

Physico-Chemical Characterization and Stability Study in Acidic and Basic Solution of Ceramic Filters from Mouka's Clay (Cameroon)

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Abstract

This paper deals with the characterisation and study of physico-chemical stability of ceramic filters from clays and rice husk obtained from the far north region of Cameroon (Logone Valley) and their application in potable water treatment. Clays from Mouka were characterized by FTIR analysis. The results showed that the filter formulated with a mixture containing 80% clay and 20% rice husk of 100 μm in size each gave the lowest filter shrinkage rate; these formulated filters were chosen for the remaining work. Leaching tests showed that with filters at a sintering temperature of 830°C the leaching was not observed under neutral (pH 6.8) and acidic (pH 5) conditions as compared to 950°C and 1000°C where the leaching was observed. In basic (pH 9) condition, all the filters obtained released ions. Leaching tests revealed that the conductivity of the leachate for the filters sintered at 830°C was lower than those sintered at 950°C and 1000°C. Meanwhile, conductivity decreases with increasing sintering temperature (temperature up to 830°C) due to the fact that ceramization starts as from 850°C that leads to an amorphous state that favours chemical stability, the leaching ions were Fe^{2+} , Ca^{2+} , Mg^{2+} and Al^{3+} . The ceramic filters sintered at 950°C were applied to the filtration of water and the performance in terms of turbidity reduction was 95% and the flow rate after 50 minutes was 100×10^{-3} L/h.

Keywords

Ceramic Filter, Chemical Stability, Clay, Filtration, Lixiviation Test

1. Introduction

According to World Health Organization, over one billion people do not have

access to safe drinking water. In Cameroon, disparities between urban and rural areas are evident. In fact, 48% and 5% of rural and urban inhabitants use surface water or other unimproved water sources. Previous monitoring programs executed in the Logone Valley in the Far North region of Cameroon reported high turbidity in water resources. Turbidity is a typical problem of surface water and shallow open wells, as observed in the studies carried out in Cameroon.

Ceramic membranes for water filtration are one of the alternative techniques for treatment of drinking water [1]. Ceramic water filtration is the process of passing water through a porous ceramic material. It is a promising way to reduce the burden of water-borne diseases; it is affordable in terms of cost and made from local resources [2]. It has been shown that ceramic filters can reduce turbidity [3] and microorganism [2]. Meanwhile, physico-chemical properties of ceramic filters such as porosity and chemical stability are important to evaluate the filter efficiency. These properties are linked to the amount of porogen and sintering temperature. Previous works in the field have shown that porosity of ceramic filters decreases with increase in firing temperature for the same composition of clay and porogen fired at different temperatures [4] and that the physico-chemical stability increases with increase in firing temperature due to the formation of more crystalline phase such as mullite [5]. But no matter the crystalline phase present in the material its stability can be influenced by the physico-chemical properties of the feed water such as pH. Recent monitoring programs executed in the Logone Valley in the Far North region of Cameroon reported that pH of surface water could be either acidic and basic with maximum values of 5 and 9 respectively. If ceramic filters based on Mouka clay have been already investigated on treating drinking water [3] there is no information about their behavior under acidic and basic conditions. The new challenge is therefore to state if under specific feed water condition ceramic filters will or not be able to leach metal pollutant in treated water. Nowadays, ceramic membranes can be obtained with high separation coefficient, but at the same time, this fact supposes low permeate fluxes. For each application, it is necessary to look for the adequate combination between selectivity and permeation. The choice of these parameters depends on the type of clay used, the type of porogen, and the ratio between clay and porogen. In this research, ceramic filters based on materials from the Logone Valley (clay and rice husk) were formulated. Study of the effect of fabrication method on parameters such as retention of suspended matter and chemical stability of the ceramic membranes was investigated.

2. Main Body

2.1. Sampling and Characterization of Raw Materials

Clay material was collected at a mining site located at Mouka (Far North region of Cameroon) altitude 323 m, latitude 10.34716°N and longitude 15.25248°E. Rice husks were collected at Yagoua (Far North region of Cameroon) market. After collection, the clay and rice husks were stored and dried at room tempera-

ture in the laboratory. The clay was ground using an artisanal (made of wood) mortar while the rice husk was ground using a grinding mill (farmer mill used to grind corn). The clay and rice husk were sieved to obtain two different diameters ($-50\ \mu\text{m}$ and $-100\ \mu\text{m}$) of powder. The clay was subjected to mineralogical analysis by FTIR. Chemical compositions of both clay and rice husks powders were analysed using a wavelength dispersive X-Ray fluorescence (Shimadzu XRF-1800).

2.2. Preparation and Characterization of Ceramic Membranes

The preparation of the porous support was achieved by mixing clay and rice husk powders with water, to produce a homogenous paste. The homogenous paste contained different clay: rice husk weight ratio of 0.6:0.4; 0.7:0.3 and 0.8:0.2. For each ratio, the mixture of clay: rice husk powders consist of sizes of $-100\text{:}-100\ \mu\text{m}$, $-100\text{:}-50\ \mu\text{m}$ and $-50\text{:}-50\ \mu\text{m}$ respectively. The volume of water necessary to get a moldable paste varied depending on the mixture. The paste was then moulded in an annular cylindrical (stainless steel) of 36 mm internal diameter, 59 mm height and a thickness of 5 mm. The obtained material was then dried first at room temperature for 24 hours, then at 50°C for 6 hours and 100°C for 42 hours in a drying oven (Crouzet, France) for complete removal of moisture. Afterwards the membranes were sintered in a muffle furnace (Nabertherm, Germany) at various temperatures (830°C , 950°C and 1000°C) for 8 hours with 2 heating rates; the first one was $5^\circ\text{C}/\text{min}$ until 500°C and then $10^\circ\text{C}/\text{min}$ until one of the 3-desired final sintering temperatures was reached. The final temperature was kept for 2 hours then the muffle furnace was cooled gradually to room temperature. Before and after sintering, the membrane dimensions were measured to evaluate the shrinkage (diameter, height). The open porosity was determined by the Archimedes method (using water as immersion fluid) [6].

The dissolution study of the ceramic membranes was performed by two experiments: study of the conductivity of water as a function of time under fixed pH and the lixiviation test.

In the first experiment, solutions were prepared at pH 5 and 9 (using hydrochloric acid and sodium hydroxide in distilled water). Distilled water was used in the case of pH 7. For each test, water was introduced into the filter and maintained constant by continuous addition. The conductivity of the filtrate was measured by sampling 20 ml of filtrate.

In the lixiviation test, the ceramic membrane was immersed in distilled water in a container at the ratio Liquid/Solid (L/S) = 10 [7] the container was then put in a shaker for 24 hours at 30 rpm. After this time the pH and conductivity of the water were measured using a multiparameter pH-conductivity instrument (Hach, HQ40d). The determination of Iron and Aluminium released in solution was done using a colorimetric method (Molecular absorption spectrometer) with a UV-Visible spectrophotometer (spectroquant Pharo 100 M). The determination of Calcium and Magnesium was done by titration with Ethylene Diamine Tetra Acetic Acid (EDTA).

2.3. Microfiltration Studies

2.3.1. Preparation of Kaolinite Suspension

Approximately 1.500 g of kaolinite sample was soaked in 3 L of distilled water for 24 hours. And let to rest for 8 hours to settle larger particles before decanting the liquid. The particle size distribution in the decanted suspension was obtained by mastersizer (Malvern hydro 2000SM). The analysis showed that 6.66% of the particles had sizes $\leq 0.93 \mu\text{m}$; 33.33% of the particles had sizes $\leq 1.088 \mu\text{m}$ and 60% of particles had sizes $\leq 1.16 \mu\text{m}$.

2.3.2. Filtration Test

Turbidity of 100 Nephelometric Turbidity Unit (NTU) was obtained by simple dilution of the later suspension with distilled water. The turbidity was determined by using a nephelometric turbidimeter (HACH RATIO 2100A). The filter was placed on a funnel on top of a collecting beaker and then filled with the suspension and maintained constantly full by continuously adding on. Filtration was carried out at atmospheric pressure. The filtrate was recovered as a function of time, turbidity and conductivity measurements were also performed. Blank tests were carried out with distilled water before using the membranes.

3. Results and Discussion

3.1. Characterization of Raw Materials

3.1.1. Chemical Characterization

Chemical properties using XRF were assessed. As shown in **Table 1**, the major chemical component of Mouka clay is SiO_2 , with 47.47 wt% for particles of average size $< 100 \mu\text{m}$. The percentage of Al_2O_3 corresponds to non-refractory clay since it does not exceed 45% [3]. There is also a small quantity of alkali with values between 0.22% and 0.76% as Na_2O and 0.99 wt% and 3.1 wt% as K_2O showing that this clay sample matures at relatively high temperatures [8]. **Table 1** also shows that the percentage of CaO is 7.51 wt% which is higher than 6 wt%, characteristic of calcareous clay [9]. The percentage of iron is 6.9 wt% which is under the limit allowable values (8 wt%) for using in the ceramic industry [10]. The percentage of other components is lower than 1%. The Loss of ignition (Loi) of clay is in the range of 8% - 18% allowable for ceramics [11]. For rice husk, the high percentage of Loi showed that this can create pores.

3.1.2. Phase Identification

The FTIR spectrum (**Figure 1**) showed absorption bands at 3693.67 cm^{-1} , 3620.63 cm^{-1} , 910.03 cm^{-1} , 791.82 cm^{-1} and 524.21 cm^{-1} indicating the presence of kaolinite [12]. The presence of illite is confirmed by absorption bands at

Table 1. Chemical composition (wt%) of the Mouka clay and rice husk.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	Mn_2O_3	P_2O_5	Loi ¹
Mouka	47.47	15.60	6.95	7.51	0.66	0.67	2.43	0.11	1.31	0.04	0.08	16.22
Rice husk	17.35	0.67	0.48	5.62	0.15	0.03	0.51	0.02	0.07	0.03	0.23	69.47

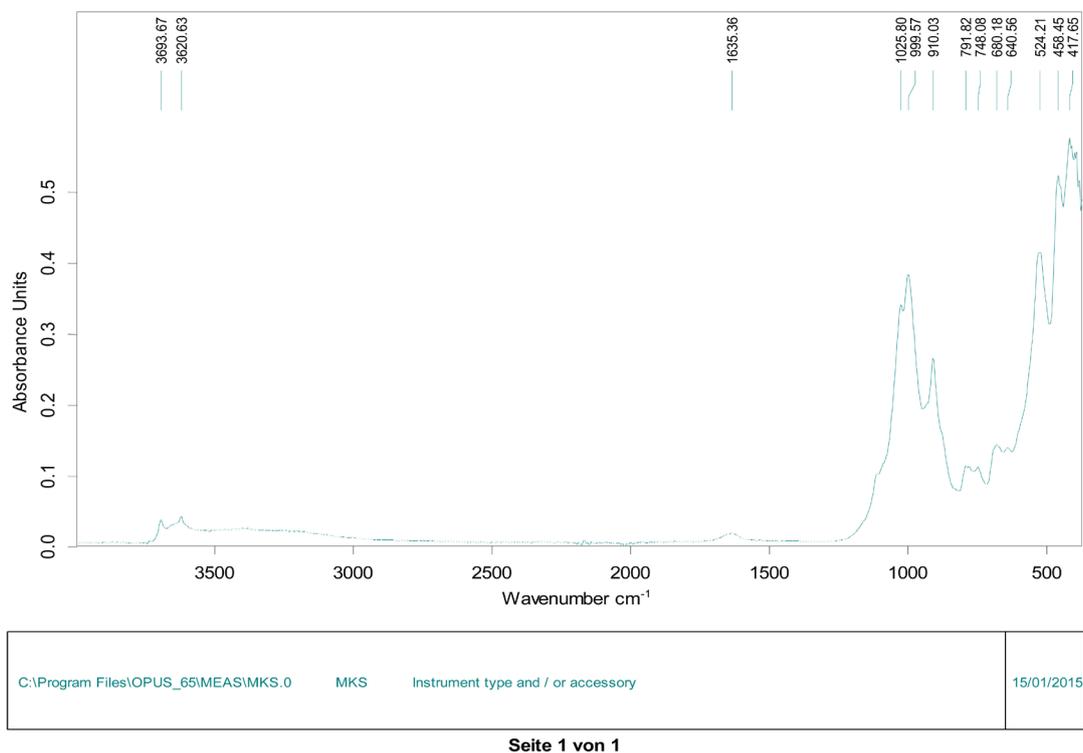


Figure 1. FTIR spectrum of Mouka clay.

1635.36 cm^{-1} , 1025.80 cm^{-1} , 910.03 cm^{-1} and 791.82 cm^{-1} . Also, hematite is present due to the absorption bands at 24.21 cm^{-1} and 458.45 cm^{-1} [13].

3.2. Structural Characterization of Sintered Membranes

The produced membranes were characterized using XRD and the results are shown in **Figure 2**; the major crystalline phases identified were mullite ($2\text{Al}_2\text{O}_3\text{SiO}_2$), anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) and quartz (SiO_2). The presence of mullite is due to the presence of illite in the clay [5]. The presence of anorthite just confirms that the clay is a calcareous one (transformation of illite to anorthite instead of spinel) [14]. The high intensities of the quartz peaks are related to the degree of crystallinity of these ceramics when compared to the clay minerals [15]. The Archimedes principle method showed that the porosity was about 30%, which is a value high enough to get efficient membranes for filtration. This porosity was created by incorporating the rice husk material in the clay before heat treatment [2] [16].

3.3. Influence of Mixture Proportions and Size of Clay and Rice Husk on Withdrawal Thickness

Table 2 shows the influence of mixture proportions and size of clay and rice husk on shrinkage (percentage of thickness diminution after firing), we observed that when the clay granulometry is $\leq 50\text{ }\mu\text{m}$ and rice husk granulometry is $\leq 50\text{ }\mu\text{m}$, the shrinkage was 46%. In addition, when the clay granulometry is $\leq 100\text{ }\mu\text{m}$ and rice husk granulometry is $\leq 50\text{ }\mu\text{m}$, the shrinkage was 29%.

When we used the mixture proportion of 70% of clay ($\text{Ø} \leq 50\text{ }\mu\text{m}$) and 30% of

rice husk of ($\varnothing \leq 50 \mu\text{m}$), the shrinkage was 46%. When the proportion of the mixture is 80% clay ($\varnothing \leq 50 \mu\text{m}$) and 20% of rice husk ($\varnothing \leq 50 \mu\text{m}$), the shrinkage was 21%. The sharp contraction observed after sintering is due to the phenomenon of expansion and densification taking place during sintering. It appears that less shrinkage occurred when the clay proportion is higher as compared to that of rice husk. The mixture containing 80% of clay ($\varnothing \leq 100 \mu\text{m}$) and 20% rice husk permitted the researcher to obtain the lowest shrinkage.

3.4. Influence of Sintering Temperature on Water of Absorption Capacity of Ceramic Membrane

The water absorption capacity in a material can be related to its porosity. The porosity is obtained based on the Archimedes method whereby the difference between the mass of the filter before and after immersion in water expressed in percentage gives the porosity. Moreover, the amount of water absorption increases with the amount of porogen added [17]. Hence, pore volume and pore structure are significant parameters that influence the leaching behavior of materials. Table 3 shows the influence of sintering temperature on water absorption

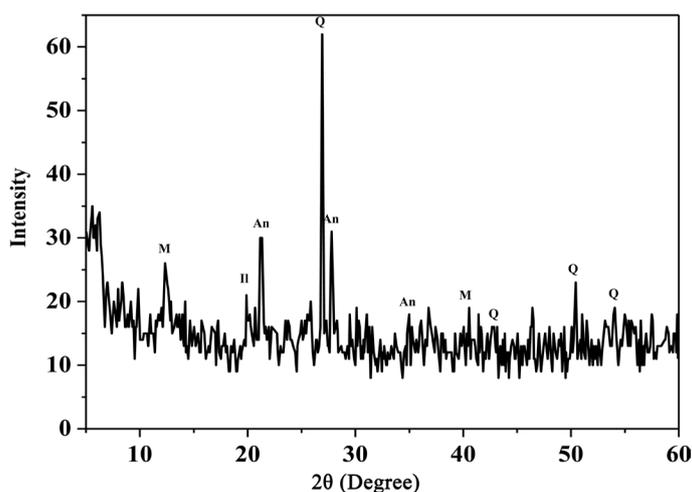


Figure 2. XRD pattern of sintered filters (950°C) (Q = quartz, M = mullite, An = anorthite, Il = Illite).

Table 2. Influence of mixture proportions and size of clay and rice husk on shrinkage.

	Granulometry of clay and rice husk Clay:rice husk		Proportion of clay and rice husk Clay:rice husk	
	50:50 μm	100:50 μm	70%:30%	80%:20%
Shrinkage	46.00%	29.00%	46.00%	21.00%

Table 3. Influence of sintering temperature on total porosity and pore volume of filters.

Temperature	830°C	950°C	1000°C
Percentage of void	30%	22%	21%
Pore volume	(17.99 \pm 0.44) cm^3	(11.30 \pm 0.50) cm^3	(8.97 \pm 0.50) cm^3

and pore volume. It appeared that the water absorption decreased when sintering temperature increased. The porosity decreased from 30% to 21% when the sintering temperature increased from 830°C to 1000°C. Also, it can be observed that the porosity decreases sharply from 830°C to 950°C but slightly from 950°C to 1000°C. The reduction of water absorption capacity is due to the densification of the membrane. In fact, the particles tend to agglomerate with each other with increasing temperature leading to a more consolidated ceramic body. These results are similar to those of [4].

3.5. Filtration Tests

The experimental results obtained from the filtration of water with initial turbidity of 100 NTU under a fixed temperature and pressure are presented in **Figure 3**. The variation of the flow rate as a function of time is presented in **Figure 3(a)**. We noted that time has a significant effect on filtration rate. Flow rate dropped sharply to 100×10^{-3} L/h during the first 50 min of filtration. The flow rate reduced more slowly between 50 min and 150 min and more slowly again beyond this time. The drop of the flow rate value, observed after 150 min, can be attributed to clogging by suspended particles in the feed water having approximately the same size as the mean pore diameter [18]. The reducing rate corresponds with the observed formation of a coating layer on the surface of the filter. The deposited layer which increased with time reduced the filtration rate. This behavior corresponds to the classical dependence of flux with the time of micro-filtration. The decreased permeate flux with the time is due to the formation of a particulate cake [3].

The change of turbidity as a function of the final filtration time is presented in **Figure 3(b)**. Turbidity is the cloudiness or haziness of a fluid caused by individual particles (suspended solids) which are generally invisible to the naked eye. The measurement of turbidity is a key test of water quality. It was observed that the turbidity of water dropped sharply four times less than 25 mins and stabilized

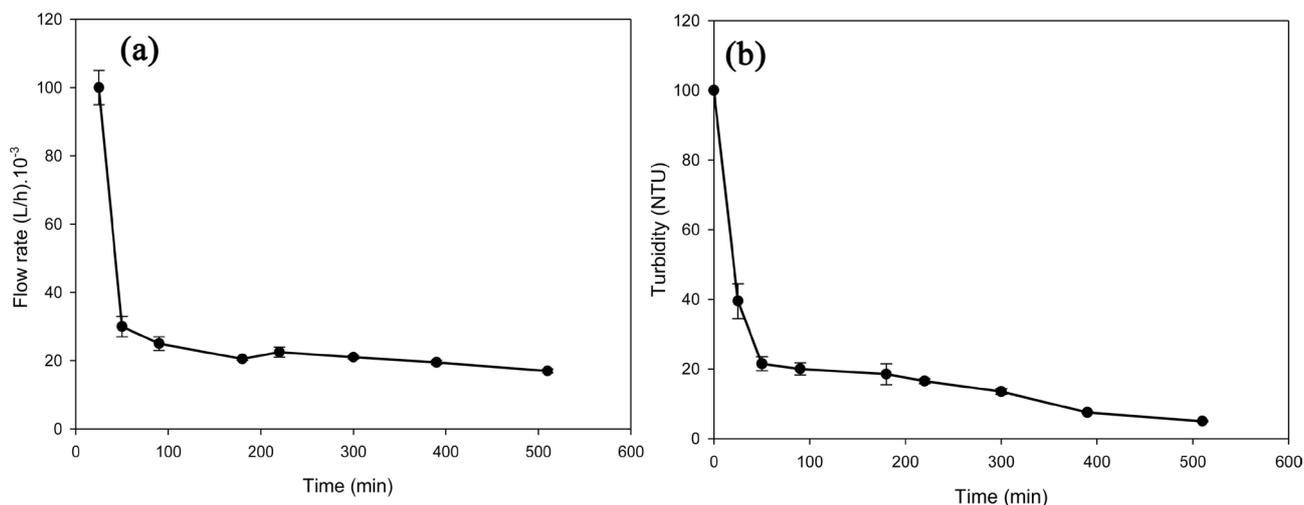


Figure 3. Reduction of flow rate (a) and residual turbidity (b) as a function of time during filtration.

beyond. The final reduction of turbidity was 95%. The decrease of turbidity during the time of filtration is due to the retention of particle in water. This retention increases with the formation of cake.

3.6. Physical Characterization: Chemical Stability

Chemical stability analysis of ceramic filters was done either in acidic, neutral (distilled water) or basic media and the results are shown in **Figure 4**. In the acidic medium, temperature of sintering and time had a great influence on the final meso-structure of the ceramic filter. The change in conductivity values over time was observed in a solution of pH 5. For a sintering temperature of 830°C, the conductivity remained constant with time. On the contrary, sintering temperatures of 950°C and 1000°C, showed an increase in conductivity values, which reached a maximum after 100 mins (213 $\mu\text{S}/\text{cm}$) and 300 min. (216.1 $\mu\text{S}/\text{cm}$) respectively. The conductivity then decreased to the initial conductivity. The increase in conductivity is due to an attack of the material in acidic media. In fact, closed pores are opened which therefore release ions into the solution due to the presence of OH groups. The decrease in conductivity observed after 300 mins is explained by OH groups in the ceramic forming hydrated components with H^+ of the acid. This renders some components of material matrix unstable in acidic media. These results show that chemical stability is higher for filters sintered at 830°C but the one for 950°C was lower than 1000°C. This is because ceramization starts from 850°C, which leads to an amorphous state that favors chemical stability.

In distilled water (pH 6.8) conductivity also remained constant with time of filtration for a sintering temperature of 830°C. For a sintering temperature of 950°C, the conductivity increased to a maximum of 76 $\mu\text{S}/\text{cm}$ (after 90 mins) whereas for a sintering temperature of 1000°C the maximum conductivity is 30 $\mu\text{S}/\text{cm}$ (after 180 mins). After those values, the conductivity slightly decreased with time. This is related to the end of the salting process due to the presence of non-crystallized elements in the structure of the ceramic material. These results can be explained by the fact that when the sintering temperature is increased the porosity decreases, and the material has fewer OH groups on the surface thus exhibiting higher resistance to degradation in water [19]. In fact, these results show that for a sintering temperature of 830°C there are low amount of OH groups, whereas a sintering temperature of 950°C produces more OH groups than a 1000°C sintering temperature.

In basic media (pH 9) the initial conductivity dropped at the beginning of filtration whatever the temperature. This decrease is because OH^- ions responsible for the basicity, are absorbed in the structure of the ceramic filter at the beginning of filtration and remain constant over time. It also appeared that the conductivity reduced until after 60 mins when using a filter with sintering temperature of 830°C. Above this time, the conductivity did not change with an increase of the filtration time. The drop-in conductivity is due to the adsorption of OH^- ions at the surface of the material.

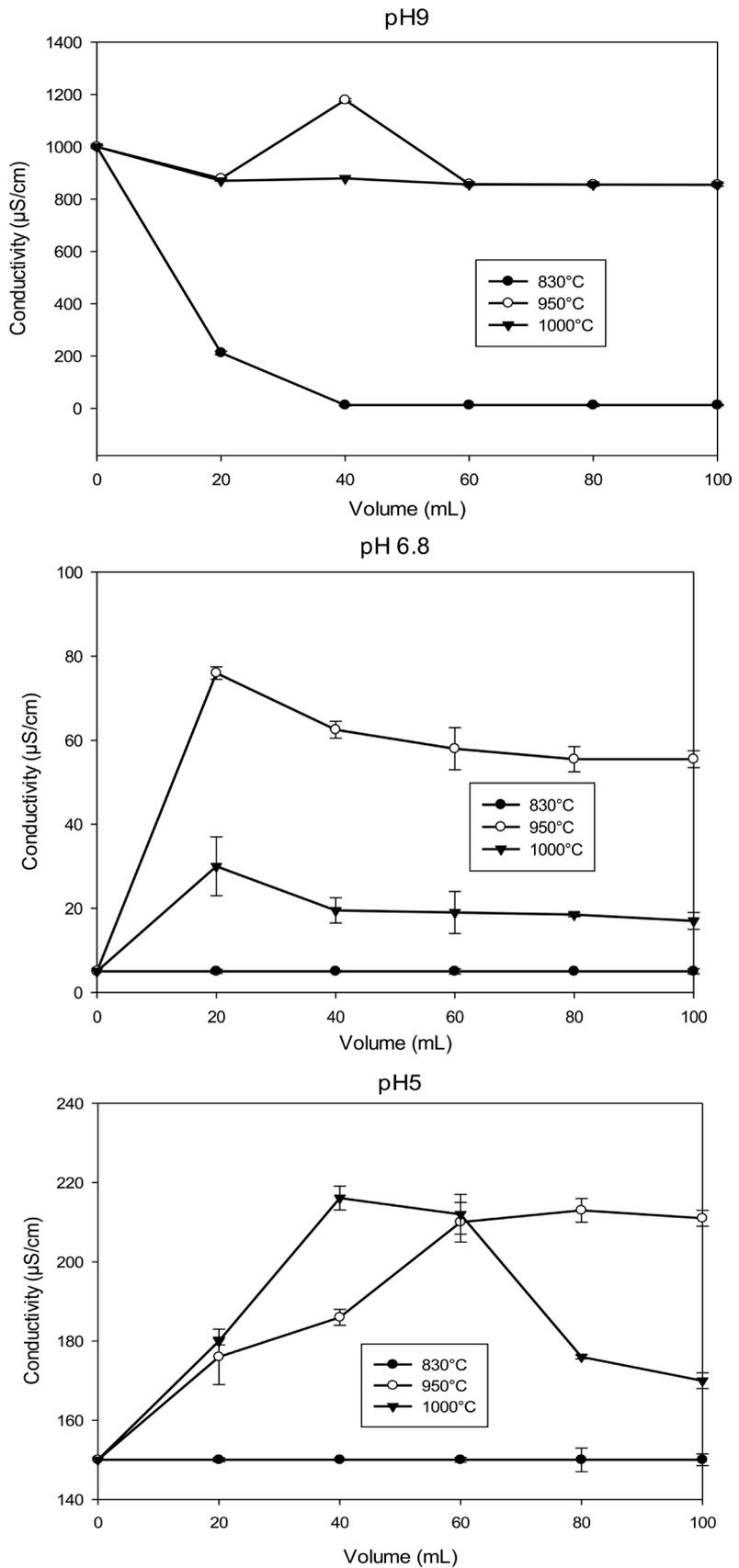


Figure 4. Filtrate's conductivity with pH.

3.7. Lixiviation Test

Figure 5 shows the pH value (**Figure 5(a)**) and the metals released (**Figure 5(b)**) in the lixiviate as a function of the sintering temperature 830 °C, 950 °C and 1000 °C. With initial pH 6.8 (distilled water), we observed an increase in pH with increasing sintering temperature; pH increases from 6.4 (a sharp drop as compared to the initial) at 830 °C to 7.18 at 1000 °C. This observation is due to the higher metal concentration in the solution, the higher H⁺ ions concentration that results from the hydrolysis of metal cations in the water, which triggers a reduction of pH equilibrium. Therefore, the sintering process reduces the leaching of the metals due to the reactions produced during sintering.

Figure 5(b) shows the metal dissolution as a function of sintering temperature. Iron, Calcium and Aluminium are the major metals released in solution. The amount of Calcium decreased when temperature increased; this is because up to 850 °C the crystallized phase of Calcium (Calcite) appeared, so the amount of Calcium in the lixiviate is less. The higher amount of Iron and Aluminium released was observed at 950 °C. This is because around 950 °C FeO is transformed into Fe₂O₃ (Hematite). At temperatures higher than 950 °C, the amount of Iron reduced gradually and for Aluminium, the formation of silicon-spinel appeared. The latter is more crystallized so the leaching behavior is reduced. In fact, as the sintering temperature increased, the material became more crystalline and therefore less subject to degradation in water. High conductivity values obtained in the case where the sintering temperature was 830 °C shows the high instability of various oxides in the material such as lime (CaO), monocalcium silicate (CaOSiO₂), iron oxide (Fe₂O₃) metakaolin (2SiO₂, Al₂O₃) and mullite (2SiO₂, 3Al₂O₃) that were released in solution meaning that at this sintering temperature crystallization phase is limited [20].

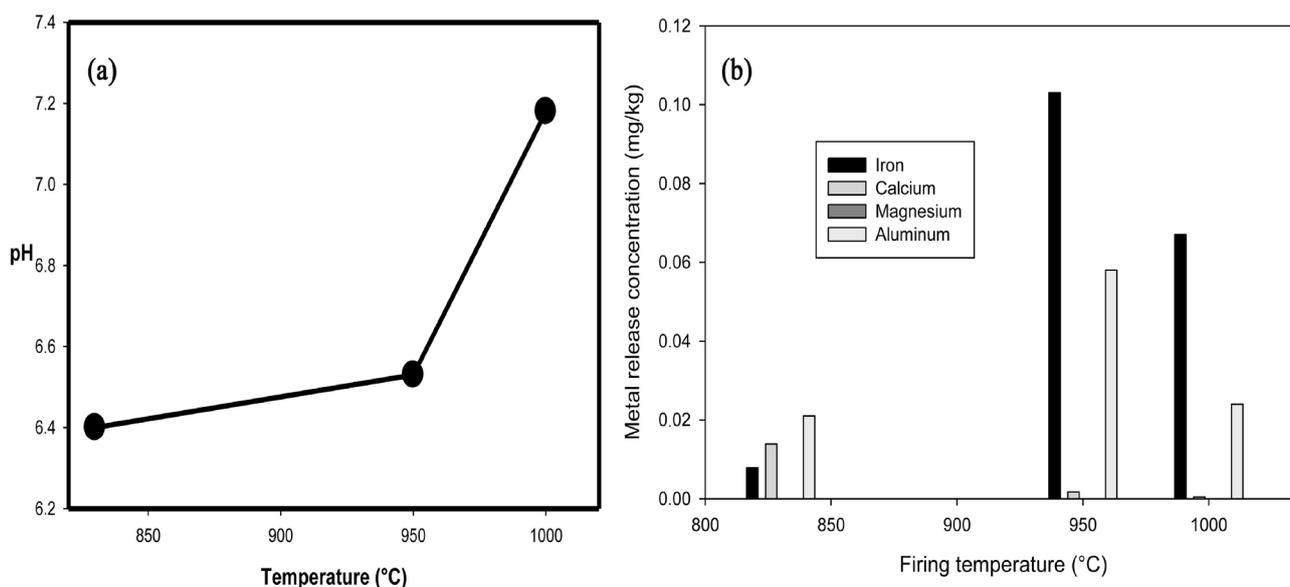


Figure 5. Evolution of pH (a) and metal release in solution (b) of filtrate as a function of firing temperature (830 °C, 950 °C and 1000 °C).

4. Conclusion

This work shows the application of ceramic membrane filters obtained with local raw materials from the Logone Valley in water treatment. The produced materials can improve the quality of water in terms of turbidity. The proportion of 80% of clay (milled at diameter < 100 μm) and 20% of rice husk (milled at diameter < 50 μm) reduced filter shrinkage after sintering. Different measures of conductivity and pH of the water and leachate showed that the conductivity of the leachate decreases when the sintering temperature increases reflecting an increase in crystallinity of the material when sintering temperature increased. The filtration tests of turbid water exhibited a reduction of turbidity of 95%. These experimental results show that ceramic filter made with Mouka clay and rice husk is an appropriate material for the development of microfiltration membranes which could be applied to drinking water treatment.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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