution of aerogel and aerogel-foam composite.

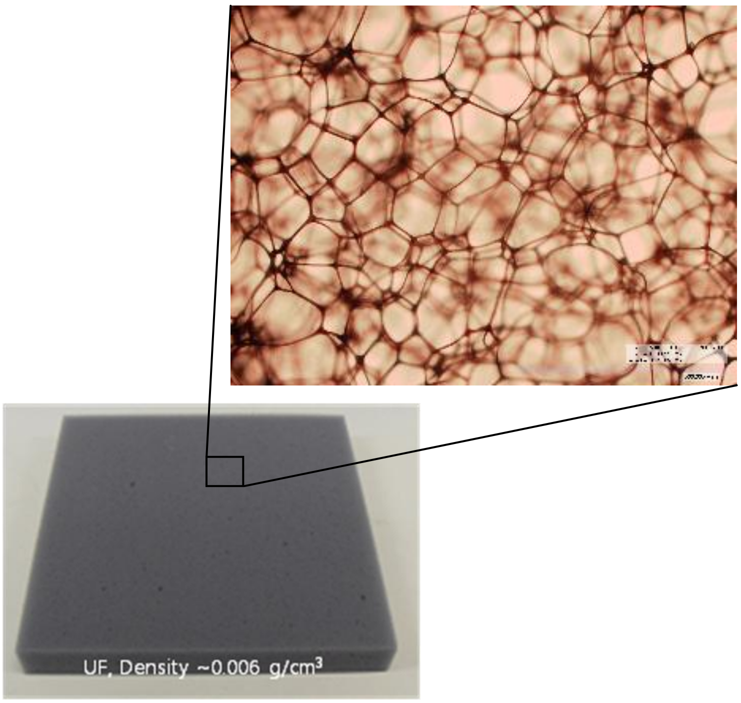

Fig. S4 The sample and optical microscopy image of Melamine foam manufactured by BASF (BASOTEC, ultrafine grade).

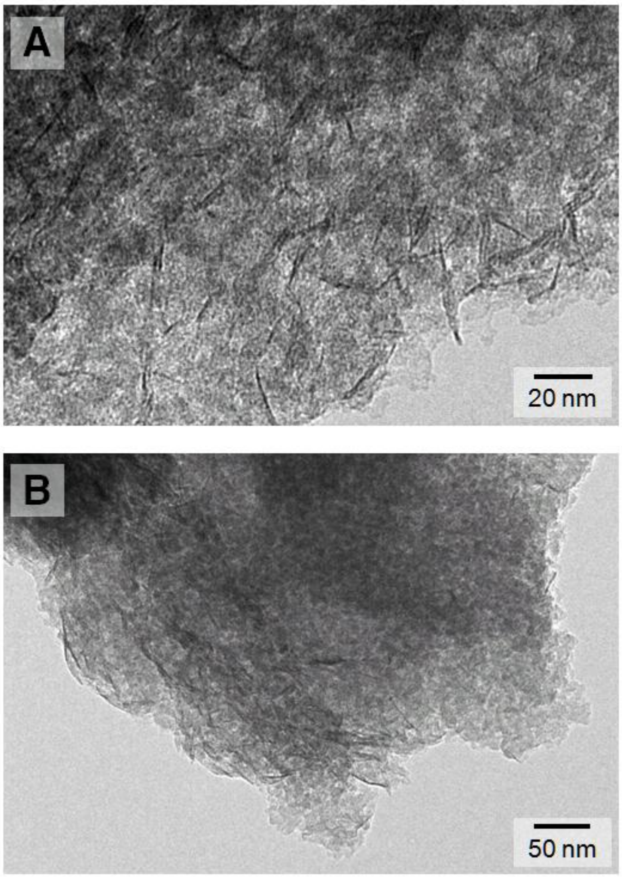

Fig. S5 TEM images of clay-catalyzed organic RF aerogels prepared at 1wt% of G-clay (A) and S-clay (B).