Supporting Information of the manuscript entitled

Deep Eutectic Assisted Synthesis of Carbon Adsorbents Highly Suitable for Low-Pressure Separation of CO₂/CH₄ Gas Mixtures

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Figure S1: ¹H NMR spectra of DES125 (a, b), DES150 (c, d) and DES200 (e, f), in neat form (a, c and d) and diluted in water for a DES content of 67 (b), 66 (d) and 64 wt% (f).



Figure S2: SEM micrographs revealed (a) the formation of non-interconnected pores resulting from polycondensation of DES100 with formaldehyde using 140 mg/mL Na₂CO₃ as catalysts (a similar structure was also found after polycondensation of DES100 with formaldehyde using 140 mg/mL Na₂CO₃ as catalysts) and (b) the formation of a heterogeneous bicontinuous structure resulting from polycondensation of DES150 with formaldehyde using 220 mg/mL Na₂CO₃ as catalysts (a similar structure was also found after polycondensation of DES150 with formaldehyde using 220 mg/mL Na₂CO₃ as catalysts (a similar structure was also found after polycondensation of DES100, DES175 and DES200 with formaldehyde using 180, 220 and 220 mg/mL Na₂CO₃ as catalysts, respectively).



Figure S3: SEM micrograph showing a detail of the heterogeneous structure of the carbon C150-220 that resulted from polycondensation of DES150 with formaldehyde using 220 mg/mL Na_2CO_3 as catalysts and subsequent carbonization.



Figure S4: Isosteric heats of CO₂ adsorption in C125-220 (blue) and C200-180 (red).



Table S1: Details on reagent concentrations used for preparation of carbons that exhibited anon-homogeneous structure. The name of the samples follows the same trend than thatdescribed for homogeneous samples in Table 1

Sample	R + H + T in DES ^a (mmols)	F ^a (mmols)	F/(R + H) molar ratio	Na₂CO₃ (mg/mL)	Na₂CO₃ (μL)
C100-220	1.00 + 1 + 1	4.0	2.0	220	31
C100-180	1.00 + 1 + 1	4.0	2.0	180	31
C100-140	1.00 + 1 + 1	4.0	2.0	140	31
C100-100	1.00 + 1 + 1	4.0	2.0	100	31
C125-140	1.25 + 1 + 1	4.5	2.0	140	35
C125-100	1.25 + 1 + 1	4.5	2.0	100	35
C150-220	1.50 + 1 + 1	5.0	2.0	220	40
C150-100	1.50 + 1 + 1	5.0	2.0	100	40
C175-220	1.75 + 1 + 1	5.5	2.0	220	44
C175-100	1.75 + 1 + 1	5.5	2.0	100	44
C200-220	2.00 + 1 + 1	6.0	2.0	220	47
C200-100	2.00 + 1 + 1	6.0	2.0	100	47

^a R stands for resorcinol, H for 4-hexylresorcinol, T for tetraethyl ammonium chloride and F for formaldehyde