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Permeation and sorption of gases in mixed matrix membranes based on hyperbranched polyimides and hollow silica microspheres

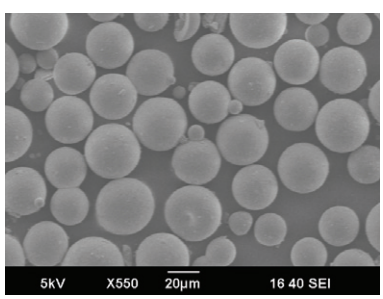
M. Lanc, K. Friess*, V. Hynek, P. Sysel, M. Celisova, F. Stepanek et al

Institute of Chemical Technology Prague, Czech Republic

Further progress in field of membrane separations requires new ideas and approaches to substantially overcome well-known limitations of permeability-selectivity trade-off, known as the Robeson plot [1]. New successful separation membranes require increasingly sophisticated membrane materials with improved performance. The preparation and characterization of mixed matrix materials (MMM) based on a polymer matrix and organic or inorganic additive is a rapidly developing field of investigation that has made possible the tailoring of new materials combining the properties of both components. The success of matrix mixed materials depends greatly on a proper balance between properties of the polymer matrix and that of the additives (chemical composition, morphology, weight (volume) ratio, compatibility [2].

Polyimides, rigid polymers with a high glass transition temperature, exhibit very good chemical, mechanical and dielectric stability at temperatures from -150 to 250 °C. These polymers are mostly used in (micro)electronics, aircraft industry and as polymeric separation membranes [3]. Higher selectivity of such materials is “compensated” by their relatively lower permeability [4]. Therefore, the aim of this work is preparation of HPI based mixed matrix membrane separation membrane with same selectivity like pure HPI but with improved permeability.

In this work, preparation and transport properties of mixed matrix membranes based on hyperbranched polyimides (HPI) and hollow silica spheres is presented. HPI were prepared from 4,4´-oxydiphthalic anhydride and 4,4´,4´´-triaminotriphenylmethane [5] Hollow silica microspheres (HSF) have been prepared via the soft template route by the hydrolysis and polycondensation of alkoxysilane precursors, namely tetra-ethoxysilane (TEOS) [6]. Octylamine has been used as a liquid template, which was sacrificed during the calcining step. The mean particle size can be systematically varied from a few 100’s nm to 10’s microns depending on the preparation conditions and the surface properties (namely hydrophobicity) of the formed silica hollow spheres can be further modified e.g. by silanisation to ensure compatibility with the polymer matrix. The size distribution of HSF was characterised by light scattering and the their morphology and internal structure by a combination of SEM (Fig. 1), TEM and laser scanning confocal microscopy.



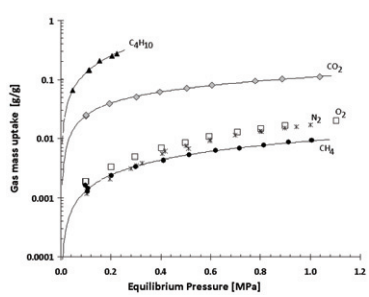


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Figure 1: SEM image of hollow silica microspheres (left) and corresponding

equilibrium gas sorption in this material at 298.15K (right).

Permeation tests of pure HPI and HPI-HSF based mixed matrix membranes with different content of HSF (0, 10 and 30 wt %), carried out in the time-lag mode at 1 bar and at 298.15K, provided data on the permeability and the diffusion coefficient for oxygen, nitrogen, carbon dioxide, methane and butane. Sorption experiment of pure HSF (Fig. 1) and of all membrane samples were performed independently at same temperature and at the absolute pressures ranging from zero to 10 bar using self-developed sorption apparatus [7,8]. Obtained solubility coefficients were compared with indirectly evaluated ones (by solution-diffusion model) from permeation experiments.

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Keywords: Mixed matrix membranes, Hyperbranched polyimides, Hollow silica spheres, Gas permeation/sorption