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Elaboration of new tubular ceramic membrane from local Moroccan Perlite for microfiltration process. Application to treatment of industrial wastewaters

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Abstract

In this study, an original microfiltration tubular membrane (M1) made from local Moroccan Perlite was used to treat three wastewater types: effluents coming from beamhouse section of tannery (effluent A), textile effluent coming from jeans washing process (effluent B), and dicing wafer effluent generated by electronic industries (effluent C). The prepared membrane is composed of two layers of Perlite with two different granulometries: a macroporous support with a pore diameter centered near 6.6 μm and porosity of about 42%, and a microfiltration layer, performed by slip casting method, with a mean pore size of 0.27 μm. The water permeability determined of the membrane is 815 L/h m^2 bar. Tangential microfiltration using Perlite membrane proved to be effective in removing pollutants from the three effluents with almost the same efficiencies than that obtained with a commercial Alumina membrane (M2) with a pore diameter of 0.2 μm and a water permeability of 1022 L/h m^2 bar. Tangential microfiltration process operated at lower pressure (1 bar) was seen to remove turbidity from the three feeds completely. Perlite membrane allowed significant reduction of Chemical Oxygen Demand COD (50–54%) and Total Kjeldahl Nitrogen TKN (56%) of beamhouse effluent. It showed a significant decrease of COD (54–57%) and a complete discoloration of textile wastewater.

Keywords: Moroccan Perlite; Ceramic membrane; Microfiltration; Treatment; Industrial effluents

1. Introduction

Due to their potential application in a wide range of industrial processes and in the pollution treatment, membrane technologies have received an increasing interest. The use of organic membranes is actually more developed but inorganic membranes display a number of performance advantages, such as better thermal, chemical and mechanical resistances, long lifetime, and catalytic properties from their intrinsic nature [1]. One of the challenges for future development of the inorganic membranes will be to produce low-cost membranes with high flux performance to treat large volumes of liquid effluent. In recent years, mineral-based porous ceramic membranes have been considered for their low costs (both raw materials and preparation process) and additional functions. Some new ceramic membrane types and applications have been developed [2–10]. Different ceramic supports materials are currently used; however, α-alumina supports are the most common [11–14] to support different filtering layers made in TiO_2, SiO_2 and ZrO_2 by sol–gel route. The selectivity of the membrane is obtained by the deposition of a thin porous layer on the surface of the support. The development of ceramic membranes based on natural materials such as clays and some powder wastes such as fly ash was investigated by several authors [15–26]. Saffaj et al. [15–20] described the elaboration and characterization of microfiltration and ultrafiltration membranes deposited on raw support prepared from natural Moroccan clay and on other support made of cordierite. Khemakhem et al. [22] investigated the development of ceramic microfiltration membranes made of Tunisian clay, the prepared microfiltration membranes were used for the treatment and the decoloration of cuttlefish effluent. Bentama et al. [23] elaborated a new microfiltration

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membranes from sintered clay, these membranes were applied to crossflow microfiltration of dilute suspensions of bentonite and talc, and of a biologic fluid model. Jedidi et al. [24] demonstrated that mineral coal fly ash obtained from coal-fired power stations could be also a good material to make low cost microfiltration membrane, elaborated membrane was applied to the treatment of the dying effluents generated by the washing baths in the textile industry.

In our previous studies, we developed a new ceramic support for microfiltration membrane, with flat disk configuration, based on natural Moroccan Perlite [27]. The support was elaborated by extrusion of the paste and sintering at 1000 °C. The structural properties of Perlite support are satisfying in terms of porosity (~42%) and pore diameter (6.64 μm). It presents a well chemical resistance in acidic medium than in basic medium. In the first part of this work, we will present the elaboration of a low cost microfiltration ceramic membrane, with tubular configuration, made of local Perlite of the Tidiennit region (Morocco). The support and the filtration layer were elaborated both from Perlite which is in abundance. Particles used to prepare filtering layer are small than those used for support manufacture. In the second part, the performances of the elaborated Perlite membrane (M1) were compared with those of a commercial ceramic membrane (M2) made from Alumina. We evaluate the applicability of these membranes of microfiltration in treating three wastewater types:

(i) Effluents coming from beamhouse section of tannery (effluent A): the tanning process can be divided into three main phases: beamhouse, tanning and finishing. The beamhouse process normally accounts for about 40% of wastewater volume in a tannery [28]. In general, the beamhouse phase includes soaking (first process used to rehydrate and wash the hides or skins), liming (treatment with sulfides and lime milk for unhairing), washing (rinse with water and sodium bisulfide), deliming (lime removal with mineral or organic acids or acid salts), bating (enzymatic treatment to complete the removal of epiderm residues and more or less completely destroy the elastic fibers) and pickling (acidification in order to permit the penetration of tanning material) [29].
(ii) Textile effluent coming from jeans washing process (effluent B): in the textile industry, most of the operations require considerable water consumption, high concentration of dying substances and others chemical species. Textile effluents are characterized by high concentrations of pollutants and a great variety of composition which results from changeability of conducted technological processes. The colored wastewaters from textile industries are often composed of a high content of various dyes as well as of dissolved organic carbon (DOC), suspended solids (SS) and colloids.
(iii) Dicing wafer effluent generated by electronic industries (effluent C): wafer dicing is the process by which die are separated from a wafer of semiconductor following the processing of the wafer. The dicing process can be accomplished by scribing and breaking, by mechanical sawing (normally with a machine called a *dicing saw*) or by laser cutting. Following the dicing process the individual silicon chips are encapsulated into chip carriers which are then suitable for use in building electronic devices such as computers, etc. Generally, dicing is a wet process. It is carried out at extremely low deionized (DI) water resistivity. Deionized water is used at wafer saw to cool down the dicing blades and provide some washing action [30]. The dicing process generates considerable amount of turbid water which must be treated and recycled.

2. Experimental

2.1. Perlite sample

Perlite, usually gray, is acid volcanic glassy rock containing 2–6% combined water. For this work, Perlite sample was collected from Tidiennit region at 17 km SW of Nador, Morocco. The sample was crushed into small fragments, ground into powder, and sieved through a 200 μm sieve. The chemical composition of Perlite, given in weight percentage (wt.%) of oxides, is the following: 70% SiO_2, 14.3% Al_2O_3, 2.8% Fe_2O_3, 3.3% Na_2O, 5.2% K_2O and 2% CaO. The characteristics of the raw Perlite powder were detailed in our previous paper [27].

2.2. Elaboration of Perlite support with tubular configuration

The process used to manufacture tubular support is similar to that already described in our previous work and used to elaborate flat disk supports from Moroccan Perlite [27]. The plastic paste was prepared from the Perlite powder (particle diameter < 200 μm) homogeneously mixed with organic additives and water. Plasticizer and binder are required to prepare a paste with rheological properties allowing the shaping by extrusion. The optimized composition of the paste is: (i) 81.7% (w/w) Perlite powder, and (ii) organic additives: 4% (w/w) Methocel derived from methylcellulose (The Dow Chemical Company) as a plasticizer, 4% (w/w) Amijel derived from starch (Cplus 12076, Cerestar) as a binder, 10% (w/w) Starch of corn (RG03408, Cerestar) as porosity agent, 0.3% PEG 1500 (Prolabo) as a binder. The mixture was aged (250 tr/min) during 30 min in order to obtain a good homogeneity. After adding an emulsion [32% water and 0.24% Zusoplast (Zschimmer and Schwartz) as lubricating agent; % based on total weight of mixture powder], the mixture was aged during 30 min in the same box. Obtained paste was stocked for two days in a closed box under high humidity. Paste was then extruded to form tubular supports with the following characteristics: length 15 cm, internal diameter 0.6 cm, external diameter 0.8 cm. The extruded pieces were dried at temperature 40 °C during 24 h and then sintered at 1000 °C in furnace (Nabertherm L9/13/P320, Germany). Process of the ceramic preparation is described in Fig. 1. The Perlite support views, after drying at 40 °C and after sintering at 1000 °C, were shown in Fig. 2.

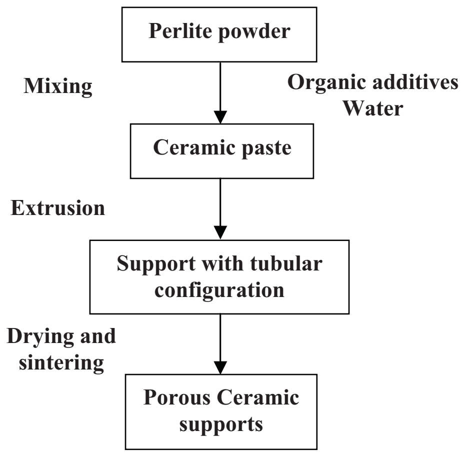

Fig. 1. Simplified diagram for elaboration of porous support by extrusion method.

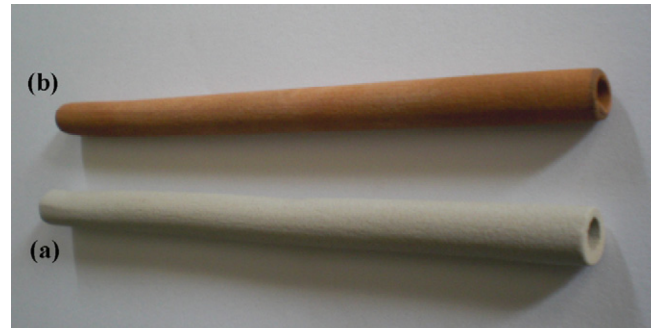

Fig. 2. Perlite support views: (a) after drying at 40 °C and (b) after sintering at 1000 °C.

Macroporous support was characterized by the use of a Mercury Porosimeter (Model Autopore III, Micromeritics Instrument Corporation, USA). The support has a pore diameter centered near 6.6 μm and a porosity of about 42%.

2.3. Preparation and characterization of the microfiltration Perlite membrane

A microfiltration Perlite layer was prepared using the suspended powder technique. Details of the method were described in the literature [31]. A deflocculated suspension was obtained by mixing 10% (w/w) of Perlite (particle diameter < 50 μm), 30% of PVA (12% (w/w) aqueous solution) as binder and 60% of Dolapix CE 64 dispersing agent (0.2% (w/w) aqueous solution). In order to remove no dispersed aggregates, obtained suspension was sieved through a 125 μm sieve. The microfiltration layer was deposited on the inner surface of the support by slip casting. After coating vertically the tubular clay support with the Perlite suspension, it was dried at room temperature and then fired at a temperature of 930 °C for 1 h. The SEM micrographs of Fig. 3 show the general view of surface and the cross section view of membrane. As it can be seen, the microfiltration layer is well coated on the support. The membrane has the advantage of having the same composition as that of his support. It therefore has a good adherence with the support made of same material (Perlite). The scanning electron micrograph view of surface shows that there is no defect or crack on the membrane layer.

Characterization by Mercury Porosimeter (Model Autopore III, Micromeritics Instrument Corporation, USA) show that microfiltration layer, performed by slip casting method, has a mean pore size of 0.27 μm.

2.4. Alumina membrane

The performances of the elaborated membrane were compared with those of a commercial ceramic membrane, provided by Pall Corporation, made from Alumina and having a

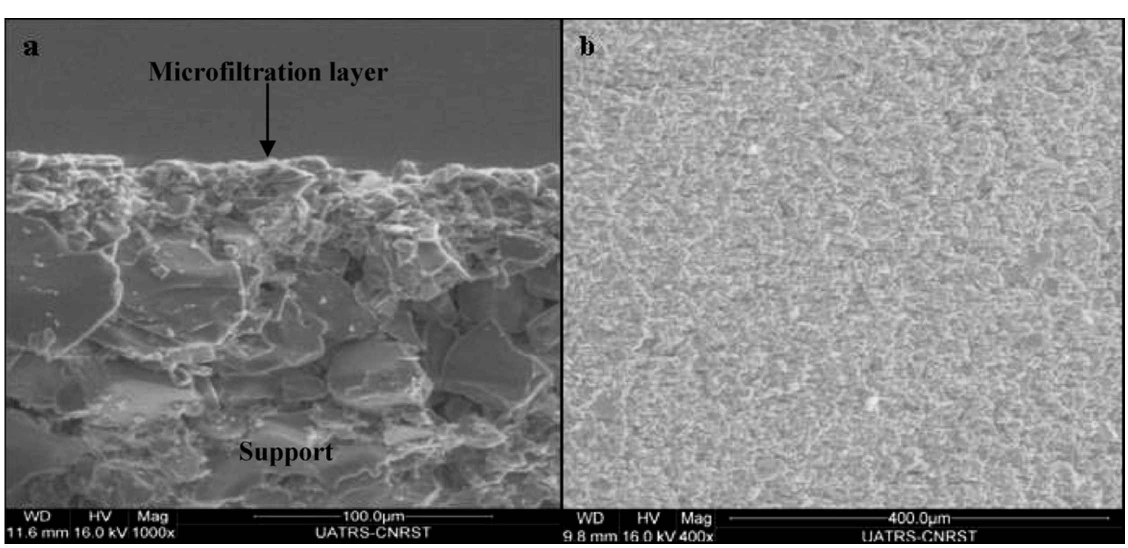

Fig. 3. Scanning electron micrographs of Perlite membrane: (a) cross section view and (b) surface view.

Table 1
Characteristics of Alumina membrane.

Parameter	Description
External diameter	10 mm
Internal diameter	7 mm
Number of channels	1
Surface of filtration	26×10^{-4} m^2
Structure	Asymmetric and composite
Number of layers	3
Thickness of outer layer	2 mm
Pore diameter of outer layer	12 μm
Thickness of intermediate layer	40 μm
Pore diameter of intermediate layer	0.85 μm
Thickness of microfiltration layer	15 μm
Pore diameter of microfiltration layer	0.2 μm

pore diameter of 0.2 μm. The characteristics of Alumina membrane are summarized in Table 1.

2.5. Filtration tests

Tangential filtration tests were performed on laboratory scale filtration pilot using a recycling configuration (Fig. 4). It was equipped with an adjustable out-flow pump, a thermostatic feed tank and a membrane module of 15 cm length. The filtration area developed by the membrane is about 28 cm^2. Effluent was fed into the membrane module by means of gear rotate pump. Inlet flow rate was measured by the flow meter. A valve was used to control the pressure in the system.

The membranes are conditioned by immersion in pure deionized water for a minimum of 24 h before filtration tests to obtain a stabilized flux right from the beginning of the experiment.

Three industrial effluents were treated in this study to evaluate the performances of the elaborated membrane: (i) effluents coming from beamhouse section of tannery (effluent A), (ii) textile effluent coming from jeans washing process (effluent B), and (iii) dicing wafer effluent generated by electronic industries (effluent C).

2.6. Pollution analysis

The microfiltration membranes have been applied to wastewater treatment coming from the beamhouse section of tannery, the dying textile industry and the wafer dicing process. The feed and permeate concentrations were obtained by the use of standard methods (AFNOR). The main analyzed parameters were Chemical Oxygen Demand (COD), Total Kjeldahl Nitrogen (TKN), turbidity, pH and conductivity. Turbidity was measured using TN-100/T-100 device. The pH and conductivity of the feed and permeate solution were measured directly by the use of a Fisher Scientific Accumet Basic AB15 pH Meter (USA) and a Conductivity Meter Model 101 (Orion Research, Cambridge, MA, USA), respectively. The color intensity of feed and permeate samples were analyzed by ATI Unicam UV2 UV/vis spectrophotometer (Cambridge, UK). The color was measured using the integral of the absorbance curve in the whole visible range (400–800 nm).

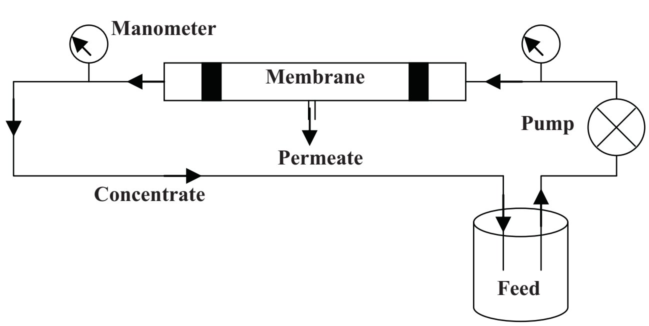

Fig. 4. Microfiltration pilot plant scheme.

The pollutants rejection R was determined by the classical relation: $R\,\% = (1 - C_p/C_f) \times 100$ where C_p is the concentration in permeate and C_f the concentration in the feed.

3. Results and discussion

3.1. Characterization of effluents

The characteristics of beamhouse wastewater (effluent A), textile wastewater (effluents B) and dicing wafer effluent (effluent C) are shown in Table 2. As it can be seen, wastewaters of beamhouse section of tannery, textile industry and dicing wafer process are characterized by high turbidity which is 219 NTU for effluent A, 367 NTU for effluent B and 132 NTU for effluent C. This is due to presence of great amount of suspended solids (4.32, 6.50 and 2.74 mg/L, respectively). The conductivity value of effluents A, B and C is 22.2, 8.7 and 0.09 ms cm^{-1}, respectively. The high conductivity of effluents A and B can be explained by the use of chemical salts in the tannery and textile processes. The conductivity of effluent C is due to the carbon dioxide gas CO_2 injected in order to decreases resistivity of deionized (DI) water used in the semiconductor industry process. The pH of textile effluent and dicing wafer effluent is slightly acid (between 5 and 6). The great value of pH (∼12), detected in the case of beamhouse tannery wastewater, can be explained by the use of chemical products such as lime, sodium sulfide and sodium hydrogen sulfide. These compounds are basic in nature and increase consequently the pH of the effluent. The highest value of COD (40,320 mg/L) measured in beamhouse wastewater may be due to the presence, in effluent, of high content of organic matter and soluble hydrolyzed proteins (KTN = 3570 mg/L). Chemical Oxygen Demand COD value is 4317 mg/L for effluent B, this high COD value is due to the presence of important concentration of colloidal dyes in

Table 2
Effluents characterization. A: beamhouse effluent, B: textile effluent, and C: dicing effluent.

Parameters	Effluent A	Effluent B	Effluent C
pH	12	5.9	5.5
Conductivity (ms cm^{-1})	22.2	8.7	0.09
Turbidity (NTU)	132	219	367
COD (mg O_2/l)	40,320	4317	<50
KTN (mg/l)	3570	–	–
Suspended matter (g/l)	4.32	6.50	2.74

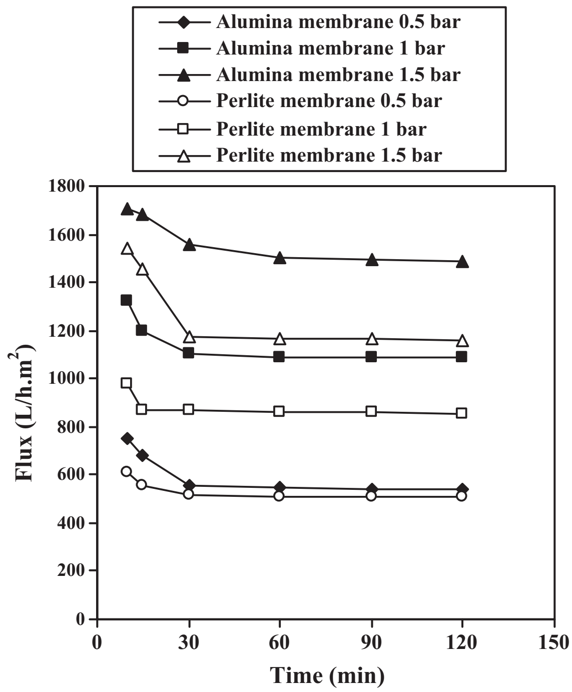

Fig. 5. Water flux vs. operating time.

effluent. The COD value is lower in the case of dicing wafer effluent (<50 mg/L); this can be explained by the presence in water of only hydrophobic mineral suspended matter.

3.2. Determination of membrane permeability

Perlite and Alumina microfiltration membranes were first characterized by their water permeability. For each membrane, fluxes are measured at different transmembrane pressures (0.5, 1 and 1.5 bar). It can be observed that the stabilization of the water flux through the membrane takes approximately 30 min (Fig. 5). Experiments show also that the water flux through the membrane depends on the applied pressure. The average membrane permeability determined using pure distilled water is 815 L/h m^2 bar and 1022 L/h m^2 bar in the case of Perlite membrane and Alumina commercial membrane, respectively (Fig. 6). It remains almost constant during all the filtration experiments. Permeability of the Alumina membrane (M2) is higher than that of Perlite membrane (M1) even if the average pore size of M2 is smaller than that of M1, this can be due probably to the thickness of filtration layer which is 15 μm and 25 μm in the case of Alumina membrane and Perlite membrane, respectively.

As can be seen, permeability is a function of the nature of membrane and of its porosity. It also depends on the pore size and thickness of the membrane.

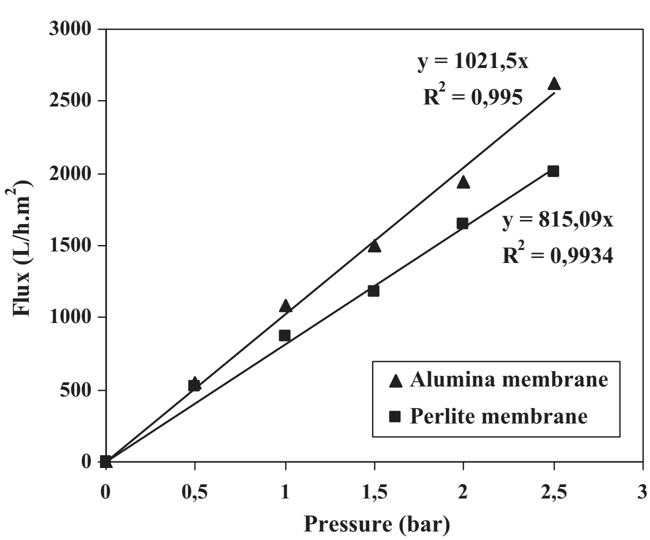

Fig. 6. Variation of water flux as a function of pressure.

3.3. Filtration test

The variation of permeate fluxes of the three effluents as a function of time is shown in Fig. 7. For the two membranes, all microfiltration experiments were carried out at pressure 1 bar. Permeate fluxes are low in comparison with those obtained with distilled water. This can be explained by the effect of contaminants which changes the dynamic proprieties of water as viscosity for example. The permeate flux of effluents A, B and C, obtained with Perlite membrane (M1) are about 132 L/h m^2, 85 L/h m^2 and 175 L/h m^2, respectively. In the case of Alumina membrane (M2), the permeate flux of effluents A, B and C, are about 165 L/h m^2, 145 L/h m^2 and 206 L/h m^2, respectively. Fluxes obtained by the use of Alumina membrane (M2) are higher than those obtained by the use of Perlite membrane (M1). This is in agreement with results of distilled water permeability tests. For two studied membranes, the permeate flux J (L/h m^2) of effluents decrease in the following order: $J(C) > J(A) > J(B)$, this is due to the nature and physical properties of filtered effluents.

For all microfiltration experiments, periodically pH and conductivity were determined. The evolution of these parameters with the time can be observed in Fig. 8 for the three effluents. The conductivity of dicing water is expressed in μs/cm and that of beamhouse and textile effluents is expressed in ms cm^{-1}. Obtained results show that pH value remains constant

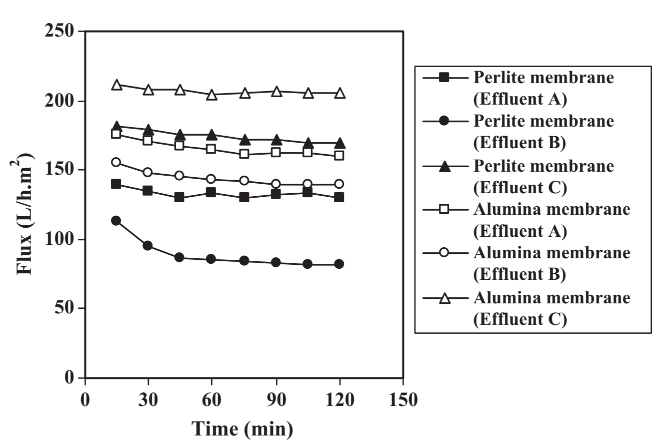

Fig. 7. Permeate flux of effluents as a function of operating time (pressure = 1 bar). (A) Beamhouse effluent, (B) textile effluent, and (C) dicing effluent.

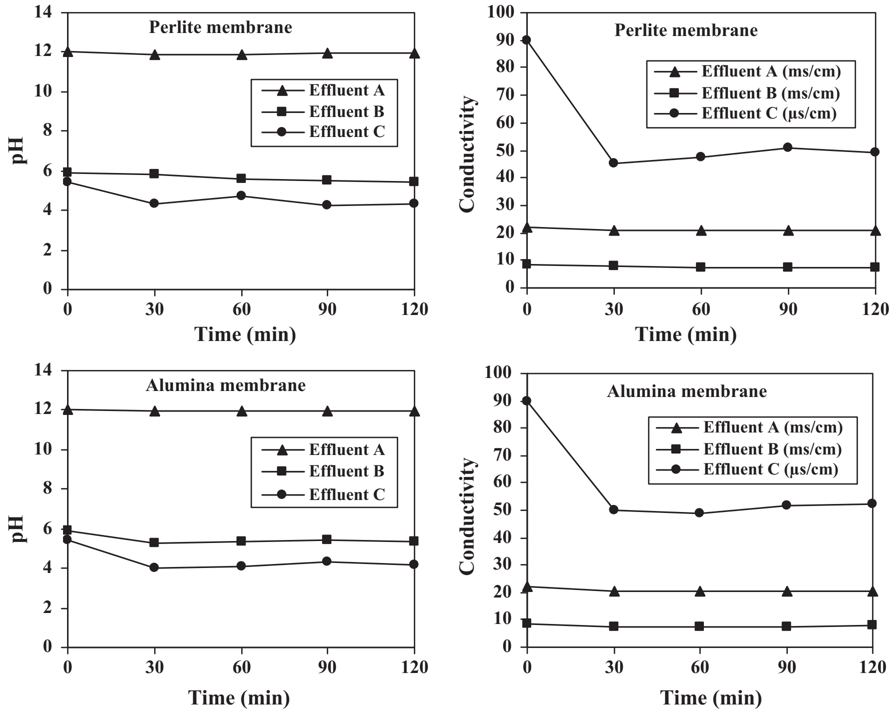

Fig. 8. Variation of pH and conductivity with the operating time (pressure = 1 bar). (A) Beamhouse effluent, (B) textile effluent, and (C) dicing effluent.

for effluents A and B but it decreases slightly for the effluent C during all the filtration tests. For the effluents A and B, the effect of membrane on conductivity is negligible because microfiltration process is not efficient to remove the soluble salts from effluent. However, the conductivity of effluent C decreases approximately to the half. But, by considering the unit of this parameter, the decrease of conductivity of effluent C can be considered no more significant. In the semiconductor dicing saws process, the CO_2 injection is a common and inert way to lower the resistivity of deionized (DI) water by dissolving carbon dioxide gas in the dicing cutting water. Probably, the lower decrease of electrical conductivity and pH, of dicing effluent after filtration with membranes, is due to the removal of an amount of carbon dioxide gas.

Turbidity was analyzed in order to show the effect of microfiltration membranes (M1 and M2) on reduction of pollutants present in beamhouse, textile and electronic effluents. As it can be seen in Fig. 9, the removal percentage of turbidity from beamhouse effluent is between 98 and 99%. In the case of effluent of dicing process, the microfiltration process was seen to remove turbidity from the feed completely. The removal percentage of turbidity is about 100% for two membranes which allow complete elimination of colloidal particles or suspended solids (Fig. 9). However, removal turbidity percentage ranging from 89 to 94% was determined in the case of textile effluent.Membrane treatment technology was shown to be also efficient in reducing organic pollutants from beamhouse and textile effluents. According to the results shown in Fig. 10, treatment by microfiltration decreases considerably the COD concentration. The reduction of COD of effluent A can reaches 50–54% and 58–65% using Perlite membrane (M1)

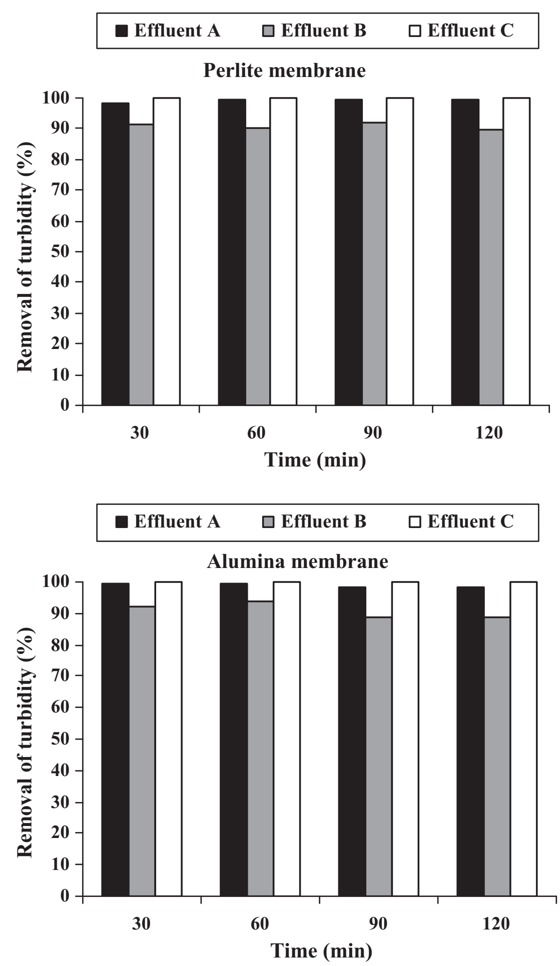

Fig. 9. Removal of turbidity as a function of operating time (pressure = 1 bar). (A) Beamhouse effluent, (B) textile effluent, and (C) dicing effluent.

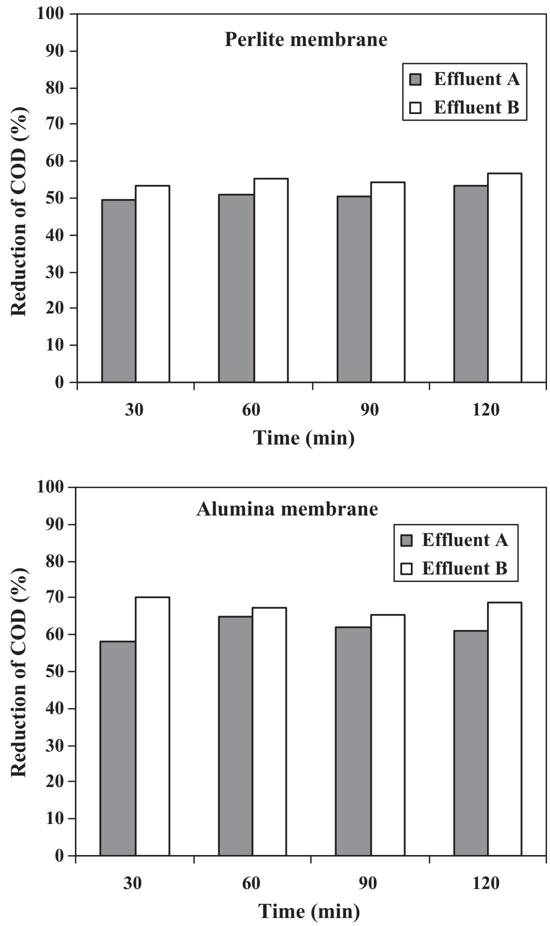

Fig. 10. Reduction of COD with the operating time (pressure = 1 bar). (A) Beamhouse effluent and (B) textile effluent.

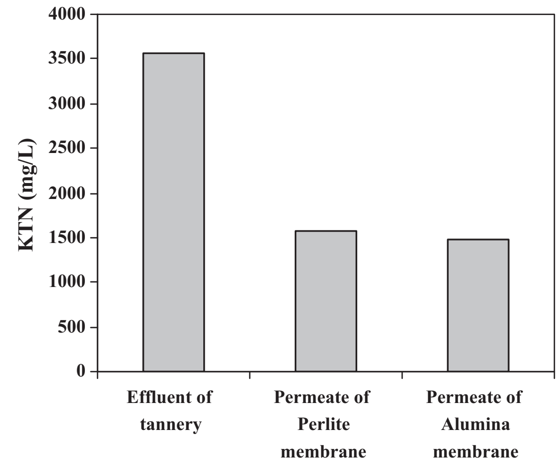

Fig. 11. Reduction of KTN content of beamhouse effluent by microfiltration process (pressure = 1 bar).

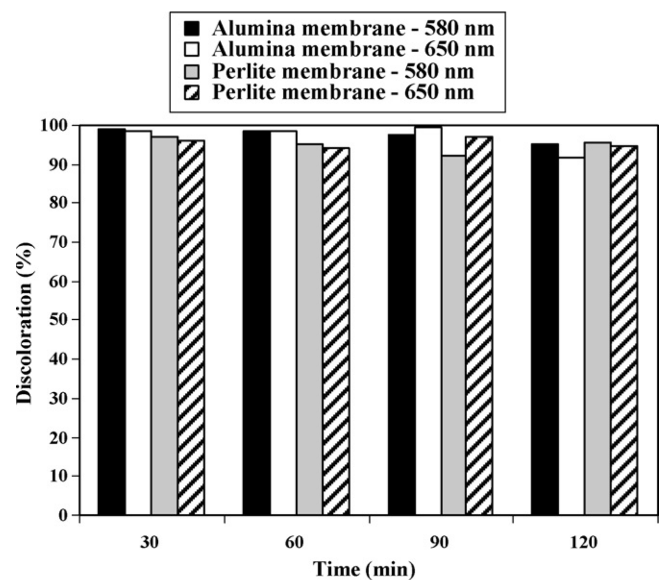

Fig. 12. Discoloration of textile effluent as a function of operating time (pressure = 1 bar).

and commercial Alumina membrane (M2), respectively. In the case of effluent B, membranes (M1) and (M2) are able to reduce COD by about 54–57% and 65–70%, respectively. The use of microfiltration treatment technology showed to be promising in removing pollution from tannery and textile wastewaters. Remained percentage in permeate effluents may be due to the soluble COD of the wastewaters which correspond mainly to hydrolyzed proteins resulting from unhairing process of

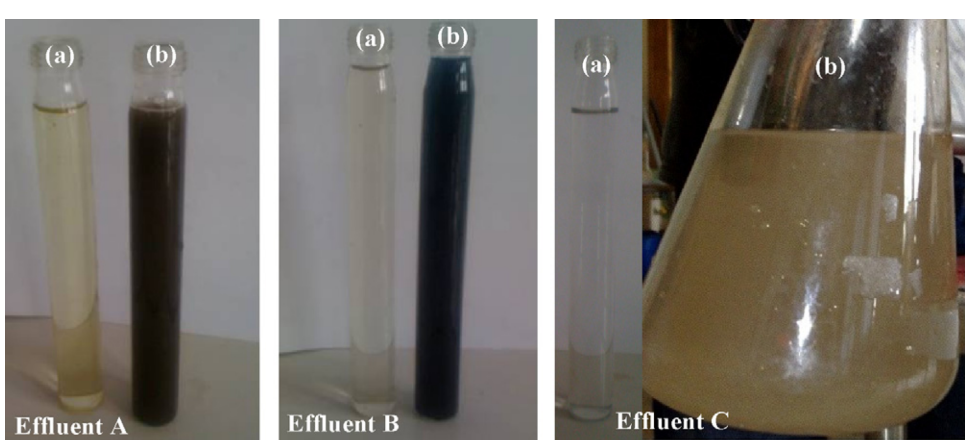

Fig. 13. Effluent after (a) and before (b) treatment using Perlite microfiltration membrane. (A) Beamhouse effluent, (B) textile effluent, and (C) dicing effluent.

tannery and to soluble matter of textile effluent. Obtained results reveal also that microfiltration decreases KTN content from 3570 mg/L to 1580 mg/L in the case of Perlite membrane and from 3570 mg/L to 1470 mg/L in the case of commercial Alumina membranes. The KTN rejection is 56% and 59%, respectively (Fig. 11). Results show clearly that rejection percentage of KTN is directly proportional to COD removal.

Regarding textile effluent, the color removal is of the greatest interest because color is considered the most important polluting parameter. Visible spectral analysis (between 400 and 800 nm) of textile effluent, diluted five times, shows two absorption bands at 580 and 650 nm. These wavelengths were used to assess the color removal process. Fig. 12 shows that the discoloration is complete by the use of tangential microfiltration; because effluent B contains colloidal particles which are responsible of coloration and turbidity. We conclude that the color is influenced by the turbidity effectively; color and turbidity have a proportional relation.As it can be seen, microfiltration using Perlite membrane proved to be effective in removing turbidity and color and in reducing COD and TKN with almost the same efficiencies than that obtained with Alumina membrane. Fig. 13 reveals that microfiltration process, using Perlite membrane, improves considerably the quality of studied effluents.

4. Conclusion

The performances of a low cost ceramic microfiltration membrane, with tubular configuration, made of Moroccan Perlite were studied. The membrane support was prepared by the extrusion of ceramic paste made with Perlite powder. The development of a microfiltration membrane requests a fine grinding of the Perlite powder. The microfiltration layer was deposited on the support by slip casting. This membrane has the advantage of having the same composition as that of his support. It therefore has a good adherence with the support made of same material (Perlite).

The membrane presents interesting retention properties with regard to the suspended matter, colloidal dyes and suspended silicone particles. Tangential microfiltration process operated at lower pressure (1 bar) was seen to remove turbidity from wastewaters completely. Perlite membrane allowed significant reduction of COD (50–54%) and TKN (56%) of beamhouse effluent generated by tannery industry. It showed a significant decrease of COD (54–57%) and a complete discoloration of textile wastewater. However, complementary treatment is necessary to remove or reduce residual pollutants due mainly to soluble matter of effluents. The combination of microfiltration with other techniques should be carried out to achieve the treatment of beamhouse and textile effluents. This is the objective of our later works.

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