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Identification of an Appropriate Formulation for Domestic Water Ceramic Filters from Soukamna Clay (Cameroon)

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ARTICLE INFO	ABSTRACT
	This paper deals with the formulation of ceramic filters having the porosity adapted to domestic potable water treatment. The filters were made from clays and rice husk obtained from the Far North region of Cameroon (Logone Valley). Nine formulations were investigated to choose those that might have the porosity standing between 35 and 50% (the ideal porosity adapted for water treatment). The nine formulations investigated were as follows: clay:rice husk mixture weight ratio 0.7:0.3; 0.8:0.2 and 0.9:0.1 with the particle size of 100:100 microns. The sintering temperatures of 900 °C, 950 °C and 1000 °C were applied for each of the mixtures. The results showed that only filters with weight ratio 0.7:0.3 sintered at 900 °C, 950 °C and 1000 °C had porosity between 35 and 50% with values of 39.41±0.96; 40.15±1.59; 40.14±1.31 respectively. Mechanical strength, permeability and iron leaching behavior were investigated for these three formulations. The formulation 0.7:0.3 with sintering temperature of 1000 °C had the higher permeability and was the more stable for iron leaching so it is the more adapted for water treatment in terms of flow rate and iron leaching behavior, pore size distribution showed that these filters were macroporous and designed for microfiltration with average pore diameter of 0.46 μm.

1. Introduction

Ceramic filters for water filtration is one of the alternative techniques for treatment of drinking water [2]. They are made of clay and porogen. 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 [3]. It has been shown that ceramic filters can reduce turbidity [1] and microorganism from water [3]. However, any ceramic filter intended to treat domestic water must have a minimal flow rate of 1 L/h [4]. To get this flow rate the porosity of the filters should be between 35 and 50% [1], this porosity will ensure a high flow rate and an efficiency against microbial pollutant [3]. Previous works have shown that filters based on clays of the Far North region of Cameroon can leach iron on the filtrate. Many parameters could influence the porosity and the leaching behavior of a ceramic filter such as the sintering temperature and the percentage of porogen. Determine the best formulation that ensures the ideal porosity and the lower leaching behavior is therefore a crucial task for a clay-rice husk mixture.

In this research, ceramic filters based on materials from Far North region of Cameroon (clay and rice husk) were formulated. Study of the effect of weight ratio (clay and rice husks) and sintering temperature on porosity of the

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filters was done. This was done to choose the formulation (weight ratio and sintering temperature) that gives the porosity between 35 and 50%. Then the study of the effect of temperature on mechanical strength, permeability and iron leaching behavior of the having porosity between 35 and 50% was done. Finally, pore size distribution was investigated on the formulation with the higher permeability and the lower iron leaching behavior.

2. Experimental

2.1 Sampling of Raw Materials

Clay material was collected at a mining site located at Soukamna (Far North region of Cameroon) altitude 321 m, latitude 10.34716°N and longitude 15.26525°E. Rice husks were collected at Yagoua (Far North region of Cameroon) market. After collected, the clay and rice husks were stored and dried at room temperature in the laboratory. The clay was grind using an artisanal (made of wood) mortar while the rice husk was grind using a grinding mill (farmer mill used to grind corn). The clay and rice husk were sieved through a 100 μm sieve.

2.2 Ceramic Body Preparation

Sieved clay and rice husk were then mixed at different ratios 0.7:0.3, 0.8:0.2 and 0.9:0.1 (by weight) followed by making dough by adding water. This leads to what is called "ceramic paste". We evaluated the influence of rice husks on the viscosity of Soukamna clay and the influence of time on ceramic paste viscosity.

The ceramic paste was then cast in a cylindrical-shaped mold to make the filter. The resulting ceramic filters were cylindrical in shape, hollow with one side open, and had a height of 5,9 cm and a thickness of 5 mm. The samples were then air dried in the laboratory (temperature of 25 °C) for 2 days after which they were further dried at 105 °C in an electric oven (Crouzet, France) for 24h (to make the samples safer and to remove excess moisture). Afterwards the samples were sintered in a muffle furnace (Nabertherm, Germany) at 3 different temperatures (900, 950 and 1000°C) for 8 hours in 2 steps; firstly, sintering temperature was set up at 500 °C at a rate of 5 °C/min and held for 2 h so that all the pore former would be burned off. Secondly, the sintering temperature was increased up to one of the 3-desired final sintering temperatures (900, 950 and 1000 °C) at a rate of 10 °C/min and held for 2 h. Then the muffle furnace was cooled down gradually to temperature below 100 °C before removing the ceramic filters.

2.3 Ceramic Filters Characterization

Lost in weight of ceramic filters was evaluated by weighting the ceramic body before and after sintering in order to evaluate the influence of ratio and sintering temperature on ceramic filters lost in weight. The ceramic body (before sintering) was weighted and the weight (M1) was recorded. After sintering the ceramic filters obtained were weighted and the weight (M2) was recorded. Then lost in weight (in percentage) was calculated using the following formula:

$$L = \frac{M1 - M2}{M1} \times 100 \qquad (1)$$

The apparent porosity (amount of voids or pores within a volume of porous material) was measured according to the Archimedes method (using water as immersion fluid) [5] as follow: the ceramic filters were weighted and the weight (W1) was recorded. They were then immersed in a beaker of water. Bubbles were observed as the pores in the filters were filled with water. Their soaked weights were measured and recorded. The filters were lightly wiped with a moistened smooth cotton cloth to remove all excess water from the surface, and the saturated weight (W2) was recorded. The porosity was then calculated using the following formula:

$$p = \frac{W2 - W1}{W1} \times 100 \qquad (2)$$

Mechanical strength of the filters was measured using the compression method [6]. The filters were clamped with 2 pieces of tests grippers and a stretching force was applied until the filters rupture, the maximum load and elastic modulus were recorded by a dynamometer. The mechanical strength was then obtained by the following formula:

$$MS = \frac{F}{S} \qquad (3)$$

Where MS is the mechanical strength (MPa), F is the minimal force that cause the filter rupture and S is the surface area of the filter.

The permeability and leaching tests were done using distilled water as water to filtrate. Each ceramic water filter was filled with distilled water and allowed to filter until they are emptied; this was done for a consecutive 10 h (during 10 days). Each time the filters are emptied, the time required and the volume of filtrate were recorded. The flow rate was then obtained by dividing the volume obtained by the time necessary to empty the filter. The flow rate of a day was the mean of each run for this day. The determination of Iron released in solution was done using a colorimetric method (Molecular absorption spectrometer) with a UV-Visible spectrophotometer (spectroquant Pharo 100 M). The Iron content of the filtrate for a day was the mean of each run for this day. The pore size distribution was determined by mercury intrusion porosimetry. The analysis was performed on a

porosimeter AutoPore IV 9500 V1.

3. Results and Discussion

3.1 Influence of Rice Husks and Time on Ceramic Paste Viscosity

As shown in Figure 1, it can be seen that clay alone (100% clay content in ceramic paste) has the highest viscosity (regardless of time) compared to ceramic paste that contains the rice husks. It is observed a decrease of 58%, 48%, 83%; 60%, 45%, 86% and 67%, 52%, 82% respectively for a clay content of 90%, 80% and 70% and for the times 60s, 120s and 180s. This observation suggests that addition of rice husks has the effect of destroying the three-dimensional structure of clay [7] and increasing the interaction forces (Van Der Waals forces in particular) between particles which promote the formation of flocs and aggregates and therefore the decrease of viscosity [8], indeed, it is the three-dimensional structure of clay that is responsible for the viscosity of a clay. However, it is noticed that viscosity for the ratio 80:20 is higher than that of the ratio 90:10; this suggests that when the amount of clay is 80 the amount of silica supplied is such that the water absorption capacity decreases. The ratio 70:30 has the advantage of having a viscosity which does not vary with time and whose value (4 PaS) is closest to those accepted in the ceramic industry (0.2-0.4 PaS) [8].
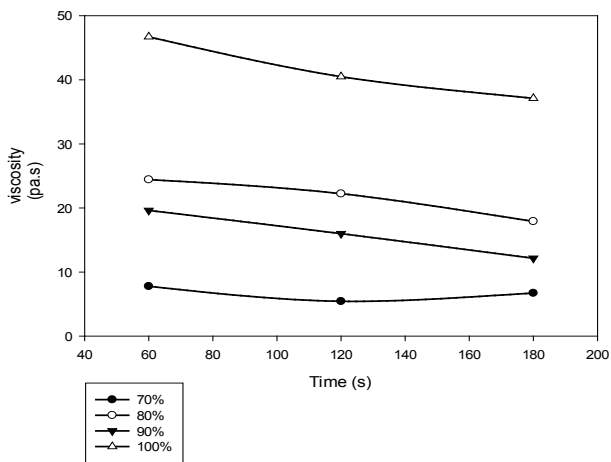

Figure 1. Ceramic paste viscosity

3.2 Ceramic Filters Characteristics

Lost in weight

As shown in Table 1, the temperature did not influence lost in weight of the ceramic filters made with ratio 70:30. This suggests that it is from 30% to above of rice husks content in the paste that rice husks stop to have an effect on clay behavior with sintering temperature. Meanwhile,

with other ratios, it can be seen that lost in weight percentage of ceramic filters raises with temperature. This is due to the development of vitreous phase in these cases [9]. This phenomenon is explained by the fact that raising temperature leads to the move of pores from center towards the surface [10]. Table 1 also shows that lost in weight drops when clay content in the paste raises. This is in accordance with the results of [11] and confirm that rice husks in the paste lead to a compaction of the ceramics.

Table 1. Lost in weight percentage of ceramic filters

% argile	900 °C	950 °C	1000 °C
70	46.09 ± 0.85	45.80 ± 0.67	47.97 ± 0.30
80	36.49 ± 0.08	39.99 ± 0.02	42.44 ± 0.28
90	30.21 ± 1.55	33.16 ± 0.71	35.31 ± 0.12

Apparent porosity

As shown in Table 1, the temperature did not influence the porosity of the filters. However, the percentage of rice husks did. The fact that sintering temperature did not affect the porosity values could be because all the three temperatures studied are on the solid phase of the heat treatment since only liquid phase is the one that is responsible of porosity reduction [11,12]. Meanwhile porosity increases with porogen. This table also showed that only filters of 70:30 at the three sintering temperatures had the target porosity. So, this weight ratio was used for the remaining work.

Table 2. Apparent porosity values of filters in function of sintering temperature and clay content

Clay (%)	900°C	950°C	1000°C
70	39.41 ± 0.96	40.15 ± 1.59	40.14 ± 1.31
80	30.15 ± 0.01	29.34 ± 0.02	28.54 ± 0.34
90	18.65 ± 0.19	21.32 ± 0.08	20.89 ± 1.74

Mechanical strength

Table 2 shows the influence of sintering temperature on the mechanical strength of the filters. It was observed that the mechanical strength remained constant with the increasing of sintering temperature. This result is in harmony with the results of porosity that showed that the porosity of these three formulations was almost the same. In fact, mechanical strength of a ceramic drops when porosity increases [13,14].

Table 3. Mechanical strength of the filters

Clay (%)	900°C (MPa)	950°C (MPa)	1000°C (MPa)
70	0.21 ± 0.03	0.22 ± 0.01	0.23 ± 0.02

Permeability

Figure 2 shows the permeate flux of the ceramic filters.

The result showed that the filters sintered at 1000 °C had the higher permeate flux. This could be because pores diameter and pores density increase with temperature [15]. So in this case filters sintered at 1000 °C, filters should have higher diameter pores size and the higher permeability.

Iron leaching behavior

Figure 3 and 4 show the evolution of iron content (mg/L) per day and the evolution of cumulative iron content (mg/L) for the filtrate for 10 days trial respectively. Figure 3 shows that the iron content of the filtrate did not exceed 0.3 mg/L which is the maximum tolerable for potable water. Iron leaching is due to the hydroxylation of iron oxides present in the filter matrix when this latter is in aqueous solution [16]. It was observed that for the first day the higher value (0.133 ± 0.004 mg/L) of Iron content was filtrate of filters sintered at 1000 °C; this is because clay used in this work is non-calcareous clay. In this type of clay amount of iron oxides increases with temperature starting from 700 °C to 1080 °C. Meanwhile

it was observed that iron content in the filtrate from filters sintered at 900°C was higher (0.111 ± 0.025mg/L) than the one from filters sintered at 950 °C (0.088 ± 0.012 mg/L). This could be because at 950 °C the Iron is in form of Hematite (Fe_2O_3) that is more stable than magnetite (Fe_3O_4) present at 900 °C [16]. From Figure 4 it was observed that filters sintered at 900 °C were the less stable with the higher cumulative iron content. The cumulative data for sintering temperature of 1000°C and 950°C are around the same. This meaning that the level of iron crystallization is almost the same for these two temperatures [17].

Due to the higher permeability lowest cumulative iron content on the filtrate, the sintering temperature of 1000 °C was definitely chosen like the adequate temperature for producing ceramic filters. Pore diameter distribution was then investigated for filters sintered at 1000 °C.

Pore diameter distribution

Figure 5 shows the pore size distribution for the ceramic filters. This shows the plot of the derivative of the cumulative curves, dV/d, versus the pore diameter of
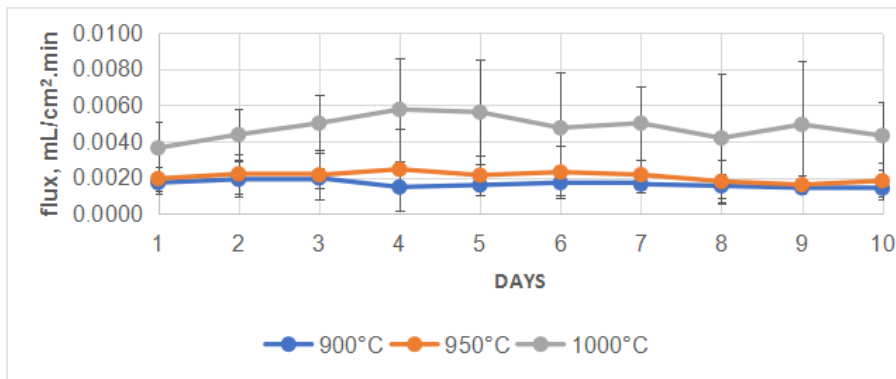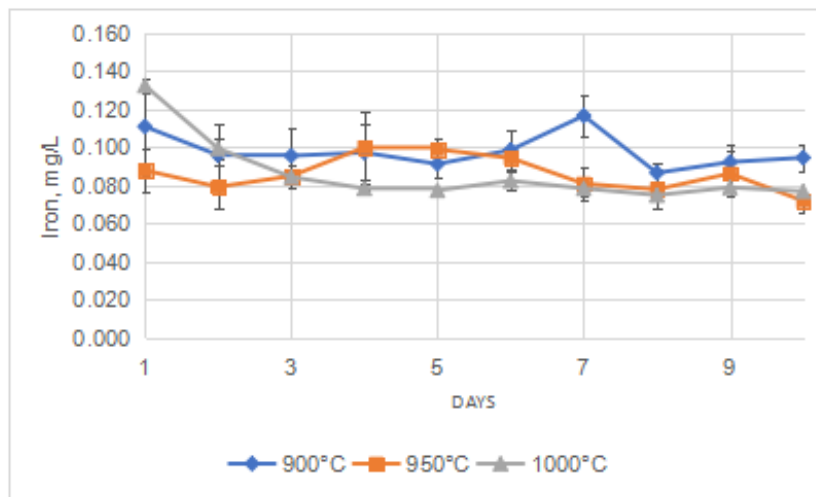

Figure 2. Permeate flux of the ceramics filters

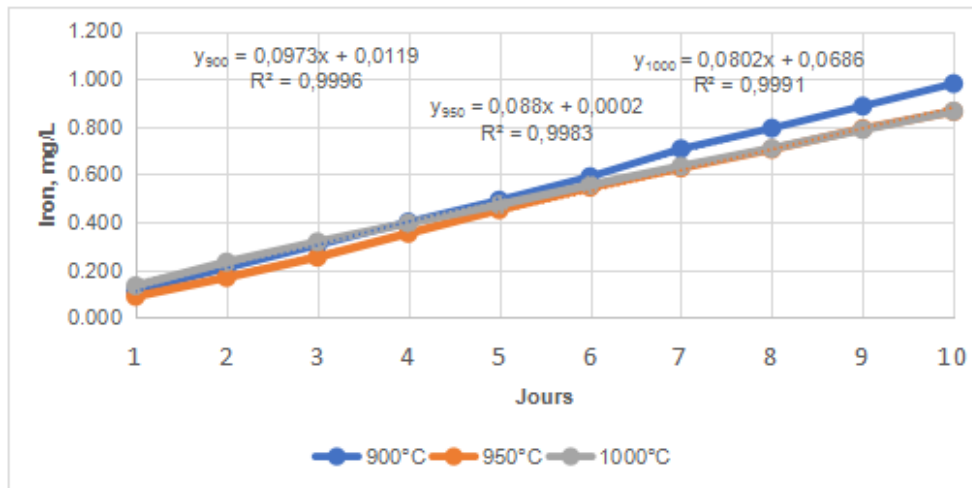**Figure 3.** Iron content of the filtrate per day

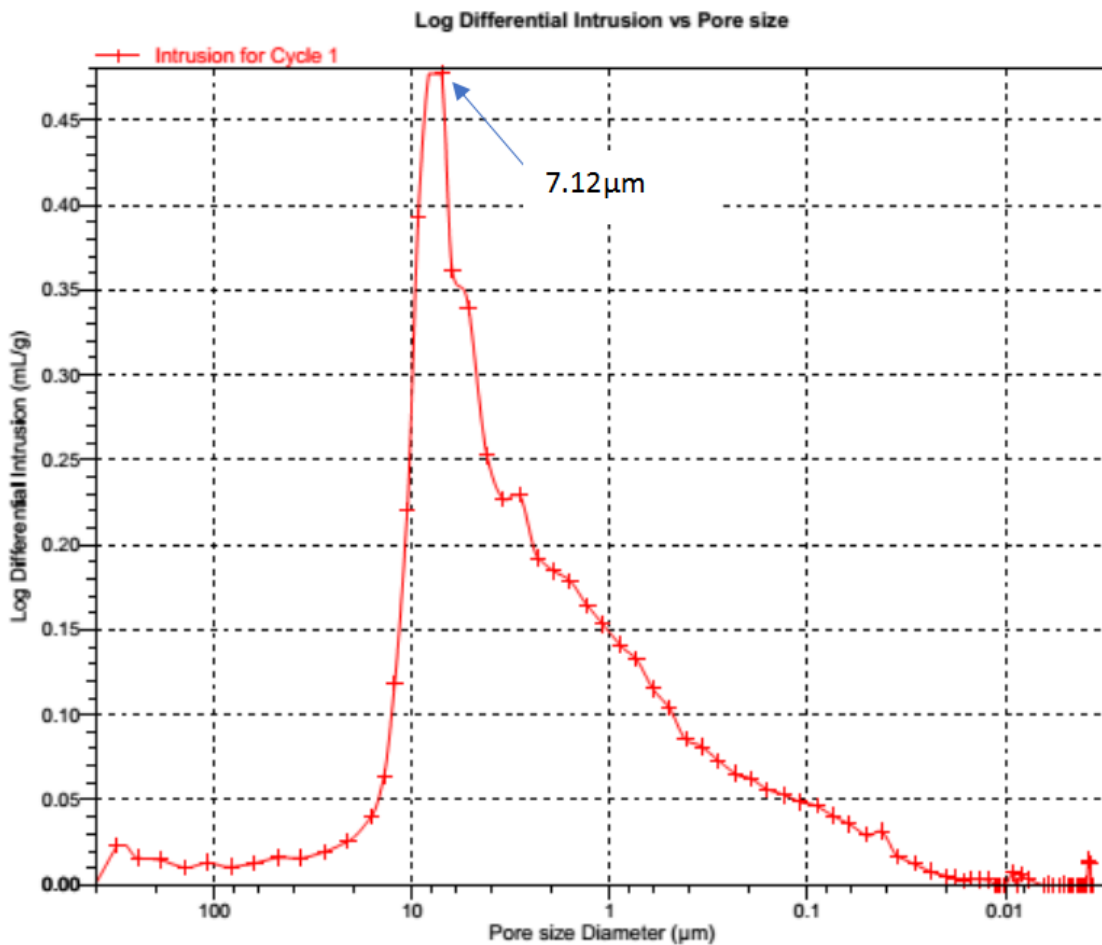**Figure 4.** Cumulative Iron content of the filtrate per day

Figure 5. Pore diameter distribution of filters

the studied ceramic filters. The dV/d function is widely used for determination of diameter, which occurs in the penetration of the maximal mercury [18]. This figure shows that there is a predominant presence of pores with diameters of 0.02 to 21.2 μm, illustrated by the appearance of a broad peak in this pore diameter range. The summit of this peak (7.12 μm) suggests that pores with this diameter occupy the maximum volume of the filter's matrix. Since average pore diameter is 0.46 μm these filters are macroporous [19] and designed for

microfiltration.

Table 4. Intrusion data summary

Total Intrusion Volume	0.4168 mL/g
Median Pore Diameter (Volume)	3.8253 μm
Median Pore Diameter (Area)	0.0592 μm
Average Pore Diameter (4V/A)	0.4592 μm
Bulk Density at 0.53 psia	1.3384 g/mL
Apparent (skeletal) Density	3.0270 g/mL
Stem Volume Used	66 %
Porosity	55.7838 %

4. Conclusions

The characterization of ceramic filters showed that to obtain apparent porosity between 35 and 50% the adequate mixture clay:porogen is 70:30 in weight. The results also showed that temperature does not influence porosity because the three temperatures studied are all in the solid phase of heating treatment. Temperature of 1000 °C led to the higher permeability due to the increasing of pore diameter and to the lower iron leaching due to crystallisation of iron with temperature. Pore size distribution on these filters showed that majority of pores has diameter between 0.02 and 21.2 μm and are designed for microfiltration. So that they are able to retain pollutant such as suspended matter that causes turbidity of water. Anyway further research should be done to evaluate the performance of these filters to reduce turbidity.

Acknowledgements

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