SYNTHESIS AND CHARACTERIZATION OF \(\alpha\)-ALUMINA BY INORGANIC SOL-GEL METHOD

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The aim of this work was the synthesis of nano alumina by an inorganic sol-gel method seeking for the greatest yielding and ease of operation. A \(2^k\) design of experiments was used. The factors were the concentration of the reactants and the order of addition, resulting in twenty reactions. The synthesis was performed in a three-input reactor with heating and the pH was measured along the reaction. Aluminum chloride, aluminum nitrate and sodium hydroxide were the reagents. After synthesis, the aluminum hydroxide solutions were centrifuged and washed to avoid contamination of the samples. The aluminum hydroxide powder were then calcined at 1200 °C for 1 h to obtain the desired nano alumina phase. The calcination temperature was determined by DSC/TG analysis. The alumina powders were characterized by XRD, DLS, zeta potential, FTIR, FESEM and BET techniques, which proved the formation of nano alumina in the desired \(\alpha\)-alumina phase. Salt contamination was observed, indicating the need to improve the washing process. In order to identify the contamination, atomic absorption analysis was performed, showing that the average contamination was below 8 mass %. The use of inorganic precursors as precipitating agents is not common to obtain nano alumina. However, this study shows that inorganic precursors can be used for the synthesis of nano-oxides by the sol-gel route. As a result, the synthesis using 12 M NaOH and the combination of aluminum chloride and aluminum nitrate drip simultaneously yielded 89% Al(OH)\(_3\) and 58% Al\(_2\)O\(_3\). The thermal analysis for this synthesis showed the formation of intermediate salts and their decomposition resulted in \(\alpha\)-alumina phase, identified by XRD and FTIR, with 88% degree of crystallinity. The DLS analysis for showed a hydrodynamic diameter of 33 nm, before calcination, in a very stable solution with a zeta potential of 43 mV, which would facilitate its synthesis on an industrial scale. After calcination, the FESEM images show agglomerated nanometric particles, resulting in a powder with a surface area of 5.4 m\(^2\)/g (BET), which shows the possibility of reducing the calcination temperature or time.

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