



## Effect of rice husk ash content on alumina or kaolin flat ceramic supports applied to carbon membrane development

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### Abstract

Carbon membranes (CM) are promising inorganic materials for gas separation processes, offering high chemical and thermal stability, along with the ability to separate gas species with similar sizes, such as CO<sub>2</sub> and CH<sub>4</sub>. In a supported configuration, CM demonstrates high mechanical strength, making it applicable to processes at high pressures. The development of supported CM with defect-free selective layers and satisfactory gas separation selectivity relies on the pore structure and roughness of the supports. Despite the high cost, commercial alumina supports are widely used for CM development [1]. In this manner, the development of supports for MC emerges as a new area to be investigated. Kaolin, a natural ore predominantly composed of kaolinite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>·2H<sub>2</sub>O), has been employed in membrane studies due to its low cost, refractory properties, and low plasticity [2]. Rice husk ash (RHA) is an agro-industrial residue containing high SiO<sub>2</sub> content [3]. Some studies report the use of RHA for modifying ceramic materials as alumina [4] or kaolin [5]. Incorporating RHA into flat alumina supports can potentially reduce material costs, while this residue may improve the physical properties of flat kaolin supports. The objective of this study was to investigate the effect of RHA in the preparation of flat ceramic supports, either alumina or kaolin-based, applied to CM preparation. Alumina was supplied by Almatis GmbH, kaolin by Minério Santa Bárbara (Brazil), and RHA from rice processing companies in Rio Grande do Sul (Brazil). Alumina powder was mixed with 0, 30, and 50 wt. % of RHA (designated as AA-0, AA-30, and AA-50, respectively), and kaolin powder was mixed with 0, 30, and 50 wt. % of RHA (designated as KA-0, KA-30, and KA-50, respectively). Flat supports were prepared using the dry pressing technique with a 6-ton load. After pressing, the supports were sintered at 1400 °C for 2 hours with a heating rate of 5 °C/min. The prepared ceramic supports were coated with a polymeric precursor solution of polyetherimide and polyethersulfone, with a ratio of 93:7 and a concentration of 18 wt. %, using the spin coating technique. After solvent evaporation, the supported polymeric membranes underwent pyrolysis at 700 °C. Gas permeation tests with pure CO<sub>2</sub> and CH<sub>4</sub> gases were conducted using the constant volume method, with the signal of pressure variation (dP/dt) at the membrane permeate side monitored through a pressure transducer connected to a data logging system.

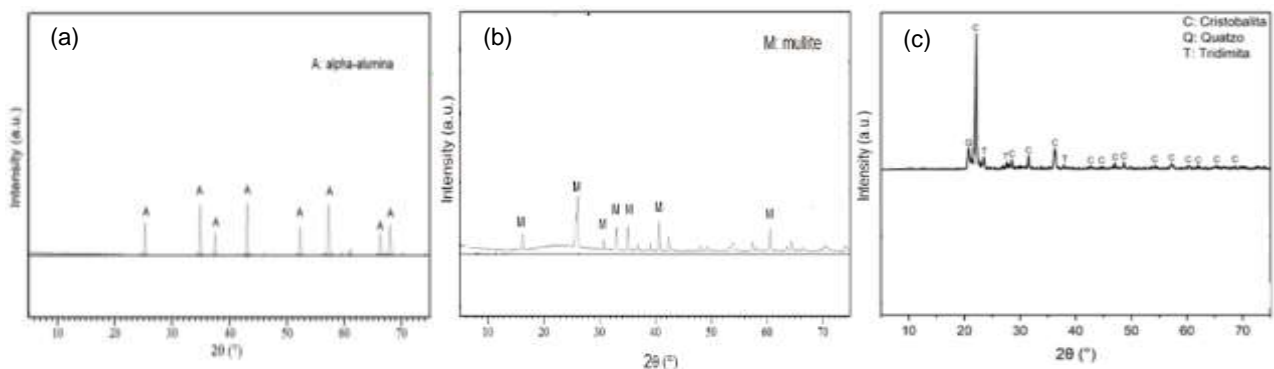


Fig. 1 – Diffractogram of raw materials: (a) alumina sintered at 1400 °C, (b) kaolin sintered at 1400 °C, and (c) rice husk ash calcined at 1200 °C

Fig. 1 presents X-ray diffractograms of raw materials. The alumina diffractogram (Fig. 1a) shows peaks at around 25,6°, 35,2°, 37,8°, 43,4°, 52,6°, 57,5°, 66,5°, and 68,2°, characteristic of alpha-alumina [6]. The kaolin diffractogram (Fig. 1b) exhibits peaks at around 16,5°, 26°, 33,5°, 35°, 37°, 40,9°, and 60,7° characteristic of mullite [7], while the rice husk ash diffractogram (Fig. 1c) shows peaks of cristobalite, quartz, and tridymite phases [8].

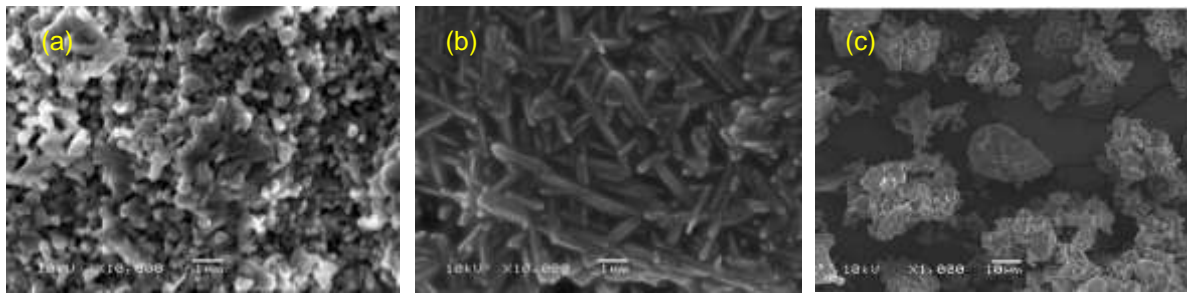


Fig. 2 – SEM micrographs of (a) alumina support, (b) kaolin support, and (c) rice husk ash powder

The SEM image in Figure 2 (a) shows the alumina support sintered at 1400 °C with densified regions resulting from particle coalescence during sintering [9]. The kaolin support sintered at 1400 °C displays needle-shaped particles characteristic of mullite [10]. Figure 3 (c) shows that RHA calcined at 1200 °C have spherical particles with some irregularities.

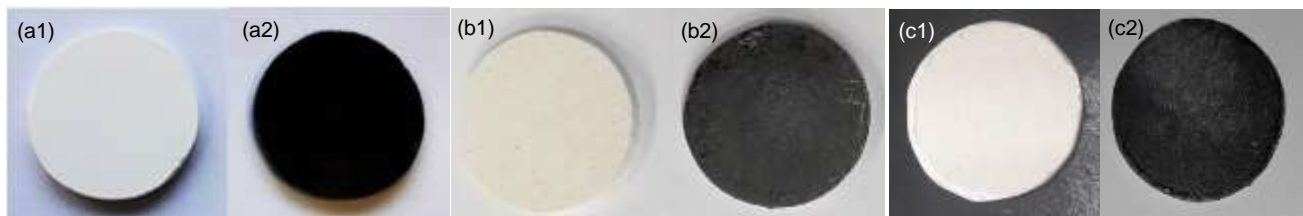


Fig. 3 – Photographs of supports obtained from (a1) alumina powder, (b1) kaolin powder, and (c1) KA-30, and CM prepared over these supports (a2), (b2), and (c2).

This work is in progress, and Fig. 3 presents some photographs of the supports and CM prepared to date. The CM could be successfully prepared on the evaluated supports. Previous work developed from our group [11] has demonstrated the potential of preparing flat alumina supports for CM development. Therefore, through the incorporation of RHA into alumina supports, it is expected to reduce the costs of these materials, while maintaining their good properties. On the other hand, achieving CM with good separation properties on kaolin supports has been challenging, due to carbon layer intrusion into large pores in the kaolin ceramic structure. Therefore, the addition of RHA to kaolin supports aims to promote greater structural uniformity by modifying the porous structure of these kaolin supports [5]. The CM produced on alumina or kaolin supports with varying RHA contents will be evaluated in gas permeation tests for CO<sub>2</sub> and CH<sub>4</sub> separation.

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