

C
540
CHIA

**SYNTHESIS, PROPERTIES AND APPLICATIONS OF
NANO GIBBSITE CRYSTALS:
A SUBSTRATE FOR ARSENATE REMEDIATION**

A THESIS PRESENTED BY

I. G. CHANAKA KAPILA KUMARA
~

to the Board of Study in Chemical Sciences of the
POST GRADUATE INSTITUTE OF SCIENCE

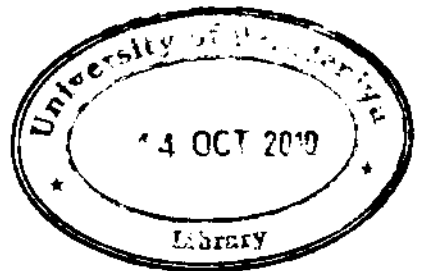
*in partial fulfillment of the requirement
for the award of the degree of*

MASTER OF PHILOSOPHY

of the

**UNIVERSITY OF PERADENIYA
SRI LANKA
2009**

635241



SYNTHESIS, PROPERTIES AND APPLICATIONS OF NANO GIBBSITE CRYSTALS: A SUBSTRATE FOR ARSENATE REMEDIATION

I. G. Chanaka Kapila Kumara

Institute of Fundamental Studies

Hanthana Road

Kandy

Sri Lanka

Gibbsite (α -Al(OH)₃, monoclinic, P21/n) structure consists of double layers of close packed OH groups. The primary aim of the present research is the synthesis and characterization of nano gibbsite crystals. Subsequently its potential use for arsenate removal from drinking water is examined. Nano gibbsite crystals were synthesized by the titration between AlCl₃ and NaOH under suitable experimental conditions. The particles were characterized by TEM, EDS, XRD and FTIR spectroscopic methods. The initial product of Al(OH)₃ is amorphous and as dialysis proceeds, it gradually transformed into crystalline structure. A 10 days of dialysis are sufficient for initiation of gibbsite crystal formation. Synthesized gibbsite crystals were hexagonal in shape and around 90 ± 25 nm in diameter. XRD and FTIR further reveal the formation of gibbsite crystals.

Synthesized Al(OH)₃ particles at pH 9 shows somewhat a different IR spectrum. The OH stretching and bending vibrations correspond to bayerite. Thus, pH plays a critical role in the selection of aluminum hydroxide morphology. Gibbsite surface bears positive charge below pH 9.3. The DTA and TGA experiments indicate formation of new phase upon dehydroxylation of gibbsite. FTIR spectroscopy data coupled with XRD analysis suggests that dehydration sequence of nano gibbsite was via chi-alumina.

Arsenate adsorption on gibbsite nano particles is pH dependent and sorption favor at acidic medium. Affinity of gibbsite for arsenate at pH 5 is nearly 4 fold greater than that of at pH 9. Monolayer coverage of arsenate on gibbsite nano crystals surface at pH 5 found to be $3.12 \times 10^{-6} \text{ mol m}^{-2}$ while that of in pH 9 to be $2.36 \times 10^{-6} \text{ mol m}^{-2}$. Model

calculations imply significant lowering of active surface sites for arsenate adsorption as pH increases. A 98% of arsenate removal observed at the pH range 4 to 8.5. Arsenate exhibit negligible changes with ionic strength variation indicating possible inner-sphere surface complex mechanism. Adsorption process causes a significant change in the OH stretching modes of gibbsite. Molecular modeling calculations using DFT, predict the bond lengths of $As-Al$, $Al-O$, $As-O$ to be 3.15 Å, 1.90 Å, 1.74 Å and bond angles of $O-As-O$, $Al-O-As$ to be 106° and 122° respectively for possible binuclear-bidentate surface complexation.