

*Surfactant mediated synthesis and characterization of
Boehmite nanowires*

A Dissertation

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MASTER OF SCIENCE IN CHEMISTRY

Under The Academic Autonomy

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

By

HARIKRISHNA SAHU

Under the supervision of

Dr. G. Hota



DEPARTMENT OF CHEMISTRY

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

ROURKELA-769008

Guide Certificate

Dr. G. Hota

Department of Chemistry,

National Institute of Technology,

Rourkela - 769008



CERTIFICATE

This is to satisfy that the thesis entitled “**Surfactant mediated synthesis and characterization of Boehmite nanowires**” being submitted by Mr Harikrishna Sahu (Roll No. – 409CY2027) for the partial fulfillment of the requirements for the award of M.Sc. degree in Chemistry at the National Institute of Technology, Rourkela, is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University or Institute for the award of a degree or diploma.

Date:

Place:

Signature of the guide:

Dr.Garudadhvaj Hota

DECLARATION

I, Harikrishna sahu hereby declare that this project report entitled “**Surfactant mediated synthesis and characterization of Boehmite nanowires**” is the original work carried out by me under supervision of Dr. G. Hota, Department of chemistry, National Institute of Technology Rourkela (NITR), Rourkela and the present work or any other part thereof has not been presented to any other University or Institution for the award of any other degree regarding to my belief.

May 5, 2011

Harikrishna sahu

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ABSTRACT

The Boehmite (AlOOH) nanowires have been prepared by surfactant mediated sol-gel synthetic technique. The resulting nanowires have been characterized by using the XRD and FE-SEM and SEM-EDAX analytical techniques. From the X-ray diffraction (XRD) analysis, it is clearly observed that the nanowire synthesized by sol-gel method has γ - AlOOH crystalline phase. The FE-SEM analysis shows the formation of wire shaped material having average diameter in the range of 200 nm to 1 micron and length up to several hundred microns. The SEM-EDAX (Element detection X-ray analysis) result suggests the presence of Al & O elements in boehmite nanowires. In order to obtain the mechanism of formation of nanowires, the effect of various reaction parameters such as pH, reactant and surfactant concentration, drying of gel etc. have been studied here. It is observed that the optimum pH ranges for the formation of nanowires is 7 to 9. The anionic surfactant SDS is more suitable surfactant than other anionic surfactants (Na-PSS, DBSA, Na-PAA) and act as a directing agent in the formation of nanowires. Surfactant concentration has no role but the aluminum nitrate concentration plays very important role in the formation of boehmite nanowires. As the aluminum concentration increases the amount of nanowires (%) formation increases. It was also found that drying of gel-cake plays an important role in the conversion $\text{Al}(\text{OH})_3$ gel to nanowires.

INTRODUCTION

In past few decades, synthesis of nanomaterials with novel properties have attracted a great deal of attentions to materials community [1,2]. This is due to their intrinsic properties, which can be determined by their composition, structure, crystallinity, size and morphology [3]. Among various type materials, nano-structured alumina is an important ceramic material and used in a wide range of application such as catalysis, adsorbent, fabrication of catalyst, templates, and nanocapacitors [4]. Apart from this alumina materials exhibits many novel properties, such as high elastic modulus, low density, high strength and toughness, thermal and chemical stability, and optical characteristics.

The $\gamma - \text{Al}_2\text{O}_3$ can be obtained by heating of boehmite ($\gamma\text{-AlOOH}$) at a temperature range 400°C to 700°C . At this temperature $\gamma\text{-AlOOH}$ release one water molecule to form $\gamma\text{-Al}_2\text{O}_3$. The morphology of boehmite does not change during transformation. [5, 6]

Many research groups have been synthesized and reported different morphologies of nanostructures boehmite such as nanofibers [7,8], nanobelts[9], nanotubes [10,11], nanoparticles [12], aligned nanowires [13], and flower like three-dimensional nanoarchitecture[14]. Among them, one dimensional (1D) nanofibers, nanowires and nanotubes have attracted much attention due to their unique electronic, mechanical and chemical properties and potential application in various areas [15].

One dimensional AlOOH nanowires have been synthesized previously e.g. boehmite nanofiber and nanotubes were synthesized with the assistance of poly (ethylene oxide) (PEO) surfactant [16] and boehmite nanorods with length of 50-200 nm and diameter of 6-20 nm was fabricated via hydrothermal method using sulfate solution [17]. Shen et al. have reported a steam-assisted solid-phase wet-gel conversion process for synthesis of boehmite nanorods [18].

However, it is desired to synthesize highly pure 1-D boehmite nanomaterials in an effective way i.e. in terms of low cost and large-scale production is still a technical challenge. In this report we have synthesized 1 D boehmite nanowires by a surfactant mediated sol-gel synthetic technique. The effect of pH, reactant concentration, nature of surfactant molecules and drying condition on the formation of nanowires has been studied extensively.

EXPERIMENTAL SECTION

MATERIALS

We have used the following chemicals without further purification for synthesis of boehmite nanowires.

- Aluminum nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), (purchased from S.d, fine chemical Ltd. India.)
- Potassium hydroxide (KOH), purchased from Merck Chemical Ltd. India
- Sodium dodecyl sulfate (SDS), obtained from Merck Chemical Ltd. India

Cleaned and dried glass wares and double distilled water have been used throughout the experiments.

METHODS

18.75 g of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was dissolved in 10 mL double distilled water. Then 0.05 g of SDS was added and stirred for getting a clear solution. 1M KOH solution was added drop wise into $\text{Al}(\text{NO}_3)_3$ solution at a constant rate of 5 mL/min under constant stirring. KOH solution was ceased to be added when the pH value of the reaction mixture reached P^{H} 9. The obtained white gel was heated at 100°C in water bath for 3 hours. After that the solution was centrifuged and washed thoroughly and the gel-cake was dried at 60°C for 5 hours.

RESULT AND DISCUSION

A. XRD Analysis:

The formation and phase evolution of boehmite nanowires prepared by sol-gel method were studied by XRD analysis using the $\text{Cu K}\alpha$ ($\lambda=1.54059\text{\AA}$) in a Philip's X-pert PRO high-resolution X-ray diffractometer (XRD) spectra is shown in figure 4.1. This figure shows well defined diffraction peaks at 2θ values of 29.3, 38.9, 48.66 and 66.12 degrees corresponding to the reflections from the (0 4 0), (1 1 1), (2 0 0), (0 0 2) planes respectively. These Peaks are characteristics of the γ -boehmite phase according to (JCPDS FILE NO.-72-0359). However the large peaks ($2\theta= 31.5, 45.3, 56.4$) are due to presence impurity of highly crystalline potassium nitrate present in boehmite nanowires.

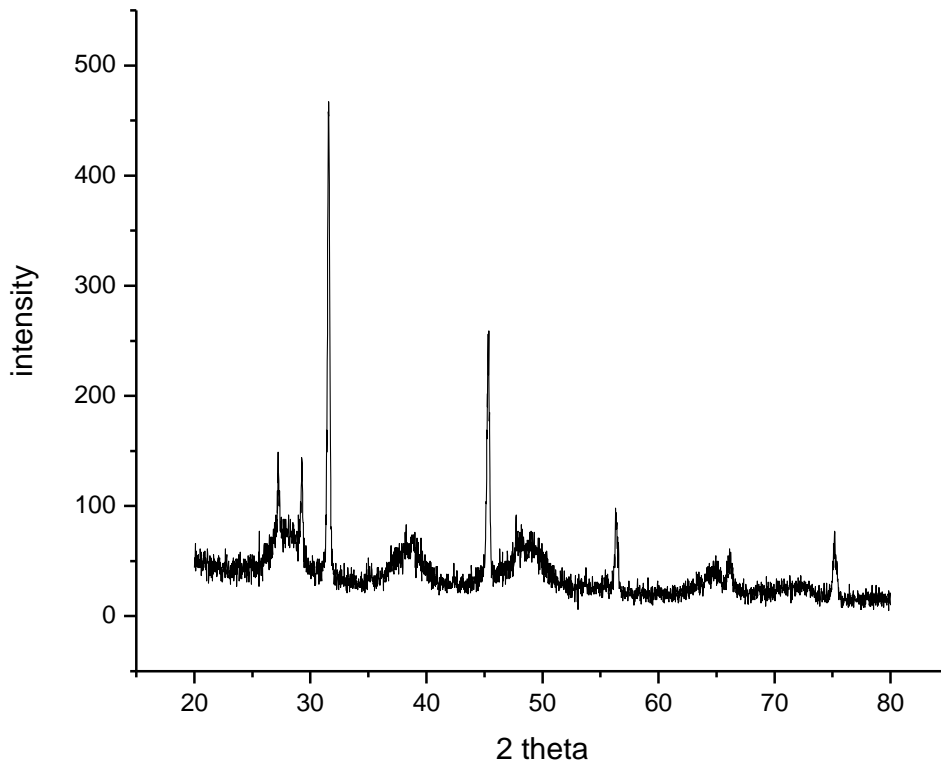


Fig-4.1 XRD pattern of boehmite nanowires

B. SEM Analysis:

The morphology and microstructure of the boehmite nanowires prepared in different reaction conditions were investigated by SEM micrographs. Figure 4.2 represents the SEM images of boehmite nanowires. The SEM images suggest that the morphology of the boehmite sample is wire shaped. And they have average diameter 200 nm to 1 micrometer and length several hundred microns. The EDAX analysis Fig-4.3 of the boehmite sample suggests that the Al and O are present.

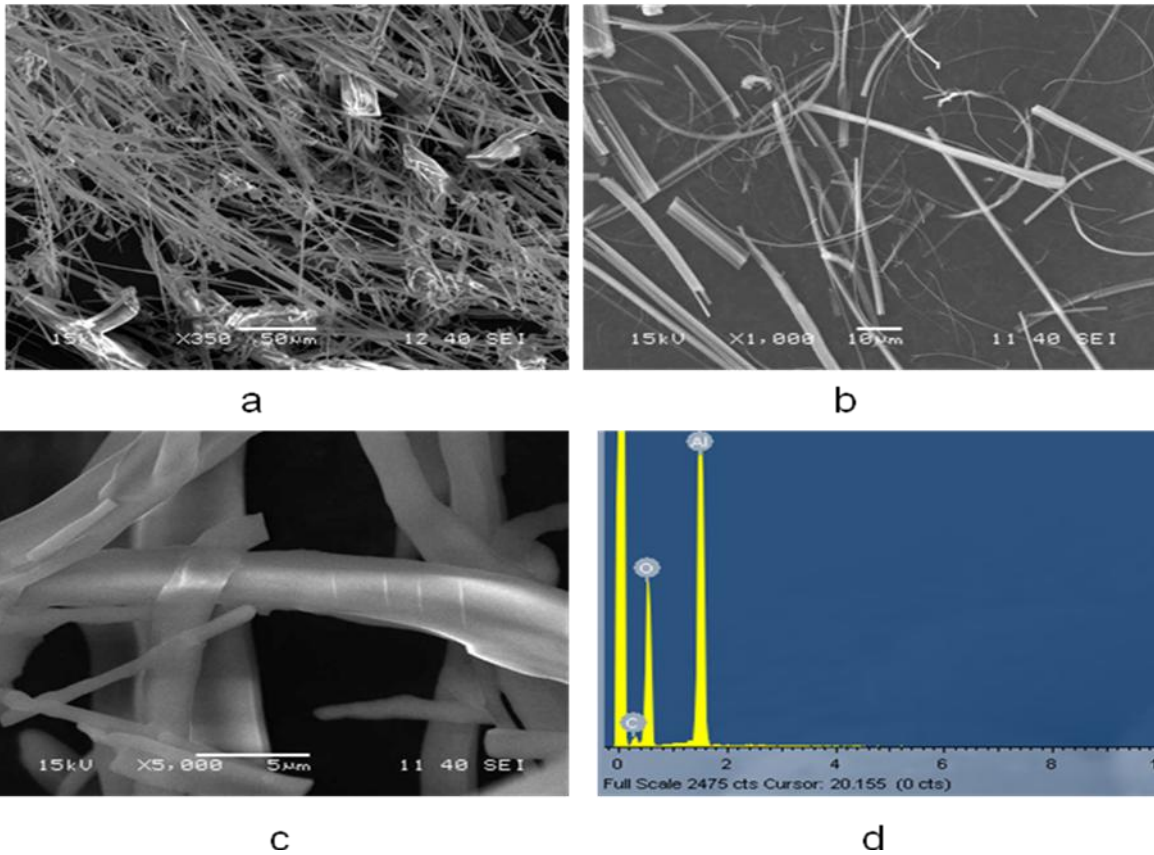


Fig 4.2. SEM micrographs of boehmite nanowires, 5 M $\text{Al}(\text{NO}_3)_3$ (a) Low-magnification (b) Medium-magnification (c) High-magnification (d) SEM-EDAX spectra

CONCLUSION

The studies as part of this project have established the precise protocol for the synthesis of Boehmite nanofibres.

- ❖ We have successfully prepared AlOOH nanowire materials using surfactant mediated Sol-gel synthesis method.
- ❖ XRD studies confirmed the formation of AlOOH nanowire.
- ❖ SEM micrographs suggest the boehmite are wire in shape having average diameter 200 nm to 1 micrometer and length several hundred microns.
- ❖ This methodology depends upon the use of surfactants as directing agents.
- ❖ To be best of my knowledge, this is the most simple method for preparation of boehmite nanowire.

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