From waste epoxy resin to efficient oil/water separation materials via microwave assisted pore-forming strategy

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Experimental Section

Materials

E-51 epoxy resin and curing agent, 4, 4-diaminodiphenyl methane (DDM) were provided by Bluestar Chemistry Company and Aladdin Chemistry Company, respectively. Other solvents were purchased from Aladdin Chemistry Company. All chemicals were directly used as received without further purification. The diameter of EP was 25 mm, while the thickness of the sample was 2 mm.

Swelling of EP and preparation of PEP

The swelling of epoxy resin in NMP was achieved by microwave-assisted heating. A Sineo UWave-2000 batch microwave oven (Shanghai, China) with a rated power of 1000 W was used at the 600 W power setting. The heating rate was 12^{o}C/min. The EP was put into 30 mL of NMP solution in microwave reactor. The temperature was set at a constant temperature for a certain time. After the reaction, the swollen EP was floated on the top of the NMP and then was collected after filtration. Here we obtained the SEP. The SEP was washed with deionized water then heated via microwave radiation to obtain the porous epoxy resin (PEP).

Preparation of emulsion

The oil/water emulsions with different droplet sizes were prepared by mixing 100 mL chloroform containing different dosages of Span 80 and 1.5 mL water for different time by the ultrasonic method. Take an emulsion with droplet size of 3.5 μm for example: 0.4 g span 80 was added into 100 mL chloroform, followed by 1.5 mL water. The

mixture was ultrasonicated for 3 min and no demulsification was observed after 8 h.

Oil/Water Separation

PEP was placed on the filtration device as filtration film. 10 mL of oil/water mixture (1:1) was added into the device. The separation process was carried out under gravity. The water content in the filtrate was measured by Karl fisher method. In order to clearly observe the separation process, oil was dyed using Oil red O, and water was dyed using methylene blue. The absorption time was observed from the test video. Oil/water emulsion was separated according to the procedure above except 15 mL of oil/water emulsion was used instead of oil/water immiscible mixture.

Characterization: SEM images were taken with a Phenom Pro X (Netherland) at an accelerating voltage of 10 kV. Pior to observation, the cryogenically fractured surfaces were coated with Au. FTIR were measured on a Nicolet 6700 spectrophotometer. Atomic force microscope (AFM) was measured on a Bruker dimension icon with Scan ASYST. Water contact angles were measured by JC2000D2H Powereach with a water droplet (3 μL) as an indicator. Thermogravimetric analysis was recorded on a 209 F1 (NETZSCH). The samples were heated from 40 to 550°C at a heating rate of 10°C min^{-1} in N_2. The compressive strength of materials was measured on a static mechanical tester (Instron 3345) at room temperature.

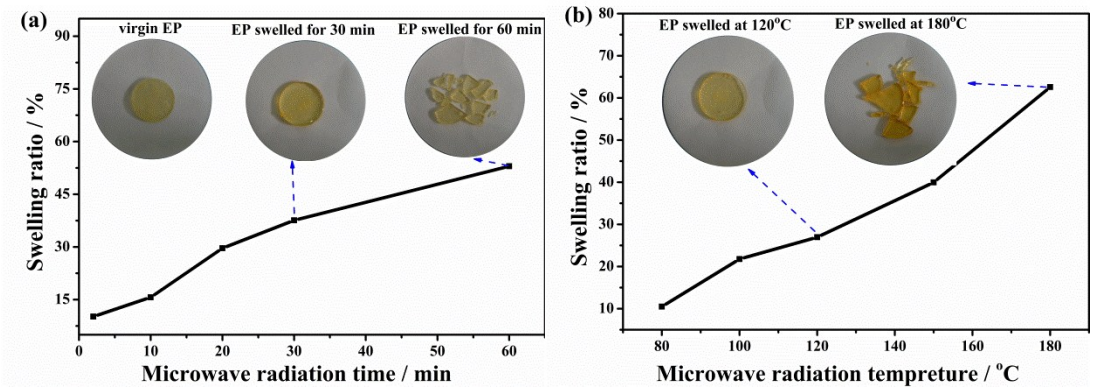


Figure S1. The effect of microwave conditions on the swelling of EP: a) microwave time (microwave temperature is 150°C, and microwave power is 400 W); b) microwave temperature (microwave time is 30 min, and microwave power is 400 W). The embedded graph is optical pictures of EP during the swelling. The swelling ratio (r) of EP was calculated as follows:

$$r = \frac{m_1 - m_o}{m_o}$$

m_o was the mass of virgin EP, and the m_1 was the mass of swollen EP.

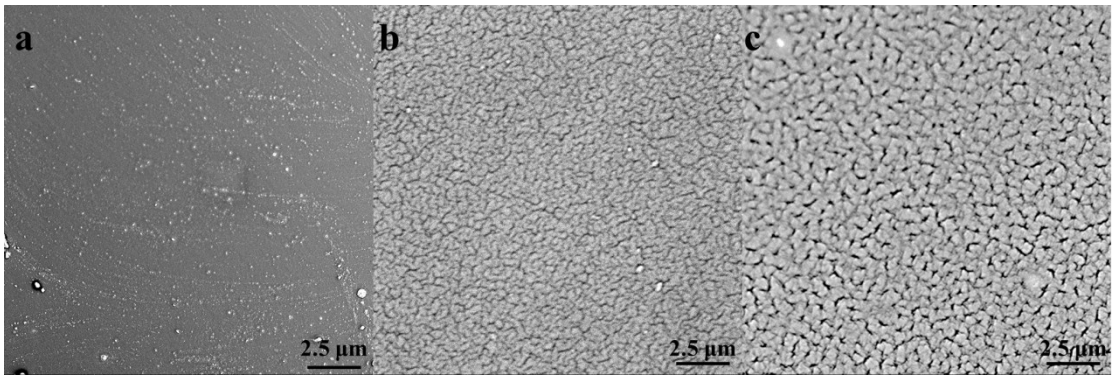
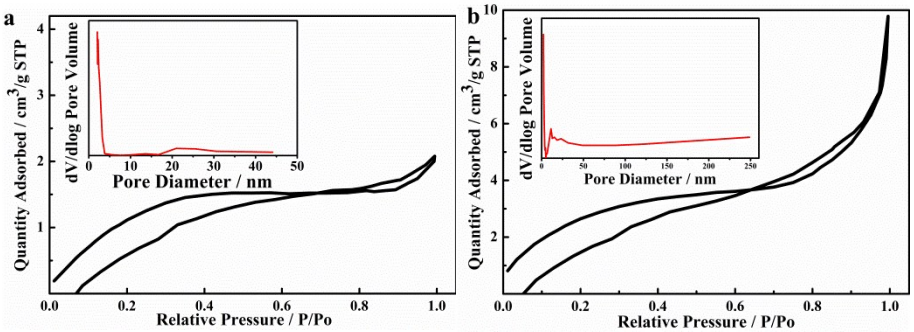


Figure S2. The SEM of cross section of EP (a), SEP (b) and PEP (c).



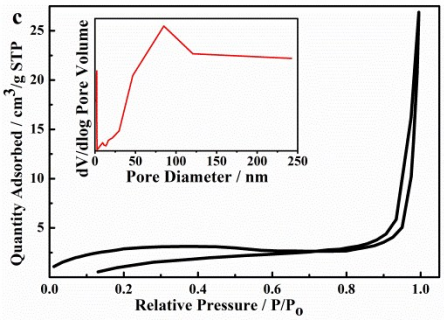


Figure S3 N_2 adsorption-desorption isotherms and pore distribution (inset) of relevant (a) EP, (b) SEP and (c) PEP.

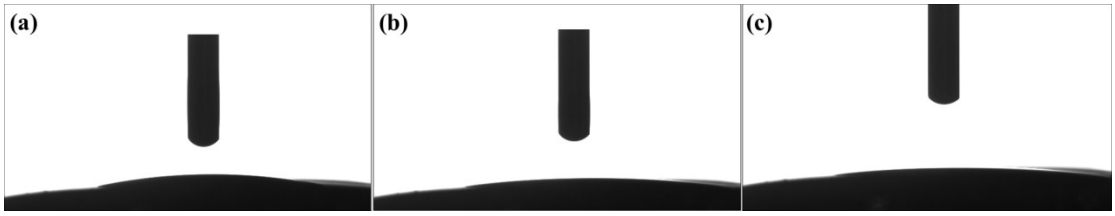


Figure S4. Wettability toward oil of the (a) EP, (b) SEP, (c) FEP. During the oil CA measurement, the chloroform droplet was 3 μL.

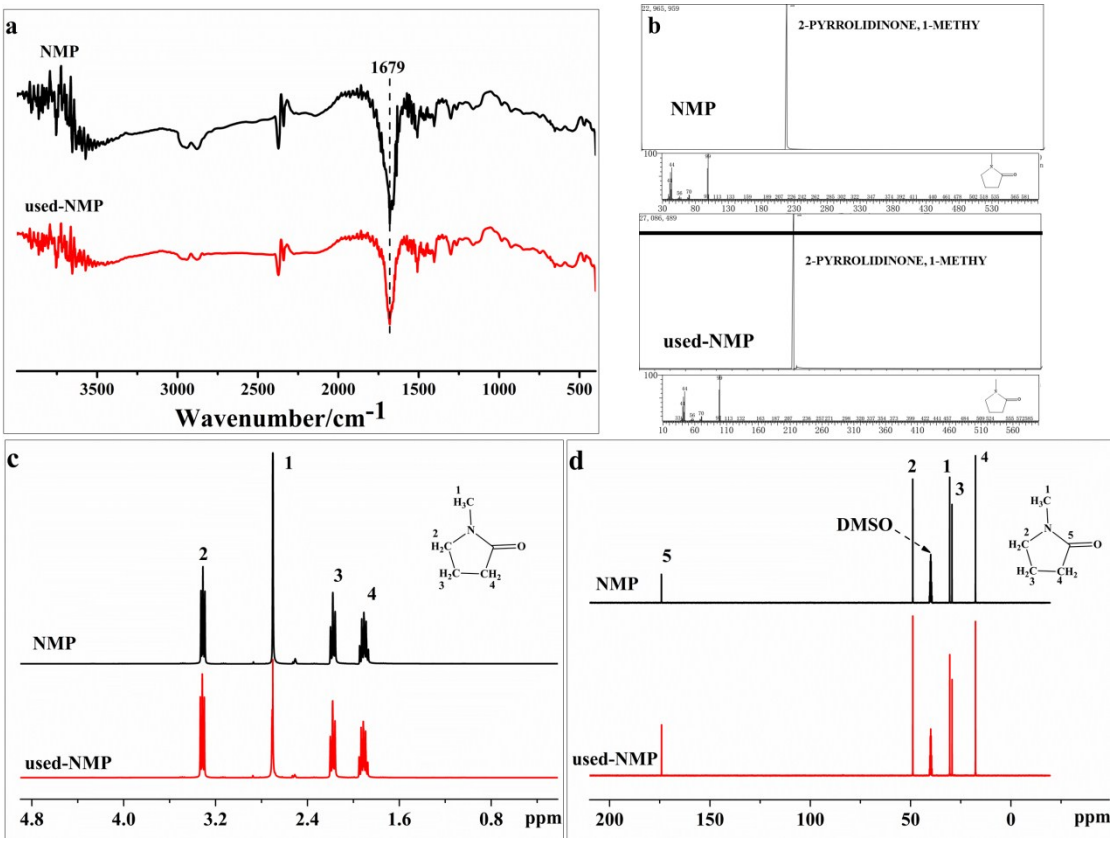


Figure S5. FT-IR spectra (a), GC-MS chromatogram (b), ^{1}H-NMR spectra (c), ^{13}C-NMR spectra (d) of virgin NMP and used NMP.

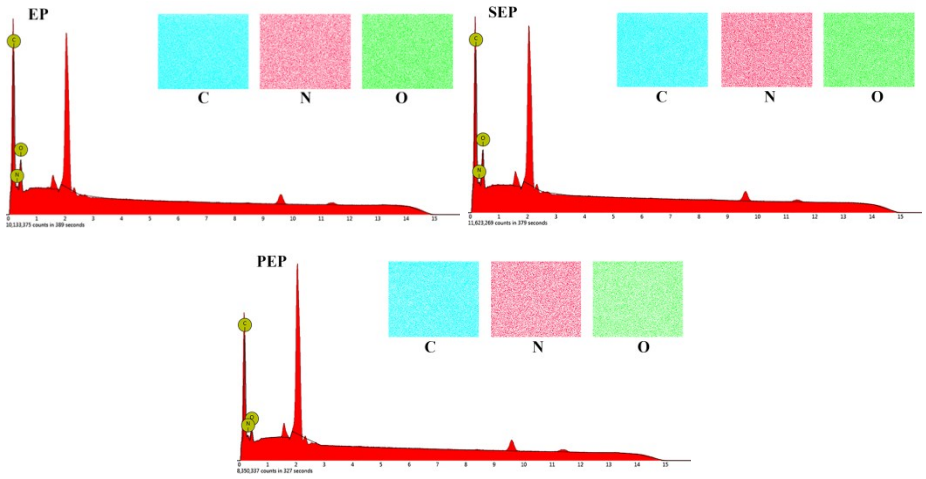


Figure S6. The EDX spectra and C, N, O mapping of EP, SEP and PEP.

Table S1. The C, N and O contents of EP, SEP and PEP.

Sample	Carbon content (wt%)	Nitrogen content (wt%)	Oxygen content (wt%)
EP	51.02	16.75	32.24
SEP	51.44	26.23	22.33
PEP	51.35	25.21	23.44

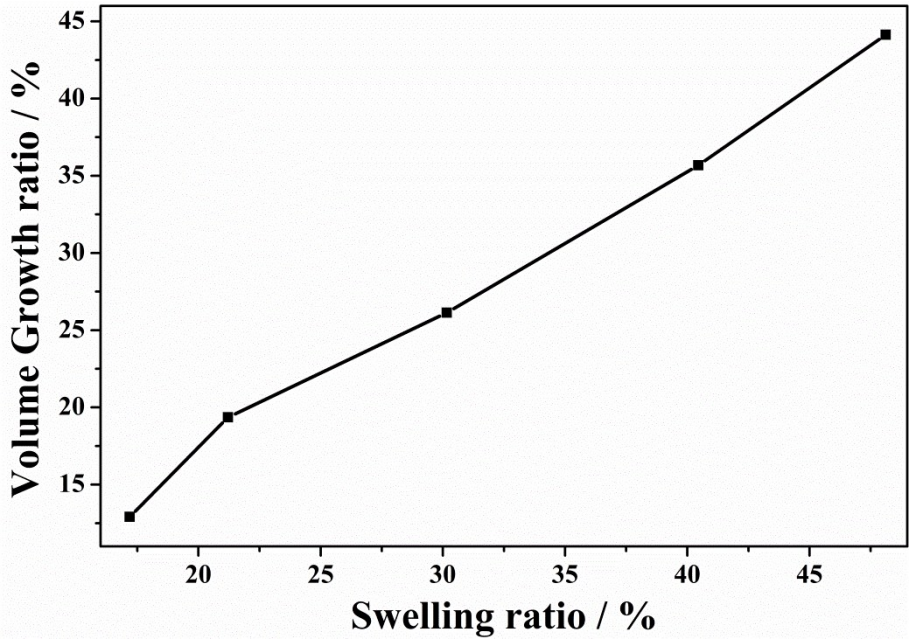


Figure S7. The swelling ratio versus volume growth ratio of SEP.

Table S2 Preparation of emulsions with different droplet sizes.

Emulsion droplet size	Span 80 (g)	Ultrasound time (min)
20 μm	0.2	10
6 μm	0.4	5
3.5 μm	0.4	3
550 nm	0.4	10
300 nm	0.4	30
150 nm	0.8	60

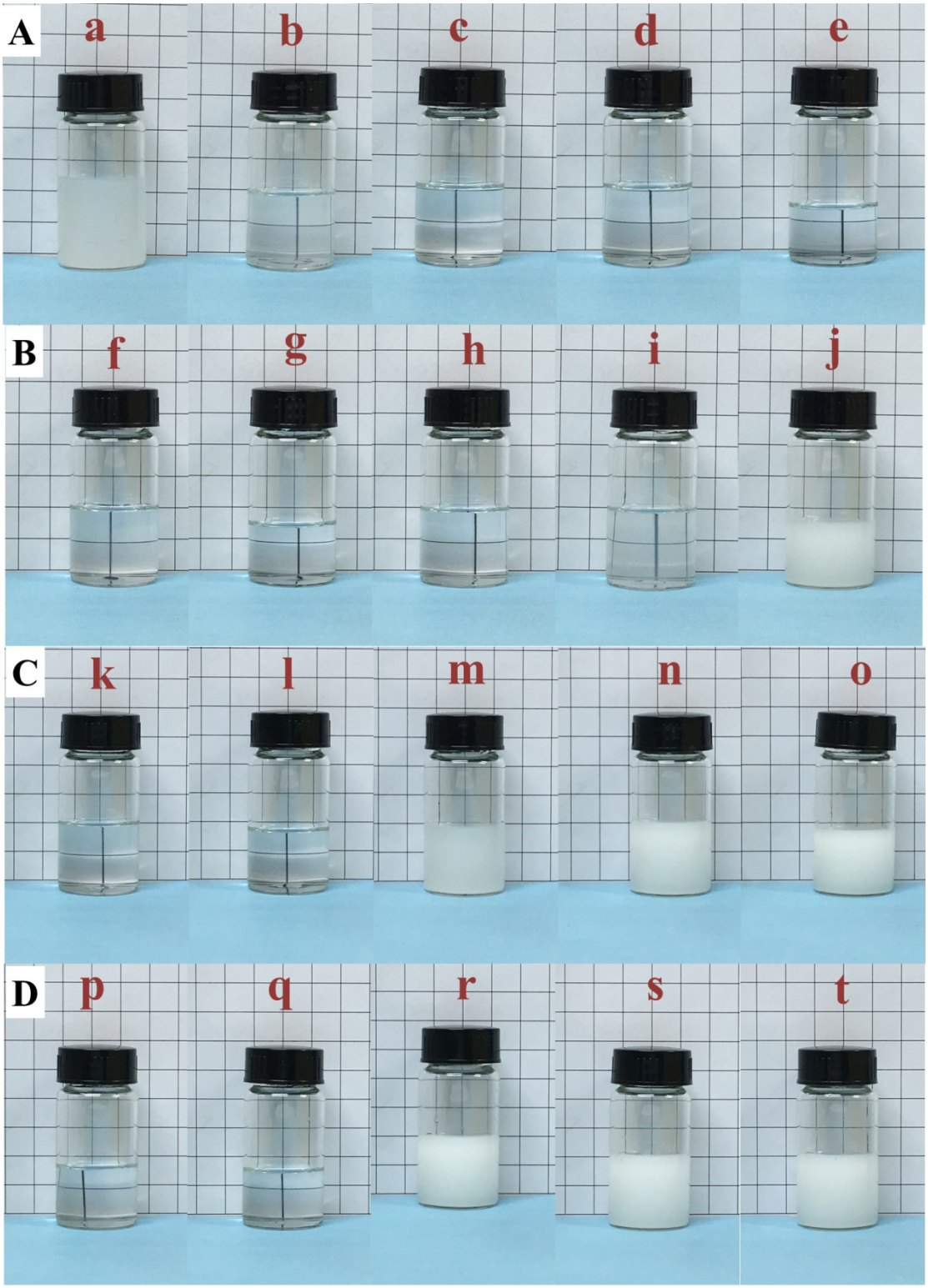


Figure S8. The optical images of the effect of PEP with different pore sizes (from A to D, 80 nm, 360 nm, 550 nm and 1.5 μm respectively) on separation of emulsion with different droplet sizes (from left to right, 20 μm, 6 μm, 550 nm, 300 nm, and 150 nm respectively).

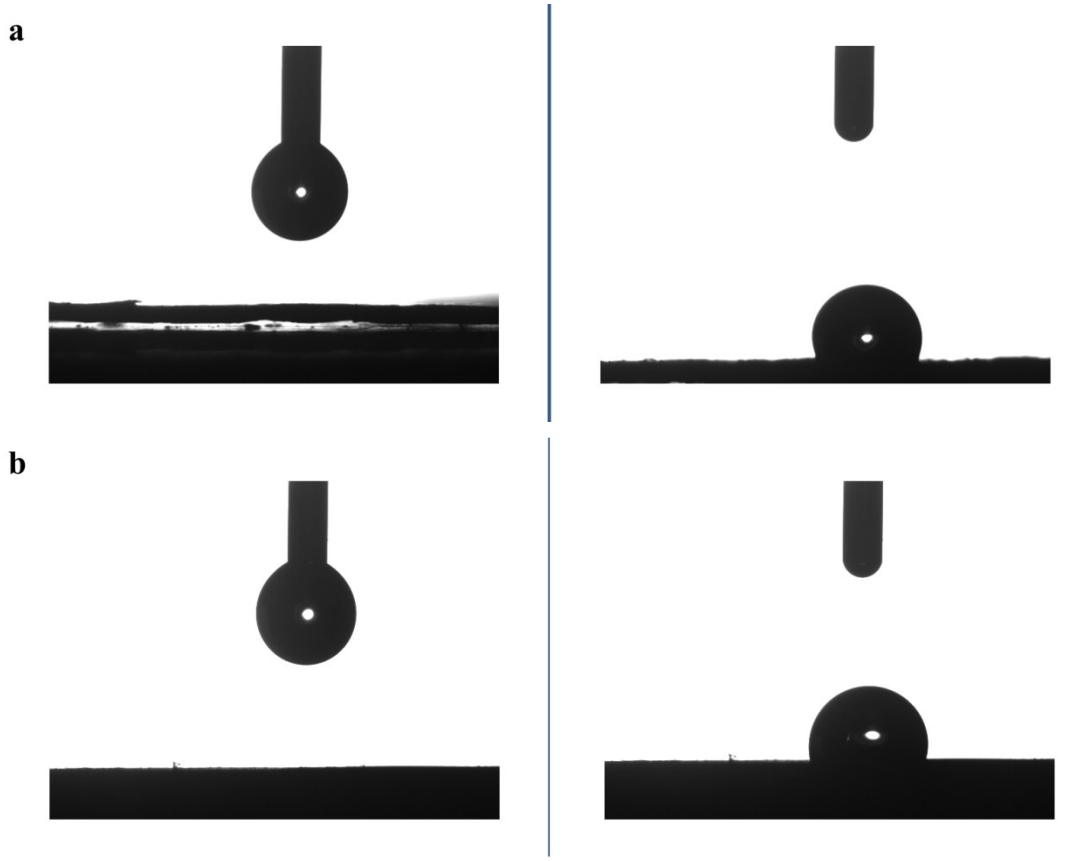


Figure S9 Water contact angle of epoxy-insulation board (a) and epoxy-heat insulation board (b) were modified by NMP under microwave radiation at 150°C for 30 min. During the water CA measurement, the water droplet was 3 μL.

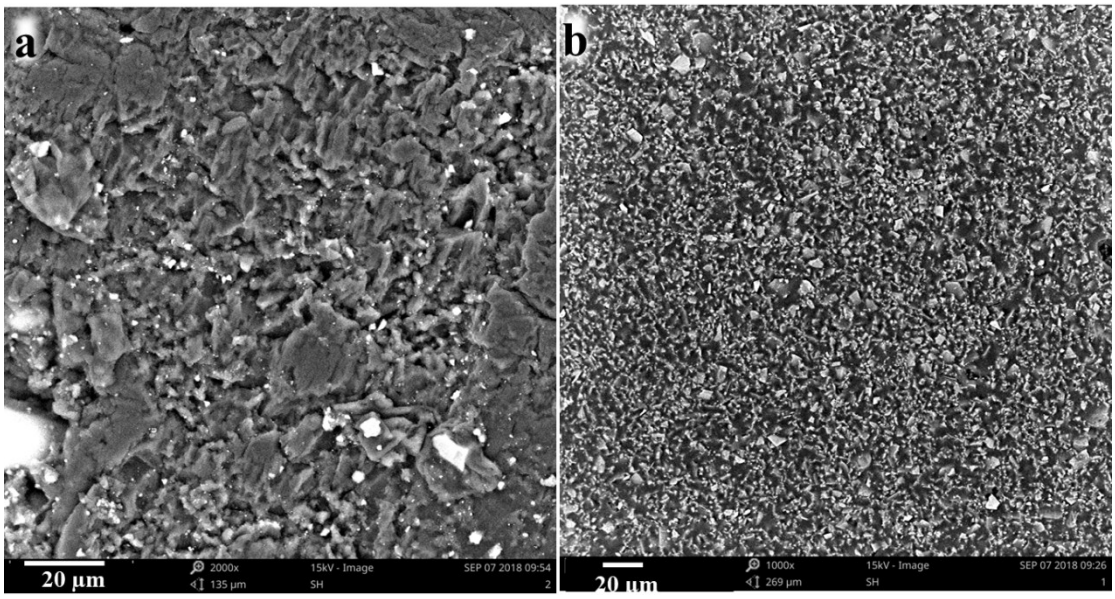


Figure S10 SEM of epoxy-insulation board (a) and epoxy-heat insulation board (b).

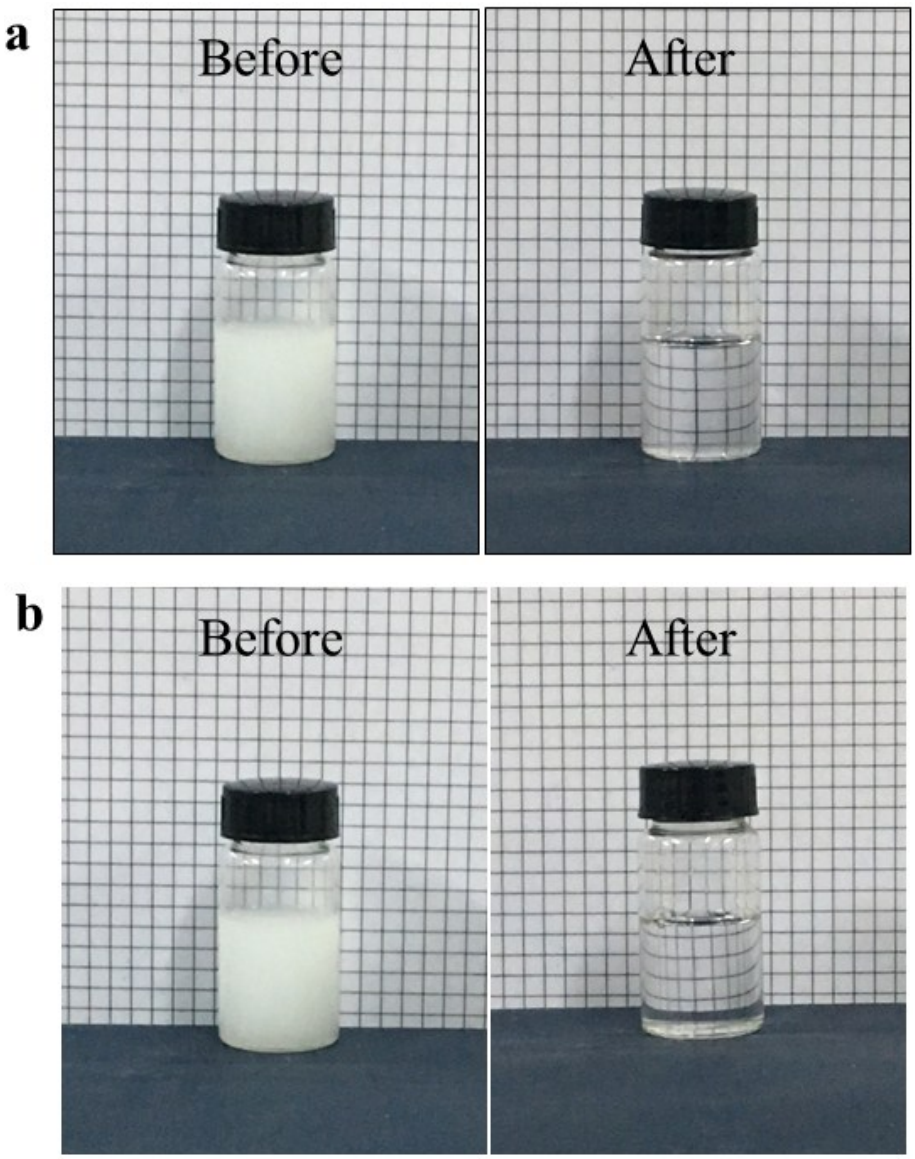


Figure S11 Photos of oil/water emulsion with an average droplet size of 3.50 μm before and after separation by modified epoxy-insulation board (a) and epoxy-heat insulation board (b).