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Synthesis and Characterization of a Novel Hydrophobic Membrane: Application for Seawater Desalination with Air Gap Membrane Distillation Process

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1. Introduction

Ceramic membranes are usually prepared from metal oxides like alumina, zirconia, titania. The development of membrane processes is generally limited because the price of the commercial membranes is too expensive, which is particularly true for the inorganic membranes. One of the challenges for future development of the inorganic membranes consists to prepare membranes of low cost made with natural non-expensive material (Khemakhem, & al., 2009; Saffaj & al., 2006). Rapid development and innovation have already been realized in this area (Cot, L.; 1998). Clay minerals are a well-known class of natural inorganic materials, with well-known structural adsorption, rheological and thermal properties (Brigatti & al., 2006; Wang & al., 2007). Research on clay as a membrane material has concentrated mainly on pillared clays (Tomul & al., 2009). Studies of membranes prepared entirely from natural clays have just started (Mao & al., 1999). These materials originally have a hydrophilic character due to the presence of the surface hydroxyl (-OH) groups, which can link very easily water molecules (Larbot& al., 2004; Picard & al., 2004).

Modification of membrane material and/or membrane surfaces has considerable influence on separation characteristics. Modification of the membrane materials surface is done to increase its hydrophilicity. The technique of modification normally involves the use of hydrophilic polymers or copolymers (Zhao & al., 2010; Alias & al., 2008), blending of hydrophilic or charged polymers with hydrophobic polymers (Mohd Norddin & al., 2008; Bolong & al., 2009), grafting of polymers (Nishizawa & al., 2005; Zdyrko & al., 2006) and the surface modification of the membrane itself (Hu & al., 2008; Yu & al., 2008). Grafting process, leading to the increase of the hydrophobic properties, can be performed by reaction between –OH surface groups of the membrane and ethoxy groups (O–Et) presented in organosilane compounds (Krajewski & al., 2004 Faibish & Cohen 2001). The grafting process leads to a monomolecular layer of organosilane compound on the membrane surface (Yoshida & Cohen 2003; Schondelmaier & al., 2002). This chapter describes the development of Novel mesoporous hydrophobic membrane obtained by grafting of fluoroalkylsilane onto microfiltration layer surface based on Tunisian clay. Ceramic membranes can be described by an asymmetric porous material formed by a macroporous support with successive thin layers deposited on it. Paste from Tunisian silty marls referred M11, is extruded to elaborate a porous tubular configuration used as supports. The support heated at 1190°C, shows an average pore diameter and porosity of about 9.20µm and 49%, respectively. The elaboration of the microfiltration layer based on Tunisian clay referred JM18, is performed by slip-casting method. The heating treatment at 900°C leads to an average pore size of 0.18µm. The obtained membrane was surface modified to change their hydrophilic character into hydrophobic one by the grafting of the triethoxy-1H,1H,2H,2H-perflluorodecylsilane C₈F₁₇(CH₂)₂Si(OC₂H₅)₃. The following grafting parameters were studied: the concentration of fluoroalkylsilane (C8) compound in the grafting solution and the time of grafting. The resulting products were investigated using Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA) and ²⁹Si CP/MAS NMR spectra. The new surfaces were examined by water contact-angle measurements and the water flux through the grafted membrane was also measured. The new hydrophobic membrane seems to be promising in the field of membrane distillation. High salt rejection rates higher than 99% were obtained for modified MF ceramic clay membrane. Air gap membrane distillation (AGMD) is a low cost process very efficient to produce fresh water from seawater.

2. Membrane preparation

2.1 Support shaping and characterisation

For this study, the supports were prepared from Tunisian silty marls (M11). The chemical composition of these materials is shown in Table 1.



Table 1. Composition of silty marls (M11).

The chemical analysis reveals that this kind of silty marls is essentially formed with a great amount of silica and calcium oxide. Fig. 1 presents the XRD patterns of raw silty marls, it shows that quartz (Q) is the main crystalline mineral present in this powder.



Fig. 1. X-ray diffractograms of the silty marls sample at different temperatures (Q = quartz, Ca = calcite, Ch = chlorite, D = dolomite).

The particle size analysis of the powder after crushing for 2 h with the assistance of a planetary crusher at a rate of 250 revolutions/min and calibrated with 100 mm was determined using a Particle Sizing Systems (Inc. Santa Barbara, California, USA Model 770 AccuSizer). The particle diameters range varied from 0.5 to 54 mm (Fig. 2).



Fig. 2. Silty marls (M11) particle size distribution.

Plastic pastes are prepared from ceramic powder of silty marls mixed with organic additives:

- 4% (w/w) of Amijel: pregelated starch, as plasticizer (Cplus 12072, cerestar).
- 4% (w/w) of Methocel: cellulose derivative, as binder (The Dow Chemical Company).
- 8% (w/w) of starch of corn as porosity agent (RG 03408, Cerestar).
- 25% (w/w) of water.

The rheological properties must be studied to obtain a paste allowing shaping by extrusion process [18]. Fig. 3 shows the different configurations of tubes extruded in our laboratory (two monochannel of different diameter and one multichannel tube).



Fig. 3. A photograph of variety of configurations of porous ceramic supports.

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed with simultaneous DSC-TGA 2960 TA instrument. The sample was heated at room temperature to 1250 8C at a rate of 5 8C/min under static atmospheric conditions. Two endothermic peaks were detected (Fig. 4).



Fig. 4. Thermal analysis curve: DSC and TGA for silty marls powder.

First peak appears at 51.38 8C, due to a weight loss of 1.81% of the initial weight. It corresponds to the departure of water (moisture or adsorption) due to attraction on the surface of the sample and zeolitic water inserted between the layers or in the cavities of the crystalline structure. Second peak which maximum appears toward 749.87 8C corresponds to the dehydroxylation.

Sintering experiments of the support were carried out in air. Two steps has been realised: the first for the elimination of organic additives at 250 °C, and the second for the sintering at 1190 °C. The temperature-time schedule not only affects the pore diameters and porous volume of the final product but also determines the final morphology and mechanical strength. By controlling the sintering temperature of the ceramic, it is possible to increase the pore size and to obtain a higher mechanical strength. We have also observed that the obtained silty marks support presents the highest mean pore diameter for the highest mechanical strength: the support fired at 1190 °C and characterised by mercury porosimetry showed mean pore diameters and porosity of about 9.2 mm and 49%, respectively (Fig. 5).



Fig. 5. Pore diameters of the silty marls supports.

It can also be observed that the porosity and the pore size parameters are strongly dependent on the sintering temperature and particle size of the powders (Table 2).

2.2 Microfiltration layer shaping and characterization

The material used for the membrane preparation is a Tunisian clay powder (JM18) taken from the area of Sidi Bouzid(Central Tunisia). This powder is crushed for 4 h with a planetary crusher at 250 revolutions/min and calibrated with 50 mm. The obtained particle diameters range from about 0.5 to 23 mm (Fig. 6).

Powders	Temperature (°C)	Pore size (µm)	Porous Volume (%)
Crushed during one	1160	9.6	58
hour and calibrated	1170	10.9	56
with 125 µm	1180	12.8	53
	1190	14.3	52
	1200	16.5	51
Crushed during two	1160	5.9	52
hours and calibrated	1170	7.3	50
with 100 µm	1180	8.5	49
	1190	9.2	49
	1200	12.5	46

Table 2.Variation of pore size and porous volume according to the powder particle sizes for the silty marls (M11).



Fig. 6. Clay (JM18) particle size distribution.

The chemical composition of the clay (JM18) is shown in Table 3.	
Elements (%)	JM18
SiO ₂	62.64
Al ₂ O ₃	17.09
FeO ₃	8.5
MgO	0.07
Na ₂ O	0.32
K ₂ O	4.8
Mn_2O_3	0.02
SO ₃	0.4
Loss on the ignition	6.16

Table 3. Composition of clay (JM18).

It reveals that this material is essentially formed with a large amount of silica 62.64%. For preparing a microfiltration layer with JM18, the suspended powder technique was used. A deflocculated slip was obtained by mixing 5% (w/w) of JM18, 30% (w/w) of polyvinyl alcohol (PVA) (12% w/w aqueous solution) as binder and water (65% w/w). The thickness of microfiltration layer can be controlled by the percentage of the clay powder added to the suspension and the deposition time. The viscosity of the slip elaborated according to the protocol described previously has been studied right before deposition. The used viscosimeter (LAMY, TVe-05) permits to use 5 speeds of rotation for the determination of the dynamic viscosity of the substance to characterize. Fig. 7 shows the rheogram of the slip used. It is done by the curve of shear stress (τ) versus speed of rotation (D). The slip has a plastic behavior of Bingham, controlled by the presence of PVA; the value of the limiting shear stress is 4 mPa. Such behavior permits the maintenance of particles in a stable suspension. The deposition of the slip on the M11 support was performed by slip-casting using a deposition time between 10 and 15 min.



Fig. 7. Evolution of the stress (τ) and the viscosity (μ) vs. deformation of clay (JM18) slip.

After drying at room temperature for 24 h, the clay membrane was sintered at 900 °C for 2h, after debonding at 250 °C for 1 h. Total porous volume and pore size distribution are measured by mercury porosimetry. This technique relies on the penetration of mercury into a membrane's pores under pressure. The intrusion volume is recorded as a function of the applied pressure and then the pore size was determined. The pore diameters measured were centered near 0.18 mm (Fig. 8).

The pore size in the microfiltration layer can also be varied using powders with different particle size distributions. Different microfiltration membranes with different layers thickness (between 5 and 50 mm) were prepared. SEM (scanning electron microscopy) images of the resulting membranes are shown in Fig. 9.



Fig. 8. Pore diameters of the clay (JM18) membrane.



Fig. 9. Scanning electron micrographs of clay (JM18) membrane (A: membrane thickness upper than 10 mm, B: membrane thickness less than 10 mm, 1: surface, 2: cross-section).

This figure gives information on the texture of the elaborated membrane surface. A defect free membrane was only obtained for membrane thickness less than 10 mm (in order to 7 mm). Crossflow microfiltration tests were performed using a home-made pilot plant (Fig. 10) at a temperature of 25 °C and transmembrane pressure (TMP) range between 1 and 4 bar.



Fig. 10. Flow schema of experimental apparatus.

The flow rate was fixed at 2.5 m s⁻¹. Before the tests, the membrane was conditioned by immersion in pure deionised water for a minimum of 24 h. The working pressure was obtained using a nitrogen gas source. The membrane was initially characterized by the determination of water permeability which was $870 l h^{-1}m^{-1}bar^{-1}$ (Fig. 11).



3. Modification of elaborated microfiltration membrane surface

3.1 Experimental

3.1.1 Materials

Tunisian clay membrane was prepared in our laboratory from the support to the finest layer and was grafted as described earlier (Khemakhem, & al., 2006; Khemakhem, & al., 2007). The triethoxy-1H,1H,2H,2H perfluorodecylsilane C8F17(CH2)2Si(OC2H5)3 (97%) from Sigma was used. Analytical grade ethanol (99%) was purchased from Riedel-de-Haën.

3.1.2 Grafting process

Grafting of membranes was performed by the use of fluorinated silanes: triethoxy-1H,1H,2H,2H-perflluorodecylsilane. Grafting it occurs with a succession of condensation reactions between the OH groups found in the surface membrane and the Si–O–alkyl groups of the silane. To realize grafting, solutions of the triethoxy-1H,1H,2H,2H-perflluorodecylsilane were prepared in ethanol at a concentration of 10⁻² mol l⁻¹. Prior to the chemical modification, the membranes were cleaned in an ultrasonic bath in the presence of ethanol and acetone successively for 5 min and dried in an oven at 100°C. Samples, planar membranes as well as tubular membranes, were completely immersed in prepared solution for different times (15min, 30min and 60min) at room temperature. The grafted membrane was then rinsed in ethanol and acetone successively and placed in an oven at 100°C for 1 h.

3.1.3 Characterization

The FTIR spectra were obtained from KBr pellet using a Perkin-Elmer BXII spectrophotometer under transmission mode. The spectra were collected for each measurement in the spectral range 400-4000 cm⁻¹ with a resolution of 4 cm⁻¹. Thermogravimetric analysis (TGA) was performed on a TGA2950 thermobalance. Samples were heated from 30 to 900°C at a heating rate of 10°C/min. The measurements of contact angles were performed at room temperature (20°C) using a OCA 15 from Dataphysics, equipped with a CCD camera, with a resolution of 752-582 square pixels, working at an acquisition rate of 4 images per second. Collected data were processed using OCA software. Distilled water was used for measurements and planar membranes realized with clay were grafted. The drop image was recorded by video camera and digitalized. Each contact angle is the average value of 20 measurements. Grafted and ungrafetd surfaces were also characterized by Scanning Electron Microscopy (Hitachi S-4500). ²⁹Si cross-polarization magic-angle-spinning nuclearmagnetic-resonance (CP/MAS NMR) spectra were gained on a Bruker DSX-300 spectrometer operating at 59.63 MHz. Water permeability was measured on grafted ceramic membranes by crossflow filtration experiments. Measurements were performed using home-made pilot plant.

3.2 Results and discussion

3.2.1 Contact angle measurements

The hydrophobic character of the resulting material was tested by measuring the contact angle of water drop. This method gives us information and a determination of the hydrophobicity of the grafted samples. Results obtained on the planar membrane are reported in Fig.12. The low contact angle of the ungrafted membrane, results from the high hydrophilic character of the membrane surface as a consequence of the high density of the hydroxyl group on the surface of the membrane. The studied grafting times were (15, 30 and 60min). We noticed that the value of contact angle increases weakly with the increase of grafting time.

A high efficiency of molecule triethoxy-1H,1H,2H,2H- perfluorodecylsilane and a high hydrophobicity of grafted surface is shown in Fig. 12. Values of the contact angle were in the



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Fig. 12. Evolution of contact angle versus time of microfiltration membrane at different grafting times.

range of 177°–179° for all prepared membranes, what means that the tested membrane possessed the hydrophobic character. The high hydrophobicity of the grafted ceramic surface is illustrated in Fig.13. We obtain non-wetting materials as we observed that there is no capillary suction of a drop of water deposited on the grafted planar membrane.



Fig. 13. Top view of a droplet of water, deposited on a membrane surface, (a) ungrafted membrane, (b) grafted microfiltration membrane.

3.2.2 Infrared absorption spectra

Infrared spectra on each grafted or ungrafted surface membrane were measured. The infrared spectra of the ungrafted clay membrane are shown in Fig. 14a. It could be confirmed that there were hydroxyl groups in the ungrafted surface clay membrane from

the band at 3100–3500 cm⁻¹. The band at 3425 cm⁻¹ corresponds to the –OH stretching vibration of the adsorbed water (Frost & al., 2000). A new band at 2978 cm⁻¹ attributed to the anti-symmetric stretching of the –CH₂ group of the triethoxy-1H,1H,2H,2H-perflluorodecylsilane was showed in Fig. 14b, indicating the presence of silane in the grafted products. The bands at 1540 cm⁻¹, 1240 cm⁻¹ and 1207 cm⁻¹ corresponding respectively to the stretching vibration of the C-C, C_xF_{2x+1} and Si-CH₂CH₂C_xF_{2x+1} groups. The pores size diameter and the size of the used particles for the elaborated membrane have an important influence on the amount of the grafted silanes. The grafting reaction conducted in the silane favors its entering into the clay layer.



Fig. 14. Infrared spectra of (a) ungrafted membrane, (b) microfiltration grafted membrane.

3.2.3 TGA analysis

Thermogravimetric analysis (TGA) was provided as a simple method to measure the content of silane and adsorbed water. This method is based on the assumption that the dehydration and dehydroxylation reactions correspond to the two discrete mass loss steps in TG curves and they do not overlap each other. The TG curves of ungrafted membrane

decreased rapidly when the decomposition temperature varied from 30 to 200°C, resulting from the loss of water molecules for the clay membrane contained (Fig. 15a). When increasing the temperature from 200 to 900°C, the curve was smooth with a mass loss of about 0.35%, suggesting that the membrane was very stable at high temperature. Threestage decomposition procedure is shown in the TG curves of the grafting membrane (Fig. 15b). The initial mass loss took place between 30 and 200°C, which was attributed to the loss of water molecules attached to the membrane. The highest mass loss in the second stage from 300 to 650°C, results from decomposition of the grafted silane. A complete mass loss was not occurred even after heating the material up to 900°C.



Fig. 15. TG curves of: (a) ungrafted membrane, (b) microfiltration grafted membrane.

3.2.4 CP/MAS ²⁹Si solid state NMR

The chemical shift of silicon is determined by the chemical nature of its neighbors, namely, the number of siloxane bridges attached to a silicon atom, and M, D, T, and Q structures are the commonly used notation corresponding to one, two, three, and four Si-O- bridges, respectively. According to the nomenclature recommended in the literature of M^i , D^i , T^i , and Qi (Glasser & Wilkes, 1989; Young & al., 2002; Gavarini, 2002), where i refers to the number of -O-Si groups bound to the silicon atom of interest, Tⁱ corresponds to R-Si(-OSi)i-OR-(3-i). Four Q peaks can be present, namely Q⁰ (-66 to -74 ppm), Q¹ (-78 to -83 ppm), Q² (-83 to -88 ppm), Q³ (-90 to -100 ppm), and Q⁴ (-107 to -110 ppm) and four T peaks can be present, namely T⁰ (-37 to -39 ppm), T¹ (-46 to -48 ppm), T² (-53 to -57 ppm), and T³ (-61 to -66 ppm) (Glasser & Wilkes, 1989).

Three distinct peaks observed in the ²⁹Si NMR spectra of the fluoroalkoxysilane indicating three distinct silicon chemical environments Fig. (16a). These peaks at chemical shifts of approximately –48.14 ppm, –54.52 ppm and –61.65 ppm are representatives respectively of T_1 , T_2 and T_3 resonances. In Fig. (16b) we reported the ²⁹Si CP/MAS NMR spectrum of used

Tunisian clay. In this spectrum we observed tow ²⁹Si NMR peaks at -71.92ppm and -81.11ppm which correspond to the tow different Q⁰ and Q¹ resonances of the different types of silicon atoms in the structure, respectively.

The ²⁹Si CP-MAS NMR spectra recorded for the modified MF membranes were given, on Fig. (16c). This spectra can be divided into two regions. The first region, from +50ppm to



Fig. 16. ²⁹Si CPMAS NMR spectra of: (a) Fluoroalkylsilane molecule (b) ungrafted membrane, (c) microfiltration grafted membrane.

50ppm, contains the signal corresponding to the silicon atom of the fluoroalkoxysilane (T1). The second region range from -60ppm to -150ppm, corresponds to the signals of the various silicon in the clay structure. However the tow peaks observed in Fig. 16a at -71.92ppm and -81.11ppm correspond, to the Q₀ and Q1 silicons in the clay structure respectively. The presence of the T₁ peak indicates that the silane of fluoroalkylsilanes has been successfully grafted to the surface of microfiltration layer. In other words, the surface of microfiltration layer has been silylated.

3.2.5 Crossflow filtration experiments

Permeability measurements were used to test the hydrophobic character of the grafted membranes. Permeability of membranes was measured for grafted and ungrafted membranes. The ungrafted membrane exhibit a permeability of 867 l hm⁻²bar⁻¹. After grafting, there is a high reduction of permeability. The permeability measured for grafted microfiltration membrane was $2.7 l h^{-1} m^{-2} bar^{-1}$ (Fig. 17). So, we can conclude a high effect of these molecules to decrease strongly the size of pores diameters and to reduce the permeability of the membrane.



Fig. 17. Values of flux for microfiltration grafted and ungrafted membranes.

4. Application for seawater desalination

The experimental set-up presented in Fig. 18 was used for the performances of the triethoxy-1H,1H,2H,2H perfluorodecylsilane grafted membranes in the AGMD configuration process. The air-gap width was 10 mm. The used water was heated in a feed tank and it was circulating through the membrane module. During experiments, permeate vapors were condensed at a cooled stainless steel surface close to the membrane (Fig. 18). The permeate water flux through the membranes was determined by measuring permeate volume as a function of time.



Fig. 18. Schematic presentation of AGMD in counter current flow configuration.

In each experiment, two liters of water were used and was circulated through the membrane module by using a variable flow peristaltic pump. The feed pressure was measured with a manometer at the inlet of the feed cell frame. The feed flow rate was measured with a digital flowmeter at the outlet of the feed cell frame, and it was kept constant during the experimental run by adjusting the pump speed. The feed and cooling plate temperatures were kept constant by using controlled heating and cooling thermostats, respectively.

The fine control of the feed temperature was achieved by using an auxiliary heat exchanger between the feed reservoir and the pump. The temperatures were measured with two thermometers located at the feed cell frame and at the cooling plates, respectively. The obtained distillate was collected directly in a calibrated graduated cylinder. The distillate flow through the membrane was determined from the temporal evolution of the liquid level in the cylinder. The temperature difference is defined as $T = T_2 - T_1$ where T_2 is the feed solution temperature and T_1 is the cooling water temperature. Salt concentration both in feed and permeate solutions were determined through conductivity measurement. The separation factor, *R* was calculated using the following expression:

$$R = \left(1 - \frac{C_p}{C_F}\right) \times 100$$

Where Cp and Cf are the NaCl concentration in the permeate and in the bulk feed solution, respectively.

Air Gap Membrane Distillation (AGMD) experiments were performed for seawater desalination using prepared hydrophobic membrane. Seawater desalination aims to obtain fresh water with free salt adequate for drinking. In our work seawater treated is collected from SIDI MANSOUR Sea, located at Sfax (Tunisia). Measurements of permeate flux and rejection rates were carried out by AGMD as a function of the temperature. The feed side temperature was thus varied from 75 °C to 95 °C, while keeping the cooling system



temperature constant at 5 °C. It can be seen that the salt retention in AGMD process with grafted ceramic membranes is higher than 98% (Fig. 19).

Fig. 19. Variation of the permeate flux as a function of the temperature. The values reported on the graph correspond to the rejection rates calculated for the different membranes after filtration of seawater.

These results proved that in the case of AGMD with aqueous solutions containing nonvolatile compounds like NaCl, only water vapor is transported through the membrane. It can be seen that the salt retention in AGMD process with grafted ceramic membranes is close to 100%.

5. Conclusion

Surface modification of "silanized" MF Tunisian clay membrane via graft polymerization of perfluoroalkylsilane (C8) was carried out. Ceramic membranes surface grafted with perfluoroalkylsilane changed the hydrophilic character into a superhydrophobic one. The effect of silane treatment on the properties of the clay membrane surface studied was found to depend on the membrane pore structure. It was proven that the presented grafted silane constitutes an ideal method for producing improved ceramic NF membranes with a decreased pore size. The data obtained with different techniques indicated a polymer grafting on the treated ceramic grains on the surface and inside the pores to form a selective barrier whose permeation flux decreases. FTIR measurements determinate the nature of chemical bonds involved in the organic layer covering the clay surface. Contact-angle experiments provided important information on the wettability and the hydrophobic character of the modified surfaces. Modification of the clay surface with C8 led to an increase of the contact angle.

Membrane distillation is an emerging technology for desalination. Membrane distillation differs from other membrane technologies in that the driving force for desalination is the difference in vapour pressure of water across the membrane, rather than total pressure.

In our study, interesting results were obtained for membrane distillation experiments conducted using MF modified clay membrane, validating the ability of these modified ceramic membranes to act as membrane contactors for desalination. An important influence of the feed temperature and NaCl concentration on the permeate flux was observed. In the same time, high salt rejection rates were obtained in AGMD process with grafted clay ceramic membranes.

The membranes for MD are hydrophobic, which allows water vapour (not liquid water) to pass. The vapour pressure gradient is created by heating the source water, there by elevating its vapour pressure. The major energy requirement is for low-grade thermal energy. It is expected that the total costs for drinking water with membrane distillation, depending on the source of the thermal energy required for the evaporation of water through the membrane. Solar energy could very much help this process in our countries which are very sunny resulting in a reduction of energy costs. Thus, membrane distillation could become competitive relative to other processes.

6. References

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