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Preparation of new tubular carbon ultrafiltration membrane for oily wastewater treatment by air gap membrane distillation

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ABSTRACT

The present study focuses on the preparation of a new tubular carbon ultrafiltration (UF) membrane for oily wastewater treatment by air gap membrane distillation (AGMD). The UF membrane was elaborated following a slip casting method by deposition of a thin carbon film over a previously prepared microfiltration (MF) carbon membrane. The mesoporous top layer was achieved using viscous suspension composed by commercial black carbon (BC) powder with an average particle size of 60 nm added to an alcoholic solution of novolac-phenolic resin (NPR). After curing at 150°C during 60 min and carbonization at 700°C during 300 min under nitrogen atmosphere, the resulting UF membrane exhibits an average pore diameter of about 4 nm and a thickness around 9 µm. The determination of water permeability shows a value of 18 L/h·m^2·bar. In addition, a hydrophobic character has been identified which allows the application of air gap membrane distillation for oily waste water treatment. High salt and oil retention of about 99% were then achieved.

Keywords: Carbon; Ceramic UF membrane; Hydrophobic character; Membrane distillation, Oily wastewater treatment

1. Introduction

The porous inorganic membranes are widely used in the industrial application, primarily due to their chemical and thermal stability, low fouling and high mechanical strength [1–4]. The ceramic membranes are generally fabricated from pure metal oxides such as zirconia, titania and alumina [2–5]. However, their use is still limited due to the high cost of materials and the high firing temperature needed in sintering process [6]. At present, attention is particularly focused on the preparation of low cost membranes made from abundant natural materials like starch [7,8], kaolin [9,10], apatite [11], clay [12–14], granitic [15] and carbon [16–18].

From the literature, several works were devoted to the preparation of new type of porous ceramic membranes based on carbon. A very recent development realized by Abd Jalil et al. [19] involved the preparation of carbon membrane applying a vacuum-assisted method as a pre-treatment of carbon thin films. The carbon membrane was prepared from a phenolic resin as polymeric precursor which was dip-coated on an alumina substrate to form a resin thin film. After curing and carbonization under inert nitrogen atmosphere, a carbon thin film was achieved. Lately, Song et al. [20] prepared a carbon-alumina mixed matrix membrane (CMS-Al_2O_3) for sea water desalination (NaCl 3.5 wt%) by pervaporation. A high water flux of up to 25 kg m^{-2} h^{-1} and a high salt rejection varying between 93–99% depending on temperature and feed salt concentration were obtained. Ayadi et al. [21] developed new conductive porous supports based on graphite powder as the main carbon source and Résol®resin as carbon precursor. The elaborated supports have been used for mineralization of synthetic dye

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(acid orange 7 (AO7)) by electrochemical advanced oxidation process (electro-Fenton process). Tahri et al. [22] prepared new carbon/carbon asymmetric microfiltration membrane applied to the treatment of industrial textile wastewater. The retention of the different pollutants in terms of color, COD, Salinity and Turbidity was 80%, 57%, 30%, 90% respectively. Wei et al. [17] developed an asymmetric carbon membrane coated by alcoholic solution of novolac phenol–formaldehyde resin containing a little hexamine on a porous support prepared from the same material carbonized at 800°C in air atmosphere. The support and the membrane layer were carbonized simultaneously. This membrane seems to be promising for H_2/N_2 and H_2/CH_4 separation.

Carbon membranes can be used in a broad range of applications because they are chemically inert and they tend to be much stronger than most ceramics. Moreover, carbon membranes possess good physicochemical properties such as hydrophobicity and antifouling properties. Another advantage of carbons membranes is related to the good stability conferred by carbon structures. They can be successfully applied in the separation of pesticides [23] and of gas [24–26]. In addition, they are good candidates for the treatment of textile industry wastewater [22,27], industrial dyeing effluent [28] and oily wastewater [29]. Carbon as ceramic materials has been used to obtain membranes with controlled pore size diameters and to improve membrane selectivity and permeability. Pan et al. [30] used the lab-made tubular coal-based carbon membrane for performance of series of experimental researches on treatment of emulsified oily wastewater. An oil rejection rate close to 97% was achieved. In other study [31], authors show that coal is a good precursor for preparation of a microfiltration carbon membrane and that treatment of oily wastewater using the carbon membrane is effective with oil rejection efficiency up to 98%. The membrane fouling mechanisms were also analyzed. A low cost tubular microfiltration carbon membrane based on coal as a cheap carbonaceous material was successfully developed by Song et al. [20] for the purpose of oily wastewater treatment. Using the prepared membrane with the pore size of 1.0 µm, the oil rejection coefficients of oily wastewater are up to 97% and the oil concentrations of the permeate are less than 10 mg/L. These results were obtained under transmembrane pressure of about 0.10 MPa and crossflow velocity of about 0.1 m/s.

According to the literature, carbonc the most common carbon membrane active layer was obtained by the deposition of a phenolic resin layer on noncarbonaceous support [32–35] by the process of “slip-casting” or “dip-coating”. These membranes are generally a carbon molecular sieve (CMS) suitable for gas separation. Little previous studies have been conducted so far to prepare asymmetric membrane totally made from carbon material. It can be noticed particularly the work recently done by Tahri et al. [29] who were successful in preparation of crack-free tubular membrane completely based on carbon material with a microporous UF top layer. The slip-casting process was performed for the deposition of the carbon mesoporous interlayer and the microporous top layer using suspension made from a mixture of an alcoholic solution of phenolic resin and carbon powder. It was found that 6 min of casting duration leads to an average pore diameter and a thickness layer of about 5.3 nm and 12.6 µm, respectively. This membrane offers a better alternative for the treatment of industrial dyeing effluent. A study performed by Ayadi et al. [36], involved the preparation of two tubular carbon microfiltration membranes S25/CM and S44/CM with a mean pore diameter of 0.5 µm and 0.8 µm and a thickness of around 23 µm and 20 µm, respectively. These membranes showed good performances in textile industry wastewater treatment especially S44/CM membrane which exhibited a stabilized permeate flux value at 1 bar of about 150 L/h·m^2 against 4.5 L/h·m^2 for S25/CM. The retention of pollutant substances was relatively high and almost similar for both membranes.

Based on these considerations, the present work focuses on the preparation of an ovel tubular ceramic ultrafiltration membrane based on carbon as a cheap carbonaceous material by slip-casting method. The performances of this UF membrane for the treatment of oily wastewater were then discussed.

2. Experimental

2.1. Materials

Graphite powder from TIMCAL Group (Switzerland) as the main carbon source and novolac-phenolic resin (NPR) (marketed by the company Irons Resins S.A, Spain) as the second source of carbon were used in this work. Organic additives as corn starch powder (RG03408, Cerestar, France), ethyleneglycol, amijel (Cplus 12072, Cerestar, France) and methylcellulose (Dow Chemical Company) were used respectively as agent of porosity, lubricants and plasticizers. Organic additives were homogenously mixed with the graphite powder and the phenolic resin solution to form a porous tubular support by paste extrusion.

Microfiltration and ultrafiltration layers were achieved from suspensions containing carbon powder having an average particles size of 1.8 µm (MF) and 63 nm (UF) and novolac phenolic resin.

The oily waste water provided by a Tunisian petroleum industry has been selected as the representative wastewater to be treated by air gap distillation.

2.2. Substrate and UF membrane preparation

The first step for the fabrication of UF carbon membrane is the elaboration of high-quality macroporous support without defect. To achieve this, a plastic paste has been prepared from a graphite powder (mean particle sizes of 44 µm) homogeneously mixed with organic additives and an alcoholic solution of NPR. A tubular support of 150 mm of length was obtained by extrusion-carbonization process and then characterized by a porosity of 37% and a mean pore diameter of 3 µm. Afterward, an intermediate MF layer was deposited by slip casting process onto the inner face of the support. Thanks to a curing–carbonization cycle at 700°C under a nitrogen stream of 1 mL/min, MF layer with a mean pore diameter of 0.8 µm and a thickness around 20 µm has been achieved. A detailed description of the microfiltration carbon membrane preparation method was given in a previous study by Ayadi et al. [36].

For UF top layer preparation, two compositions of suspension formed by a commercial BC powder and an alcoholic solution of NPR were prepared in empiric way for which different casting times (4, 6, 8, 10 and 12 min) were tested. The appropriate composition was determined basing on SEM observations (Table 1). The mixture was homogenized by a magnetic stirrer for 10 min at a power of 180 W to avoid resin cross-linking. The optimized suspension was slip-casted on the inner face of the MF carbon membrane. Then, the deposited layer was carbonized in nitrogen environment at 700°C according similar temperature program than that used for MF membrane preparation (Fig. 1) [36].

2.3. Analytical methods

A HitashiS-4500 scanning electron microscope (SEM) was used to characterize the membrane morphology, to study the state of the surface and to determine the membrane thickness of the elaborated UF carbon membrane. Water permeability was performed by measuring of the water permeate flux as a function of transmembrane pressure using home-made pilot plant.

The average pore diameter of the prepared membrane was evaluated by the nitrogen adsorption/desorption method (BET) using a Micrometrics gas analyzer Asap 2010 machine.

Contact angle measurements were performed on planar samples by the water-drop method on a KRUSS apparatus. This technique is efficient to evaluate the hydrophobic character of the membrane material. To minimize error, an average value of three results of the tested membrane contact angle was considered as the resultant value.

Table 1
The optimized composition of UF suspension

Components	Weight percent (Wt %)
BC powder (60 nm)	2
NPR alcoholic solution	10

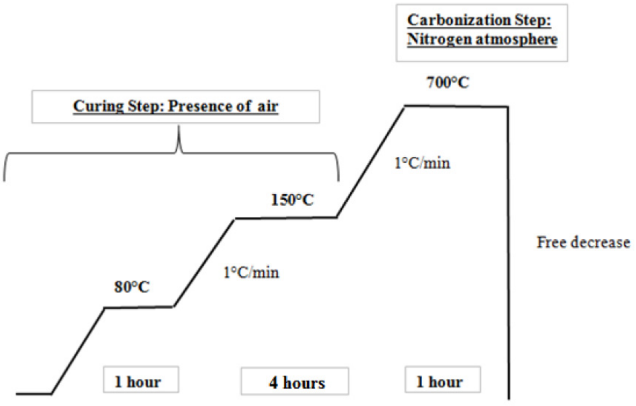

Fig. 1. Temperature-time schedule applied for MF and UF membranes preparation.

3. Results and discussion

3.1. Scanning electron microscopy analysis

To obtain the best homogenous carbon ultrafiltration active layer, two main parameters have been considered: casting time and suspension's viscosity related to the concentration of the NPR. For this, two compositions were selected by varying the percentage of NPR (10% and 15%) while a fix percentage of BC powder of 2%was considered as the best to form an homogenous structure of UF membrane (results not shown here). The resulting UF membranes were named "A" (10% NPR and 2% BC powder) and "B" (15% NPR and 2% BC powder). For SEM analysis, different samples of each type of UF membranes were investigated considering a mean value of thickness was retained.

As shown in Figs. 2 and 3, the thickness of the active layer increased with the casting time following the order 12 > 10.8 > 9 > 6 > 3 µm for "A" and 11 > 10 > 8 > 5.5 > 2.7 µm for "B" for casting time of 12, 10, 8, 6 and 4 min, respectively. It seems that the casting time of 4 min is not enough to have a good adhesion of the active layer on the support.

For a casting time of 10 and 12 min, "A" and "B" membranes exhibit non-homogeneous layers with several holes in the surface. It was clearly observed that samples obtained after 8 min of casting duration present less defects (cracks and pinholes) and have homogenous and smooth surface structure. With 6 min casting time, significant defects were observed.

It can be revealed that membrane "A"realized with 8 min casting time exhibits the best morphological properties. A thickness of about 9 µm was achieved.

3.2. Slip characterization

The slip viscosity is a primary parameter that should be taken in consideration to have a good adhesion on the support and to avoid fast absorption of the suspension during the casting. A viscosimeter (LAMY, TVe-05) was used to determine the dynamic viscosity of the UF top layer before deposition on the inner surface of MF layer. The rheological data of the suspension used for membrane "A"preparation (obtained after 8 min of casting duration) is shown in Fig. 4 which represents the evolution of the shear stress (Tau)with the shear rate (D). It can be observed that the suspension has a plastic behavior of Bingham and that the value of the limiting shear stress is 11 Pa. Masmoudi et al. [27] reported that the stability of the suspension can be maintained by the plastic behavior of Bingham.

3.3. Determination of membrane pore size

The pore size distribution of the UF top layer was obtained from nitrogen adsorption/desorption isotherm. Fig. 5. shows similarity to type IV adsorption isotherm with hysteresis behavior, according to IUPAC classifications. It is worthy to mention that type IV isotherm is characteristic for mesoporous materials (pore size in the range from 20 to 500 Å), in which capillary condensation and the multilayer adsorption (for higher pressures) take place. Fig. 6. shows a relatively narrow distribution with an average pore size of about 4 nm which proves that an ultrafiltration layer was achieved.

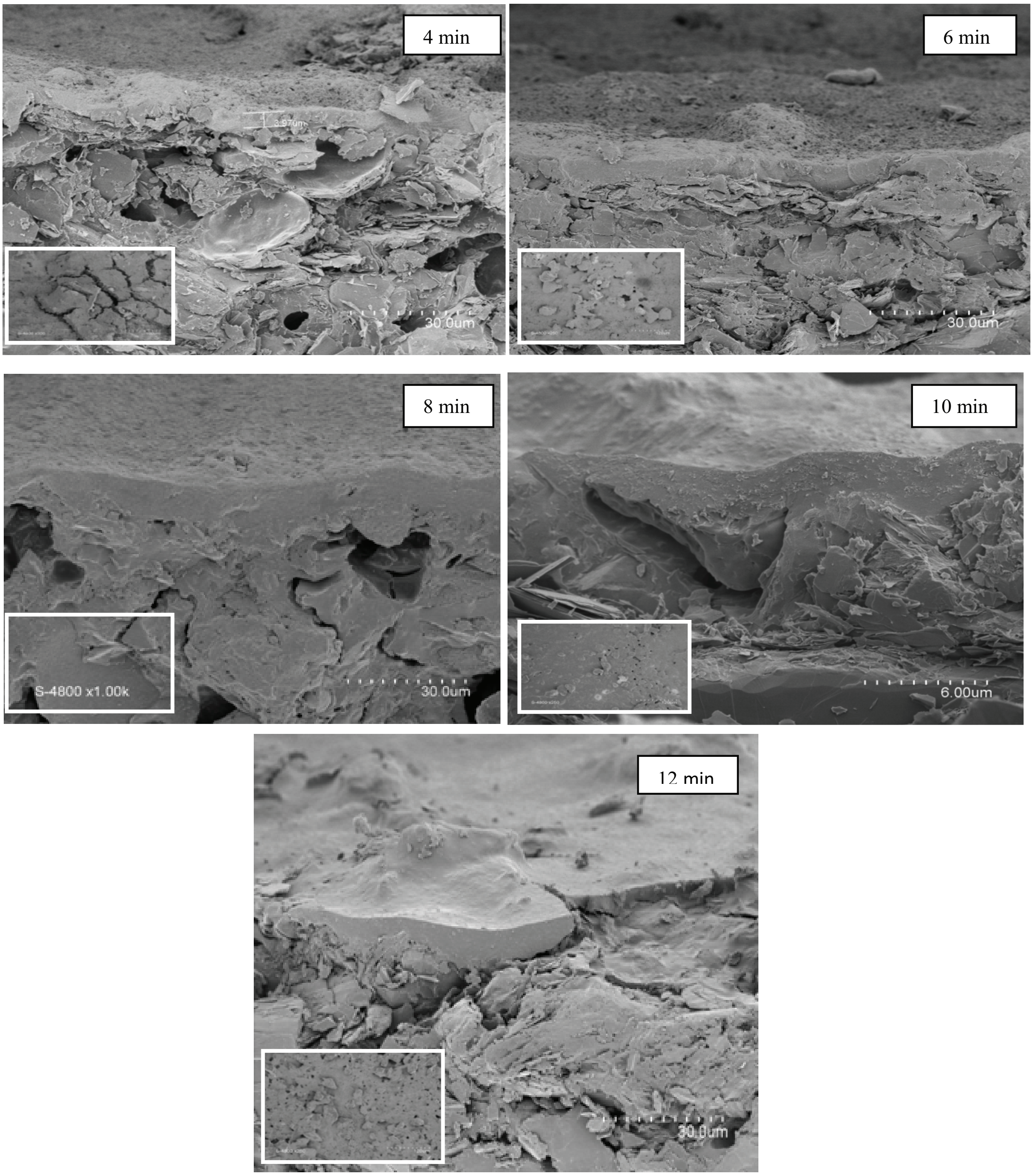

Fig. 2. SEM micrographs of membranes "A" at different casting time. Surface morphology is shown in the insert.

3.4. Determination of membrane permeability

For a fast stabilization of the permeate flux, the carbon-membrane was conditioned in distilled water during 24 h before filtration. The water permeability value (Lp) of UF membrane was achieved using deonized water. The evolution of the water permeate flux (J_w) with the transmembrane pressure (TMP) varying in the range 0–6 bar was determined (Fig. 7). A noticeable linear increase of the permeate flux with transmembrane pressure was observed. According to the Darcy's law (Eq. (1)), the water permeability value was 18 L/h·m²·bar.

$$J_w = \mathrm{Lp}\Delta \mathrm{P} \tag{1}$$

where J_w (L/h·m²) is the liquid flux through the membrane, Lp (L/h·m²·bar) is the membrane permeability and ΔP (bar) is the applied transmembrane pressure.

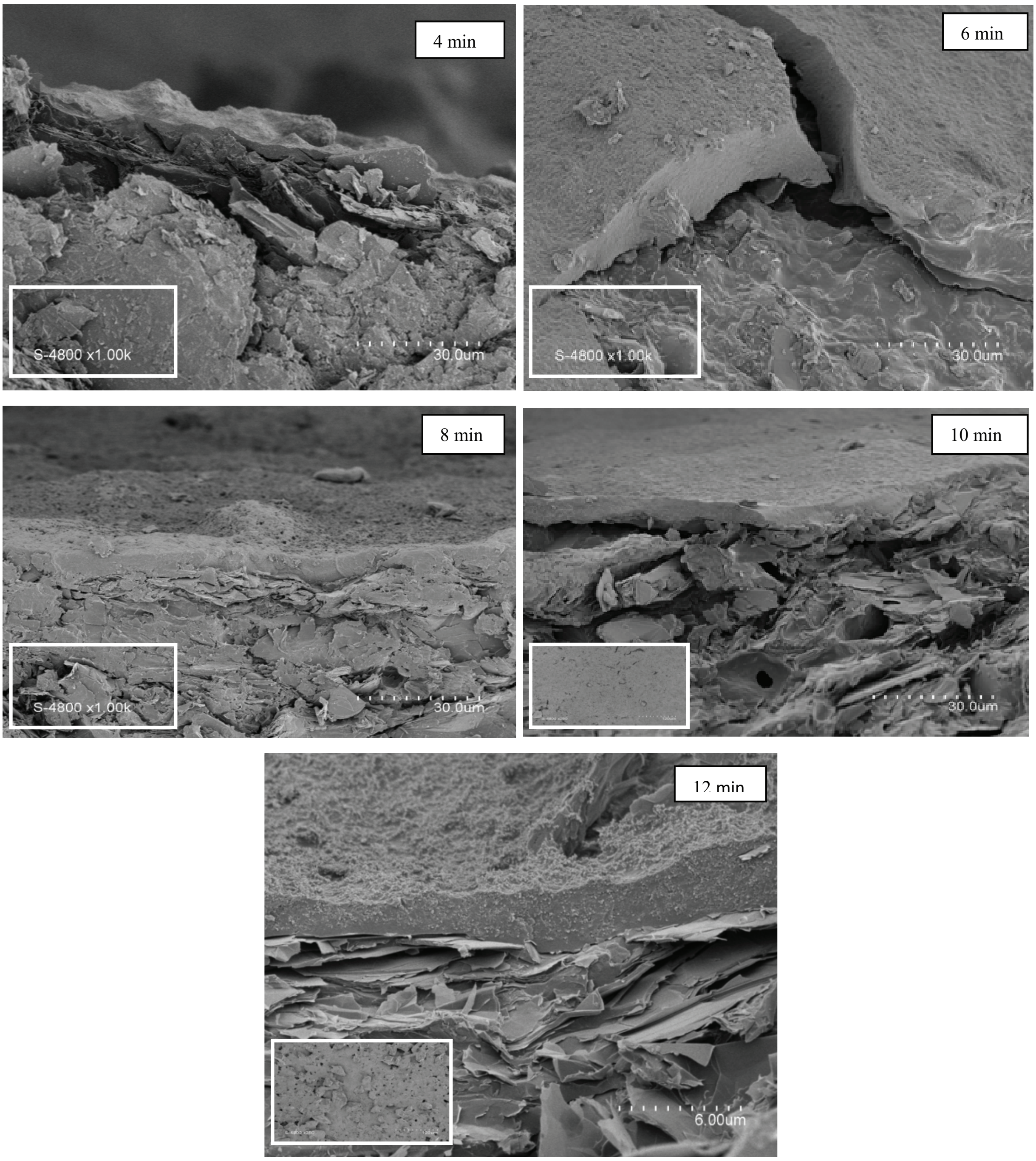

Fig. 3. SEM micrographs of membranes "B" at different casting time. Surface morphology is shown in the insert.

3.5. Contact angle measurement

The contact angle technique was estimated to measure the hydrophilic/hydrophobic character of the material. The measurements of the contact angle of water drop were done twice to give a mean contact angle value with an error of 1°. Each measure required between 10 to 15 µL and was be taken after 10 s. The UF membrane contact angle was found to be equal to 120° (Fig. 8) showing a good hydrophobicity which allows the application of air gap membrane distillation process for the separation of the aqueous phase from the oily wastewater.

3.6. Application to oily waste water treatment by air gap membrane distillation

3.6.1. Oily waste water characterization

Oily waste water was provided by a Tunisian petroleum industry. It contains much amount of free floating

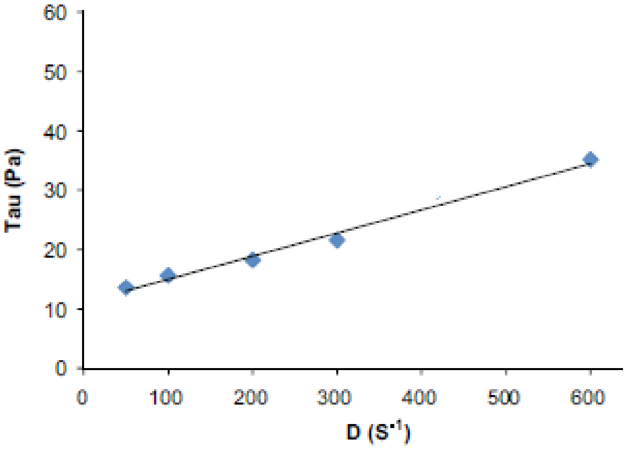

Fig. 4. Evolution of the shear stress (tau) versus shear rate (D).

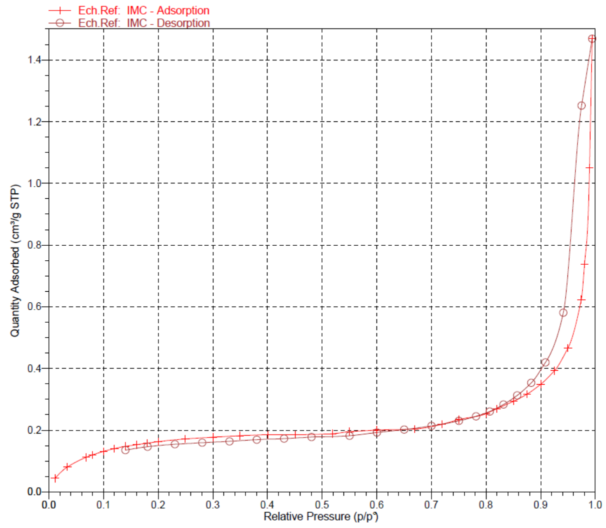

Fig. 5. Nitrogen adsorption–desorption isotherm of top layer.

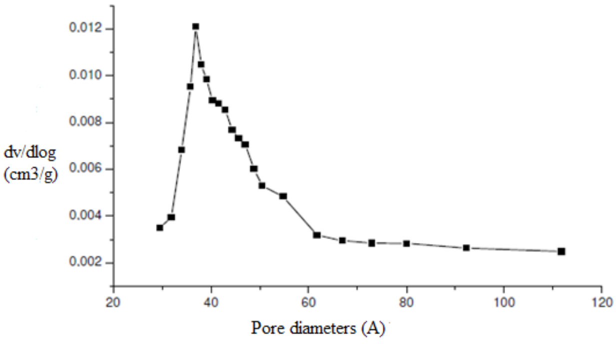

Fig. 6. Nitrogen adsorption–desorption pore size distribution of UF carbon membrane.

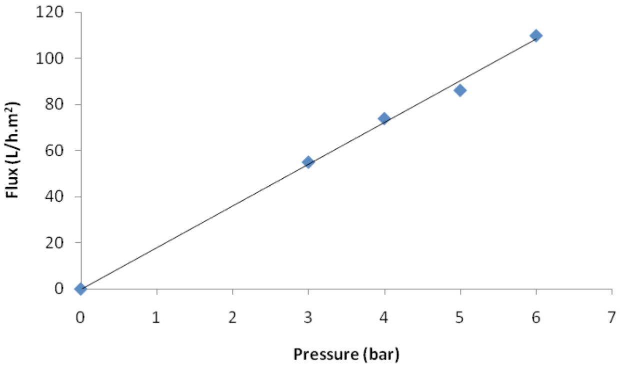

Fig. 7. Evolution of the permeate flux (L/h·m^2) of the carbon membrane with the applied pressure.

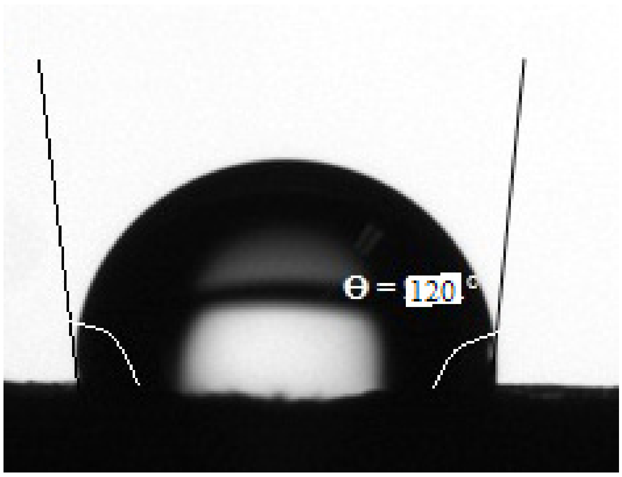

Fig. 8. Top view of a water droplet deposited on a carbon UF membrane surface.

oil on the top and solid particles at the bottom which can cause the clogging of the membrane pores, resulting in more severe fouling. For this reason, the oily waste water doesn't be used directly for application but it should be pretreated by a conventional methods. For that, a gravity settling method was used for extraction of large suspended solid particles. Using skimming method, the floating oil (formed by the coalescence of the bigger oil droplets) was removed from water and then a filtering method through 1 mm porous filter paper was carried out. The optical microscopy showing the oil droplet size is illustrated by Fig. 9. The drop sizes of oil droplets are all smaller than 5 µm. The characteristics of the oily wastewater are given in Table 2.

3.6.2. Air gap membrane distillation (AGMD)

Air gap membrane distillation (AGMD) experiments were performed for the treatment of oil-in- water emulsion through the UF grafted membrane to obtain pure water. The oil filtration experiment was conducted with a real oil-in-water (o/w) emulsion (previously described). The oil/water mixture was heated in a feed tank, up to 70–95°C while maintaining the coolant temperature, in the permeate side, fixed at 5°C and was then circulating through the membrane module by a variable flow peristaltic pump. The cross-flow velocity was kept at 2.6 m/s. The temperatures were measured with tow thermometers located at the feed

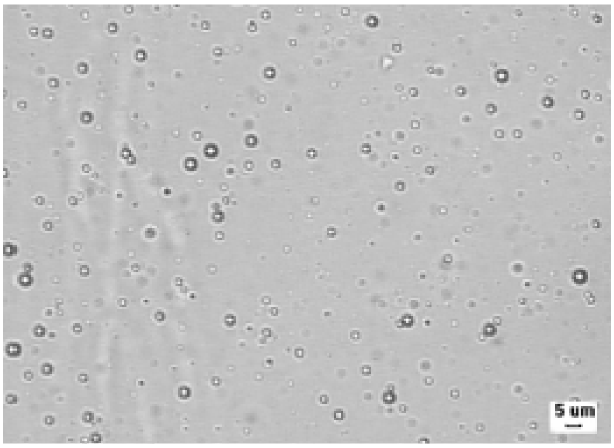

Fig. 9. Micrograph of oil-in-water emulsion.

Table 2
Principle physicochemical characteristics of the oily wastewater

Parameters	Values
Oil content (mg/l)	125
Density (kg/m^3)	900
Salinity (g/l)	9.4
COD (mg/L)	1300

cell frame and at the cooling plates, respectively. The permeate vapor was condensed at the cooled surface which is near to the membrane at a distance of 10 mm (distance necessary to reduce the loss of energy by conduction through the membrane). The permeate flux measurement was achieved using a graduated test-tube and a stopwatch.

Fig. 10 illustrates the variation of the permeate flux between feed and permeate sides as a function of the temperature. The permeate flux is expressed in L/d·m^2 with a precision of ± 5 L/d·m^2.

The permeate flux increases exponentially from 60 L/d·m^2 to 240 L/d·m^2 with the increase of the feed temperature from 70°C to 95°C, respectively. This result can be explained by the exponential increases of the water vapor pressure with the temperature following the Antoine's equation:

$$\log\left(\frac{\mathrm{Ps}}{\mathrm{P°}}\right) = \mathrm{A} - \frac{\mathrm{B}}{\mathrm{T}+\mathrm{C}} \tag{2}$$

where Ps is the vapor pressure, P° is the standard pressure, A, B, C are the coefficients of Antoine and T is the temperature.

From the literature, many researches proved that the use of hydrophobic membrane leads to an exponential dependence of permeate flux with the temperature due to the exponential increase of the water vapor pressure [37–39].

The accurate oil concentration in feed and permeate solutions were evaluated using a UV-vis spectrophotometer (UV-9200) at a wave length of 245 nm. After treated by UF carbon membrane, a significant decrease of the oil concentration from 125 mg/L for the feed solution to 5 mg/L for the permeate solution was obtained.

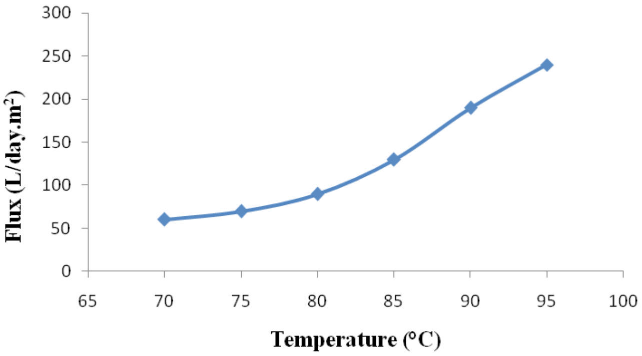

Fig. 10. Variation of the permeate flux with the feed temperature by AGMD using carbon membrane.

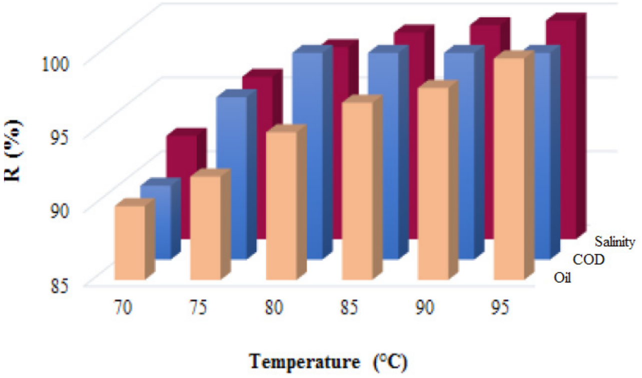

Fig. 11. Evolution of the rejection rate R_x (%) of salt, oil and COD with the temperature.

The salt and oil rejection ratio during the AGMD under different temperatures were calculated according to the following expression:

$$R_x = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \tag{3}$$

where R is the rejection rate, x is attributed to oil, salt or COD, C_f and C_p are the concentration in the feed and in the permeate, respectively.

Fig. 11 shows a very significant salt rejection rate of about 99.8% at 95°C which proves that only the water vapor was transported by the membrane. Similar results were obtained by Khemakhem et al. [40,41] who showed that during the treatment by the AGMD process of the aqueous solutions containing high concentration of salt (NaCl), only water vapor passed through the hydrophobic membrane. In addition, the increase of feed temperature from 70°C to 85°C led to an increase in the oil rejection rate from92.4% to more than 99%, respectively. Beyond 85°C a very little variation of the retention rate was illustrated. Fig. 11 reveals also a good retention of COD which varies from 90% at 70°C to up to 99% beyond a temperature of 80 °C due to the strong affinity of the surface for adsorption of organic matters promoted by the high hydrophobicity of the membrane surface [36]. Based on these outcomes , it can be concluded that the UF carbon membrane can be successfully used in the treatment of oil-water emulsion using AGMD process at a temperature of 85°C.

4. Conclusion

The preparation and application of a new ceramic membrane consisted of an asymmetric structure composed of a macroporous graphite support, a carbon MF transition layer and a carbon UF separation layer, are studied in this work. Following are the major conclusions.

- Achievement of carbon UF membrane with a strong hydrophobicity (contact angle equal to 120°) having a thickness around 9 µm, a pore size of 4 nm, and a water permeability of about 18 L/h·m^2·bar.
- The treatment of oil-in-water emulsion using the UF carbon membrane is feasible and effective
- Over 99% of the oil rejection efficiency was achieved at 85°C with a relatively high permeate flux of about 130 L/d·m^2 which can reach 420 L/d·m^2 when the temperature increased to 95°C.

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