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The Wolfson Department of Chemical Engineering

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6FDA-HAB polyimide–amide-based Carbon Membranes for CO_2/N_2 Separation

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MSc Seminar

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Abstract: Membrane-based post-combustion CO_2 capture relies on the solution-diffusion mechanism, governed by the coupled effects of gas solubility (sorption selectivity) and diffusivity (molecular sieving). Carbon molecular sieve (CMS) membranes derived from polymeric precursors can enhance both to surpass the Robeson upper bound and resist CO_2 plasticization, yet practical outcomes remain limited by two bottlenecks: (i) pore fusion and collapse during high-temperature pyrolysis, and (ii) insufficient synergy between accessible fractional free volume (FFV) and CO_2-philic sites. To overcome these barriers, we design and synthesize a rigid, non-coplanar 6FDA-HAB polyimide-amide (PI-amide) precursor (M_w = 49,431 g/mol) via two-step chemical imidization and fabricate tailored CMS membranes through controlled pyrolysis at 600-800 °C (CMS-600, CMS-700, and CMS-800). Bulky -$C(CF_3)_2$- linkages in 6FDA disrupt chain packing to boost FFV and generate diffusion channels during pyrolysis, while the rigid ortho-hydroxyl biphenyl backbone of HAB and interchain amide hydrogen bonding ensure high char yields (48.7-52.7%) and suppress pore collapse. Multi-scale characterizations (1H NMR, FTIR, GPC, TGA, XRD, Raman, XPS, and physisorption) reveal that elevating the pyrolysis temperature yields more ordered sp^2-hybridized turbostratic carbon (sp^3/sp^2 decreasing from 0.52 to 0.30 and I_{D1}/I_G from 2.82 to 2.66) with narrowed d-spacing, retained CO_2-philic pyrrolic-N sites, and rich microporosity (BET surface area of 696 m^2/g). Decoupling solubility (S_i) and diffusivity ($D_i = P_i/S_i$) via competitive Langmuir and dual-mode models in pure- and mixed-gas (10/90 CO_2/N_2 and 35/65 CO_2/CH_4) tests demonstrates that preferential CO_2 sorption in ultramicropores competitively excludes N_2, raising CO_2/N_2 solubility selectivity from 6.73 (pure gas) to 16.18 (mixed gas) alongside a diffusivity selectivity of 5.31 in CMS-800. Consequently, the optimized membrane achieves a CO_2 permeability of 10,546 Barrer and a CO_2/N_2 selectivity of 78.86 (higher than the 2019 Robeson bound) with 88-90% thermal reheat stability, establishing a robust precursor-to-carbon molecular design strategy for next-generation carbon capture.