Supporting Information

Aerogel Microparticles from Oil-in-Oil Emulsion Systems

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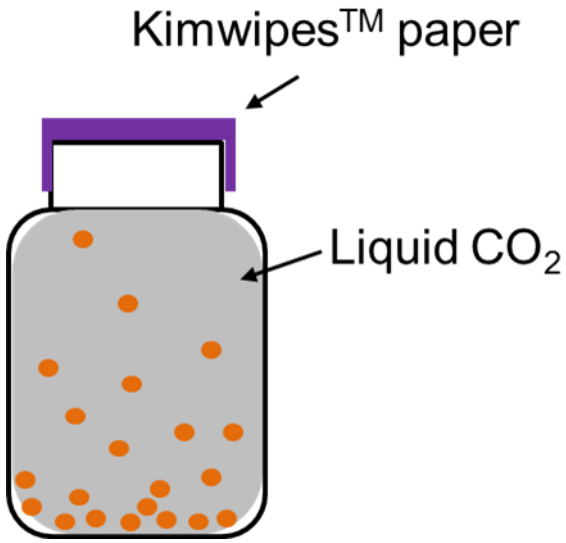
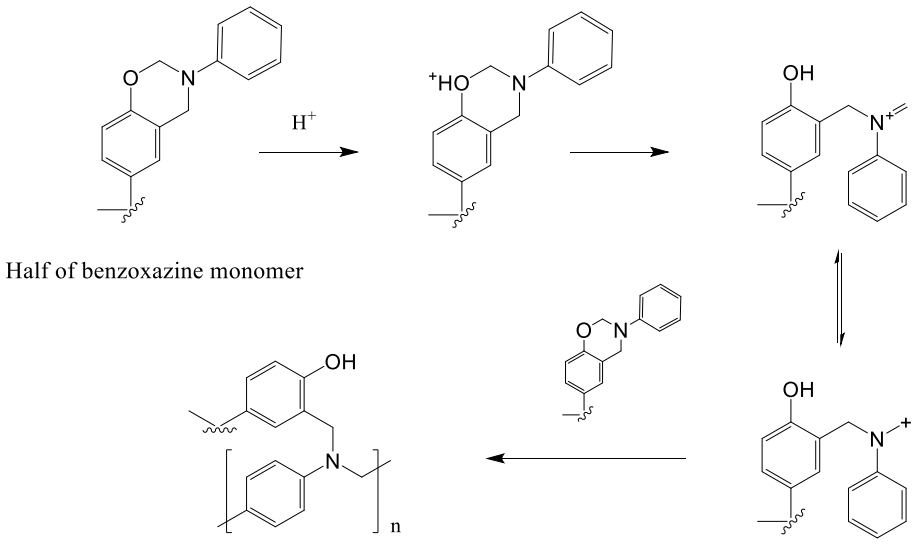


Figure S1. Container with gel micropaticles when filled with liquid CO2 in the vessel of supercritical dryer.

Note: In the supercritical drying process, acetone in the gel needs to be exchanged with liquid CO2 in the supercritical dryer. Neck of the container was covered by KimwipesTM paper. Liquid CO2 can flow into the container when the vessel is fulfilled by liquid CO2. In this process, the particles can be retained in the container because that pores of KimwipesTM paper are smaller than the diameter of the gel particles. After this step, the supercritical dryer was heated to 50 ℃ and waited for 60 min before pressure release.



Scheme S1. Mechanism of HCl-catalyzed polymerization of benzoxazine.[1]

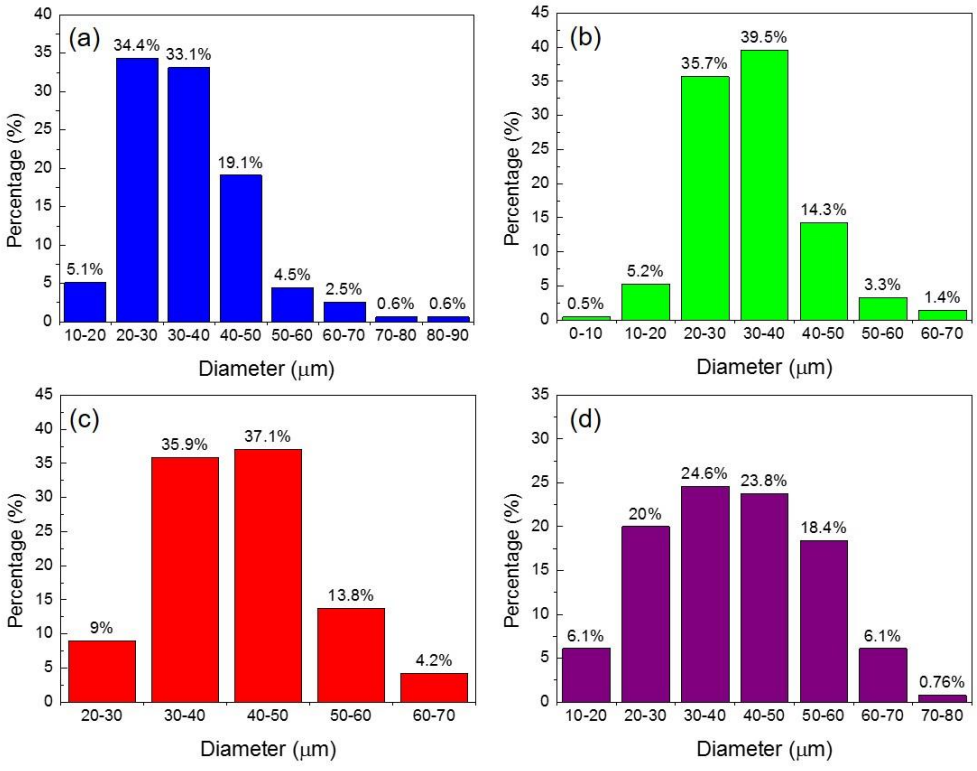


Figure S2. Diameter distribution of (a) polybenzoxazine gel microparticles, (b) polybenzoxazine aerogel microparticles, (c) polyimide gel microparticles, and (d) polyimide aerogel microparticles.

Table S1. Values of *i* and *n* for different microparticles.

	i	***n***
Polybenzoxazine gel microparticles	1	8
Polybenzoxazine aerogel microparticles	0	6
Polyimide gel microparticles	2	6
Polyimide aerogel microparticles	1	7

Table S2. Diameter and shrinkage of gel and aerogel specimens.

	D_0 (mm)	**D (mm)**	**δ_d**	**D_0' (μm)**	**D′(μm)**	**δ_d**
Polybenzoxazine	12	9.41	21.6%	34.6	32.7	5.5%
Polyimide	16	14.93	6.7%	41.8	40.0	4.3%

Reference

1. Mahadik-Khanolkar, S.; Donthula, S.; Sotiriou-Leventis, C.; Leventis, N. Polybenzoxazine Aerogels. 1. High-Yield Room-Temperature Acid-Catalyzed Synthesis of Robust Monoliths, Oxidative Aromatization, and Conversion to Microporous Carbons. *Chem. Mater.* **2014**, *26*, 1303-1317.