

**STUDIES ON ACTIVATED MESOPOROUS ALUMINA DERIVED
FROM SYNTHESIZED ALUMINIUM ISOPROPOXIDE
FOR REMOVAL OF ARSENIC**

Ph.D. DISSERTATION

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ABSTRACT

Highly activated mesoporous alumina possessing a high removal efficiency (ca. 100 %) for arsenic has been prepared via synthesized aluminium isopropoxide. Aluminium isopropoxide with the percent yield of 88 % was synthesized by using aluminium shavings (mesh sieve: 50-80 mesh) and pure isopropyl alcohol. The synthesis was achieved using mercury (II) chloride (0.5 mol %) as a catalyst under nitrogen atmosphere at 95 ± 2 °C. The optimum refluxing time was about 7 hours.

Characterization of the synthesized aluminium isopropoxide by FT - IR, ¹HNMR and XRD analyses showed it to be tetrameric aluminium isopropoxide. This solid alkoxide serves as a molecular precursor for the preparation of alumina gels.

Two different types of alumina gels were derived from solid tetrameric aluminium isopropoxide by sol - gel hydrolysis. An opaque alumina gel (11%) was obtained when the alkoxide was subjected under a heterogeneous condition using 95% (w/w) isopropyl alcohol - water system at ambient temperature. A transparent alumina gel (33%) was achieved under a homogeneous condition using 80% (w/w) benzene-water system at ambient temperature.

Characterization by XRD and SEM indicated that the transparent alumina gel was more of the crystalline type ($2\theta = 10$ to 80). The transparent alumina gel was more porous in its morphology texture showing a mixture of boehmite and bayerite, whereas the opaque alumina gel was more amorphous ($2\theta = 10$ to 60) consisting mainly of hydrated alumina. Both alumina gels when subjected to selective heat

treatment (200,400, 600, 800, 1000 and 1200) °C gave rise to different forms of transition aluminas. The δ - Al_2O_3 derived from the transparent alumina gel heated at 800 °C was found to show high sorption property with respect to iodine, compared to other transition aluminas. The mesoporous nature of δ - Al_2O_3 was inferred from Raman spectrum (200-380 cm^{-1}) and from SEM photomicrograph (2-50 nm) and also from the speciation of iodine removal capacity.

Sorption studies for the removal of arsenic using the dosage and concentration methods showed that the δ - Al_2O_3 possessed the highest sorption capacity even when compared to a commercial alumina. Maximum sorption capacity of arsenic has been achieved at pH 5.5. It was found that δ - Al_2O_3 , sorption capacity for arsenic conformed the Freundlich and Langmuir isotherms favourable linear relationship with correlation factors > 0.9800 were found. The Freundlich constant (1/n) was about 0.9 and the Langmuir monolayer parameter (X_m) was 188.7 mg g^{-1} . The efficiency and effectiveness of δ - Al_2O_3 for arsenic removal was evaluated by using a model As (V) solution where 92-93% was removed at the break point of a fixed bed column unit in 15 min as regard to a feed in of 1000 ppm at the flow rate of 40 mL hr^{-1} .

A down flow fixed bed δ - Al_2O_3 packed column showed that arsenic contaminated rural drinking water feed (98 ± 2 ppb) was effectively and efficiently removed by about 100%. It indicated that the prepared δ - Al_2O_3 with meso porosity possessed the speciation property for arsenic removal.

Keywords : *tetrameric aluminium isopropoxide, opaque like alumina gel, transparent alumina gel, arsenic removal efficiency ca. 100 %, activated mesoporous δ - Al_2O_3*