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Fluorine-induced microporous silica membranes: Dramatic improvement in hydrothermal stability and pore size controllability for highly permeable propylene/propane separation

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## Abstract

A molecular sieving membrane was fabricated using triethoxyfluorosilane (TEFS), which contains Si-F bonds and is categorized as a pendant-type alkoxy silane. The hydrothermal stability and hydrocarbon (C<sub>3</sub>H<sub>6</sub>, C<sub>3</sub>H<sub>8</sub>) permeation properties were evaluated for TEFS membranes. When a fluorine-induced silica membrane had a Si-F bond in the amorphous structure, the reaction of steam and Si-F groups during steam treatment formed Si-OH groups, which slightly decreased the gas permeance. Even though gas permeance slightly decreased under a steam atmosphere, a TEFS membrane calcined at 350 °C had networks that were looser and more uniform than those of a conventional SiO<sub>2</sub>. In addition, the formation of adsorption sites (Si-OH groups) under steam treatment enhanced both interactions with the  $\pi$ -bonds (C=C double bond) of C<sub>3</sub>H<sub>6</sub> and the C<sub>3</sub>H<sub>6</sub>/C<sub>3</sub>H<sub>8</sub> permeation properties (C<sub>3</sub>H<sub>6</sub> permeance:  $2.2 \times 10^{-7}$  mol m<sup>-2</sup> s<sup>-1</sup> Pa<sup>-1</sup>, C<sub>3</sub>H<sub>6</sub>/C<sub>3</sub>H<sub>8</sub> permeance ratio: 42 at 35 °C). The hydrothermal stability was dramatically enhanced by calcination temperatures as high as 750 °C due to the presence of fewer Si-OH and Si-F bonds in the amorphous structure, although the network pore size of a TEFS membrane was the same whether it was calcined at 750 °C or at 350 °C.

**Keywords:** Amorphous silica; Fluorine; Hydrothermal stability; Affinity control; Gas separation

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