

# **Treatment of Subterranean Mining Water through Filtration Using Ceramic Bilayer Membranes Based of Recycled Diatomites and Kaolin**

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## **Abstract**

The treatment of underground wastewater is presented. Used at the San Martín 5 mine in the San Roque sector of the municipality of Sardinata (Norte de Santander-Colombia); through the process of filtration through bilayer porous membrane (diatomite-kaolin / kaolin) based on recycled diatomite from the brewing industry and kaolin. The water samples were looked at before and after the filtration process, taking as reference the maximum values allowed by resolution 0631 of 2015. The analyzed parameters were Suspended solids, BOD5, COD, pH, Fe, dissolved solids. The porous cylindrical ceramics, 5 cm in diameter and 0.9 cm thick, were formed by casting using the plaster mold, where the layer of 100% kaolin with a thickness of approximately 0.3 cm was first deposited. applied the second layer from pulps with weight percentage of 50% recycled diatomite from the brewing industry, 40% kaolin, and 10% calcium carbonate. Once the ceramics are carried out, they are

subject to the drying process at room temperature for 24 hours, then by means of the forced circulation oven Memmert UF-110 at 100 ° C for an equal time. The sintering of the ceramic 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 physical-ceramic analyzes were performed in accordance with ISO 10545-3. The results show that the ceramic membrane is, in fact, 52% for the reduction of total solids, while the elimination of iron is 38%, on the other hand, it is also evident that the ceramics are not effective in the treatment of dissolved solids with high concentrations.

**Keywords:** Mining water treatment, Bilayer porous ceramic, Recycled diatomite, Kaolin

## **1. Introduction**

Mining plays a relevant role in terms of environmental problems; as it is the pollution of rivers, lakes, groundwater that affects the ecology and at the same time human health, the class of pollutants present in the environment depends on the degree of concentration, on the other hand, climatic factors play an important role with respect to the availability of water and the assimilation of pollutants in the environment. Consequently, the disappearance of biological diversity present in the tributaries that are affected by deposits and discharges of activities related to mining exploitation is reflected. [1-2]

Therefore, water pollution is one of the crises with the greatest emphasis because water is the primordial base of the existential life. Therefore, interest arises in the application of micro filtration and ultrafiltration membranes in processes for the separation of contaminants for the treatment of large amounts of wastewater. So it becomes a successful way to solve water pollution and maintain the welfare of the environment [3]. Thus, for example, the use of ceramic membranes prepared from a silicate and clay-mineral for the removal of oils in wastewater from metallurgical, petrochemical, transport, textile and oil refinery activities, among others, is Research topic, due to its simplicity, profitability, longer duration, thermal and mechanical stability. [4]

The use of ceramic membranes is a low-cost process, in addition to advantages such as high thermal and chemical stability, pressure resistance, long service life, good fouling resistance and ease of cleaning [3]. Bearing in mind that Diatomite is a macro structured material, micro-porous and of low cost, that allows to be used in the adsorption of pollutants since it has characteristics such as high porosity, mechanical resistance, thermal and chemical stability, so they are a suitable material

in the process of filtering heavy metals present in contaminated water in mining extraction processes. [5]

For this reason, the groundwater treatment of the San Martín 5 mine, located in the municipality of Sardinata (Norte de Santander - Colombia), is presented at the laboratory level, using transverse filtration through two-layer ceramic membranes of recycled diatomite from the industry. brewer and kaolin.

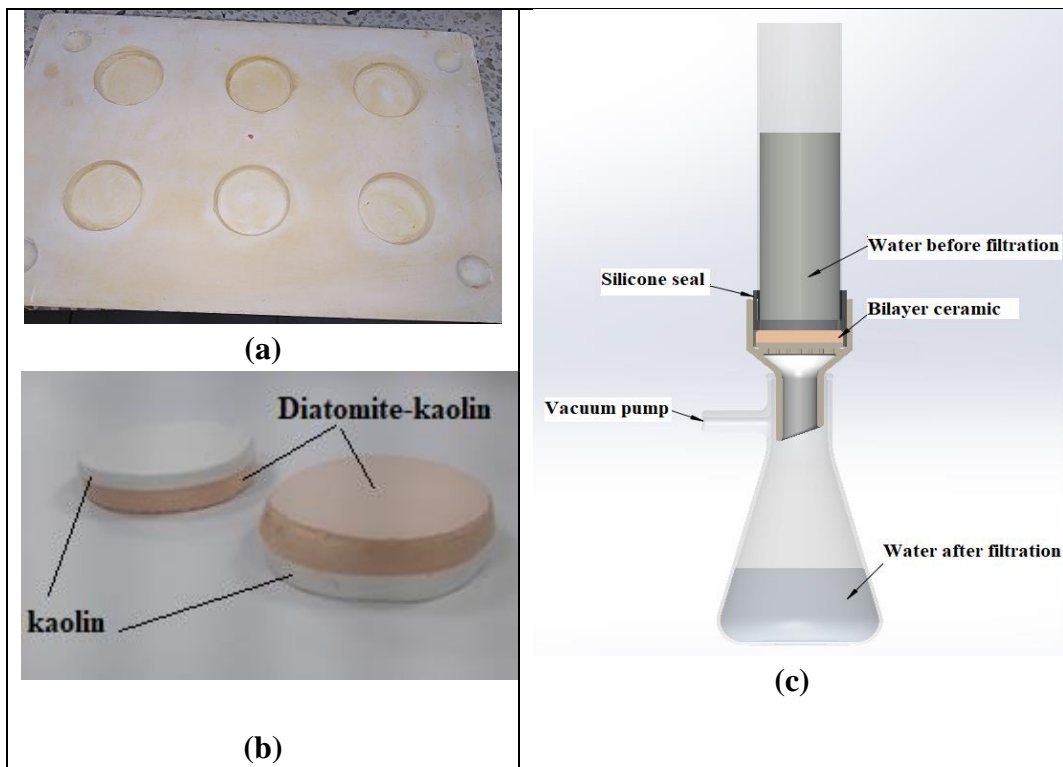
## **2. Materials and methods**

Once obtained the diatomites discarded by the brewing industry in their filtration processes, they underwent an activation process, which consisted of a thermal treatment, followed by ultrasound agitation, and finally drying. The calcination was carried out using Vulcan D-130 electric muffle, at a maximum temperature of 1000 ° C, with a heating ramp of 10 ° C / min. The application of the ultrasound was performed using the digital cuvette CE-5600A, with frequency of 30kHz, during 8 minutes. For the drying, Memmert forced circulation oven UF-110 was used at 100 ° C for 24 hours. Both the activated diatomite powders, as well as those of kaolin and calcium carbonates were screened using ASTM No. 200 mesh (<75 µm), they were subjected to a physical chemical analysis using Perkin Elmer atomic absorption spectrometer (PinAAcle 900F) and Perkin Elmer UV-Visible spectrophotometer.

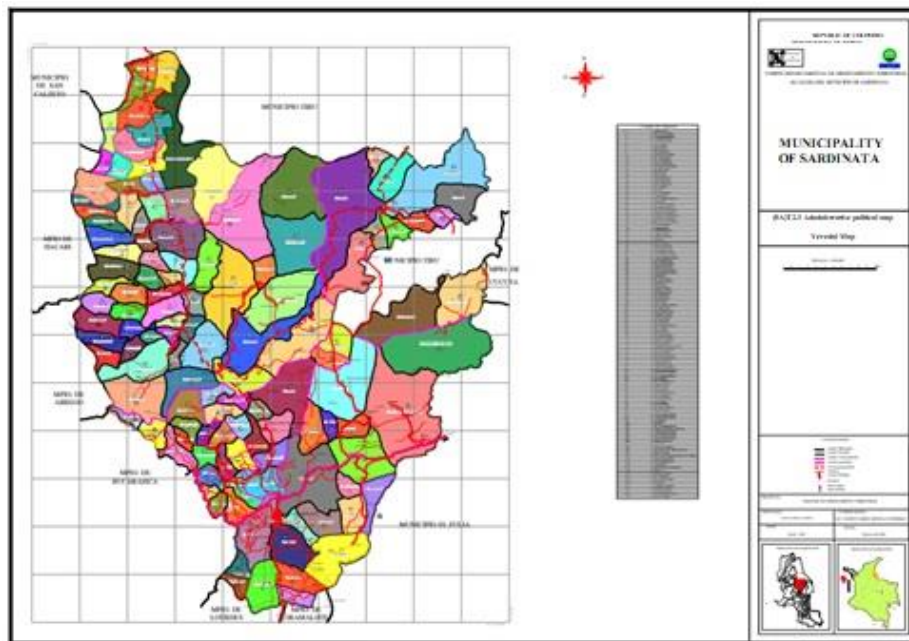
The conforming of the bilayer porous ceramics sees figure 1b) was done by casting in plaster mold (see figure 1 a). The first layer that was applied was that of kaolin (ratio 1: 0.5 (kaolin: water)), with a thickness of approximately 0.3 cm. Next step was applied on the kaolin layer, the diatomite-kaolin-carbonate slip with weight percentages of 50%, 45% and 5% respectively, and with an approximate thickness of 0.6 cm. Once the samples were formed, they were subjected to the drying and sintering process, with a firing curve of room temperature at 600 ° C at a rate of 5 ° C / min, then brought to 1000 ° C at a rate of 10 ° C / min, staying 2 hours at this temperature, then the cooling process begins.

In Figure 1b), the bilayer cylindrical membranes are presented. The surface 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 bilayer membrane following the experimental diagram presented in figure 1c), this process was done in triplicate.

The water samples were taken at the outlet of the San Martín 5 mine, which is discharged directly into the Resaca stream of the San Roque sector of the municipality of Sardinata, Norte de Santander-Colombia, with geographical coordinates of 8.40999 ° N and 72,77692 ° W, the sector is located north-west about 18 km from the urban area. The procedure for taking samples was carried out as established by the IDEAM (Institute of Hydrology, Meteorology and Environmental Studies) [6], they were kept at 4 ° C to preserve them and later analyze them in the water laboratory of the Francisco de Paula Santander, in figure 2, shows the geographical location of the sampling site.



**Figure 1.** a) plaster mold used in shaping the membranes, b) bilayer ceramic membranes, c) experimental design of the water filtering process.



**Figure 2.** Geographic location of the San Roque sector, municipality of Sardinata, Norte de Santander-Colombia, sampling site.

In table 1, the parameters studied are presented to the water samples before carrying out the filtration process, taking as reference Resolution 0631 of 2015, which establishes the parameters and the permissible limit values in point discharges to bodies of surface water and public sewage systems, for which protocols established according to the reference were followed [7].

**Table 1.** Parameters studied to the waters before the filtration process

<b>PARAMETER</b>	<b>MAXIMUM PERMISSIBLE VALUE (mg / L)</b>	<b>SHOW BEFORE THE TREATMENT</b>
Dissolved suspended solids	50	<b>3221</b>
Hydrogen potential	6.0 a 9.0	<b>2.92</b>
Total Iron	2	<b>7.3</b>
Total Suspended Solids	mg/L 50,00	<b>21</b>
Total Chlorine	N/A	<b>0.1</b>
Conductivity	mS/cm	<b>3.73</b>
DBO5	50	<b>13.8</b>
DQO	150	<b>22.3</b>

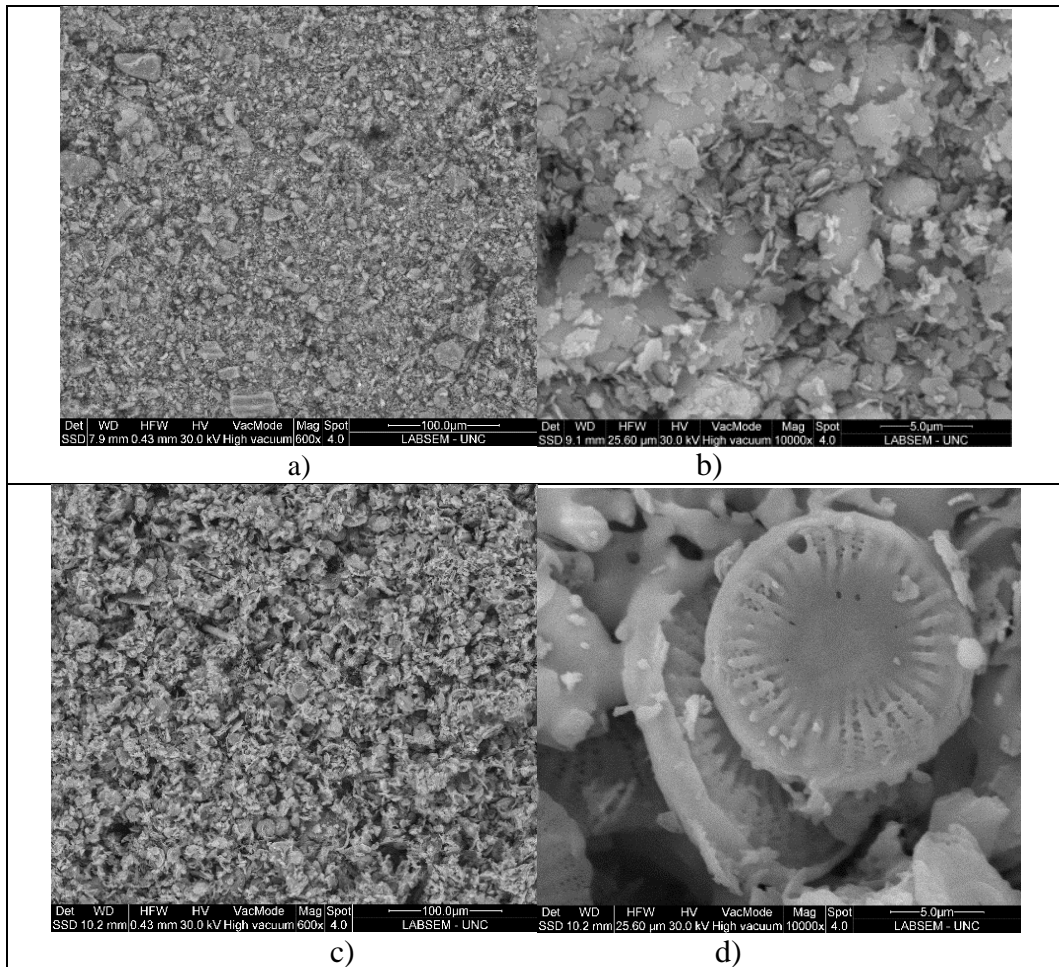
### 3. Results and Discussion

The results of the physical-chemical study of raw materials (recycled diatomite and kaolin powders) made using atomic absorption spectrometry (AAS), UV-Visible spectrophotometry and other complementary methods, reported that the pH of the kaolin was 5.03, while for the diatomite was 8.68, from the above it is inferred that the diatomites are alkaline, which is consistent with that reported in the reference [8]. It was also found that the percentage of organic matter of diatomite and kaolin was 0.63% and 1.87% respectively, from this it is inferred that the process of calcination and treatment with ultrasound to diatomite was efficient. Regarding the cation exchange capacity (CEC), it was found that the diatomite has a 13.26 meq / 100g, while for the kaolin it was 10.53 meq / 100g.

The physical-ceramic properties of the bilayer membranes (see figure 1 (b)) were found using ISO 10545-3. Finding an average density of 0.94 g / cm<sup>3</sup>, an approximate loss of mass in drying of 5.5% and by cooking of 5%, the linear shrinkage by cooking of 4%, and an average percentage of water absorption of 76.25%.

The morphology of the sintered samples of the porous ceramics using SEM is presented in figure 3. In this, micrographs are presented at 600X (Fig. 3a) and 10000X

(Fig. 3b) of the superficial part corresponding to the kaolin layer and in figure 3c) and 3d), those of the diatomite-kaolin layer. Here we can see laminar particles of kaolin spinels, which are formed at approximate temperatures of 750°C, as well as complete and fractured diatoms with radial symmetry and diameter of tenths of microns, which have porosities of the order nanometer. In general, the morphology observed in samples using SEM, is typical of porous membranes [9] and ceramics providing porosity, stability at high temperatures, mechanical properties, low creep rate, low expansion coefficient and thermal conductivity, as well as corrosion resistance provided by the diatomite and kaolin [10-11] structures, these properties contribute significantly to the filtration and water treatment processes as evidenced in the reference. [12-13].

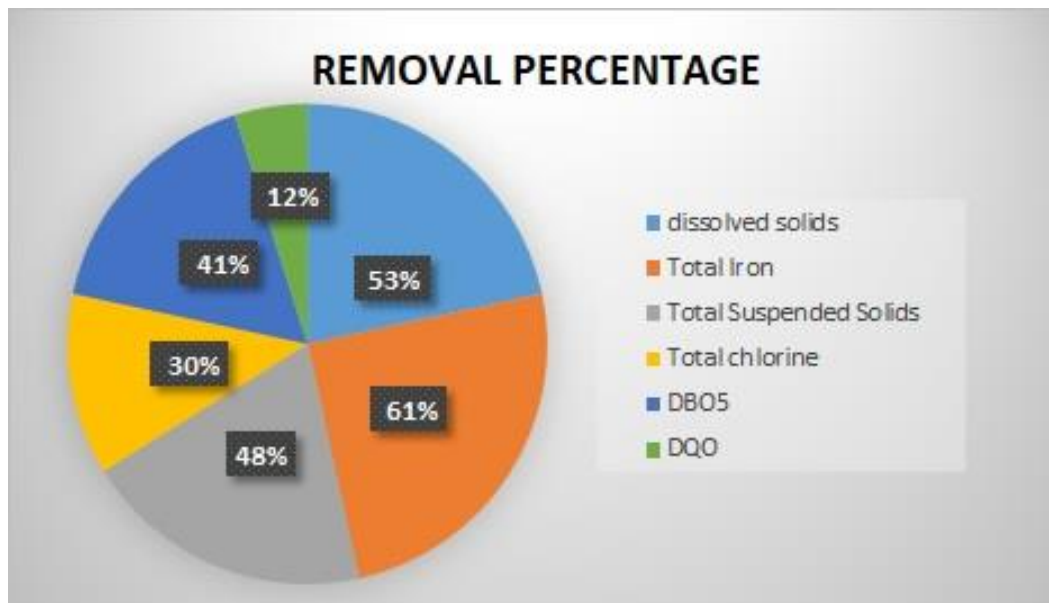


**Figure 3.** Surface morphology of the porous ceramic sample using SEM. a) and b) correspond to the kaolin layer at 600X, and 10000X. c) and d) correspond to the diatomite-kaolin layer at 600X and 10000X

Table 2 shows the results of pH, dissolved solids, total solids, chlorine, BOD5, COD, conductivity, total iron of the water sample before and after passing through the filtration system, the above is consistent with the reference [14], who worked with crude diatomite and modified as tertiary treatment of municipal wastewater, measured water quality parameters obtaining similar results, it is appreciated that, the filtration process is efficient with the removal of iron (see figure 3), which is justified in the cation exchange capacity of the ceramic membranes. Regarding dissolved solids (see figure 3) although these decrease by 53%, however, this value is higher than the maximum allowed according to resolution 0631 of 2015, in terms of pH was increased by 57%, giving compliance to the Estimated values in Res. 0631 of 2015, this could be due to the concentration of calcium carbonate present in the diatomite-kaolin layer. In general, it can be inferred that sintered porous ceramics are efficient for wastewater treatment of mineral extraction processes. The above is due to the porosity of the bilayer membrane which allows the adsorption of the iron ions present in the water samples are removed when passing through the sintered filters, being an efficiency up to 61%, although it is not complying with Resolution 0631 of 2015; similarly, a suspended solid removal was presented.

**Table 2.** Result Obtained After the Filtration Process

<b>PARAMETER</b>	<b>MAXIMUM PERMISSIBLE VALUE (mg / L)</b>	<b>SHOW BEFORE THE TREATMENT</b>	<b>SHOW AFTER THE TREATMENT</b>
Dissolved solids	50	<b>3221</b>	<b>1513</b>
Hydrogen potential	6.0 a 9.0	<b>2,92</b>	<b>6,77</b>
Total iron	2	<b>7,3</b>	<b>2,83</b>
Total suspended solids	mg/L 50,00	<b>21</b>	<b>11</b>
Total chlorine	N/A	<b>0,1</b>	<b>0,07</b>
Conductivity	mS/cm	<b>3,73</b>	<b>3,66</b>
DBO5	50	<b>13,8</b>	<b>8,2</b>
DQO	150	<b>22,3</b>	<b>19,6</b>



**Figure 4.** Percentages of removal of bilayer ceramics for groundwater.

#### 4. Conclusions

Recycled diatomite's from the brewing industry were activated by means of calcination and ultrasound, which were mixed with kaolin to form porous bilayer membranes with the aim of treating underground mining waters by transversal filtration, obtaining efficient results in the increase of pH and conductivity allowing comply with the permissible values for subsequent dumping. On the other hand, it was found that they are not effective for the treatment of BOD5 and COD

The cross-sectional filtration method of ceramic membranes based on diatomites and kaolin is efficient as shown in Figure 4 and the results obtained, however, showed that the sample did not comply with that established in resolution 0631 of 2015 for the parameter of suspended solids dissolved since it did not remove the established by the normal, which indicates that it should be 50mg / L, however this method manages to remove up to 53.02% of the total of the contaminated sample.

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