BOOK OF ABSTRACTS ▶▶▶

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"Greening Energy to Shape a Sustainable Future"

September 12-16, 2022 | Porto, Portugal

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Development of Polyethersulphone Mixed Matrix Zeolite Membranes Functionalized with Ionic Liquids and Deep Eutectic Solvents for Co_2 Separation

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ABSTRACT
Mixed matrix membranes (MMM) combine the flexibility of polymers and the strength and durability presented by inorganic solids. In an economically point of view, the advantages of membrane separation are low capital investment and space requirements, high process flexibility and lower energy consumption, helping for a more cost-effective separation process and providing a high separation degree. The molecular sieves based on nano-sized silicoaluminophosphates (SAPO) appear as one of the main materials in MMM for gas separation because the pore size of chabazite (CHA) (0.38nm) is near the kinetic diameter of gases like H_2 (0.29nm), CO_2 (0.33nm), N_2 (0.36nm), CO (0.37nm), CH_4 (0.38nm) and reduced crystal size improves the dispersion and decreases interfacial defects.
Besides, the addition of ionic liquids (IL) or Deep Eutectic Solvents (DES) with high affinity and selectivity to CO_2, onto the particle surface and then dispersing it in a polymer membrane can enhance the separation characteristics, resulting in better permeation and selectivity properties.

Keywords: Mixed matrix membranes; CO_2 separation, ionic liquids; deep eutectic solvent; permeability, selectivity.

1. INTRODUCTION
Matrix mixed membranes (MMM) containing zeolites have potential for separating CO2 owing to their superior thermal, mechanical, and chemical stability, good erosion resistance, and stability at high CO2 pressures. Zeolites have inorganic crystalline structures with uniform-sized pores of molecular dimensions. The molecular sieves based on silicoaluminophosphates (SAPO) (0.38nm) is near the kinetic diameter of gases like H_2 (0.29nm), CO_2 (0.33nm), N_2 (0.36nm), CO (0.37nm), CH_4 (0.38nm). The production of dopped SAPO-34 with transition metals (Fe, Ni, Co, Mn), named MeAPSO-34, leads to a zeolite with higher negative surface charge [1]–[6].
The use of Ionic Liquids (IL) and Deep Eutectic Solvents (DES) arises to improve the zeolite-polymer matrix interaction promoting a link between the zeolite surface (inorganic) and the polymer matrix (organic) due to its organic-inorganic dual nature. Those additives can be used via direct addition in the casting solution or through a post-impregnation process, and reportedly reduces interfacial voids, which increase the separation performance of MMMs (Asghari et al., 2018; Hu et al., 2018; Huang et al., 2015; Hudiono et al., 2011).

2. MATERIALS AND METHODS
The synthesis of the SAPO-34 samples, a gel having the molar composition 1 Al_2O_3; 1 P_2O_5; 0.6 SiO_2; 1.5 Morpholine; 0.5 TEAOH; 70 H_2O was prepared, aged for 24 hours under mixing and then dried at 90°C for 24 hours. The samples were prepared by adding water to dried gel in the proportion 1:1 and heated at 200°C for 24 hours.
20%w/w Polyethersulfone (PES) membrane was prepared using 20%w/w zeolite, based on the polymer mass, in NMP and mixed for 24 hours. The resulting solution was casted with a knife of 0.2 mm, then dried at 90°C for 8 hours and at 160°C for 24 hours under vacuum. The samples were characterized by gas permeation tests to evaluate the separation performance. The membranes tested were placed in a kitasato, and vacuum was applied for a period of 1 hour to remove the air from the pores. The additive was then introduced with a syringe through a septum and the membrane stays under vacuum for 1 hour. The membrane was then removed, and dried using a tissue paper.

3. RESULTS AND DISCUSSION
SAPO-34 was synthetized starting from the same molecular composition 1 Al_2O_3; 1 P_2O_5; 0.6 SiO_2; 1.5

Morpholine; 0.5 TEAOH; 70 H_2O, using the dry-gel synthesis following the proportion 1:1 in mass for 24 hours.

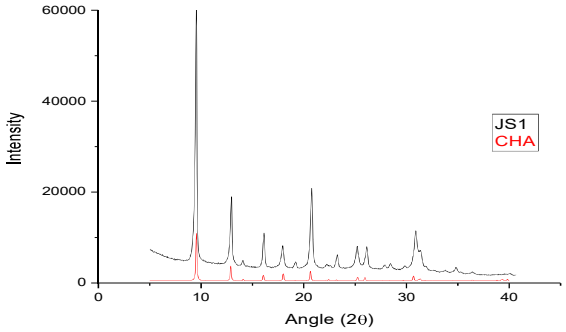

Fig. 1. XRD for JS1 sample.

The results presented in Fig. 1 showed that the SAPO-34 was obtained as presented in XRD when compared to a CHA standard x-ray dispersion analysis. The LDS analysis shows that the average particle size obtained was smaller, 0.198 µm.
The average value obtained for permeability were 17.40 GPU to N_2 and 55 GPU to CO_2 with CO_2/N_2 selectivity of 3.20, which can be related to values in literature. The initial tests using the DES ChCl/Urea 1:2 as a post-impregnant showed an increase between 4-12 times of CO_2/N_2 selectivity, specially decreasing the N_2 permeability post-impregnation.

4. CONCLUSION

It is stated for many authors that the use of mixed matrix membranes presents a great potential for further investigation and development in separation performance. The methodology for nano-sized SAPO-34 preparation was defined and the solid with the desired characteristics obtained to be used as filler in PES polymer membranes. The use of post-sealing impregnation can be a healing procedure for poor compatibility after synthesis. This work presents an innovation as preliminary results showing that 1:2 ChCl/Urea DES used as a post-sealing compound can reduce N_2 permeation and improve CO_2/N_2 separation performance by 4-12 times in neat PES membranes. Those results will be tested using PES/SAPO-34 membranes to verify if can also improve the separation performance in mixed matrix membranes.

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