Study of the influence of the aluminum source (acetate or sulfate) on the synthesis of the ceramic membrane and applications of emulsion oil water: use and reuse

Estudo da influência da fonte de alumínio (acetato ou sulfato) na síntese da membrana cerâmica e aplicações da emulsão óleo-água: uso e reuso

Estudio de la influencia de la fuente de aluminio (acetato o sulfato) en la síntesis de la membrana cerámica y aplicaciones de emulsión aceite agua: uso y reutilización

Antonielly dos Santos Barbosa
ORCID: https://orcid.org/0000-0002-5739-8772
Universidade Federal de Campina Grande, Brazil
E-mail: antoniellybarbosa@yahoo.com.br
Antusia dos Santos Barbosa
ORCID: https://orcid.org/0000-0002-8998-1727
Universidade Federal de Campina Grande, Brazil
E-mail: antusiasb@hotmail.com
Meiry Gláucia Freire Rodrigues
ORCID: https://orcid.org/0000-0003-2258-4230
Universidade Federal de Campina Grande, Brazil
E-mail: meiry.rodrigues@ufcg.edu.br

Abstract
The objective of this work was to prepare ceramic membranes and to evaluate the effect of the raw material on the ceramic membrane and on the efficiency of the emulsion separation oil/water. The ceramic membranes were manufactured using the uniaxial dry compaction method, from the thermal decomposition of aluminum sulfate or aluminum acetate, to evaluate the effect of the raw material (aluminum acetate or aluminum sulfate) on the efficiency in the emulsion oil/water separation. Ceramic membranes were characterized by measurements of X-ray diffraction patterns, scanning electron microscopy, mechanical strength, bubble point and water flow. In this study, membranes were produced with different characteristics. The values found for the permeate for the A1 membrane were 9.20 mg / L due to characteristics such as porosity and mechanical strength (44.63 % and 1.3 MPa), while the values A2 membrane was 6.52 mg / L, 18.86 % and 6.7 MPa. In conclusion, the membranes prepared are effective in removing the oil from the oily waste water. According to the results, the treatment of oil-water emulsions by microfiltration facilitates a significant reduction in the concentration of permeate oil.
Keywords: Aluminum sulfate; Aluminum acetate; Ceramic membranes; Emulsion oil/water; Reuse.

Resumo
O objetivo deste trabalho foi preparar membranas cerâmicas e avaliar o efeito da matéria-prima na membrana cerâmica e na eficiência da separação em emulsão óleo / água. As membranas cerâmicas foram fabricadas pelo método de compactação uniaxial a seco, a partir da decomposição térmica de sulfato de alumínio ou acetato de alumínio, para avaliar o efeito da matéria-prima (acetato de alumínio ou sulfato de alumínio) na eficiência na separação óleo / água da emulsão. As membranas cerâmicas foram caracterizadas por medidas de padrões de difração de raios-X, microscopia eletrônica de varredura, resistência mecânica, ponto de bolha e fluxo de água. Neste estudo, foram produzidas membranas com características diferentes. Os valores encontrados para o permeado para a membrana A1 foram 9,20 mg / L devido a características como porosidade e resistência mecânica (44,63% e 1,3 MPa), enquanto os valores da membrana A2 foram 6,52 mg / L, 18,86 % e 6,7 MPa. Em conclusão, as membranas preparadas são eficazes na remoção do óleo das águas residuais oleosas. De acordo com os resultados, o tratamento das emulsões óleo-água por microfiltração facilita uma redução significativa na concentração do óleo permeado.
Palavras-chave: Sulfato de alumínio; Acetato de alumínio; Membranas cerâmicas; Emulsão óleo/água; Reutilização.

Resumen
El objetivo de este trabajo fue preparar membranas cerámicas y evaluar el efecto de la materia prima sobre la membrana cerámica y sobre la eficiencia de la separación de la emulsión aceite / agua. Las membranas cerámicas se fabricaron mediante el método de compactación seca uniaxial, a partir de la descomposición térmica de sulfato de aluminio o acetato de aluminio, para evaluar el efecto de la materia prima (acetato de aluminio o sulfato de aluminio) sobre la eficiencia en la separación aceite / agua de la emulsión. Las membranas cerámicas se caracterizaron mediante

mediciones de patrones de difracción de rayos X, microscopía electrónica de barrido, resistencia mecánica, punto de burbuja y flujo de agua. En este estudio, se produjeron membranas con diferentes características. Los valores encontrados para el permeado para la membrana A1 fueron 9,20 mg / L debido a características como porosidad y resistencia mecánica (44,63 % y 1,3 MPa), mientras que los valores de membrana A2 fueron 6,52 mg / L, 18,86% y 6,7 MPa. En conclusión, las membranas preparadas son eficaces para eliminar el aceite de las aguas residuales aceitosas. Según los resultados, el tratamiento de las emulsiones aceite-agua por microfiltración facilita una reducción significativa de la concentración de aceite permeado.

Palabras clave: Sulfato de aluminio; Acetato de alumínio; Membranas cerámicas; Emulsión aceite/agua; Reutilizar.

1. Introduction

Water is an essential resource in the world used for economic, social and cultural development. This feature is considered to be abundant. With the industrial revolution and the increase in population, its demand has increased and scarcity is now an irrefutable result (Singh, 2015).

The biggest energy problem is the limited supply of fossil energy, in addition it generates environmental impacts throughout the energy life cycle, from mining and processing to emissions, waste disposal and recycling (Evans et al., 2009). Energy sustainability will be achieved through the development of sustainable technologies to replace non-renewable fossil fuels. Membranes technologies play an important role in water and energy sustainability.

The biggest waste generated in the oil industries is the water produced (Lodungi et al., 2016). One of the biggest challenges faced is the management of water produced by the oil and gas industries and the protection of human health and the environment is the management of produced water. This water is problematic to treat due to its complex physicochemical composition. Membrane technology plays an increasingly important role in produced water treatment (Ebrahimi et al., 2018).

The applied membrane techniques may be different depending on the reuse of the water produced (Çakmakce et al., 2008; He, & Jiang, 2008; Zaidi et al., 1992; Ashaghi et al., 2007; Bilstad, & Espedal,. 1996; Li et al., 2006).

Membrane technology has become a high-effectiveness separation technology for industrial processes (Burggraaf, & Cot, 1996; Samaei et al., 2018). Inorganic membranes exhibit excellent thermal, chemical and mechanical stability suitable for separation applications. These processes are operated under severe conditions, such as, high temperatures, high pressures and aggressive chemical environments that polymer membranes cannot handle (Li et al., 2006). In the last few years, inorganic membrane separation technology has knowledge an accelerate growth and innovation. Various membrane separation processes have been developed and new processes are continually being studied in both the academic and industrial research areas.

Membrane separation processes are alternative methods for the oil-water separation (Padaki et al., 2015; Madaeni et al., 2012; Barbosa et al., 2019; Barbosa et al., 2020). The present study is part of this line of research and represents another contribution to the field, presenting the preparation of the ceramic membrane (γ-alumina) from the decomposition of aluminum sulphate and aluminum acetate, which were used as an alternative source of matter for the production of alumina. The effect of experimental parameters such as the type of raw material used in the manufacture of the ceramic membranes and the mechanical stability of the membranes was investigated. As an application to evaluate its performance, the ceramic membranes were used for the separation of oil-water emulsion.

2. Methodology

2.1 Starting materials

Inorganic membranes with two raw materials (aluminum sulfate or aluminum acetate) was produced by uniaxial dry compaction method on a laboratory scale. The results obtained in this research are quantitative, contributing to the literature.

Aluminum acetate ($Al(OH)(CH_3OO)_2$) and Aluminum sulfate ($Al_2(SO_4)_3.18H_2O$) were purchased from Vetec.

2.2 Thermal decomposition of aluminum sulfate and aluminum acetate

The aluminum sulfate was submitted to the following heat treatment: heating from room temperature until 1000 ºC with a controlled heating rate of 5 °C/min and then 2 h to obtain gamma-alumina (powder) (Pelovski et al., 1992). The aluminum acetate was submitted to the following heat treatment: heating from room temperature until 850 ºC with a controlled heating rate of 5 °C / min and then 2 h to obtain gamma-alumina (powder) (Sato et al., 1984). Thermal decomposition of aluminum sulphate and aluminum acetate was carried out using a muffle furnace.

The steps to be followed in the thermal decomposition of aluminum sulfate and aluminum acetate are shown in the diagram in Figure 1.

Figure 1. Thermal decomposition of aluminum sulfate and aluminum acetate.
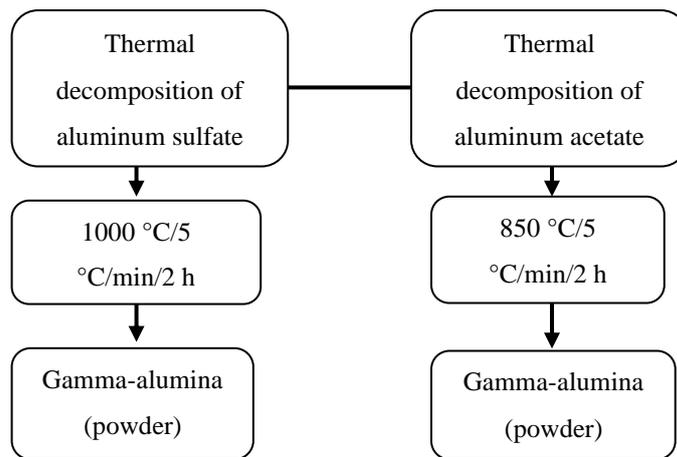
Source: Authors.

2.3 Production of ceramic membrane (γ-alumina)

Inorganic membranes with two raw materials (aluminum sulfate or aluminum acetate) was produced by uniaxial dry compaction method.

There was a mixture of γ-alumina with additives in a total of 200 ml of dispersion in the following composition: 40 % of alumina obtained above; 0.2 % of para-aminobenzoic acid (dissolved in ethyl alcohol), 0.5 % oleic acid (lubricant), and 59.3 % ethyl alcohol. The mixture was milled in a ball mill for 1 h and then dried in an oven for 24 hours at 60 °C.

3.0 g of the undersieved powders were uniaxially pressed using a 5-ton hydraulic press to produce a disk-shaped ceramic membrane. Subsequently, the green compacts were submitted to the following heat treatment: heating from room temperature until 700 ºC for 2 h. After cooling, flat cylindrical ceramic membranes with final diameter of 26 mm and thickness of 3.0 mm were produced. The steps to be followed in the Production of the ceramic membrane (γ-alumina) from aluminum sulfate or aluminum acetate are shown in the diagram in Figure 2.

Figure 2. Ceramic membrane (γ-alumina) of the aluminum sulfate and aluminum acetate.

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┌─────────────────────┐          ┌─────────────────────┐
│  (γ-alumina) of the │──────────│  (γ-alumina) of the │
│   aluminum sulfate  │          │   aluminum acetate  │
└─────────────────────┘          └─────────────────────┘
          │                                 │
          ▼                                 ▼
┌─────────────────────┐          ┌─────────────────────┐
│      Additives      │          │      Additives      │
└─────────────────────┘          └─────────────────────┘
          │                                 │
          ▼                                 ▼
┌─────────────────────┐          ┌─────────────────────┐
│   Ball mill for 1 h │          │   Ball mill for 1 h │
└─────────────────────┘          └─────────────────────┘
          │                                 │
          ▼                                 ▼
┌─────────────────────┐          ┌─────────────────────┐
│    Stove for 24 h   │          │    Stove for 24 h   │
└─────────────────────┘          └─────────────────────┘
          │                                 │
          ▼                                 ▼
┌─────────────────────┐          ┌─────────────────────┐
│       Pressed       │          │       Pressed       │
└─────────────────────┘          └─────────────────────┘
          │                                 │
          ▼                                 ▼
┌─────────────────────┐          ┌─────────────────────┐
│   Heat treatment    │          │   Heat treatment    │
└─────────────────────┘          └─────────────────────┘
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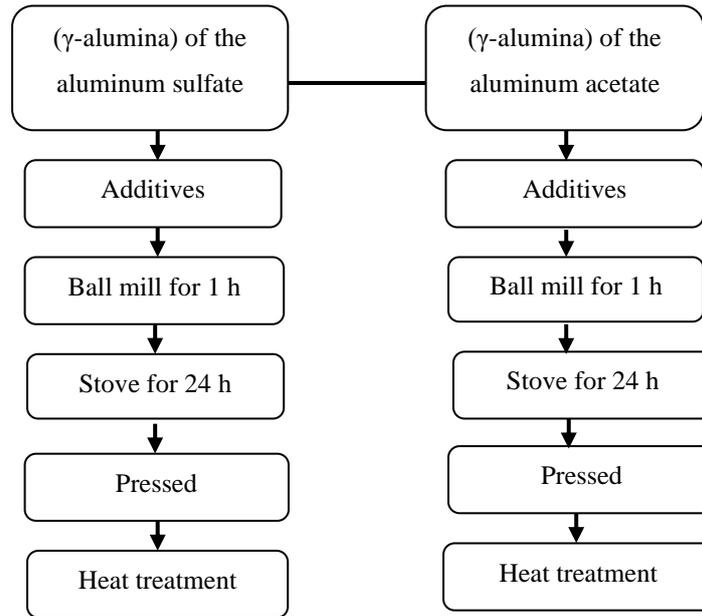
Source: Authors.

Ceramic membranes obtained after thermal decomposition of aluminum sulfate and aluminum acetate are shown in Figure 3.

Figure 3. Ceramic membranes produced.
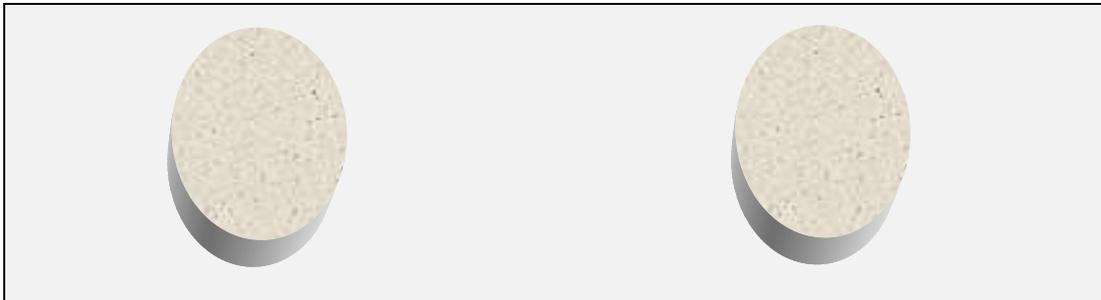
Source: Authors.

2.4 Characterization techniques

2.4.1 X-ray Diffraction (XRD)

X-ray diffraction analysis of samples was performed using a diffractometer Shimadzu XRD 6000m, Kyoto, Japan) with Copper Kα radiation, operated at 30 mA and 40 KV, with a goniometer velocity of 2 °/min and a step of 0.02 ° in the range of 2θ scanning from 2 ° to 50 °.

2.4.2 Scanning Electron Microscopy (SEM)

Powders and membranes morphology was determined by scanning electron microscopy (Philips XL 30, Amsterdam, Netherlands).

2.4.3 Bubble point

The bubble point method used is standardized by ASTM F316-03. It consists of filling the porous structure of the membrane with a liquid and measuring the air pressure needed to displace the liquid inside the pores. The mathematical relationship between pressure and pore size is given by Washburn equation 1:

$$\Delta P = \left(\frac{4\gamma \cos \varphi}{d_P}\right)$$

Eq. (1)

Where ΔP is the pressure drop (bar), dp is the pore size (µm), φ is the contact angle between the fluid and pore walls, and γ is the surface tension of the liquid (isopropyl alcohol).

2.4.4 Water flux measurements

The performance of the ceramic membranes with regard to permeate flow was analyzed in a laboratory-scale continuous-flow separation system.

The permeate samples were run at 5 min intervals for a total period of 60 min for each membrane. The flow was calculated according to equation 2.

$$J = \frac{V}{A.\Delta t}$$

Eq. (2)

where J is the water flux (L/m^2.h), V is the permeate volume (L), A is the membrane area (m^2) and Δt is the permeation time (h).

The system permeation / separation is shown schematically in Figure 4.

The permeation/separation unit consists of a feed tank (glass beaker) (I) with a capacity of 2L; a peristaltic pump (II) Cole Parmer; and a stainless steel module for ceramic supports (ceramic membranes) and ceramic membranes (III).

In figure 4 it can be seen that the fluid pumped into the module is divided into two streams, the permeate and the concentrate. The system operates with constant removal of permeate (to analyze the flow and concentration of the remaining oil) and/or concentrate. The system features two gauge pressure indicators with a scale up to 10 bar, one installed before the module inlet (5-1) and another installed at the concentrate line outlet (5-2), they are accessories for regulating the fluid pressure through of the membrane, whose adjustment is made through the regulating valve (6), installed in the return of the concentrate line to the tank.

Figure 4. Permeation/separation system used to assess the permeability and selectivity of ceramic membranes.
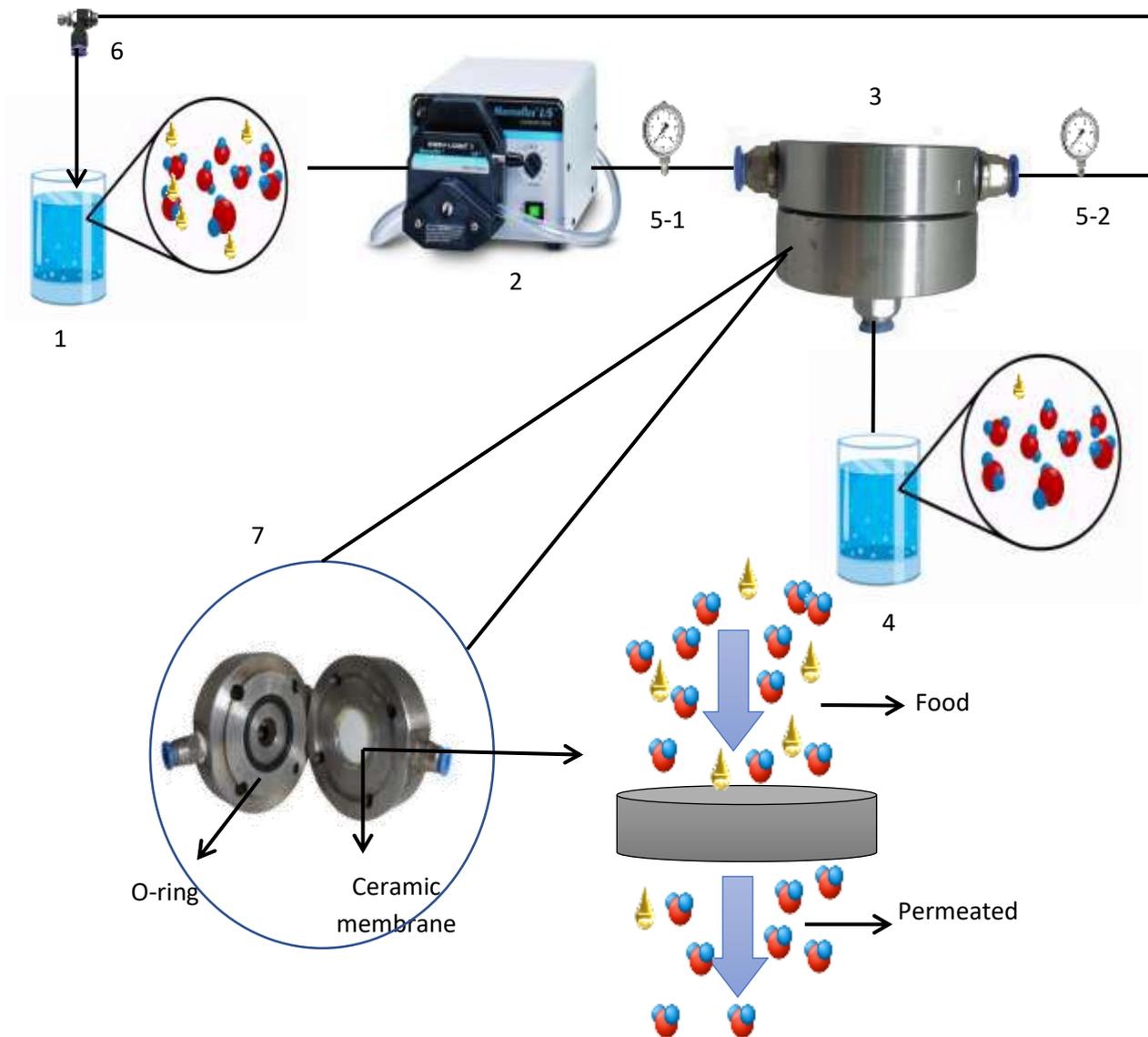
Source: Authors.

1 – Feed tank - glass beaker (2L), system feed (distilled water or oil/water emulsion);

2 – Peristaltic pump;

3 – Permeation/Separation Module (in stainless steel);

4 – Glass beaker (0.5 L), permeated volume;

5-1 – Pressure gauge before entering the module;

5-2 – Manometer at the concentrate outlet;

6 – Regulating valve (concentrate outlet).

7 – Expansion of the Permeation/Separation (in stainless steel) module;

2.4.5 Treatment of oil-in-water emulsion

The same system used in the water flux measurements, previously shown was employed for the separation of emulsion oil/water. Oily wastewater was prepared emulsifying 0.05 g of lubricant oil (LUBRAX, SL SAE 20W/50 API SL) in

500 mL of distilled water under stirring (high-speed stirrer) for 20 min to produce stable emulsion. The membrane filtration was carried out at a pressure of 2 bar for ceramic membranes. The oil concentrations of the feed and permeate streams were analyzed. The concentration of oil present in the aqueous phase was determined by analysis of absorbance using a UV–visible spectrophotometer (Zhong 2003). The oil rejection coefficient R was calculated as a percentage according to the following expression 3.

$$\%R = \left(\frac{C_0 - C_f}{C_0} \right) x100 \qquad \text{Eq. (3)}$$

where C_0 is the oil concentration in the feed, and C_f is the oil concentration in the permeate.

2.4.6 Evaluation of reused membranes

Membranes were reused in cycles of pure water flux and oil/water emulsion separation. The membranes were flushed with distilled water in a continuous flux system for 60 min to remove residual material from the membrane. Then, they were oven dried at 60 ° C for 24 h. The materials were reused for one more cycle (60 min permeation cycle) in pure water flux and separation of oil/water emulsion.

3. Results and Discussion

Among the various applications of porous ceramic materials, porous ceramic membranes (Wegmann et al., 2008; Qi et al., 2013; Busca, 2014) are the most viable. In the preparation of porous ceramic membranes, it is important to maintain a precise control of the average pore size, mechanical resistance and permeability, reducing material processing costs.

Metastable forms of aluminum oxide called "transition alumina" are used in manufacturing ceramic membranes.

X-ray diffraction patterns of A1membrane (decomposition of aluminum sulfate) and A2 membrane (decomposition of aluminum acetate) prepared by uniaxial dry compaction method are shown in Figures 5a and 5b.

Figura 5. (a) XRD of A1 membrane and (b) XRD of A2 membrane.

(a)

(b)
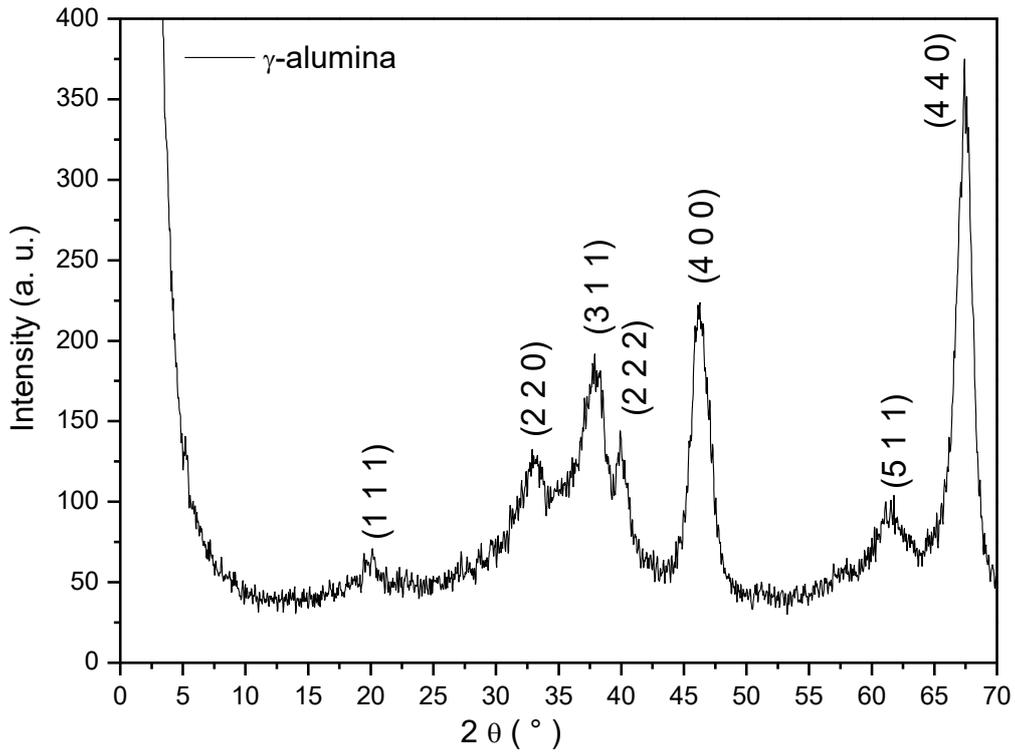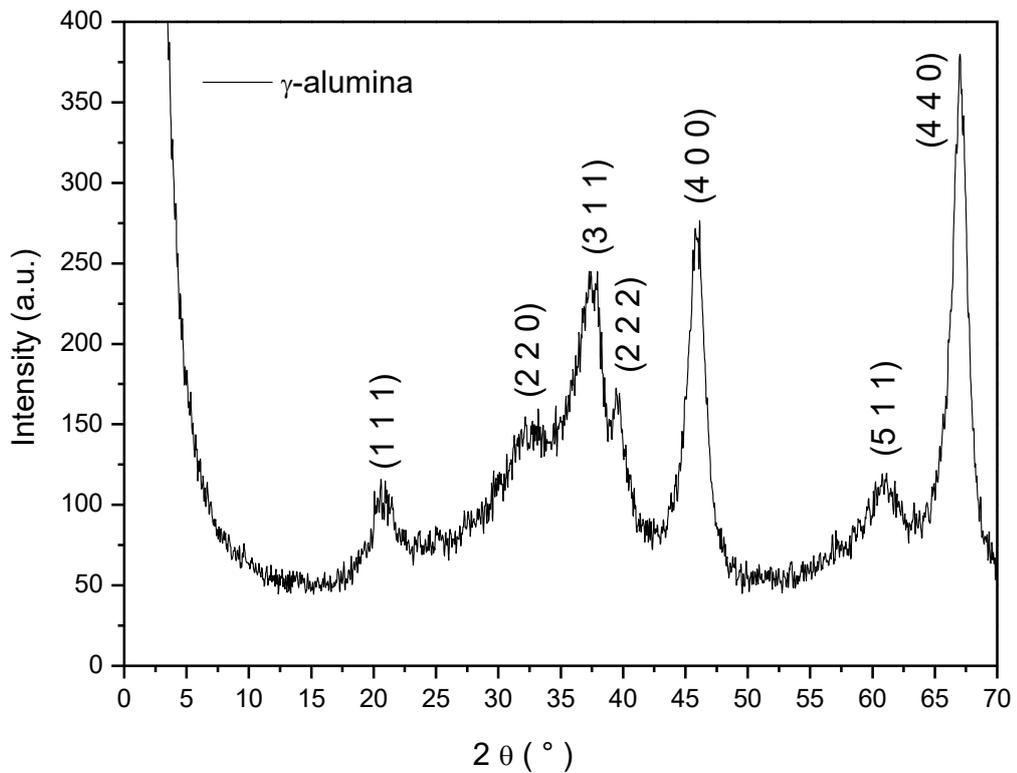
Source: Authors.

The XRD patterns (Fig. 5) indicate peaks at $2\theta = 19°$, $2\theta = 32$ - $45°$ and $2\theta = 60$ - $67°$ (JCPDS 10 - 0425). Evidencing

the formation of γ-alumina in the decompositions carried out, without the presence of contamination.

The results obtained from Scanning Electron Microscopy for the membranesA1 and A2 can be observed by means of Figures 6a and 6b.

Figura 6. Micrographs of the A1 membranes (a) and A2 membrane (b).

(a)   (b)
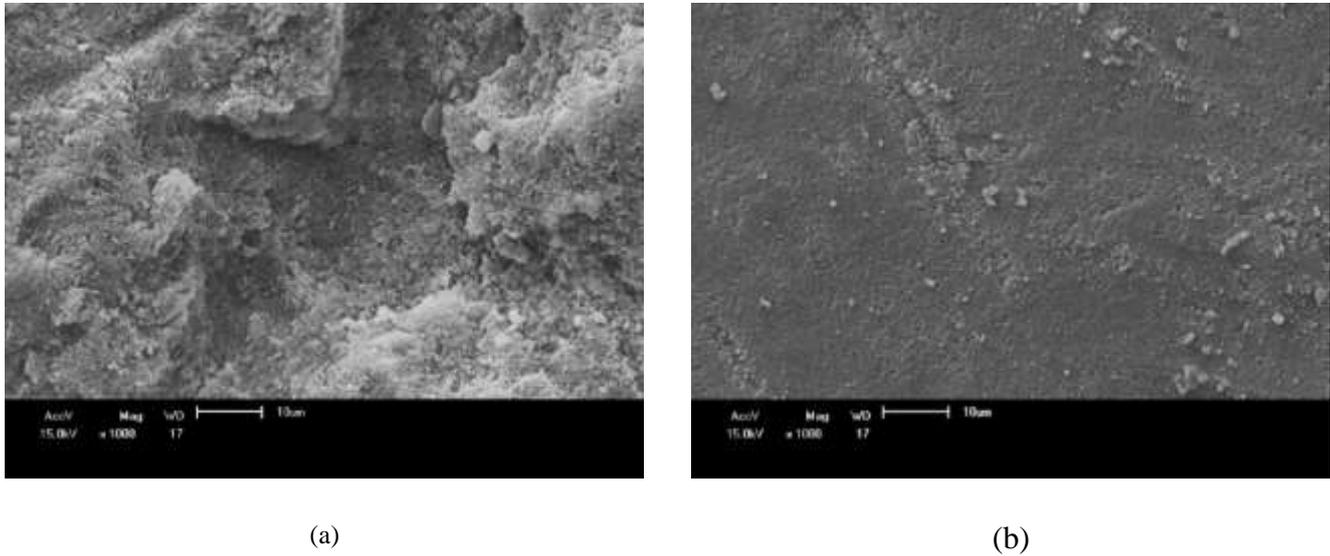
Source: Authors.

According to the micrographs shown in Figure 6, the A1 membrane and A2 membrane, it is possible to observe a homogeneous microstructure, where it was not possible to observe cracks in the surface of the membranes.

The values of average pore diameter and tensile strength of the membranes are shown in Table 1.

Table 1. Values of average pore diameter and mechanical strength of the membranes.

Membranes	Temperature sintering (ºC)	Average pore diameter (μm)	Mechanical strength (MPa)
A1	700	1.11	1.3
A2	700	0.19	6.7

Source: Authors.

According to the average diameter of the pores presented in Table 1, for the membranes, we can classify it as ultrafiltration membranes, according to (Santos et al., 2015). And because of its narrow range of pore size distribution, the carrier is likely to have high selectivity. According to the International Union of Pure and Applied Chemistry (IUPAC), the ceramic membrane can be classified as mesoporous because it has a pore diameter in the region of (2nm < dp < 50nm) (Sikdar et al., 2017).

Characterization by diametric compression test results is shown in Table 1. The tensile strength of the A1 membrane was of 1.3 MPa and 6.7 MPa for A2 membrane. It can be noted that an increase of 500 % in the mechanical strength is observed at the different natures of precursors. Because of its porosity being higher when compared to the A2 membrane, a lower resistance was obtained.

Values of the pore diameter of the ceramic membranes calculated from Eq. 1 are shown in Table 2.

Table 2. Bubble point of membranes.

Membranes	Bubble point (bar)	Dp (µm)
A1	0.75	1.11
A2	4.25	0.19

Source: Authors.

Table 2 shows that the ceramic membrane with the largest pore diameter, according to the bubble point method, was the A1 membrane, presenting 1.11 µm pore diameter. When comparing the A1 membrane with the A2 membrane, a decrease in the pore diameter is observed, considering that the A2 membrane has much smaller pores.

Water flux measurements

The pure water flux may be modified due to membrane structure and subsequently by the preparation conditions (Burggraaf, & Cot, 1996). Figure 7 presents pure water flux of the A1 membrane and A2 membrane as a function of time.

Figure 7. Water flux measurements of the A1 membrane and A2 membrane.
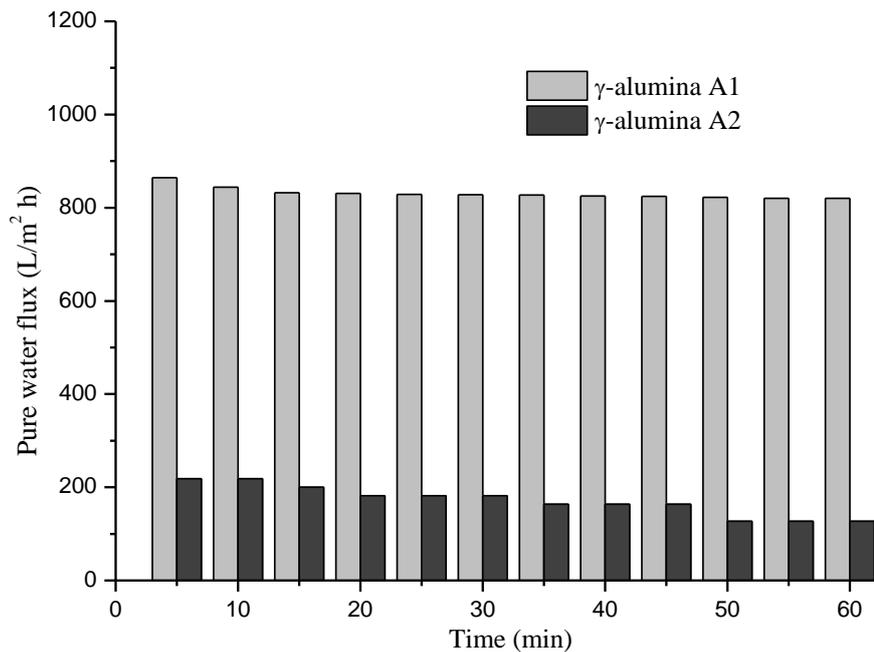
Source: Authors.

The efficiency of the water flow measurements using membranes showed that two ceramic membranes were effective. It is detected for the A1 membrane, that flow decreases slightly over time whereas for A2 membrane it was verified a more stable behavior over time. This behavior shows that the water flux of the ceramic membrane was influenced mainly for various reasons, such as pore diameter, according to results presented previously.

The characteristics of the membranes are different and were presented previously. Highlighting, the pore diameter and the mechanical strength. Therefore, it was to be expected that the results would be different. The A2 membrane showed lower

the pure water flux compared to the A1 membrane. This result can be attributed to pore diameter.

Treatment of oil-in-water emulsion

Membranes were used for the treatment of oil/water emulsions because of their antifouling properties and excellent chemical stability (Barbosa et al., 2019; Barbosa et al., 2020; Gallucci et al., 2011; Barbosa et al., 2018; Bayat et al., 2016; Suresh et al., 2016; Zhong et al., 2013).

Figure 8 presents the concentration of the permeate as a function of the time of the A1 membrane and A2 membrane and the percentage of oil rejection.

Figure 8. Removing oil water emulsion of the A1 membrane and A2 membrane over time as a function of time (a) and the percentage of oil rejection (b)
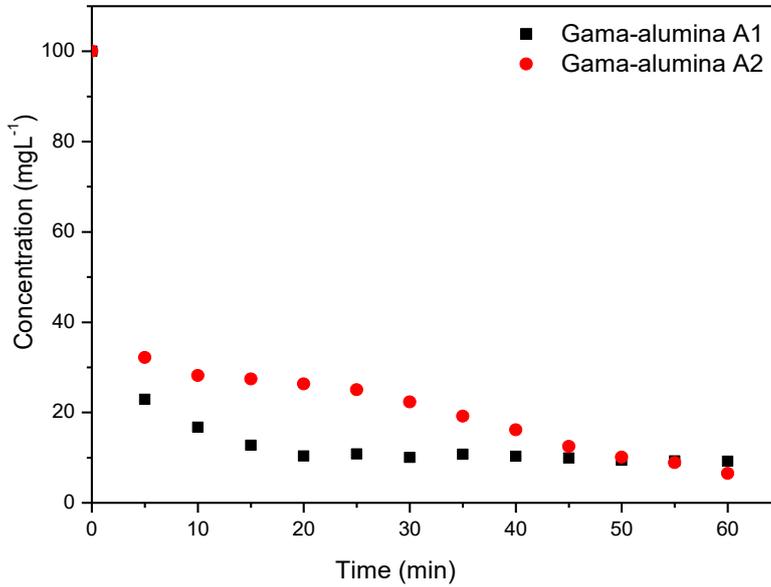
(a)
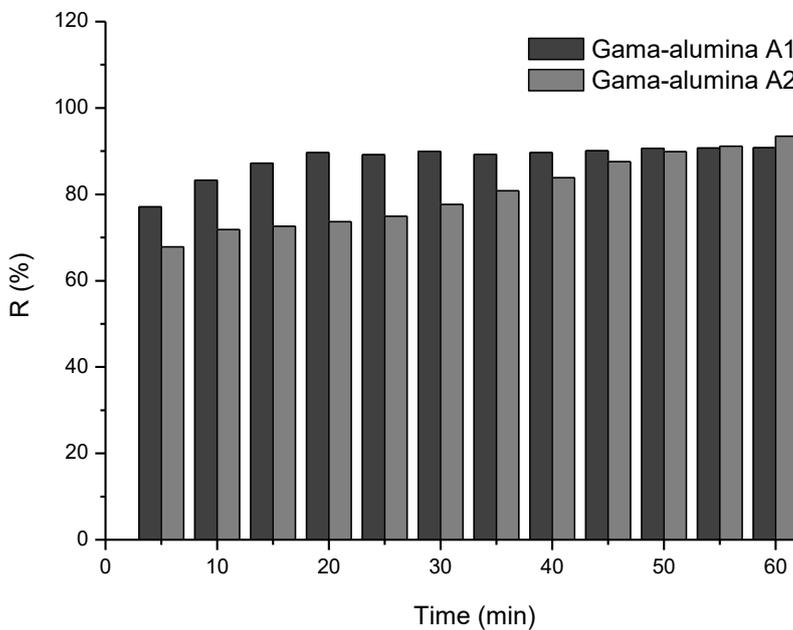
(b)

Source: Authors.

From the results presented in Figure 8 (a), it can be seen that the membranes were efficient in the water-oil emulsion removal process. A removal of 90.80 % for the A1 membrane is evident while a removal of 93.48 % for the A2 membrane. The maximum rejection of 93.48% is obtained for the A2 membrane due lowest pore size. The research findings precisely

evidence the better performance of the A2 membrane for oil rejection.

The Table 3 presents characteristics of two membranes prepared in this work and results of the process.

Table 3. Characteristics of the process feed and permeate. Experimental conditions were 2.0 bar, temperature, 27 °C, running time, 60 min

Membrane	Average pore size (μm)	Mechanical strength (MPa)	Feed (mg/L)	Permeate (mg / L)	Rejection (%)
A1	1.11	1.3	100.00	9.20	90.80
A2	0.19	6.7	100.00	6.52	93.48

Source: Authors.

It was found that the oil concentration for the A1 membrane is 9.2 mg/mL. And it was found that the oil concentration for the A2 membrane is 6.52 mg/mL. The tests performed in this work showed that oil concentration of the membranes is within the limit stipulated for oil used as reference (CONAMA, Conselho Nacional do Meio Ambiente, 2011).

Evaluation of reused membranes

The reused membranes were investigated for their performances as a function of the oil rejection capacity. Figure 9 shows the percentage values of oil rejection.

Figure 9. Removing oil water emulsion in a second cycle of the A1 membrane and A2 membrane over time as a function of time (a) and the percentage of oil rejection (b)

(a)

(b)
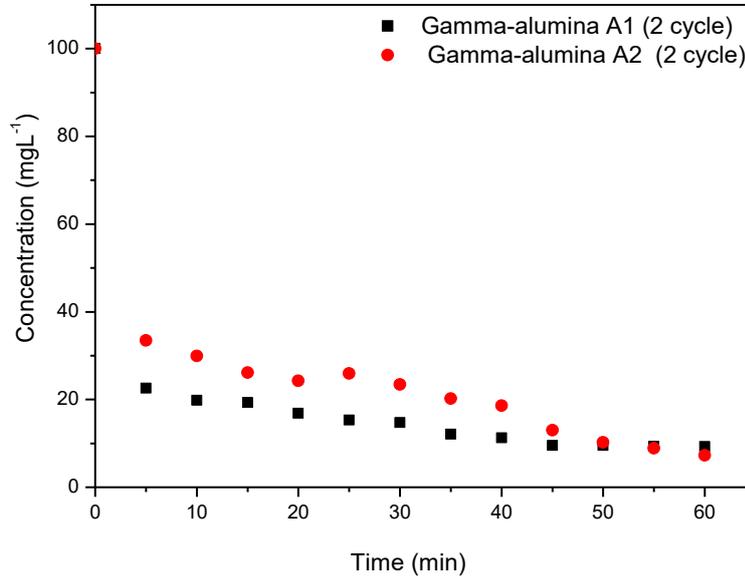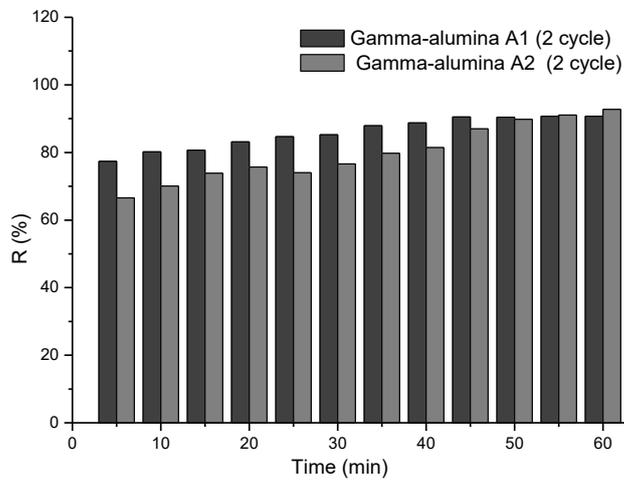
Source: Authors.

The best result presented for oil rejection for the A1 membrane and A2 membrane according to Figure 6 was 90.73 % and 92.74 %, respectively. According to the results we can observe that after the second cycle of ceramic membranes, they continue to present a satisfactory oil rejection potential.

4. Conclusion

It is possible to synthesize membranes through alternatives routes as the proposed procedure: thermal decomposition aluminum sulfate or aluminum acetate, producing high purity alumina, making the process of synthesis of these materials viable.

The values found for the permeate for the A1 membrane were 9.20 mg/L due to characteristics such as porosity and mechanical strength (44.63 % and 1.3 MPa), while the values A2 membrane was 6.52 mg/L, 18.86 % and 6.7 MPa.

The present study represents the development of suitable strategies to prepare ceramic membranes used in oil/water separation, as well as future applications in dye removal.

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