Synthesis, characterization, and adsorption properties of ionic liquid-modified hypercrosslinked polystyrene resin

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Supporting Information

Materials

Low crosslinked chloromethylated polystyrene beads was supplied by Xi'an Sunresin Technology Co., Ltd. (Shaanxi,China), its crosslinking degree was 6%, chlorine content was measured to be 17.3%, its Brunauer–Emmett–Teller surface area was 24.69 m^2/g with an average pore width of 16.2 nm. Macroporous adsorption resins Seplite D101 and Seplite AB-8 were also purchased from Xi'an Sunresin Technology Co., Ltd. (Shaanxi,China). Other chemicals were of analytical reagent grade. Anhydrous zinc chloride, nitrobenzene, acetone, hydrochloric acid, sodium hydroxide, imidazole, N,N-dimethylformamide and ethanol was purchased from Tianjin Chemical Reagent Co., Inc. (Tianjin, China), and distilled water was prepared in our laboratory. HPLC-grade methanol was from Shandong Yuwang Industrial Co., Ltd. (Shandong, China). Standard EGCG and EC were bought from Chengdu preferred Biological Technology Co., Ltd. (Sichuan, China) with a purity of 98.0%.

Adsorption experiments

Kinetic studies were conducted using about 1.000 g of the resins and 250 mL of EGCG or EC aqueous solutions with 288 K. The flasks were shaken under 100 rpm. Subsequently, 0.5 mL each of EGCG or EC aqueous solutions was sampled at preset intervals to analyze the residual concentration. The adsorption capacities of the resins to EGCG and EC were evaluated using the following equation:

$$q_t = (C_0 - C_t) \times \frac{V_0}{W}$$

where q_t is the adsorption capacity (mg/g) toward EGCG and EC at contact time t; C_0 and C_t are the initial and contact time t concentrations of the EGCG and EC solutions (mg/L), respectively; V_0 is the adsorption solution volume (mL); and W is

the dry weight of the resins (g).

Equilibrium adsorption isotherms were obtained at three different temperatures. Approximately 0.2000 g of resin sample was mixed with 50 mL of a series of EGCG or EC aqueous solutions. The concentrations of EGCG and EC were set at 400 mg/L to 900 mg/L and 500 mg/L to 1000 mg/L, respectively, with 100 mg/L interval. The flasks were shaken under 100 rpm until the adsorption process reached equilibrium. The residual concentrations were then determined by HPLC[26].

Adsorbate analysis

EGCG and EC concentrations in aqueous solution were analyzed using an Agilent 1200 series apparatus equipped with a binary pump, diode array detector, and manual injector, and then separated on a SinoChrom ODS-BP column (250 mm × 4.6 mm, i.d., 5 μm). The mobile phase consisted of methanol (A) and 1% acetic acid in water (B) in a linear gradient program as follows: 85% to 70% (B) at 0 min to 5 min, 70% (B) at 5 min to 15 min, 70% to 62% (B) at 15 min to 25 min, and 62% (B) at 25 min to 35 min at a flow rate of 1.0 mL/min. Column temperature was controlled at 30 °C. The detection wavelength was 280 nm and injection volume was 20 μL. Two well-fitted regression equations, $y = 22822\,x - 208.8$ ($R^2 = 0.998$) and $y = 11.96\,x + 22.47$ ($R^2 = 0.999$), were obtained, where y is the peak area of EGCG or EC and x is the EGCG or EC concentration.

Figure S1 N_2 adsorption and desorption isotherms of the imidazole modified hypercrosslinked resins HP-IL04, HP-IL08, HP-IL12, HP-IL16 and HP-IL20.

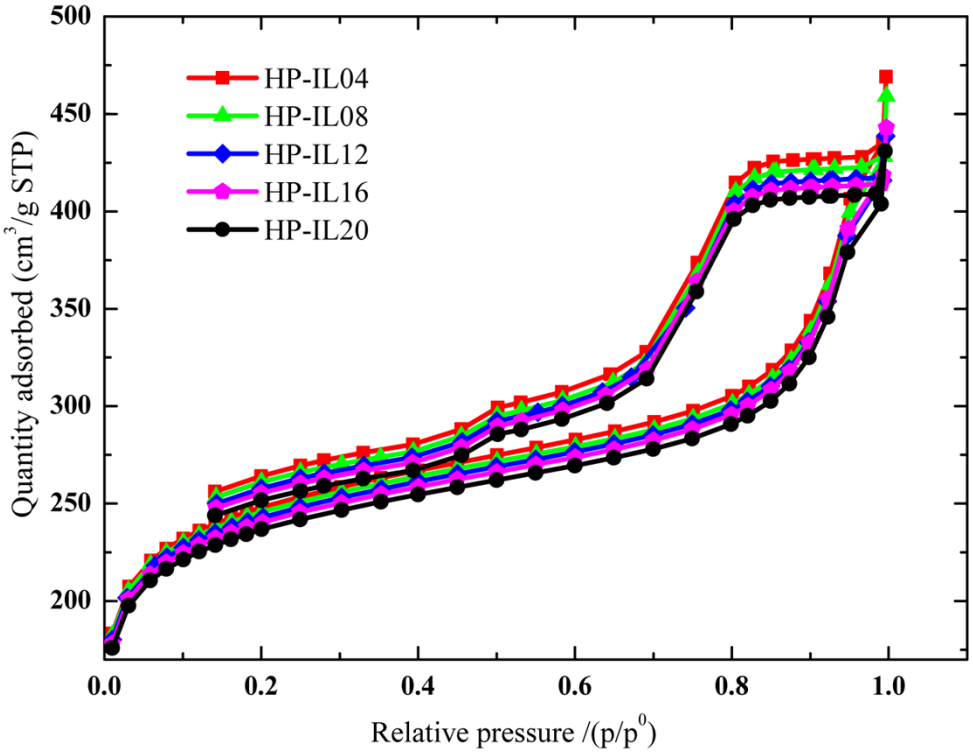

Figure S2 The pore diameter distribution of imidazole modified hypercrosslinked resins HP-IL04, HP-IL08, HP-IL12, HP-IL16 and HP-IL20.

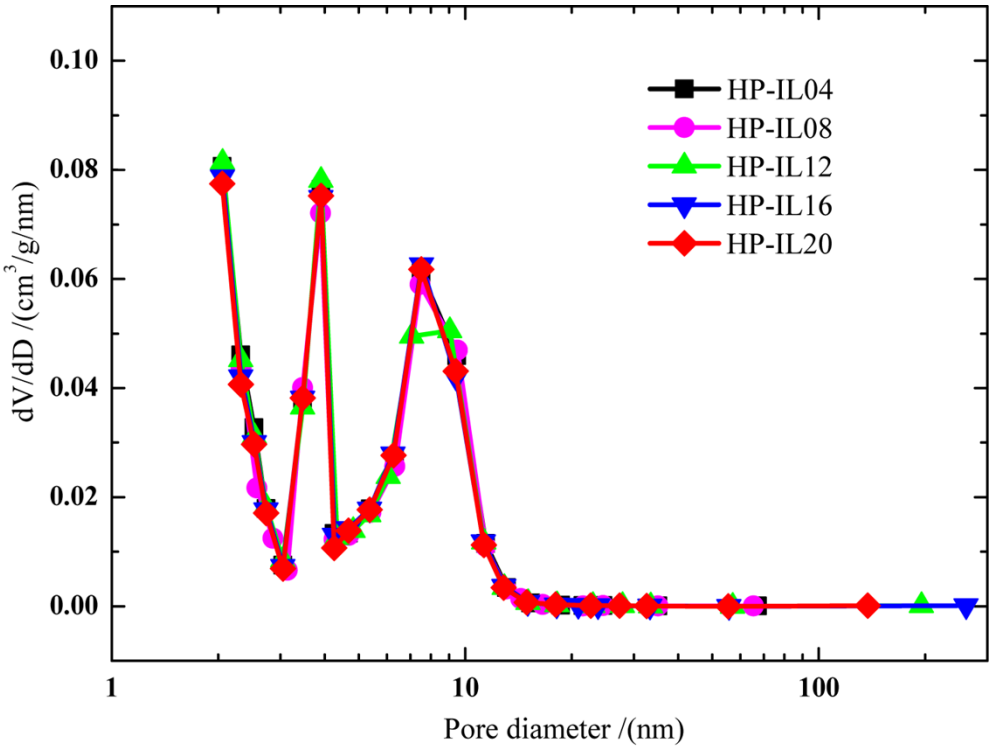

Figure S3 FTIR spectra of the imidazole modified hypercrosslinked resins HP-IL04, HP-IL08, HP-IL12, HP-IL16 and HP-IL20.

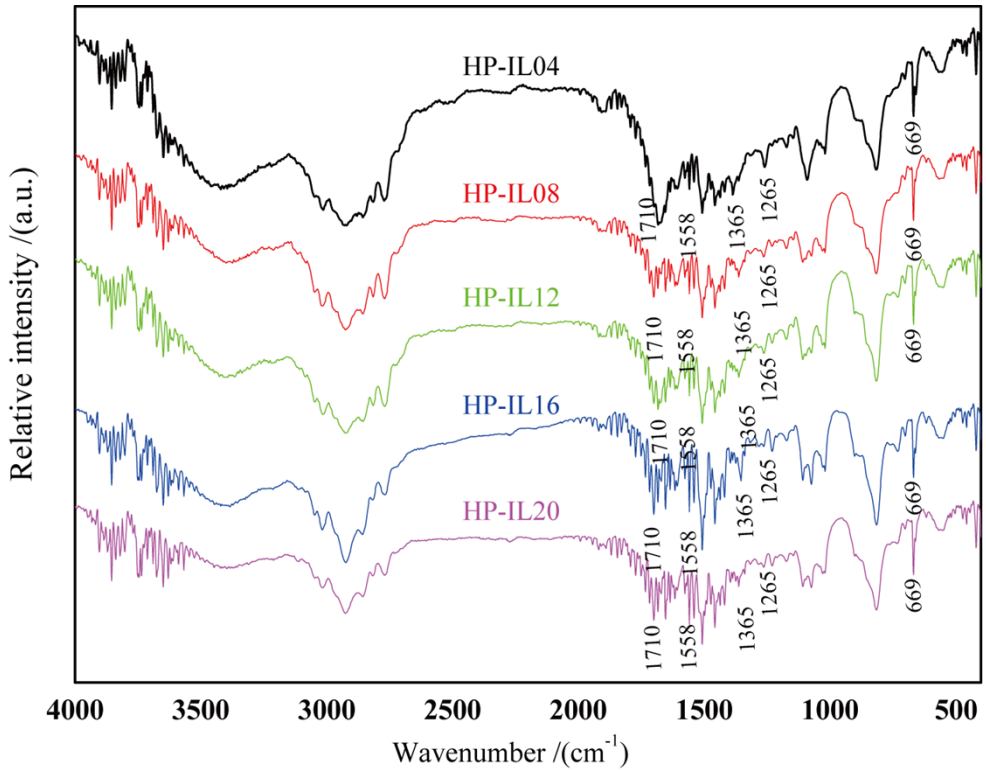

Table S1. Freundlich isotherm parameters of EGCG and EC onto MARs.

		EGCG			EC		
		K_F/ [(mg/g)(L/mg)$^{1/n}$]	1/n	R^2	K_F/ [(mg/g)(L/mg)$^{1/n}$]	1/n	R^2
HP-IL16	288 K	139.77	0.68	0.9904	137.00	0.38	0.9227
	298 K	135.64	0.58	0.9933	135.64	0.31	0.8907
	308 K	134.29	0.49	0.9907	145.47	0.32	0.8850
D101	288 K	77.48	0.41	0.9493	76.71	0.62	0.9873
AB-8	288 K	72.97	0.47	0.9479	68.03	0.57	0.9847