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# Application and Treatment of Dye Congo Red, Metal and COD Retention by Modified Membranes Texture



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#### ABSTRACT

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In materials research, the development of ceramic products is extremely interesting in the field of the membrane supports because of their mechanical resistance, chemical inertia, long working life and thermal stability. Research is currently directed towards the exploitation of materials at a lower cost. The application of ceramic membrane was believed can be utilized not only for medical and oxygen separation but also for various industry such as food and beverages industry, textile industry, metal industry and environmental. The purpose of this work is the development of modified ceramic support based on kaolin for liquid waste processing. The study of the water flow and filtration through the supports made possible to highlight a variation of retention rate varying from 99 to 100% in function of composition, pore diameter, ion interaction and molecular weight. Tests of filtration of Congo red solutions, salts solution, lead solutions and industrial waste water were performed on modified support which was synthesized starting from a natural local barb tine. Mixed with aluminium oxide. Using Filtration of tangential type under an applied pressure of 5 bar.

#### Keywords:

Clay; material oxides; dye; membrane; filtration; retention rate; effluent

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#### 1. Introduction

The wastewater treatment using membrane has rapidly increased during recent years and has emerged as an important separation and purification method in various process industries to prevent water resources pollution by toxic element such as Mercury (Hg), cadmium (Cd) And dyes and are new used to obtain effluents without contamination [1].

A membrane is a physical barrier allowing selective transport of mass species. There are three large groups of membranes biological, organic (polymeric) and inorganic membranes. Inorganic membranes can be further classified into ceramic and metallic membranes [2]. The synthesis and characterization of ceramic membranes has been intensely studied due to their potential applications

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in the domains of gas separation [3-7], pervaporation [8,9], reverse osmosis [10] and nanofiltration [11-13], and the development of membrane reactors using catalytically active or passive inorganic. Membranes [14-21] disk and single-tube are the most common geometries of the microporous inorganic membranes.

The ceramic membrane can be used to sterilize the milk and whey, to purify the drinking water, to dewatering of products and the clarification of juice produced. For the textile industry, the ceramic membrane can use for desalination, separation of yeast and disposal of fat emulsions [22].

Silica sol deposition on alumina support layer membranes creates microporous silica which can be compared to reach very high helium permeation and selectivity [23].

Filtration separates constituents dissolved in the water from detritus and other solids that may alter the chemistry of the sample before it can be analyzed. However, filtration in the field adds the possibility of sample contamination if not done carefully [24].

The studies of inorganic membrane prepared entire by from clay and alumina, zircan silicate and the titan have just started [25,26], the industry importance was attracted by this membrane sample which can be applied in liquid separation and gas. The sea products conservation industry is very important in Algeria, it generates a huge amount of wastewater, which is generally rejected in the littoral and is then responsible for an enormous pollution, by supporting the eutrophication phenomenon.

In this study, we investigate the development of ceramic membranes, based on Algeria natural materials which are in abundance and need lower firing temperature. The applications of this membrane prepared from kaolin mixed with a percentage of Al<sub>2</sub>O<sub>3</sub> and coated with thin layer of alkoxide triethoxid-alumina in filtration process were used for the treatment and discoloration of effluent (dyes, heavy metals, salts and industrial waste) from wastewater [27-30]. This support membrane can be regenerated manually through gentle scraping or chemically using hydrochloride acid (PH=3) or sodium hydroxides solution (PH=10) [31].

### 2. Materials and Methods

## 2.1 Support

In the elaboration of the membrane support starting from Clay sample kaolin type of local origin [28] paste from Algeria material. The support prepared has the same configuration of tube monochannel, the equal length(L) 30 cm and equal diameter (D) 3 cm in the laboratory.

The different support was obtained by mixture of kaolin from Algeria and alumina  $Al_2O_3$  with 15% percentage of weight and heated at 1100 °C. after a casting, the supports were symbolized by SMS, [32-34] but the elaboration must be improved by adding an intermediate thin active layer with smaller pore sizes to the support SMS. The active layer was of alkoxide triethoxid-alumina, and prepared by sol - gel method in laboratories must be casting to 750°C and at this level temperature the layer which was deposited on a durable porous alumina. The results of the mixture were placed in a stainless steel. Then, the solution of  $H_2O$  and acid solution  $HNO_3$  are added which was then heated at 750°Cfor 24 hours [35].

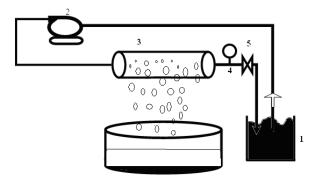
For this study, the support SMS prepared from kaolinin the chemical composition presented in the Table 1 and 15% of weight of alumina  $Al_2O_3$ , and the SMA is a same of SMS but with a thin layer. the chemical analysis reveals that this support ceramic is essentially formed with a great amount of silicat and alumina, The cross section microstructures of sintered samples were observed using scanning electron microscopy(SEM) given images of the supports are reported as shown in Figure 1, which gives information on the texture of the surface of elaboration supports membrane ceramic SMA with an energy dispersive X-ray spectrometer phase analysis of the support SMS was identified



by X-ray diffraction. The thermogravimetric analysis (ATG) differential scanning Calorimetry (DCS) the support SMA were performed with simultaneous DSC-TGA. the sample was heated from room temperature to 1000°C at a rate of 5°C/m under static atmospheric conditions. The performance of cross flow filtration with different supports SMS and SMA were tested by tangential filtration performed on laboratory scale filtration pilot plant Figure 1 at a temperature set at 25°C and transmembrane pressure equate 5bar and the flaw rate was set to 500 L/h .the membrane SMS and SMA was initially characterized by the determination of water permeability.

**Table 1**The chemical composition of kaolin

The chemical composition of Raolin			
Oxides	The mass percentage (%)		
SiO <sub>2</sub>	49,5%		
$Al_2O_3$	35,5%		
TiO <sub>2</sub>	<0,3		
$K_2O$	1,55		
$Na_2O$	<0,1		
CaO	<0,1		
MgO	<0,4		
PF	11,8		



**Fig. 1.** Schematic diagram of the experimental apparatus filtration flow diagram [30]. 1. Feed tank;2. Feed pump; 3. Tubular membrane ceramic; 4. Pressure gage; 5. Pressure valve; 6. Filtrate recovers

## 2.2 Characterization of the Support

Figure 2 and 3 presents the XRD patterns of raw thermally treated clay of supports SMS, it shows that all the peaks in difractogramme due to KAOLINITE (1), Muscovite (2) and Quartz (3) are the main minerals present in the raw clay. After casting at a temperature of 1100 °C peaks of (1and 2) disappear also, were as peaks of Mullit (4) appear due to the transformation of metakaolinite. The peaks of quartz (3) confirms the thermal stability of this phase that remain the peaks of Silica Morphed (5) and the alumina gives the polymorph structure presented with the peaks  $Al_2O_3\gamma$  (6) and  $Al_2O_3\beta$  (7).



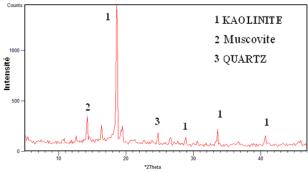
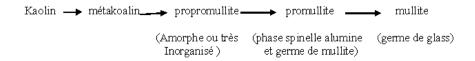


Fig. 2. X-ray diffractogram of the mixture of kaolin and  $15\% \text{ Al}_2\text{O}_3$  raw powder



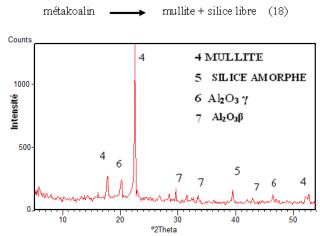


Fig. 3. X-ray difractogramme of the mixture of kaolin and 15%  $Al_2O_3$  heated to 1100°C

## 2.2.1 Thermal analysis

At 65 C°, there is an endothermic peak of Figure 4 Companied with mass loss, which were caused by the removal of residual physically adsorbed water in the simple. The second peak indicated organic components evaporated existing between 300°C and 400°C. And finely around 520°C accompanied with 1.5% mass loss, which is due to the transition from kaolinite to meta-kaolinite and structural water. The exothermic at 850°C the peak corresponds to the formation of  $Al_2O_3$ -Si spinel  $Al_2O_4$  by decomposition of meta –kaolinite.

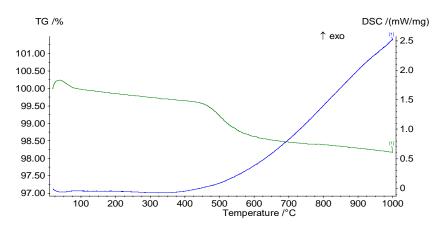


Fig. 4. Thermal analysis curve: DSC and TGA for SMC support

#### 2.2.2 Microstructure

The morphology of the surface and cross-section were recorded at 10Kv voltage and amplification 3000 (Figure 5) . The image of Figure 5 also shows that the support surface was uniformly structure and was well covered by the alumina particles with a different heat done for the mullite and the transition transformation of amorphous glassy phase, And there was a range formed for all simple with the average pore size about 1.5- 2.5  $\mu$ mand in hexagonal system. This porosity corresponded a transformation of the new crystalline phase for the first mullite to second mullite [36,37].

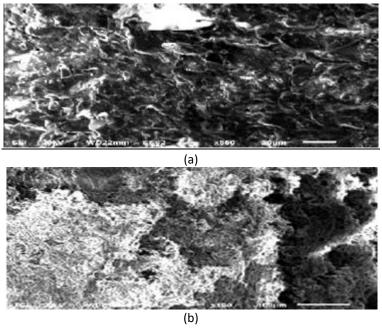


Fig. 5. SEM image of support SMS (kaolin and 15%  $Al_2O_3$ ) (a) surface section; (b) cross-section active

## 3. Filtration Tests

Figure 6, 7, and 8, presents the variation of the retention rates of Congo red solution ( $10^{-4}$  M), lead solution ( $10^{-4}$  M) as a function of time Under a pressure of 5 bar for the supports SMS and SMA. 3.1 The Permeability as a Function of the Pressure of the Different Membranes



Figure 6 shows that with increasing pressure values of the solution permeate flux increases linearly. A maximum permeate flux of supports SMS and SMA about 280 L/m $^2$  h, 1350 L/m $^2$  h was obtained at pressure values of 6 bar ,at an initial permeate flux of SMS and SMA support respective represented 80 L/m $^2$  h and 210 L/m $^2$  h at 2 bar .the difference obtained is due to the variation of the morphology of the surface between SMS and SMA supports and the nature of layer geometry (smaller pore size ).

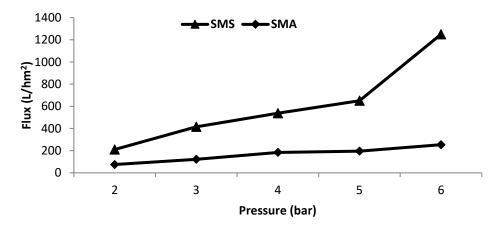


Fig. 6. Flux of water permeates versus the pressures of SMS and SMA

#### 3.2 Retention Rate

The support SMS and SMA was checked to ensure that the permeability remains almost variable the filtration efficiency in removing of the dye and heavy metal from the feed solution was evaluated through the composition rejection which was calculated using the classical rejection coefficient

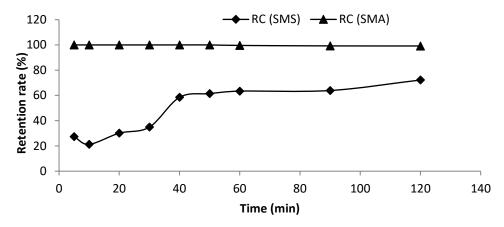
$$R_t\% = 1 - [C_P/C_0] * 100 \tag{1}$$

where  $C_0$  is the initial concentration of the dye in the feed solution and  $C_p$  is the dye concentration in the permeate. In this test, it was found the permeate quality in terms of removal of dye (Congo Red) and heavy metal (lead Pb<sup>2+</sup>), COD (chemical organic demand) the reduction of this samples are shown in Figure 7, 8 and 9, respectively. The concentration is calculated by the law of beer lambert  $A = C_2L$ .

## 3.2.1 Retention rate of dye solution (Congo red)

Figures shows that the retention rate of Congo red solution as a function of time the results presented an important remark between SMS and SMA supports. That the good rejection is obtained with SMA support a 100 % in 120 min, but support SMS the retention increases a function of time the Rt% = 35 % to 60% in 60 min and these values stand near 70 % after 120 min. The important parameter of this rejection consisted in the RC molecules are smaller than the SMA pore and the retention will be attributed to by the adsorption of dye and interaction between solute ions and material charge [38].





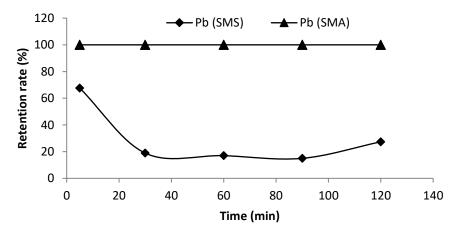
**Fig. 7.** Retention rate of Congo red solutions (10<sup>-4</sup>M) versus time of the SMS and SMA supports ceramic membranes

## 3.2.2 Retention rate of lead (Pb<sup>2+</sup>)

The Figure below shows the variation of the retention rate according to the time(min) through the SMS and SMA supports as obtained from removal lead solution. The concentration of lead in the solution is 1000 ppm.

The coefficient where retention rate obtained with SMA support 100% in 120 min of treatment filtration that the SMS support retention rate decries between a 75% to 20 %.

These results are in agreement with a previous study [39] that explained this phenomenon in terms of whey adsorption.



**Fig. 8.** Retention rate of lead solution versus time of the SMS and SMA supports ceramic membranes

## 3.2.3 COD reduction

The supportt SMS can be used for the treatement of wastewater treatment produced by sea products conditioning in our case. The application of the test filtration a SMS in the effluent produced can give the good results. The used samples were collected from a reject product factory located in towns such us relizane, Timouchente and mostaganem (ALGERIA) effluents represented in the Table 2 with the characterization initial of the raw effluent.



**Table 2** the initial of the raw effluent characterization

Effluent	Conductivity (µS/cm)	рН	COD (mgO <sub>2</sub> /l)
BCR Factory	1336	7,19	360
Hydro Canal	2481	9,87	280
TUchlore	4765	7,78	184
UR.W.W	1841	7,71	988

The results of COD retention rate (COD is chemical organic demand determinated by calorimetric method) conform the efficiency of this filtration prepared SMS to effluent treatment. Figure 9 shows a noticeable elimination of suspension matter illustrated by the change of the samples of effluents colours as well as elimination of the turbidity .the results of the COD with R.U efluent is very important there is reduction the 988 mg d'O $_2$  / L to175 mg d'O $_2$  / L after 120 min of treatment. But for the effluent of BCR, SOACHLOR and Hydro- canal the notice of COD values obtained respective360 mg d'O $_2$  / L, 280 mg d'O $_2$  / L et 184 mg d'O $_2$  / L however the reduction is very low to a point commented of COD above 80 mg d'O $_2$  / L. The VARIATION of COD evaluates according to the following order, where the reduction of COD is right in the environmental standard 120mg O $_2$  / L. Urban WASTEWATER > BCR factory> HYDRAUCANAL > TUchlore.

The results of the retention permeation salts in the versus time is represented with the Figure 10. The retention rate of the salted solutions (MgSO<sub>4</sub>, MgCl<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub> and NaCl) at  $10^{-4}$ M of concentration through SMS and SMA supports variates between 8% and 50% in the pH of solution obtained 5,8 to 6.2.

The SMA supports have a higher salt retention that the SMS support as clearly that the selectivity of retention rate for the SMA support increase with the sample salt solution containing  $Na_2SO_4$  and  $MgCl_2$ , that the retention rate obtained for  $MgSO_4$  solution is equal to 8% after 120 min after filtration.

The beginning results of retention rate given by the SMS support follow the order: Na<sub>2</sub>SO<sub>4</sub>> MgSO<sub>4</sub>=NaCl> MgCl<sub>2</sub>. And with SMA support Na<sub>2</sub>SO<sub>4</sub>> MgCl<sub>2</sub>> NaCl> MgSO<sub>4</sub>. We Can observe that the retention rate with bivalent ions is higher than monovalant ions.

The retention rate with SMS support is less and it strongly with SMA supports, it depends on the salt nature [40]. It has been postulated that the decrease in rejection rate of salt solution cult result from pore swelling and the increase due to repulsion forces between couterions ion in the electrical layer at the pore walls and / or from Hofmeister effects [41-44], That is a partial dehydratation of natural moleculars in the presence of ions because water would preferentially solvate ions. Therefore, the idea of sandwich was proposed in the study of [45,46], to compensate for the imperfection of the pore size distribution of the available commercial membranes.

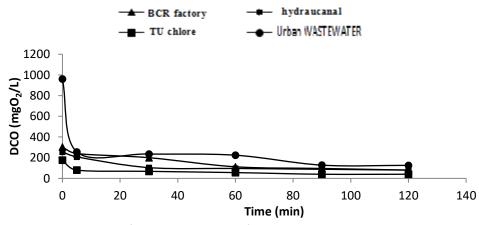


Fig. 9. Reduction of COD versus time of the SMS support ceramic membrane



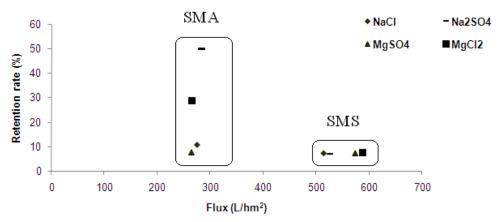


Fig. 10. The salt rejection of the permeated of membranes SMS and SMA

### 4. Conclusions

In this work, a novel low ceramic supports were prepared from local mineral kaolin and alumina oxide. It is confirmed that the sintering of support was fixed at 1100°C, and the filtration layer deposited onto the support SMS was obtained by slip-casting method.

The support SMS and SMA shows that the permeat flux given is high about 1286,21 L/ hm<sup>2</sup> with SMS support and 270,3 L/ hm<sup>2</sup> for SMA. The tast of filtration can be employed to separate the dye, salts and heavy metals from aqueous solution.

The result obtained for retention rate for congo red with SMS support between 75% to 20 % and with SMA support rejection is 100% as well a same for the retention of lead (heavy metal). The SMA supports have a higher salt retention than SMS support as clearly that the selectivity has an important effect and depended to the charge repulsion that can coexist with adsorption phenomena. These supports have a retention rate COD a range 80 %, the increase in COD water filtrate may be explained with the desired pore sizes of filtration.

These membrane support SMS and SMA can also be used in wastewater produced by surface treatment in industry and have an interesting application in physical and biological decontamination.

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