

A Novel Class of CO₂ Gas Separation Membranes based on Organic Ionic Plastic Crystals

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Membrane synthesis

Polyvinylidene fluoride (PVDF) mats were made using electrospun PVDF fibres as described previously.¹ Pellets of 12.5mm diameter were punched from the resulting mats, for use in the membrane. The OIPCs were synthesized and characterised following our previously reported procedures¹⁻³ and stored under argon prior to use.

[C₂mpyr][BF₄]/PVDF membranes were made by preparing a solution of 1:5 w/w OIPC in methanol and casting the solution onto the PVDF mats. Typically, the mass of the PVDF fibres was 4mg and the mass of OIPC used was 19mg, giving a total dry mass of 23mg after removal of the methanol. The membranes were dried in a vacuum oven overnight at 55°C before pressing. The thickness of the membranes was ca. 150µm, prepared by pressing the pellet on a 1 ton press for half an hour at 70°C.

[P₁₂₂₁₄][PF₆]/PVDF membranes were made following the same procedure but using ethanol as the solvent, followed by pressing the pellet on a 1 ton press for half an hour at room temperature.

Gas permeability measurements

Permeation measurements were performed using apparatus designed based on ref 4. The gas contacting surfaces were all stainless steel with Swagelok fittings and Viton o-rings. The feed- and permeate-side pressure transducers were both Keller Series 35X HT absolute-range transducers with a 4-20mA output and a compensated temperature range of 20-120°C. The feed tank was 2200mL with a 0-10 Bar(abs) transducer and the permeate tank was 35mL with a 0-1Bar(abs) transducer. A type-k thermocouple was used to measure the temperature, and the vacuum used was 4x10⁻²mBar. This apparatus was housed in a Binder BF 115 Model incubator with temperature control from 5°C above room temperature to 100°C, with 0.2°C accuracy. Transducer and thermocouple outputs were recorded using an MCC USB-2404-UI model data acquisition unit with 24-bit resolution.

In a typical experiment, the sample was mounted in the sample holder which was positioned between the feed and permeate tanks. Vacuum was then drawn on both sides of the membrane in preparation for the first permeation experiment. The apparatus was then allowed to equilibrate at the desired experimental temperature and left under vacuum overnight before introducing the penetrant gas and beginning the experiment.

A single gas was tested at a time. Between experiments using the same gas, a time greater than or equal to ten times the 'time-lag' was allowed to elapse,^{5,6} during which time a vacuum was maintained on both sides of the membrane. Where the gas type was changed between permeation experiments, the membrane was kept under vacuum overnight before the next permeation experiment was started.

Differential Scanning Calorimetry

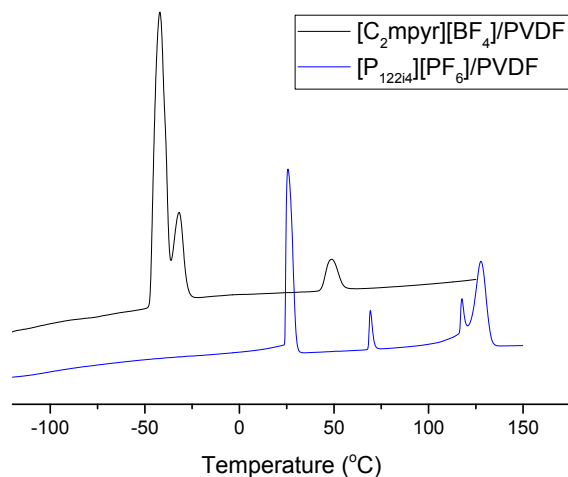


Figure S1. DSC traces for the two OIPC/PVDF membranes.

DSC traces were recorded using a Mettler Toledo DSC1 STAR[®] System at a scan rate of 10 degrees per minute. The scans shown were obtained after one heating and cooling cycle. The heating and cooling cycles for the second and third scans showed no changes.

References

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