

# Water Treatment Using Porous Ceramics Based on Recycled Diatomite and Kaolin

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## Abstract

The treatment of waters using filtering process through sintered porous ceramics based on diatomite and kaolin is reported. The water samples were prepared in low = 50%, medium = 100% and high = 150% concentrations, taking as reference point the maximum value allowed according to the resolution 2115 - 2007. The treated parameters were alkalinity; hardness to calcium, magnesium and total; chlorides; sulfates and total iron. The porous ceramics were formed by slip casting in plaster mold, from pastes with weight percentage of 50% recycled diatomite from the beer industry, 40% kaolin and 10% calcium carbonate. The particle sizes of the ceramic powders were the passed through ASTM 200 sieve. The drying of the ceramics was carried out at room temperature for 24 h, then by forced circulation oven Memmert UF-110 at 100 ° C for an equal time. The sintering of the ceramics was carried out in Vulcan D-130 electric muffle, at a maximum temperature of 1000 ° C for two hours, with a heating ramp of 10 ° C/min. The morphology of the ceramics was studied using scanning electron microscopy (SEM), while the physic ceramic analyses was evaluated according to ISO 10545-3 standard. Our results, reported what porous ceramics efficiency for the removal of chlorides up to 50%, while elimination of sulfates was approximately 43%, on the other hand, the total iron was removed up to 95%, likewise,

it was evidenced that ceramics are not efficient in the treatment of hardness and alkalinity of waters with high concentrations.

**Keywords:** Water treatment, Porous ceramics, Diatomite, Kaolin

## **1. Introduction**

The monitoring of water quality is a growing challenge for the community, due to the pollution of rivers, lakes and groundwater, as a result of the increase in population, urbanization, industrial growth, agricultural and mining activities, negatively impacting water resources [1-3].

Currently exist well-established processes for water treatment, ranging from collection, coagulation, flocculation, sedimentation, filtration and disinfection [4]. In each of these stages, the use of chemical substances and new materials, where recycling materials have been used for their synthesis, have evolved such processes favoring the cost-benefit ratio [5-6].

Nowadays, the brewing industry uses large amounts of diatomite, during the filtration processes, which, once used, turn into industrial waste contributing negatively to the environment [7-8]. The diatomaceous earths (diatomite), is formed of skeletal remains of monocellular organisms called diatoms, which have properties such as high porosity and permeability, chemical resistance, high specific surface, high adsorption capacity [8-9] among others, converting them into materials potentials in the filtration processes of beers, wines, juices, oils and pharmaceuticals [7].

Ceramic membranes offer better properties compared to organic ones in terms of thermal, chemical and mechanical stability, as well as resistance to microbial degradation [10]. In addition, they are suitable for applications in many fields, such as wastewater treatment in the textile, mining, food and pharmaceutical industries [11-12]. Porous ceramics offer advantages such as high porosity, mechanical resistance, thermal and chemical stability, properties that favor their use in industrial applications such as filters and support for catalysts [13].

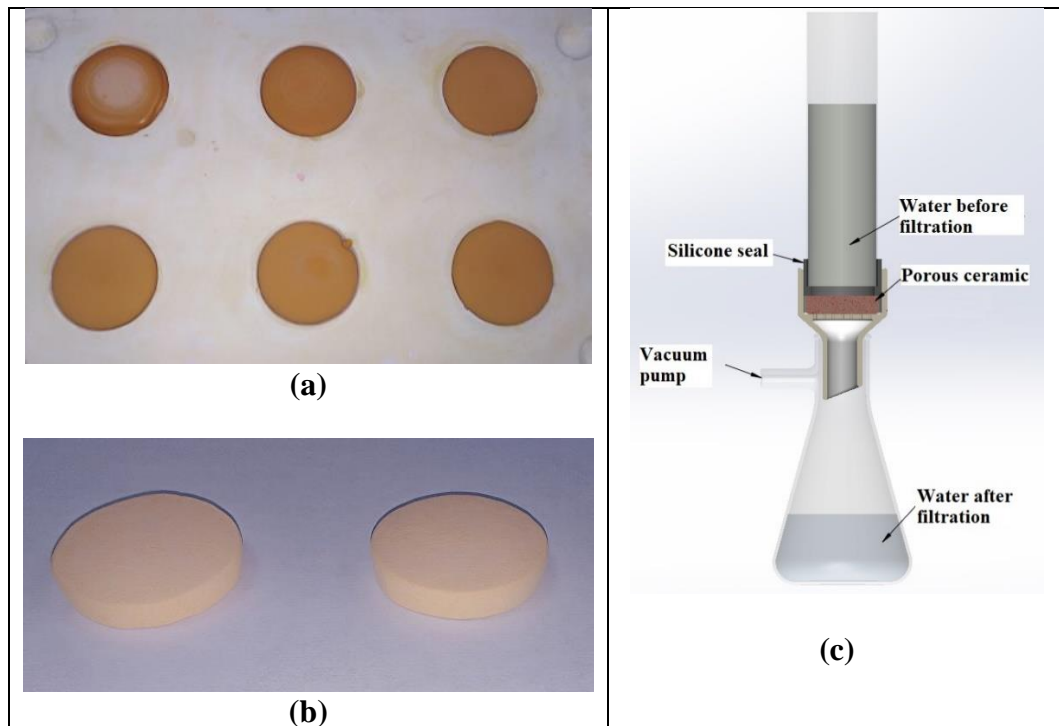
Therefore, the water treatment with low, medium and high concentrations of alkalinity; hardness to calcium, magnesium and total; chlorides; sulfates and iron, is reported. The water treatment process was by filtration by means of porous ceramics elaborated based of recycled diatomite of the brewing industry and kaolin.

## **2. Materials and methods**

The diatomite was supplied by the beer industry, which were subjected to a calcination process at 1000 ° C by Vulcan D-130 electric muffle, followed by an ultrasound treatment, subsequently they were dried using a forced circulation oven

Memmert UF-110 at 100 ° C during 24 hours. The particle size of diatomite powders, kaolin and calcium carbonate were less than 75  $\mu\text{m}$  (ASTM through sieve No 200). The porous ceramics were elaborated by slip casting in plaster mold, from pastes with weight percentage of 50% recycled diatomite from the beer industry, 40% kaolin and 10% calcium carbonate, where sodium silicate was used as deflocculant. Both diatomite and kaolin were subjected to a physical-chemical analysis using Perkin Elmer atomic absorption spectrometer (PinAAcle 900F) and Perkin Elmer UV-Visible spectrophotometer.

In figure 1 (a), the samples formed by slip casting in plaster mold are presented, while the sintered samples are presented in figure 1 (b), observing a cylindrical geometry with an approximate diameter of 5 cm, and a thickness of 1 cm, which were first dried at room temperature, subsequently using drying oven, then sintered by electric muffle at temperature of 1000 ° C, for two hours, with a sintering speed of 10 ° C / min. The morphology of the samples was studied using SEM (FEI-Quanta FG 650). The treatment of the waters was carried out by transversal filtration through the ceramic membrane following the experimental diagram presented in figure 1 (c), this process was done in triplicate.



**Figure 1.** (a) slip casting process of the samples of porous ceramics, (b) porous sintered ceramics and (c) experimental setup of filtration process of the prepared waters

Table 1 shows the relationship of water samples prepared in low, medium and high concentrations, these concentrations were defined taking as reference the maximum values allowed by resolution 2115 of 2007, where the maximum value allowed by the standard was definite with the medium concentration, while the low and high concentration, was defined as 50% and 150% respectively of the maximum value allowed by the standard. The water analysis before and after of being filtered was carried out in the water laboratory of the Francisco de Paula Santander University, for which the established protocols were followed according to the reference [14].

**Table 1.** Water Samples Prepared with Low, Medium and High Concentrations, taking as reference the resolution 2115 of 2007.

<b>Parameters</b>	<b>Low concentration mg/L</b>	<b>Medium concentration mg/L</b>	<b>High concentration mg/L</b>	<b>Permitted value resolution 2115/2007 mg/L</b>
<b>Alkalinity</b>	100.00	200.00	300.00	200.00
<b>Total hardness</b>	150.00	300.00	450.00	300.00
<b>Calcium hardness</b>	60.00	120.00	180.00	120.00
<b>Magnesium hardness</b>	90.00	180.00	270.00	180.00
<b>Chlorides</b>	125.00	250.00	375.00	250.00
<b>Sulfates</b>	125.00	250.00	375.00	250.00
<b>Total Iron</b>	0.15	0.30	0.45	0.30

### 3. Results and Discussion

The results of the physical-chemical study of the raw materials (recycled diatomite powders and kaolin) are presented in table 2, these were made using atomic absorption spectrometry (AAS), UV-Visible spectrophotometry and other complementary methods. From these it is observed that the pH of the raw materials is between 5 and 8.68, found that the recycled diatomite of the beer industry are alkaline, while the kaolin is acid, however, as reported by Gashaw Dessalewet al. [7], the greatest amount of nutrients is available in the soil when the pH ranges between 5 and 8. On the other hand, the concentration of organic matter was higher for the kaolin (1.87%) than for the recycled diatomite (0.63%), this is due to the calcination (1000 ° C) to which the diatomite samples were subjected, to eliminate the organic matter present, as well as increase its permeability and porosity to be used successfully in water treatment [15]. Likewise, it is appreciated that the cation exchange capacity (CEC) is between 10.53 and 13.26 meq / 100g, which represents medium exchange capacity. It is also observed a considerable difference between

the concentration of phosphorus and sulfur from diatomite recycled compared to kaolin, in opposite it is seen that the iron present in the kaolin sample is more than three times that the found for the diatomite.

**Table 2.** Physical-chemical properties of the materials of the porous ceramics

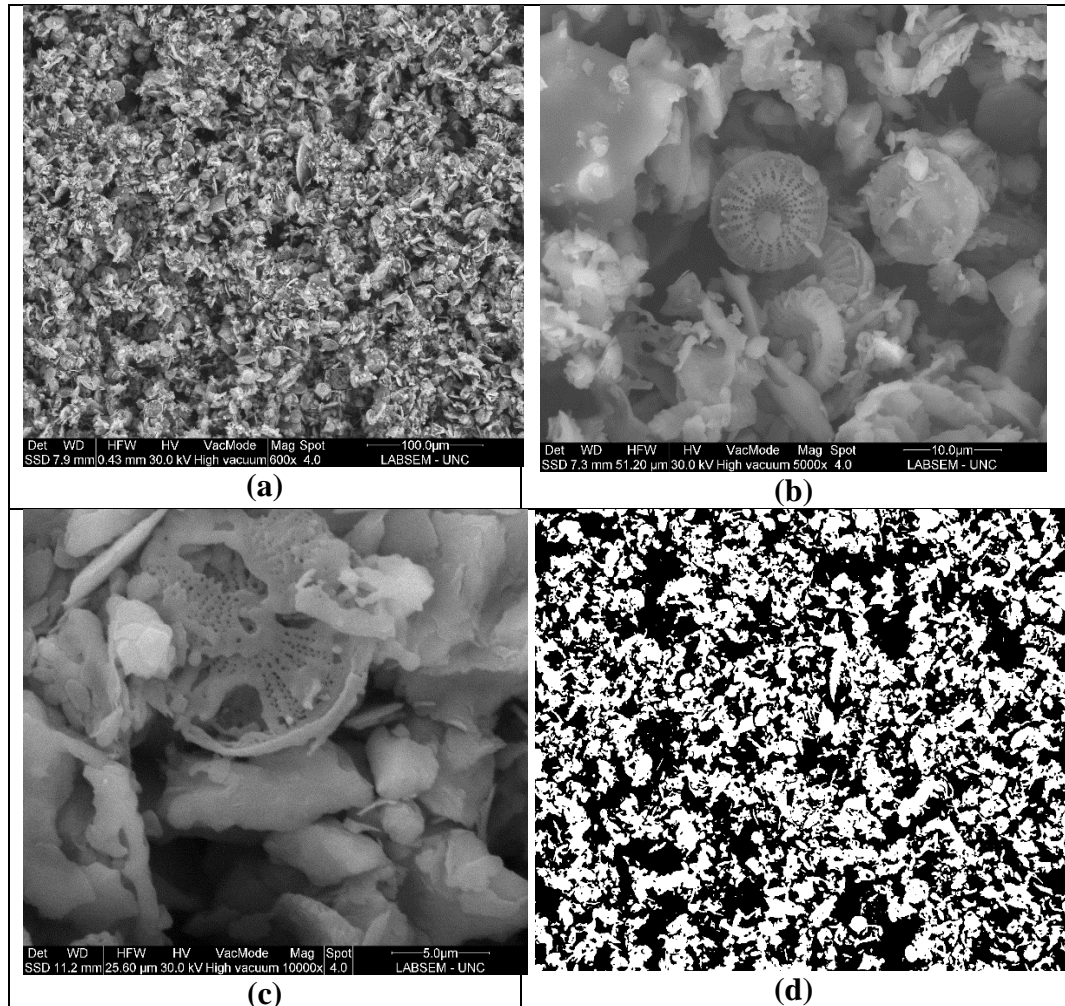
Parameters	measurement units	Diatomite	Kaolin	Methods
pH		8.68	5.03	Potentiometer 1:1
Organic material	%	0.63	1.87	Walkley Black
Phosphorus	ppm	294.00	8.00	Bray II
Potassium	meq/100 g	0.07	0.15	Extraction of ammonium acetate
Calcium	meq/100 g	1.48	1.21	pH 7.0
Magnesium	meq/100 g	0.29	0.01	AAS
Sodium	meq/100 g	0.48	0.09	Emission
Cation exchange capacity (CEC)	meq/100 g	13.26	10.53	Titration 0.1N HaOH
Iron	ppm	3.48	11.52	AAS
Manganese	ppm	0.17	3.14	AAS
Copper	ppm	0.16	0.51	AAS
Zinc	ppm	0.10	0.31	AAS
Boron	ppm	1.25	0.15	Colorimetric extraction with monocalcium phosphate
Sulfur	ppm	19.58	2.92	Turbidimeter

**Source:** UFPS soils laboratories.

The physical-ceramic properties of the ceramic membranes (see figure 1 (b)) were found using the ISO 10545-3 standard, finding an average density of 0.98 g/cm<sup>3</sup>, an approximate loss of mass in drying of 18% and of sinterization (fire) of 5%, the linear contraction by fire of 1.2%, and an average percentage of water absorption of 75.32%.

The sintered samples morphology of the porous ceramics using SEM is presented in figure 2. Complete and fractured diatoms with radial symmetry and diameter of tenths of microns are appreciated, which possess inside porosities of the nanometric order. Likewise, molten particles of kaolin spinels are observed, which are formed at temperatures around 950 ° C [16]. The superficial porosity using the ImageJ software to the 600X microphotograph (see figure 2 d)), reports an average value of 53.04%. In general, the morphology observed in the samples using SEM is typical of the porous membranes and ceramics, providing porosity, stability at high temperatures, mechanical properties, low creep rate, low coefficient of expansion

and thermal conductivity, as well as resistance to corrosion by the diatomite and kaolin structures [16], these properties contribute significantly to the filtration and water treatment processes.



**Figure 2.** Morphology of the porous ceramic sample using SEM a)600X, (b)5000X, (c) 10000X and (d) surface porosity using ImageJ to the microphotography (a).

Table 3 shows the results of the total alkalinity of the water samples before and after passing through the filtration system. It is appreciated that, for the samples with high concentration, the filtration process is not efficient, although the total alkalinity decreases by approximately 16.8%, however, the water after filtering reports a value higher than the maximum allowed. The above is consistent with the values found for hardness (see table 4), since these parameters are directly proportional, that is, the higher the concentration of carbonates, the greater the alkalinity. In general it can be inferred that sintered porous ceramics are not efficient for the treatment of hard water, as expected, since the hardness is soluble in water

and can only be reduced by precipitation or softening with lime and sodium carbonation [ 4]. The analysis of chlorides and sulphates, are presented in Tables 5 and 6, it is observed a high efficiency of sintered ceramics in the removal of chlorides, since as the concentration increases in water the higher the retention percentage, reaching an approximate value of 51% for samples with high concentration. Likewise, good efficiency in the removal of sulfates is observed, removing up to 43% for water samples with high concentration. It is also observed (see table 7) that the removal of iron ions present in the water samples are removed when passing through the porous ceramic sintered, with an efficiency of up to 100%, for high concentrations. This is due to the high CEC and porosity of the ceramic, which allows the adsorption of chlorine, sulfate and iron ions [17].

**Table 3.** Results of the analysis of total alkalinity, water samples.

Water samples	Before filtration	After filtration	Permitted value resolution 2115/2007
Low concentration	100.00 ± 2.00	89.75 ± 2.25	200 mg/L
Medium concentration	200.00 ± 2.50	169.15 ± 3.25	
High concentration	300.00 ± 2.50	249.50 ± 6.25	

**Table 4.** Results of the total hardness analysis, water samples.

Water samples	Before filtration	After filtration	Permitted value resolution 2115/2007
Low concentration	150.00 ± 8.50	111.95 ± 7.25	300 mg/L
Medium concentration	300.00 ± 12.50	266.15 ± 5.45	
High concentration	450.00 ± 10.45	401.65 ± 7.50	

**Table 5.** Results of the chlorides analysis, water samples.

Water samples	Before filtration	After filtration	Permitted value resolution 2115/2007
Low concentration	125.00 ± 6.40	83.15 ± 2.25	250 mg/L
Medium concentration	250.00 ± 7.75	151.15 ± 5.13	
High concentration	375.00 ± 5.03	181.95 ± 8.50	

**Table 6.** Results of sulfate analysis, water samples.

Water samples	Before filtration	After filtration	Permitted value resolution 2115/2007
Low concentration	125.00 ± 6.40	101.30 ± 1.25	250 mg/L
Medium concentration	250.00 ± 7.75	200.15 ± 15.45	
High concentration	375.00 ± 5.03	213.56 ± 14.50	

**Table 7.** Iron analysis results, water samples.

Water samples	Before filtration	After filtration	Permitted value resolution 2115/2007
Low concentration	0.15 ± 0.01	0.04 ± 0.01	0.30 mg/L
Medium concentration	0.30 ± 0.01	0.03 ± 0.01	
High concentration	0.45 ± 0.01	0.00	

#### 4. Conclusions

Diatomite recycled from the brewing industry were activated by means of calcination, which were mixed with kaolin and calcium carbonate to sinter porous ceramic discs with the objective of water treatment by transversal filtration. The waters were prepared in the laboratory at low, medium and high concentrations, taking as a point of reference the maximum values allowed according to resolution 2115 of 2007. The surface morphology of porous ceramics showed diatom frustules complete and fractured, as well as the kaolin spinel lamellas, with meso-porosities and optimal water absorption percentage for applications in filtration and water treatments.

In general, it can be inferred that sintered porous ceramics were effective for the removal of chlorides, sulphates and iron, during the waters filtration process, which is due to the high specific surface, porosity and CEC, which allows the fixation of the chlorine, sulfate and iron ions, on the other hand, were found not to be effective for the treatment of the alkalinity and hardness of the water, as expected, because that these parameters they are soluble in water and can be reduced by precipitation or softening adding for example lime and sodium bicarbonate among others.

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