Supporting Information for

Ultralow-Density, Transparent, Superamphiphobic Boehmite Nanofiber Aerogels and Their Alumina Derivatives

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Experimental;

Materials. Boehmite nanofiber (BNF) sol F1000 (8.5 wt % in acetic acid aq., determined by dry mass) was kindly supplied from Kawaken Fine Chemicals Co., Ltd. (Japan), and distilled water was purchased from Hayashi Pure Chemical Industry, Ltd. (Japan), respectively. Hexamethylenetetramine (HMT) and trimethoxy(1*H*,1*H*,2*H*,2*H*-heptadecafluorodecyl)silane were obtained from Tokyo Chemical Industry Co., Ltd. (Japan). All of the chemical reagents were used as received. Umm Shaif Crude Oil was supplied from the Japan Coast Guard.

Sample Preparation. First, x mL of BNF sol, $20-x$ mL of water and HMT were mixed at 80 °C in a glass sample tube for 3 min. The obtained sol was transferred into a tightly sealed container, which was then placed in a forced convection oven at 80 °C to complete gelation (in 20 min) and aging for 4 h. The obtained gels were washed with methanol by soaking for several times to remove unreacted HMT. The washed sample was subjected to solvent exchange with 2-propanol three times in an identical way to washing. To dry the BNF wet gels, 2-propanol in wet gels was exchanged with supercritical carbon dioxide at 80 °C and 14 MPa in a custom-built autoclave (Mitsubishi Materials Techno Corp., Japan), followed by slow depressurization to atmospheric pressure. The obtained BNF aerogels were typically in the shape of cylinder with 30 mm in diameter and 10 mm height. To transform into Al_2O_3, BNF aerogels were calcined for 6 h at different temperatures. For the preparation of a superamphiphobic BNF gel, the cylindrical BNF aerogel and 10 μL of trimethoxy(1*H*,1*H*,2*H*,2*H*-heptadecafluorodecyl)silane were put in a gastight container with 500 mL volume and heated at 80 °C for 12 h.

Characterizations. A field emission scanning electron microscope (JSM-6700F, JEOL, Japan) was employed to observe the microstructure. Mechanical properties of aerogels were measured by a material tester (EZGraph, Shimadzu Corp., Japan). For uniaxial compression tests, cylindrical samples were compressed using a load cell of 5 kN with a rate of 0.5 mm min^{-1}. For light transmittance measurements, a UV/vis spectrometer V-670 (JASCO Corp., Japan) equipped with an integrating sphere ISN-723 was employed. Direct-hemispherical transmittance was recorded, and obtained transmittance data at 550 nm were normalized into those of 10 mm-thick samples using the Lambert–Beer equation. Specific surface area was estimated from N_2 adsorption measurement (BELSORP-mini II, Bel Japan Inc., Japan) by the Brunauer–Emmett–Teller (BET) method. The X-ray diffraction (XRD) analysis was carried out with RINT-Ultima III (Rigaku Corp., Japan) operated at 1.6 kW using Cu Kα (λ = 0.154 nm). Helium pycnometry (Ultrapyc 1200e, Quantachrome Instruments) was used to determine the skeletal density (true density). Contact angle was measured with Drop Master DM-561Hi (Kyowa Interface Science Co., Ltd., Japan). Volume of water and *n*-hexadecane droplet was fixed at 2.5 μL and 3 μL, respectively, and contact angle was determined at 5 s after attachment of the liquid to gel surface. The elemental analysis of the samples was carried out by X-ray photoelectron spectroscopy (XPS) (MT-5500, ULVAC-PHI, Inc., Japan). The monochromatized Mg Kα radiation (1253.6 eV) was used. The core levels were calibrated by reference to the first component of the C ls core level peak (unfunctionalized hydrocarbons) set at 284.6 eV. Thermogravimetric–differential thermal analysis (TG–DTA) was performed with a Thermo plus TG 8120 (Rigaku Corp., Japan) instrument at a heating rate of 5 °C min^{-1} while continuously supplying air at a rate of 100 mL min^{-1}.

Movie list;

Movie S1. Boehmite nanofiber sol is quickly turned into a gel by adding basic aqueous solution (NaOH aq. in this movie).

Movie S2. The fluoroalkylsilane-treated BNF aerogel sample floating on Umm Shaif Crude Oil by surface tension.

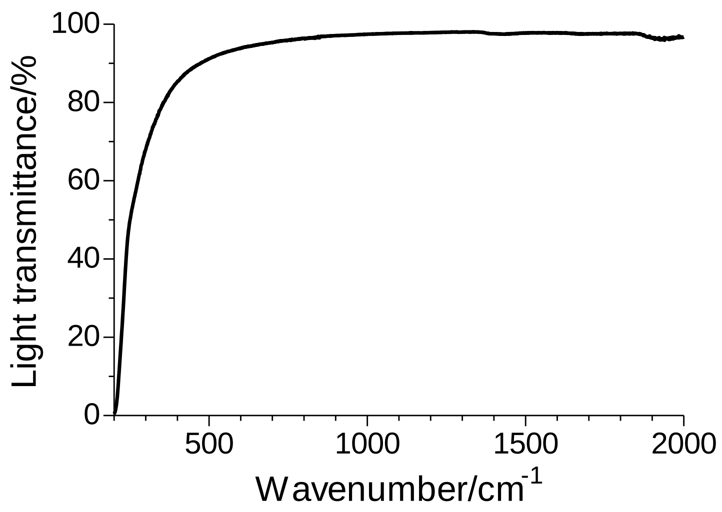

Figure S1. Visible-light transmittance of the BNF aerogel obtained from 0.21 wt % BNF sol (10 mm thick).

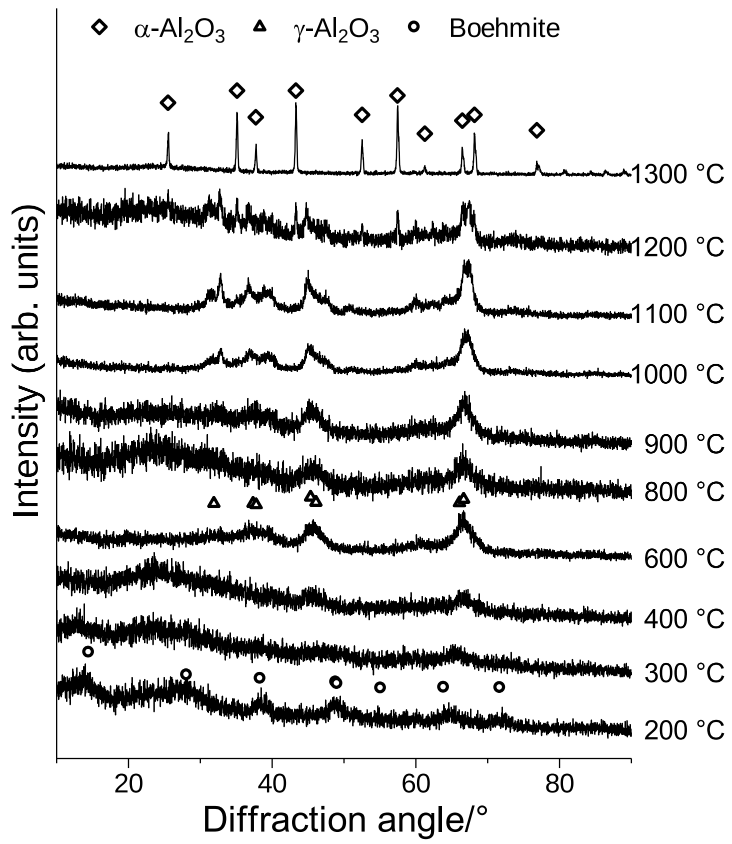

Figure S2. XRD patterns of the aerogels obtained from 0.43 wt % BNF sol and calcined at different temperatures.

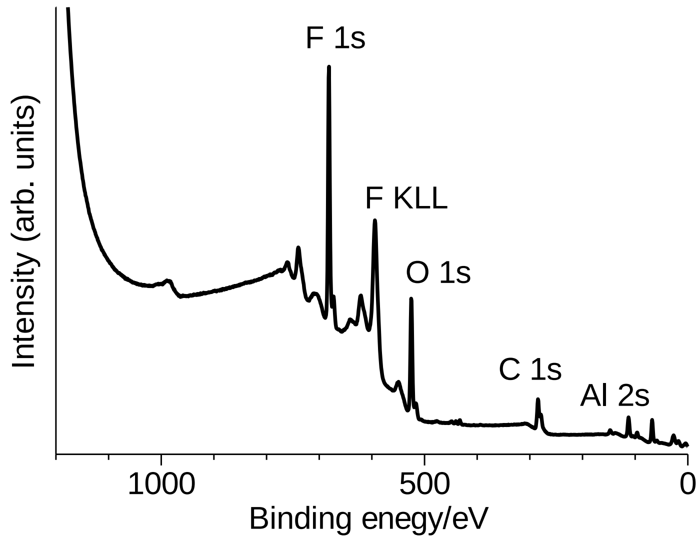

Figure S3. XPS survey spectrum of the BNF aerogel treated with fluoroalkylsilane, revealing the presence of F. The atomic ratio of F/Al on the surface of the aerogel is ~2.7.

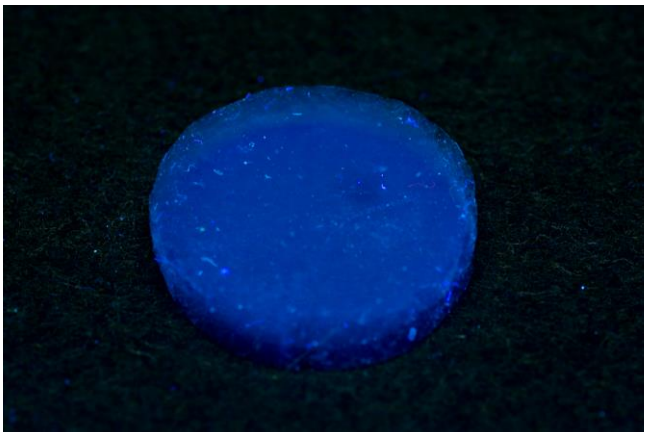

Figure S4. Photoluminescence (excited at $\lambda = 254$ nm) of the γ-Al_2O_3 monolith derived from the BNF aerogel (30 mm in diameter and 5 mm height).