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Elaboration of low-cost ceramic membrane based on local material for microfiltration of particle from drinking water

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ABSTRACT

A low-cost, ceramic membrane was prepared from local Cameroon clay (collected from Mouka village), small amount of binding materials and organic material (rice husks). The support was prepared by paste casting. It was sintered at temperature of about 1100 °C. The raw materials and the prepared support were characterized using thermal analysis, X-ray diffraction and scanning electron microscope. Subsequently, porosity, flexural strength, chemical stability, pure water permeability, phenol and particle elimination was investigated. At this temperature, the flexural strength, chemical stability and porosity of the membranes were found to be excellent. Based on these results, average pore diameter was 0.459 µm with porosity of 36.83 % and flexure strength of 22 ± 5 N. At this average size, the support was appropriate in microfiltration and can retain micro-organism. The results revealed a maximum retention of 98 % for particle suspension after continuously running for 2h without backwashing; high permeate flux of 0.83 $cm.mm^{-2}.min^{-1}$ was obtained in the permeability of pure water at frontal mode filtration.

Keywords: clay material, drinking water, Microfiltration, Ceramic membrane, particle.

INTRODUCTION

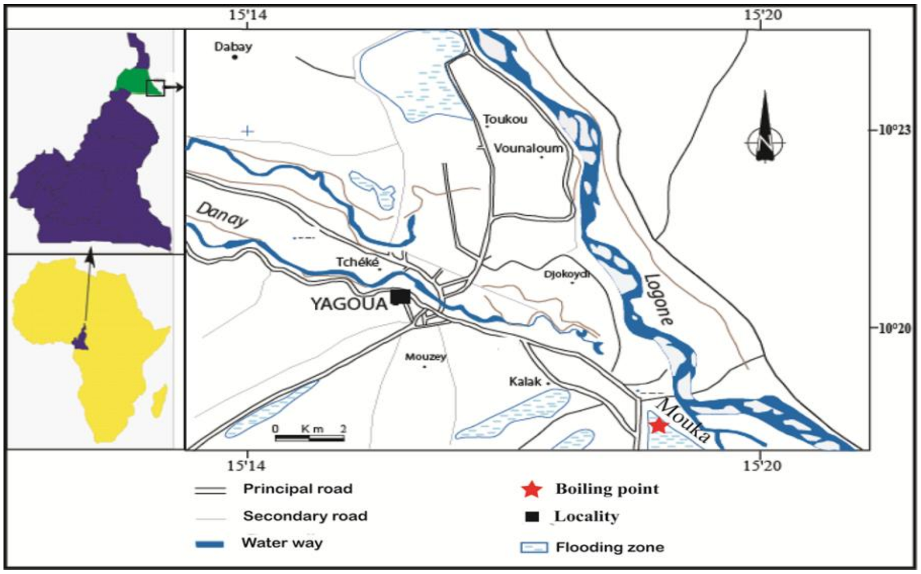
During the last few decades, the applications of ceramic membrane have increased due to its excellent chemical, thermal and mechanical stability and higher separation efficiency [1–5]. In the near future, the exploitation of new type of ceramic materials and simple fabrication techniques could play a significant role for the preparation of low cost membranes [6]. Generally, Membrane processes are more and more used in the pollution treatment. Unfortunately, these membranes are too expensive for economic application in environmental technology which requires high permeation fluxes and low costs to treat great volumes of dust-contained hot gas, industrial wastewater and drinking water. Afterward alumina-based ceramic membrane for industrial application is limited due to its higher cost and sintering temperature [7, 8]. As a result, 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 [9–17].

The development of ceramic membranes based on natural materials and some powder wastes such as fly ash was investigated by several authors [18–27]. Clays are in abundance and need lower firing temperature in comparison with metal oxide (alumina, zirconia, titania, and silica) [28]. Rice husks are abundant waste in Mouka village Cameroonian. This vegetal waste was good enough for the preparation of low-cost ceramic membrane due to its high percentage of silica. Mittal et al. [29]; Nandi et al. [30] showed effective retention of ceramic membrane in oily wastewater. Furthermore, the removal of microorganism for the purification of drinking water using different types of ultrafiltration and microfiltration membrane having different pore size was studied by Mavrov et al. [31] and the results indicated a significant reduction of microbes. Majouli et al. [32] remove suspension matter (4 g/L) from drinking water using membrane of percentage removal between 95 and 100 %. Fangshu Qu et al. [33] used membrane to remove organics matter for the purification of drinking water. Barredo-Damas et al. [34-35] treat efficiently textile wastewaters based on membrane. Llanos et al. [36] develop ceramic process membrane to separate heavy-metal ion from waste-water. In this aspect, the application of membrane filtration is effective in much case for water treatment.

The purpose of this study was to elaborate low-cost porous ceramic support based on abundant natural resources such as clay material, rice husks and another mineral powder as melting. The choice of rice husks as a porogenic agence was based on its qualities: relatively low-cost, it abundance in the region and its tenor in silicate element.

MATERIALS AND METHODS

Localization of precursor sample zone: This clay material was obtained from Mouka village, precisely in Mayo-Danay division in the Far North Region of Cameroon as indicated in figure 1. This clay material was sampled at a depth of 1.5 m. The rice husks were collected from the Yagoua town. The geographical coordinate of Mouka village is shown in figure 1.



- North 10.05700° -East 15.25264° -Altitude 332m

Figure 1: Geographical location of the sampling site.

Purification and fractioning of the precursor: After collecting the clay material, stones and other heavy particles were removed from the samples by sieving. The clay material was crushed and about 500 g of the powder was dispersed in 20 L of deionized water. After sedimentation of the clay particles, the top part or supernatant was sieved through a 50 µm-sieve to remove the larger non-clay fractions and to obtain the fine clay fraction. The samples were dried at 100 °C and stored in a desiccator. The organic additive used in the paste formulation was only the rice husks. Prior user, the rice husks were crushed and sieved through a 50 µm-sieve to obtain the fine fraction of particles that were used as organic pore-forming agent or as porogenic agent. The sepliolite rock was crushed and sieved through a 50 µm-sieve. This rock played the role of the melting in the mixture powders.

Equipment used: Chemical compositions of natural clay powders and the rice husks were analyzed using a wavelength dispersive X-Ray fluorescence (Shimadzu XRF-1800). The particle size distributions of the obtained powder clay were measured using a laser diffraction particle size analyzer (Malvern Mastersizer 2000). Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed with simultaneous DSC–TGA 2960 TA Instrument under argon. Both the clay and rice husk samples were heated from room temperature up to 1200 °C at a rate of 5°C/min under static atmospheric conditions; a-Al_2O_3 as external reference. A Linseis dilatometer type L75 to determine the thermal expansion of pastille section samples shaped by uniaxial pressing (100MPa). For a good rigidity of the disk, the press uniaxial model (Perkin Elmer N°: 15.011) was used, pressing at about 10 MPa. For mixing the powder, the Rotary mixer model (Heindolph Reax type 2 N°S: 541-21001-002) was used at a rate of 65 rpm for about 50 min. The diameter of the support was determined by a slide gauge. The microscopic analysis was performed using a scanning electron microscope Hitachi S-4500 for powders analysis and for imaging of the support. Porosity measurement was carried out by the use of a Mercury Porosimeter (Model Autopore III, Micromeritics Instrument Corporation, U.S.A.). The suspension turbidity was measured by a turbidimeter / Nephelometer model (Hach, Ratio XR 43900).

Formulation and shaping: The plastic pastes were prepared from the clay material powder were homogeneously mixed with rice husk, melting and water. Plasticizer and binder were required to prepare a paste with rheological properties allowing the shaping by extrusion. The optimized composition of the paste was: (i) 61.54% (w/w) clay powder, sepionite rock as melting 15.38 % (w/w) and (ii) organic additives: the rice husks as porosity agent 23.08 % (w/w). The mixture was aged in order to obtain a good homogeneity mixture powder. After adding water, the mixture was aged during 25 min in the same box. Obtained paste was stocked for at least 24 h in a closed box under high humidity. Paste was then extruded and calendered into a thin film which was segmented to form flat disk supports with a diameter of 5 cm and to tubular box form. The extruded pieces were dried at ambient temperature for 24 h and transferred in an oven at 50°C for 24 h then sintered at 1100°C in a furnace.

Sintering program: The firing temperature, fixed at 1100 °C, is reached following the program shown in figure 2 according to the thermal analysis.

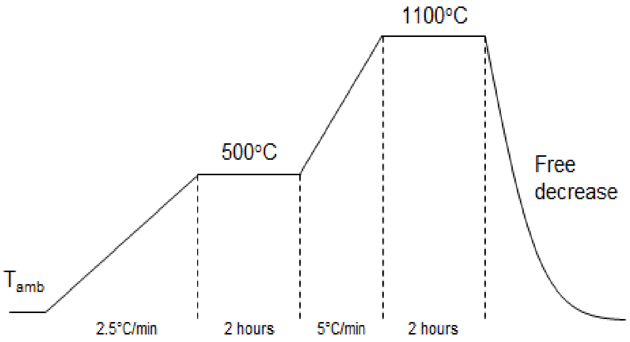


Figure 2: Temperature-time schedule used for the support sintering.

Filtration test: The frontal-flow mode of microfiltration was adopted in this study. The experimental set-up is shown schematically in figure 3. Before the filtration tests, the membrane had been conditioned by immersion in pure distilled water for 24 h. All filtration experiments were conducted at ambient temperature. The pure water flux J_t $(cm^3.mm^{-2}.min^{-1})$ was observed to be high initially and reduced to a steady value after one and two hours of operation of support studied. Permeate flux of the support was determined using standard equation [37, 38].

$$J_t = \frac{V_{(t)}}{S.t} \quad \textbf{(1)}$$

$V_{(t)}$: is the volume of the feed in function of times; : is the functional surface and t : is the time. The clarification of a suspension of clay powder at 0.9 g/L (pH: 7.72, turbidity: 55.53 NTU and at atmospheric constant transmembrane pressure) was done on the support. Suspension turbidity was measured. This test was conducted to provide guidance to know the ability of the media for microfiltration. The steady flux used in this paper was the filtrate flux at the end of 2 h. The turbidity rejection efficiency R_f (%) was calculated according to the following expression [39].

$$R_f(\%) = \frac{T_f - T_p}{T_f} \times 100 \quad \textbf{(2)}$$

T_f : is the turbidity in the feed, and T_p: is the turbidity in the filtrate.

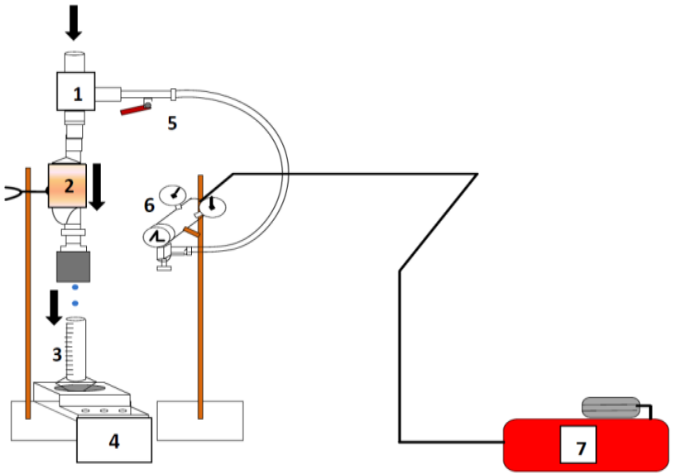


Figure 3: Schematic filtration pilot used for particular suspension: (1) feed and permeate tanks; (2) membrane; (3) permeate; (4) scales; (5) tap; (6) pressure gauge; (7) compressor pumps.

RESULTS AND DISCUSSION

Chemical composition of the powders: The chemical composition of the powders is given in table 1. The majority of the used powder consists of SiO_2, Al_2O_3. These elements are of 68.24 % for clay material, 81.50 % for the melting and 25.57 % for rice husks. These elements are fundamental for ceramic study. The other percentage is a mixture of different alkali metals for powder. The rice husks have 64.4 % of loss on ignition. That is the organic material or porosity agent.

Table 1: Chemical composition of the used precursor powder

Element (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	TiO_2	Mn_2O_3	P_2O_5	LOI[a]
Clay	43.76	24.48	8.71	1.26	0.58	0.91	0.21	0.08	1.54	0.02	0.10	18.30
Melting	69.35	12.15	0.78	1.91	0.06	4.48	3.89	0.12	0.15	0.03	0.02	1.67
Rice husks	24.89	0.68	1.23	0.64	0.87	1.67	0.19	0.04	0.24	0.05	2.22	64.4

[a] Loss on ignition

Thermal analysis: The thermo-gravimetric analysis (TGA) and differential (DTA) of clay material, the melt and the rice husks are shown in Figure 4. The dilatometric analysis of mixture was realized. Thermo-gravimetric analysis of Mouka clay material (Figure 4a) indicated an important weight loss between 25 and 200 °C. This loss is due to the removal of adsorbed surface water [32; 40]. The differential thermal analysis (DTA) shows two endothermic peaks: the peak at around 657 °C corresponds to the loss of structural hydroxyl groups and is due to the transformation of kaolinite to metakaolinite according to the following reaction [6; 41; 42], The second endothermic peak at 1059 °C can be attributed to the formation of crystalline mullite. This peak is smaller because of the effect of dilution in matrix [6; 29; 32,].

Al_2

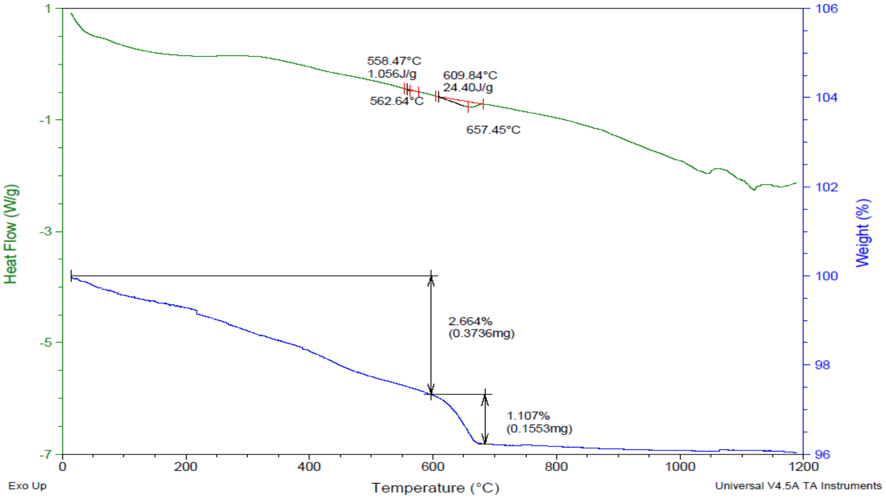


Fig. 4a

Thermo-gravimetric analysis of the melting material (Figure 4b) indicated too, an important weight loss of about 14 % between 25 and 600 °C. The first significant weight loss of 7.18 %, and appearing at 200 °C, corresponds to the departure of water (moisture or adsorption) due to attraction on the surface of the sample and water inserted between the layers or in the cavities of the crystalline structure [39]. The second weight loss of 6.75% appearing at 453 °C corresponds to the decomposition of carbonates [43, 44].

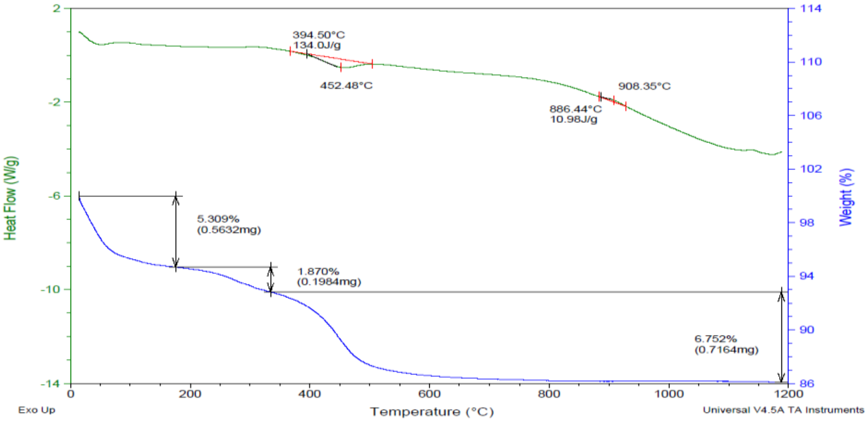


Fig 4b

Figure 4c shows TGA/DSC curves of the rice husks. A total weight loss of about 79 % was observed up to a temperature 500 °C, beyond which there was no noticeable weight loss. One can observe three well-defined regions for the weight loss; one below 100 °C and another from 100 to 500 °C. At temperatures below 100 °C, the weight loss could be due to the evaporation of adsorbed water. Between 100 and 500 °C, the weight loss might be due to the combustion of organic constituents of the soil. The DSC result shows an endothermic peak at 75 °C, corresponding to the evaporation of water, and an exothermic peak at 310 °C due to combustion of the organic precursor. A large exothermic peak has been observed at 437.36 °C, which can be associated with crystallization of SiO_2. These thermal analyses were originally of the sintering program applied in this study.

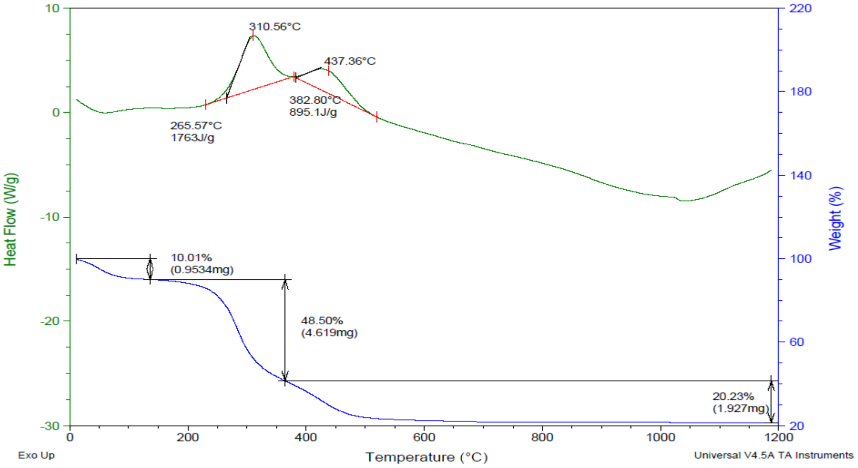


Fig.4c

Figure 4: TGA / DTA curves of (a) Mouka village clay material; (b) the melt and (c) the rice husks.

Figure 5 shows the dilatometric measurements of the mixture powders used in this study. The sintering process begins at about 900 °C. The addition of the melts decreases the sintering temperature of the raw clay [45].

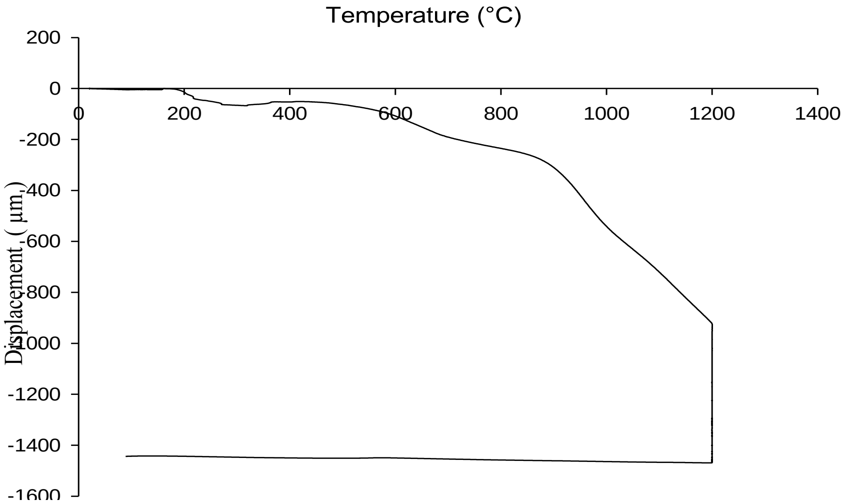


Figure 5: Dilatometric measurements of the mixture formulation powders used in this study.

Texture clay powder characterization and particle size distribution: The SEM (scanning electron microscope) micrographs of raw Mouka clay shown in figure 6 indicate that the clay grains don't have a given structure and the clay does not have a particular form but its distribution remains homogenous.Particle size of the clay material determines the porosity and the average pore size of the membrane. Moreover, the pore growth mainly depends on the initial particle size of the raw materials [46]. The raw clay material used in this work was analyzed for particle size distribution (PSD) as shown in figure 7. The PSD of Mouka clay material shows a narrow distribution in the range between 0.1 and 5µm. An overall observation conveys that the majority of the particles are inferior or equal to 5µm.

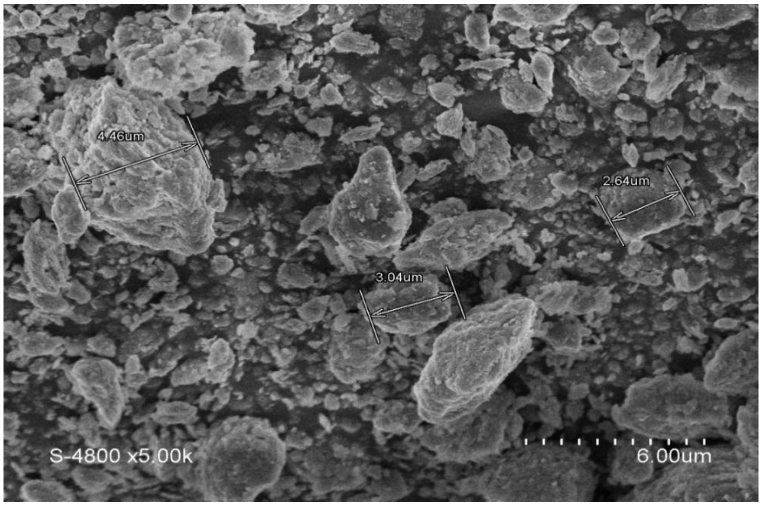


Figure 6: SEM micrographs of Mouka raw Clay**.**

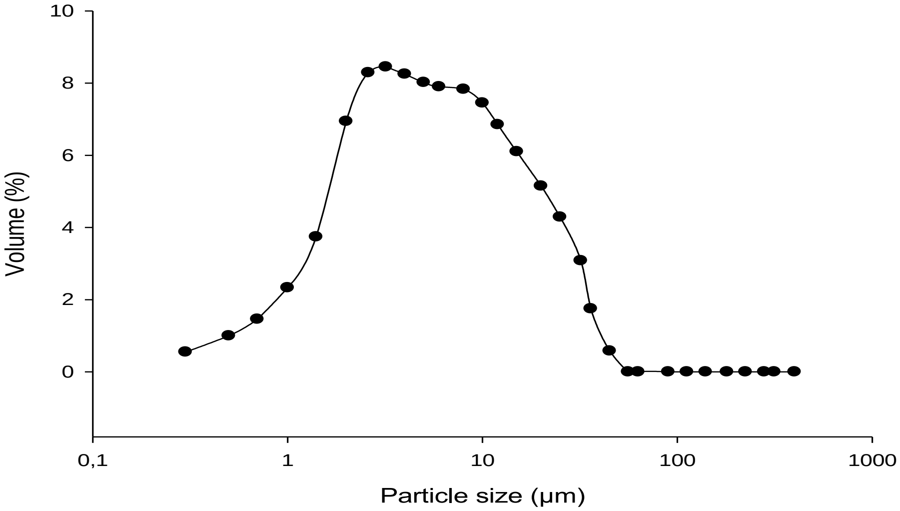


Figure 7: Particle size distribution of the Mouka clay material.

Elaboration of porous supports: The principal advantage of the rice husk is that they are eliminated by combustion during the thermal treatment. Sintering is a method for making objects from powder, by heating the material in a sintering furnace below its melting point (solid state sintering) until its particles adhere to each other. The diameter of the elaborated flat support decreases from 50 cm to 40.4 cm after sintering at 1100 °C; this is due to the shrinkage phenomena which attain 19.4 % after sintering. The change of the coloration is due to the oxidation of the Fe^{2+} ions [32].

Surface and cross-section texture: The observation of microstructure by SEM (Figure 8) reveals that the surface layer is homogeneous. The membrane sintered at 1100 °C is more consolidated due to the fact that for sintering temperatures over 900 °C the particles agglomerate together creating denser ceramic body. This more consolidation justify the good mechanic resistance of the membrane which was (22 ± 5) N. The important tenor of element S_2O_3 into the mixture is responsible of this structure and the good mechanic stability.

Mercury porosimetry: The mercury porosity in figure 9 showed that the average pore diameters and porous volume of the support (firing temperature of 1100 °C) are about 0.41µm and 36.84 %. The pore size values indicate that this membrane can be utilised in the microfiltration range. This result is very important because the microfiltration in the support is without another active layer. The good formulation mixture and the temperature of sintering were jointly responsible for these results.

Chemical stability: In order to determine its corrosion resistance, the support sintered at 1100 °C, was soaked at 50 °C using nitric acid (pH = 3) and soda solutions (pH = 12). And the weight of the support was measured each day. The results reported in fig. 10 show that Mouka clay supports presents a good chemical resistance in acidic medium and in basic medium. For 7 days of treatment, the weight loss does not exceed 0.1 wt. % in two solutions. This porous support presents a good chemical resistance than perlite supports synthesized by Majouli et al. [32], who has similar result in acid where the weight loss does not exceed 0.2 wt.% However, in basic medium, the weight loss can reach 6 wt.%.

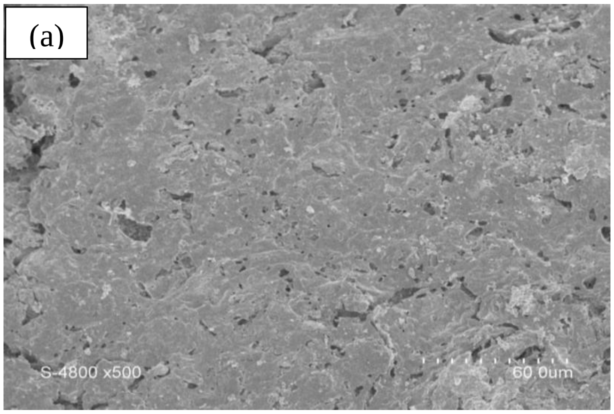


Figure 8 (a): SEM images of surface.

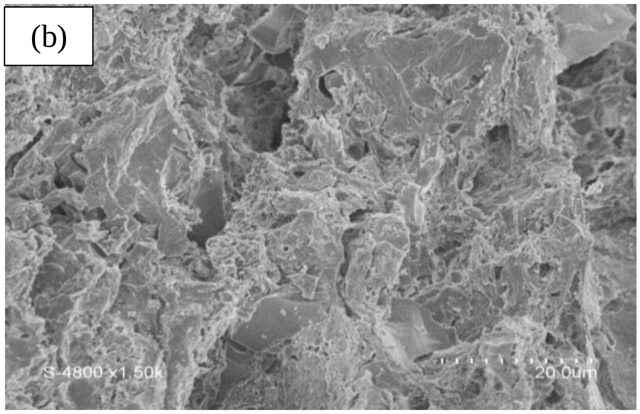
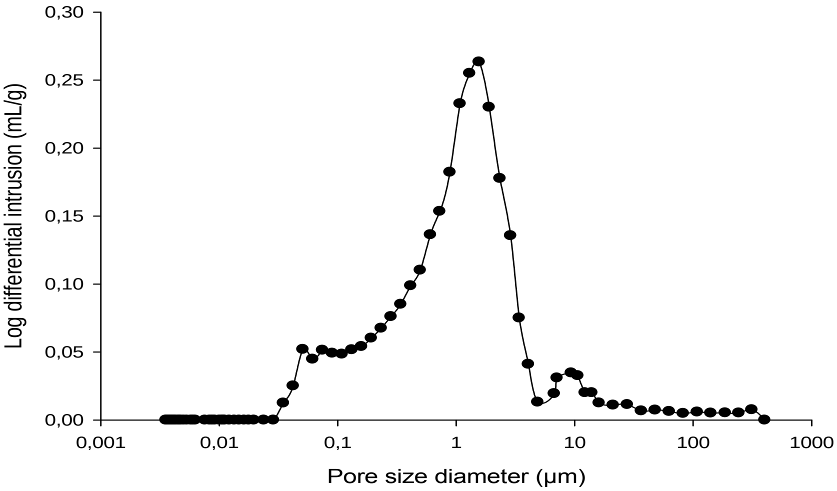


Figure 8 (b): SEM images of cross-section of membranes.



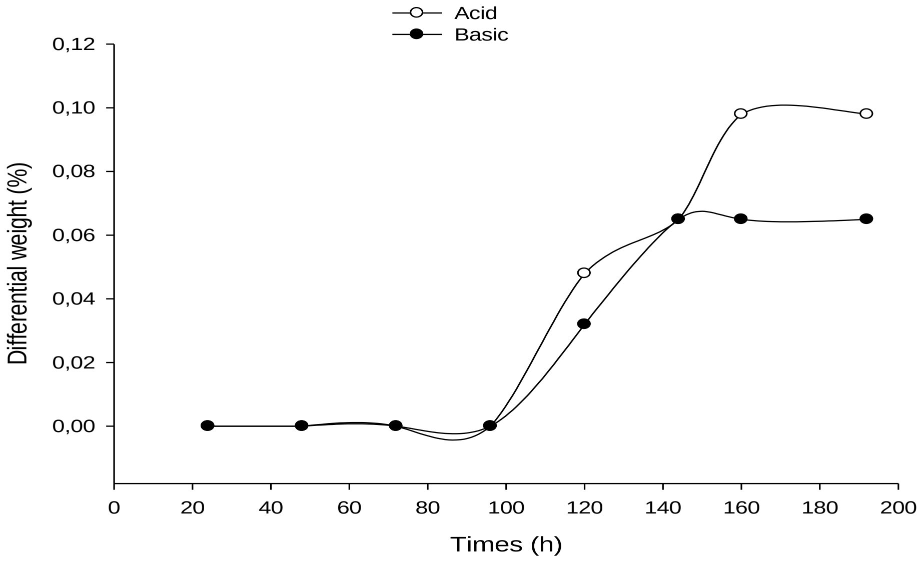


Figure 10: Weight loss of support in nitric acid and soda solutions as a function of time.

Pure water filtration: Figure 11 shows that pure water volume through the support depends on the time. It shows a quasi-linear increase in volume with the increase in time. This observation was predictable when the support is porous and the feed liquid contains no element in solution which can reduce the volume of the filtrate by stopping progressively the pores.

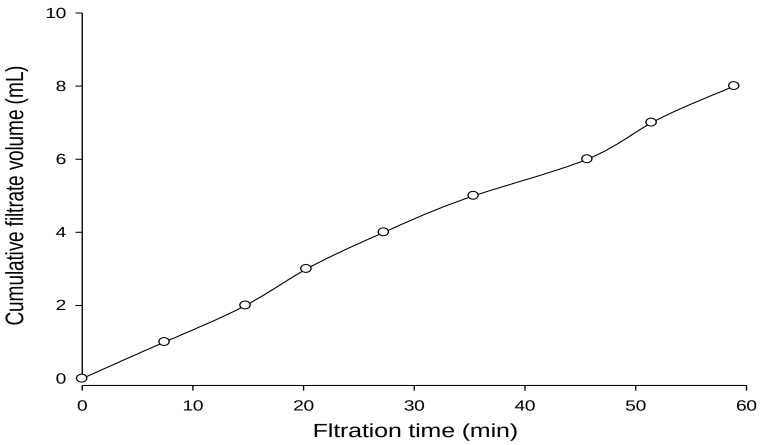


Figure 11: Pure Water cumulative filtrate volume versus filtration time.

Microfiltration of the particles: The data in figure 12, suggest that after the second filtration cycle, there is a significant increase of rejection which may attain an asymptotic value for the particle. This trend seems to be in accordance with the previous observations about irreversible fouling taking place practically during the first and second cycle. Generally the reason for increasing rejection efficiency with the increase in particle was due to the formation of larger droplets by coalescence of the smaller particle and formation of larger particle. Moreover, particle retained by the support build up a secondary layer on the support surface which further reduces the pore size of the support. Thus, rejection was increased with time and cycle. Similar trends were reported by Mittal *et al.,* [15]; Mourouzidis-Mourouzis and Karabelas [47]. The maximum rejection in this work was found to be 98.54 %.

However, the effect of deposited amount causing reversible fouling appears to be greater at higher pressures, which along with the actual particle rejection, seems to provide another indication for the presence of a surface fouling layer, being subject to compaction effects by increased pressure.

Permeate fluxes: The variation of permeate fluxes of particle solution and phenol solution as a function of time at atmospheric pressure is shown in figure 13. As it can be observed, flux decreases with time; this can be explained by the fouling phenomenon which increases by the use of frontal filtration. As shown in Figure 13, a continuous decline in permeate fluxes was observed within the first 80 min. Afterward, from 95 min the permeate flux of particle started stabilizing due to cake layer formation.

However the pure water presented higher permeates fluxes and constant. This highest flux behaviour and constant probably related to the strict absence fouling and cake and pore blockage resistances due to the solution.

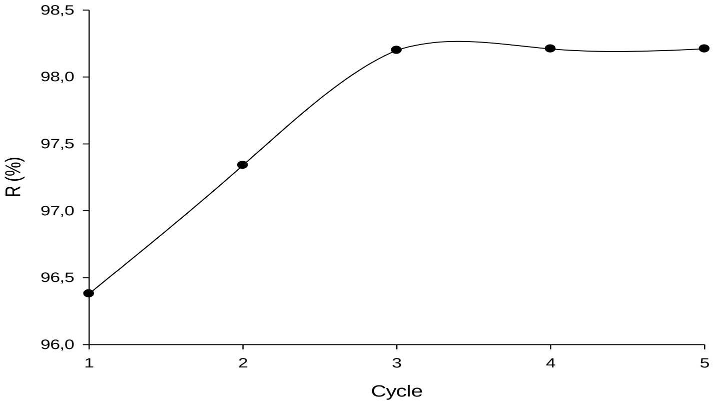


Figure 12: Estimated particle rejection at each filtration cycle.

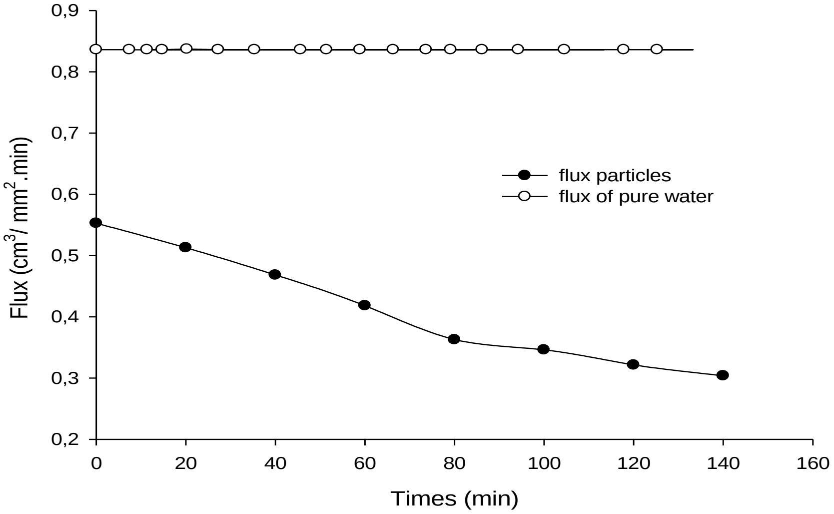


Figure 13: Permeate flux of particle as a function of time.

APPLICATIONS

The filtration tests show that the membranes manufactured based on clays can be applied for the treatment of waste water.

CONCLUSIONS

A low cost ceramic support for microfiltration based on natural Mouka clay of Cameroon and powdered rice husks as a porogenic agent was developed. The support was synthesized by extrusion of the paste and sintering at 1100 °C. Thermal analysis and SEM analysis shows that the effect of sintering was remarkable at this temperature. The structural properties of Mouka clay support were satisfactory in terms of porosity (36.83 %) and pore diameter (0.459 µm). The support provided good flexural strength (22 ± 5 N) and chemical stability (≤ 0.10 % weight loss in both acid and basic medium).

These experimental results show that mineral Mouka clay and rice husk are appropriate material for the development of microfiltration support. One application of this support is to produce drinking water laded of colloids; which could be applied to industrial wastewater treatment.

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