

CONVERSION OF RICE HUSK ASH INTO ZEOLITIC MATERIALS

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Abstract— The objective of this work was to synthesize zeolitic products using rice husk ash (RHA) as silicon source. For the synthesis, two mineralizing agents: NaOH and KOH were tested. Chemical composition of the synthesized samples as well as their cation exchange capacity (CEC) were determined. The samples were also characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Formation of structure LTA (zeolite A) with NaOH as mineralizing agent was verified. The zeolitic materials presented high capacity of cation exchange, in the range of 5-6 meq/g.

Keywords— rice husk ash, zeolite, synthesis

I. INTRODUCTION

Due to high cation exchange capacity (CEC), as well as small size particles, type A zeolite is widely used in water treatment, in particular for incorporation into the composition of detergents for the sequestration of calcium from wash water (Strack, 1981, Allen *et al.*, 1983). However, to make available this application, the cost of the zeolite must be low, demanding the utilization of cheap raw materials for the production process. In the state of Rio Grande do Sul (Brazil), the rice production is about 5,137 million t per year. Since the husks represent 20% of this value, the annual production of this residue in the state is approximately 1.027.400 t. These husks are burnt for energy production, generating great amounts of ashes ($\approx 200,000$ t per year). As the RHA presents > 90% by weight in silicon, its use as silicon source in the synthesis of zeolite A constitutes an alternative for reduction of costs. The RHA can be used as adsorbent in the process of gold extraction, in the production of silicon carbides (SiC), as load in polymers, as additive for cement, in the manufacture of concrete, and as support for preparation of nickel-based catalysts (Foletto *et al.*, 2005). The ashes have been also used for zeolite synthesis: ZSM-5 (Rawtani *et al.*, 1989, Kumar and Das, 1992, Chareonpanich *et al.*, 2004), mordenite (Bajpai *et al.*, 1981), ZSM-48 (Wang *et al.*, 1998), NaX (Hamilton *et al.*, 1993, Dalai *et al.*, 1985), beta (Praseyoko *et al.*, 2005), and omega (Fajula *et al.*, 1987).

This work had as objective to synthesize zeolitic materials using rice husk ash as silica source. Two different alkalis were used and their influence on the zeolite formation was examined.

II. MATERIALS AND METHODS

A. Composition of the Reaction Mixture

For the formation of the zeolite in alkaline pH, the molar composition proposal by Thompson and Huber (1982) was taken as reference: $1\text{Al}_2\text{O}_3:2.1\text{SiO}_2:3.9\text{M}_2\text{O}.128\text{H}_2\text{O}$, where M represents sodium or potassium. The Si/Al ratio used in the reaction medium was 1.05. As silicon source, rice husk ash generated from an industrial burner of a local industry (INDUBER, Santa Maria, RS, Brazil) was used. As alkali source, NaOH and KOH were used. As aluminum source, sodium or potassium aluminates were used. They were prepared by dissolution of 54 g aluminum wire (1mm diam., 99.99%, Aldrich) in 2615 mL of sodium hydroxide solution (12% by weight) or in 2584 mL of potassium hydroxide solution (11% by weight), using a heating system with reflux. The time of reflux was 30 minutes.

B. Crystallization

The preparation of the reaction mixture was carried out in accordance with the following procedure: 2.07 g of ashes were conditioned in stainless steel autoclaves, provided internally with Teflon vessels of 40 cm³. 40 g of sodium or potassium aluminate solution was added on this ash; the autoclaves were closed and introduced into a pre-heated oven at the temperature of crystallization (100 °C). The crystallization was performed for different periods: 3, 6 and 12 h. The formed solid was separated from the supernatant liquid by filtering and was washed with deionized water to remove the excess of alkali. Later, it was dried at 110 °C for 10 h. The samples are designated as N3, N6, N12, K3, K6, K12 (N and K represent the used mineralizing agents, NaOH and KOH, whereas e 3, 6 and 12, indicate the period used for the reaction process).

C. Samples Characterization

The RHA and the samples of aluminosilicates synthesized were characterized by X-ray diffraction (Shimadzu diffractometer, model XD-7A, with radiation Cu-K α) and by scanning electron micrograph (SEM; Model 2000FX, JEOL Co.). The chemical composition was determined by atomic absorption spectrometry (Analytik JENA, Vario 6, Germany). For determination of the cation exchange capacity, 0.5g sample was placed in contact with NH₄⁺ excess (by the use of ammonium acetate), washed with ethanol and later calcinated for release of ammonia. The set free ammonia was collected in water and this solution was then titrated with H₂SO₄, expressing the values in meq/g of sample.