



## Preparation of alumina tubular membranes for treating sugarcane vinasse obtained in ethanol production



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### ABSTRACT

In this work asymmetric tubular membranes were successfully prepared by the co-use of the slip casting and dip-coating techniques. These membranes were used for treating a sugarcane vinasse sample obtained in ethanol production. This study is supported by a series of experimental tests, including scanning electron microscopy, mercury intrusion porosimetry, and Archimedes tests. Parameters such as color, turbidity and chemical oxygen demand (COD) of vinasse were taken into consideration for evaluating the performance of the membranes prepared herein. On one hand, it was observed that a slip-cast support prepared in this study exhibited an expressive capacity for separating the solid particles present in vinasse from water. On the other hand, the deposition of a boehmite coating on this sample led to a further increase in the membrane performance. Reductions as high as 31, 98 and 89% for COD, turbidity and color were obtained when vinasse was permeated through an asymmetric membrane prepared in this study.

### 1. Introduction

It is well established that ethanol is a promising alternative energy source for crude oil [1,2]. Together, USA and Brazil produce 85% of world's ethanol. In 2015–2016, the Brazilian ethanol production reached 30.23 billion liters [3]. The large production of ethanol in Brazil is strongly related to the development of new sugarcane varieties and agricultural technologies, coupled with a suitable weather and fertile soils [4]. However, despite the important role that ethanol obtained from sugarcane plays in today's society, the ethanol industry is also responsible for significant environmental impacts. A great part of these impacts are related to the co-production of vinasse in the distillation step of ethanol. It has been reported that for each liter of ethanol that is processed from sugarcane, about 12–15 l of vinasse are co-obtained [5,6]. Vinasse is a water-based liquid waste rich in nutrients and usually exhibits a high content of organics and solids, strong odor and dark color [7].

Among the alternatives developed over the past years for vinasse disposal, fertirrigation is the most commonly used. Nevertheless, this approach becomes detrimental when performed with no dose control or

using the same application area for extended periods of time. These incorrect practices can lead to soil acidification and groundwater contamination [8]. The vinasse transport is another important problem to be faced. Since the volume of vinasse co-obtained in ethanol production is large, its transport to distant facilities is a difficult task. In this context, the concentration of sugarcane vinasse is a promising approach for improving the use of this liquid waste. Moreover, the increase of vinasse fertilization quality by concentrating it may both reduce its transport costs and broaden its application range.

The membrane technology has achieved over the last decades a great commercial and strategic importance. The growing interest in this technology is majorly related to its relative simplicity, ease of use, low energy consumption, and application in the separation of liquid and gas mixtures [9]. The membranes can be roughly classified as organics and inorganics [10]. The inorganic membranes usually show higher thermal, mechanical and chemical stabilities when compared to the organic ones [11,12]. This improved stability of the inorganic membranes allows using these materials under operating conditions that organic membranes could not withstand [13]. An asymmetric inorganic membrane usually consists of a macroporous support with successive

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layers deposited on it. The support provides mechanical strength to prevent the membrane failure under operation. Moreover, it should also show a low resistance to the permeate flux. Alumina has been widely used as the macroporous support for preparing asymmetric membranes [14,15]. The intermediate layers bridge the gap between large and small pores observed, respectively, in the support and top layers [16].

In this study asymmetric tubular membranes were prepared for treating a sugarcane vinasse sample obtained in the ethanol production. This approach may represent an alternative method to evaporation [17,18]. Thus, the permeate stream arising from the membrane module could be recycled, whereas the retentate stream could be used in fertirrigation. The temperature in which vinasse is produced (typically between 70 and 100 °C [19]) makes the ceramic membranes particularly attractive because the organic ones do not withstand such temperatures. The asymmetric membranes obtained herein were fabricated by combining the slip casting and dip-coating techniques. As far as we know, this is the first time that inorganic membranes prepared by the combination of these two methods are used for treating sugarcane vinasse.

## 2. Materials and methods

### 2.1. Sugarcane vinasse

The vinasse sample used in this work was obtained from an ethanol refinery located at the State of Minas Gerais (Brazil), which produces ethanol from sugarcane juice. This sample was initially homogenized and stored at 4 °C. It was subsequently examined according to its color (2120C/spectrophotometric-single wavelength method), pH (4500HB/potentiometric method), electrical conductivity (2510B/conductivity laboratory method), turbidity, and chemical oxygen demand (COD) (5220D/colorimetric method) following the recommendations of APHA (American Public Health Association), AWWA (American Water Works Association), and WEF (Water Environment Federation) [20]. The vinasse color was evaluated with a Hach DR 2800 spectrometer. Its turbidity, electrical conductivity and pH were assessed using, respectively, a Hach 2100AN turbidimeter, a DDS-120W conductivimeter, and a Qualxtron QX1500 pHmeter. The concentrations of calcium, potassium, sodium, and magnesium in the vinasse sample were evaluated by atomic absorption spectroscopy with a Perkin-Elmer AAnalyst 300 apparatus.

### 2.2. Slip-cast supports

CT3000SG (99.8%/Almatis Brasil) and A2G (99.2%/Alcoa Brasil) alumina samples were used as the starting powders for preparing the slip-cast supports. Ammonium polyacrylate (PAA/Lamberti Brasil) and carboxymethylcellulose (CMC/Krosaki Harima Corporation) were used as the dispersing and binder agents, respectively. Potato starch (Aldrich) was eventually employed as the pore-forming agent. Starch stands out among the materials commonly used as sacrificial templates for preparing porous ceramics because it is environmentally friendly, inexpensive, readily available, and easy to process [21,22]. The slip-cast tubular supports were prepared as follows. Slurries of deionized MilliQ water, PAA, CMC and alumina were initially obtained at room temperature. They were subsequently mixed in a U.S. Stoneware 753RM ball mill for 1 h. The as-prepared slurries were de-aired under vacuum for 5 min and then poured into porous molds made of gypsum plaster. They were kept in the mold for 10–30 min. The excess of water was then removed from the molds by draining them at room temperature for 10 min. After removing the samples from the mold, they were kept at room temperature for 24 h, and at 60 °C for additional 24 h.

The supports were heat-treated in air at 1100–1500 °C for times ranging from 1 to 3 h. The heat treatment step was carried out in air with a Thermolab furnace (Pt30%Rh/Pt6%Rh-thermocouple) at

**Table 1**  
Conditions used for fabricating the slip-cast supports.

Sample	Composition (wt%)					Heat treatment temperature (°C)
	CT3000SG	A2G	Starch	PAA	CMC	
F1	98	–	–	1.8	0.2	1100
F2	–	98	–	1.8	0.2	1500
F3	49	49	–	1.8	0.2	1300
F4	83	–	15	1.8	0.2	1300

heating and cooling rates of 2 and 10 °C min<sup>-1</sup>, respectively. Tubular supports (500 mm long) with inner and outer diameters of about 10 and 12 mm, respectively, were prepared according to the conditions given in Table 1. The composition and heat treatment temperature for each sample was defined on the basis of a previous work where we assessed the condition that leads to samples with the best mass transport capacities [23].

### 2.3. Asymmetric membranes

Two distinct chemical routes were employed for depositing alumina films on the slip-cast supports; the first one was based on the dispersion of CT3000SG in water, whereas the second one was focused on the preparation of a boehmite sol. The CT3000SG-containing slurry was prepared as follows. 49 g of CT3000SG was dispersed in a previously prepared solution of deionized MilliQ water and PAA (0.6 g L<sup>-1</sup>). The as-obtained slurry was stirred at room temperature for 24 h, and then de-aired under vacuum for up to 4 h. The boehmite sol was obtained following a procedure similar to that reported by Yoldas [24]. Briefly, a solution of aluminum-tri-sec-butoxide (ATSB/97%/Aldrich) and deionized MilliQ water was initially prepared by keeping the ATSB: H<sub>2</sub>O molar ratio at 1: 100. The as-prepared solution was kept under stirring at 85 °C for 20 min. Next, 0.07 mols of nitric acid (NA/ > 65%/Aldrich) was dropwise added into this solution. It was subsequently kept under stirring at 85 °C for additional 24 h. Lastly, polyvinyl alcohol (PVA/Aldrich/M<sub>w</sub> = 30,000–70,000/87–90% hydrolyzed) was added into the as-obtained sol for adjusting the boehmite: PVA molar ratio to 1:1.

Both the CT3000SG-containing slurry and boehmite sol were deposited on the slip-cast supports at a withdrawal speed of 5.7 mm min<sup>-1</sup>. The immersion time of the supports in these solutions was kept at 2 min. Two distinct heat treatment programs were used for sintering the samples coated with CT3000SG and boehmite. CT3000SG-coated samples were air-dried at room temperature for 24 h and then at 110 °C for 24 h. The samples were subsequently sintered in air at 1100 °C for 1 h at heating and cooling rates of 2 and 10 °C min<sup>-1</sup>, respectively. Boehmite-coated supports were air-dried at room temperature for 24 h and then at 110 °C for 24 h. Next, they were heat-treated in air at 700 °C for 2 h at heating and cooling rates of 2 °C min<sup>-1</sup>.

### 2.4. Characterizations

X-ray powder diffraction (XRD) was performed with a Philips-Panalytical PW 1710 diffractometer using Cu K $\alpha$  radiation and a step size of 0.06°. Laser granulometry was performed in a CILAS 1064 granulometer using distilled water as the dispersing medium. Archimedes tests were performed as described in ASTM B962-13 [25]. Scanning electron microscopy (SEM) was carried out in JEOL JSM-6360LV and JEOL JSM-7500F microscopes. Mercury intrusion porosimetry (MIP) was performed in a Micromeritics Poresizer 9320 apparatus.

### 2.5. Permeation setup

Permeation tests were carried out in a setup similar to that described in details elsewhere [26]. They were performed using a

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