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POTS Modification on FAU Membrane for Oil/Water Separation

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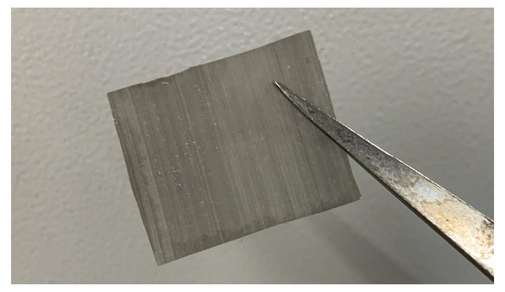
Introduction

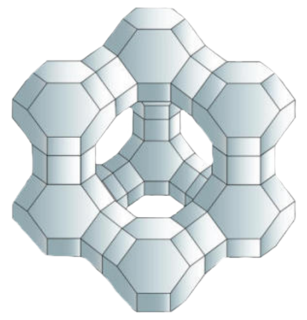
Oil spillage has caused serious environmental issues around the world. Therefore, separating oil and water has become a vital concern, and effective ways for separation need to be developed. Existing traditional ways, including gravity separation, skimming, flotation, absorption and electrocoagulation, are finding difficult to achieve low costs and high efficiency.[1][4] Therefore, interests in hydrophobic material have raised among scientists to separate oil/water as it can only allow oil to pass through but not water.

Numbers of coatings or membranes are found out to be effective for oil/water separation, including MFI and COF. [2][4] However, costs of synthesis, separation efficiency and flux have the potential to be further improved. The FAU zeolite membrane can bear high pressure and is suitable for post-synthesis to change its properties. [3] The stainless-steel wire net is a promising material with low cost and large pores. The large pores can provide a high flux for oil/water separation, whereas the low cost is crucial for industrial application. Thus, an interest has been drawn to a stainless-steel wire net supported FAU zeolite membrane for oil/water separation. However, FAU zeolite membrane is a hydrophilic material.

In order to change FAU zeolite membrane from hydrophilic to hydrophobic, post-synthesis may be a feasible solution. Perfluorooctyltriethoxysilane (POTS) is a chemical compound that is largely used to modify the surface of a material to gain a hydrophobic property, thus it may be able to modify the FAU zeolite membrane through post-synthesis to achieve a hydrophobic property without changing FAU crystalline structure. [4] The hydrophobic property can therefore allow FAU membrane to separate oil/water, hopefully remains the advantages of FAU membrane and stainless-steel wire net.

This research is conducted to test the capability for changing FAU membrane's hydrophilic property to hydrophobic for oil/water separation by using POTS post-synthesis modification.





QUICK FACTS OF FAU Zeolite Membrane

> **FAU** has an octahedron structure

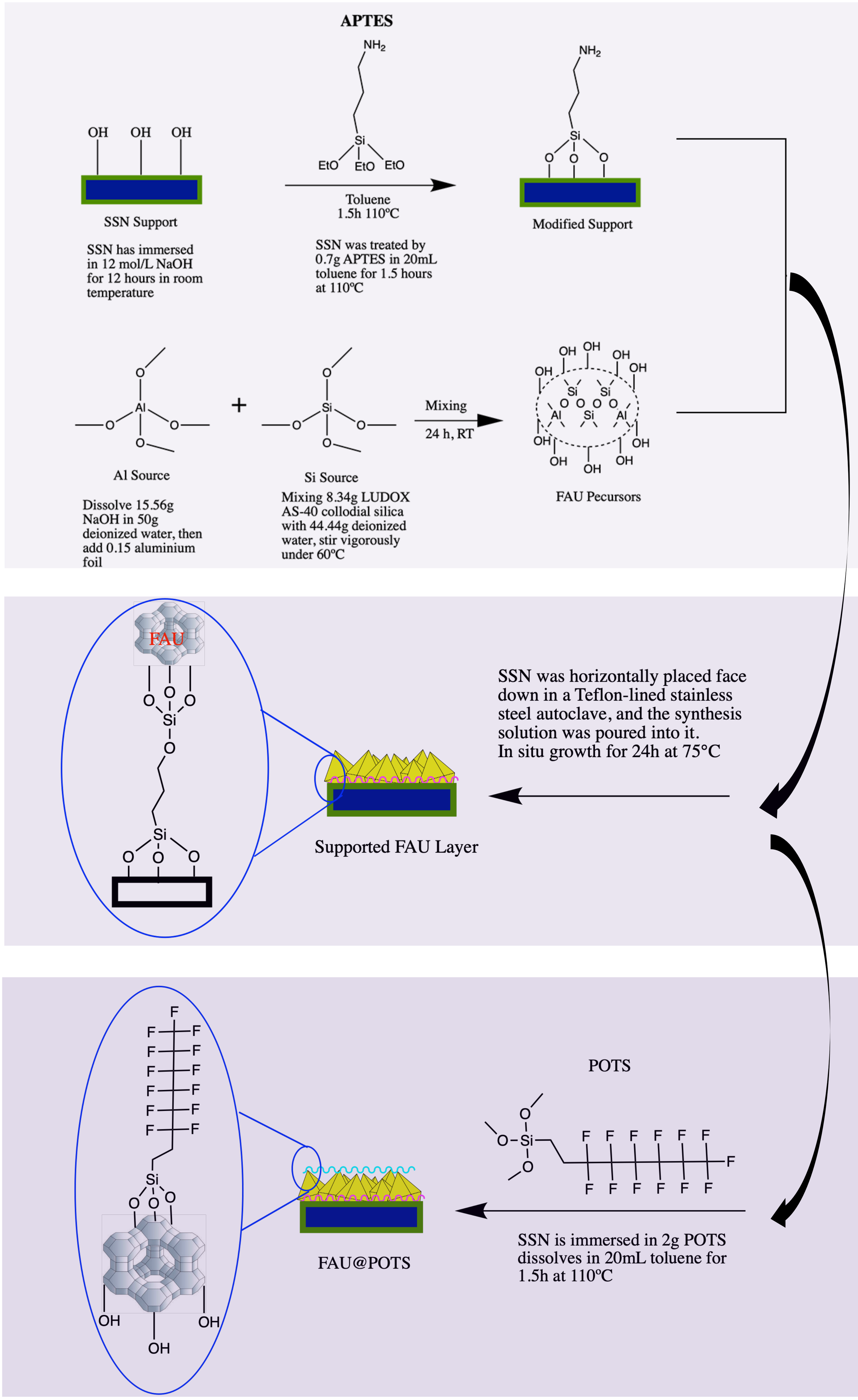
> **FAU** has a Si/Al molar ratio 1-1.5

> **FAU** has a pore size 0.74nm

> **Each** Si/Al in FAU is bonded to four oxygen atoms

METHOD-SYNTHESIS

Chemicals are used as received: LUDOX AS-40 colloidal silica (40% SiO2 in water, Aldrich) as Si source; aluminum foil (Fisher Scientific) as Al source; sodium hydroxide (>99%, Merck); 3-aminopropyltriethoxysilane (98%, Abcr); toluene (Acros); doubly distilled water; perfluorooctyltriethoxysilane (POTS, Energy chemical, 97%). Stainless steel net (SSN, 500 mesh, Local supermarkets, China) were used as supports.



METHOD-TESTING

Place the SSN FAU Membrane **in between** the two hollow tubes

- so liquids must past the membrane

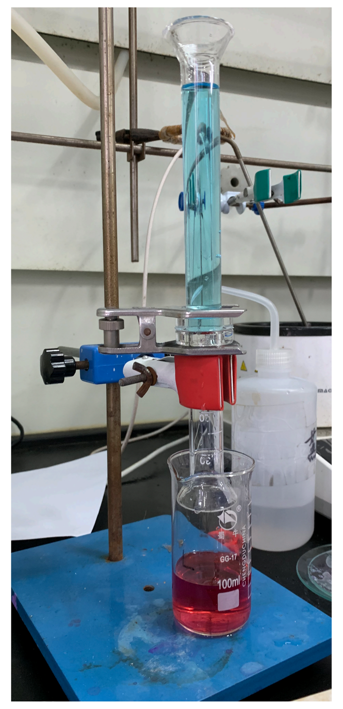
Use a clip to fix two the tubes in position

Pour a mixture of oil/water in

- Dichloromethane/Water
- Petroleum Ether/Water

$$Separation\ Efficiency = \frac{Percentage\ of\ Oil\ In}{Percentage\ of\ Oil\ Out}$$

$$Flux = \frac{Volume\ of\ Oil}{Area\ of\ the\ membrane \times Time\ Taken}$$



RESULTS

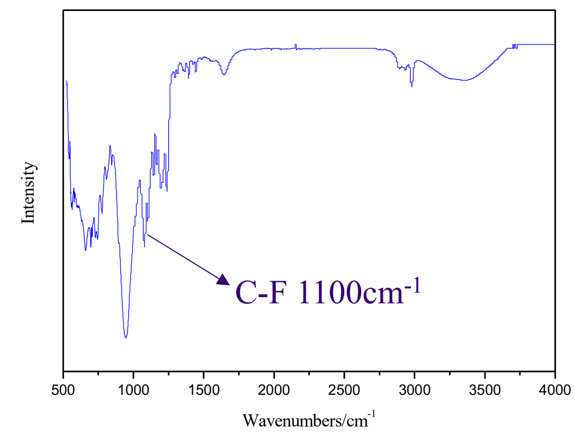


Fig. 1. Infrared spectrum for FAU powder with POTS modification

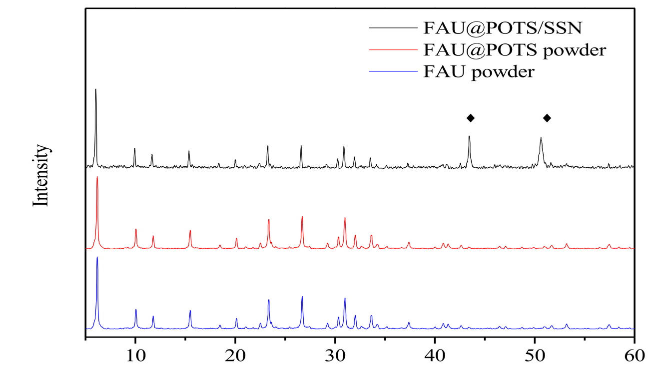


Fig. 2. X-ray diffraction pattern (XRD) for FAU powder, FAU powder with POTS modification, and FAU membrane with POTS modification on stainless-steel wire net

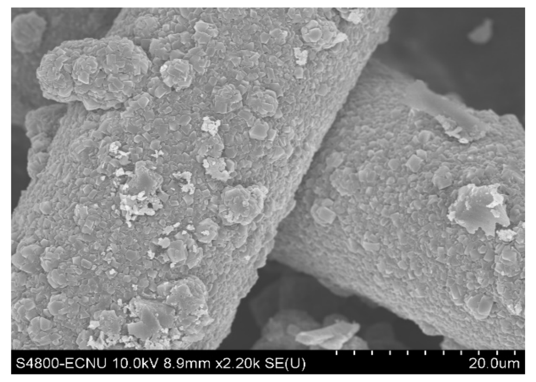


Fig. 3. Scanning electron microscope (SEM) for FAU membrane with POTS modification on stainless-steel wire net

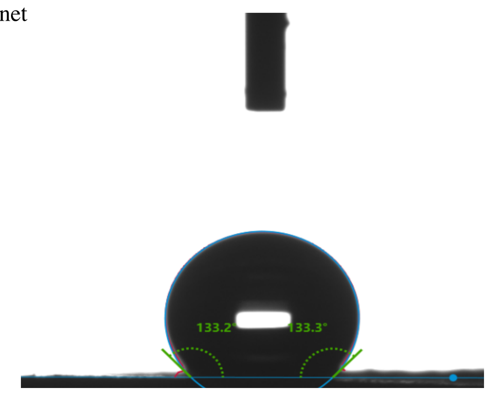


Fig. 4. Contact angles for FAU membrane with POTS modification on stainless-steel wire net

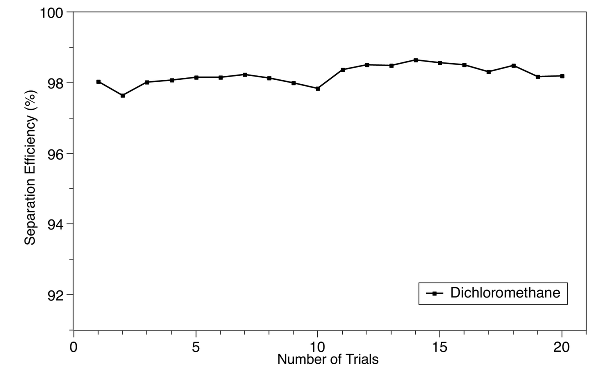


Fig. 5. Separation efficiency of FAU membrane with POTS modification on stainless-steel wire net to separate dichloromethane/water in 20 cycles

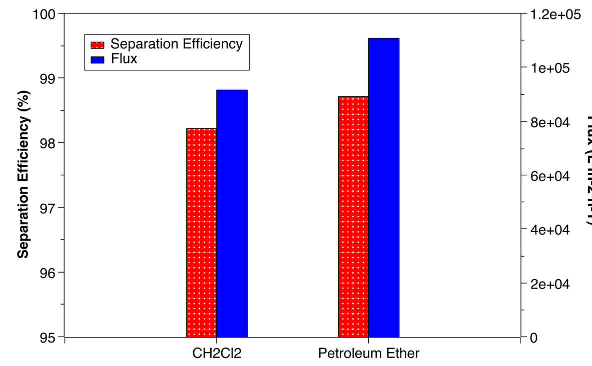


Fig. 6. Separation efficiency and flux of FAU membrane with POTS modification on stainless-steel wire net to separate dichloromethane/water and petroleum ether/water mixtures

DISCUSSION

From Fig.1 IR Spectrum
The infrared spectrum shows the C-F bond presents in the POTS modified FAU membrane. As FAU membrane nor APTES modification contain no C-F bonds, the C-F bond must come from POTS modification, thus the modification is successful.

From Fig. 2. XRD
According to the XRD graph in the results section and the another XRD graph from the published paper *Seeding-free synthesis of dense zeolite FAU membranes on 3-aminopropyltriethoxysilane-functionalized alumina supports*, it is clear that the type of zeolite membrane synthesized is FAU membrane as these two XRD graphs share similar peaks at same angles. Also, since the peaks on the XRD graph for FAU powder and FAU@POTS powder are very similar, the crystalline structure for FAU did not change by adding POTS modification, which means that it still has the properties of a molecular sieve membrane. By comparing the XRD graph for FAU@POTS and FAU@POTS/SSN, it can be said that the crystalline structure has not changed by using SSN as a support since the peaks from FAU@POTS remains on the FAU@POTS/SSN graph, and the two new peaks, however, indicate the structure of SSN.

From Fig. 3. SEM
The SEM graph shows that there are molecular sieve on the stainless-steel wire net.

From Fig. 4. Contact Angle
The contact angles, respectively 133.3° and 133.2°, are both higher than 90°. A contact angle higher than 90°, however, shows that this material is hydrophobic. Therefore, with POTS modification, the membrane reaches a hydrophobic characteristic.

From Fig. 5. Separation Efficiency in 20 Cycles
The flat line on the 20 cycles graph shows that the separation remains constant after a number of trials.

From Fig. 6. Separation Efficiency and Flux

- Two kinds of oil/water mixture were used to test the separation efficiency and flux using the equipment and methods described in the method-testing part. Dichloromethane has a higher density than water (1.324g/mL), which means that it will sink down in the mixture; petroleum ether, however, has a lower density than water (0.6500g/mL), which means that it will float on the mixture. Both oil/water mixtures work well with the membrane, thus this membrane is capable to separate oil that has lower/higher density than water.
- The average separation efficiency for both dichloromethane and petroleum ether is higher than 98%.
- Average flux for dichloromethane and petroleum flux , respectively, are 91,500 Lm-2h-1 and 111,000 Lm-2h-1.

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