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Synthesis and gas transport properties of substituted bicyclodianhydride-based polyimides

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Three novel polyimides were synthesized from a 9,10-dimethyl-2,6(7)-diaminotriptycene (T) with a commercially available *bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic dianhydride* (BC) and its halogenated derivatives. The non-halogenated T-BC polyimide derivative was made as a reference material to evaluate the effect of the halogen groups in T-$BCCl_4$ and T-$BCBr_4$ on its gas transport properties. Pure-gas permeability coefficients of He, H_2, N_2, O_2, CH_4, and CO_2 were measured at 35 °C and 2 atm. The BET surface area based on nitrogen adsorption at 77 K of T-BC was 570 m^2 g-^{1} while those of tetrachloro-functionalized T-$BCCl_4$ and tetrabromo-functionalized polyimide T-$BCBr_4$ were reduced significantly to 340 and 30 m^2 g-^{1}, respectively. The decrease in BET surface area in the halogenated polyimides resulted from reduction in their pore volumes relative to that of T-BC due to occupation of free volume space by the halogens. The freshly prepared T-BC membrane had a pure-gas O2 permeability of 66 Barrer and O_2/N_2 selectivity of five. The permeability decreased significantly by replacing the hydrogen groups by the chloro- or bromo groups in the cycloaliphatic dianhydride building block. For example, the permeability of O_2 decreased by 3-fold from 66 in T-BC to 20 Barrer in T-$BCBr_4$, while the permeability of nitrogen was reduced from 13 to 3.4 Barrer. As expected for lower permeability polymers, the O_2/N_2 selectivity increased concurrently from 5 in T-BC to 6 in T-$BCBr_4$. Long-term testing over 365 days resulted in only ~ 15% loss in gas permeability's and without significant changes in selectivity's, which demonstrated that these polyimides were resistant to physical aging. These combined results suggest that BC-based polyimides are promising candidate membrane materials for gas separation applications.

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