Synthesis and Characterization of Alumina/Iron Oxide Mixed Nanocomposite

Project Report

Submitted in partial fulfillment of the requirements for the

award of

MASTER OF SCIENCE IN CHEMISTRY

Under The Academic Autonomy

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

By

BIBHU CHANDAN PATTANAYAK

Under the supervision of

Dr. G. Hota

DEPARTMENT OF CHEMISTRY

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

ROURKELA-769008
CERTIFICATE

This is to certify that the project report entitled "Synthesis and Characterization of Alumina/Iron Oxide Mixed Nanocomposite” being submitted by Mr. Bibhu chandan Pattanayak(408CY111) is a bonafide work done by him in my laboratory under my supervision.

N.I.T Rourkela  
05 May’10

Supervisor  
Dr. Garudadhwaj Hota
DECLARATION

I, Bibhu chandan Pattanayak hereby declare that this project report entitled “Synthesis and Characterization of Alumina/Iron Oxide Mixed Nanocomposite” is the original work carried out by me under the 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, 2010

Bibhu chandan Pattanayak
ACKNOWLEDGEMENT

On owe my deepest sense of gratitude, I would like to give a reverence honor and heartfelt thanks to Dr G. Hota, Department of Chemistry, NIT Rourkela, whose valuable attention, inspiration, and all above his guidience show me a bright way to this Nanoscience throughout my project.

I would like to give a grateful thanks to Ms. Abhipsa Mahapatra with full of affection for her constant support helps me to fly up my project work in any situation.

I would like to give a hearty thanks to my lab met Ms. Shanti, Ms. Soumya and to Mr. Dayanidhi for their inspirative support throughout my project work.

A many sincere thanks to all of my faculties, Department of Chemistry, NIT Rourkela whose encouragement, inspiration, guidance helps me to build up a initial construction towards research.

Finally all credits goes to my parents and my friends for their continued support.

And to all mighty, who made all these things possible.........
ABSTRACT

The Aluminium & Iron oxides mixed nanocomposites have been prepared by hydrothermal as well as combustion synthesis process. The resulting composite has been characterized by using the XRD, FE-SEM, FT-IR, and BET analytical techniques. From X–ray diffraction (XRD) analysis, it is clearly observed that the composite synthesized by hydrothermal method has \( \alpha{-}\text{Fe}_2\text{O}_3 \) and \( \gamma{-}\text{AlO(OH)} \) crystalline phase where as \( \alpha{-}\text{Fe}_2\text{O}_3 \) and \( \alpha{-}\text{Al}_2\text{O}_3 \) phase has been found in combustion synthetic method. The FE-SEM analysis indicates that the formation of needle shaped particles for hydrothermal synthesis where as the porous & flake like morphology is found in case of combustion synthesis. The SEM-EDAX (Elemental detection x-ray analysis) results suggest the presence of Al, Fe & O elements in the alumina/iron oxide mixed composites. The FT-IR analysis indicates the formation of \( \alpha{-}\text{Fe}_2\text{O}_3 \) and \( \gamma{-}\text{AlO(OH)} \) in hydrothermal synthesis. The BET study suggests that the mixed composites synthesized by hydrothermal method are nonporous in nature and the surface area is found to be 35 \( \text{m}^2/\text{gm} \). The results of combustion synthesis illustrate that the fuel to oxidizer ratio is the most effective factor for the formation & surface morphology of mixed nanocomposite.
INTRODUCTION

Nanocomposite material has broadened significantly to encompass a large variety of systems such as one-dimensional, two-dimensional, three-dimensional and amorphous materials, made of distinctly dissimilar components and mixed at the nanometer scale less than 100 nm. The general class of nanocomposite materials such as organic/organic, organic/inorganic & inorganic/inorganic is a fast growing area of research. Recently, significant research effort has been taken on the ability to obtain controlled nano-sized composite materials via innovative synthetic approaches. The properties that strengthen the general behavior of nanocomposite towards their substantial improvement include: mechanical properties, dimensional stability, decreased permeability to gases, chemical resistance, surface appearance etc. Aluminium oxide (alumina, Al₂O₃) is one of the most useful oxide ceramics, as it has been used in many fields of engineering such as coatings, heat-resistant materials and advanced ceramics. This is because alumina is hard, highly resistant towards bases and acids, allows very high temperature applications and has excellent wear resistance. Apart from this alumina is also used in heterogeneous catalysis because it is thermally stable and allows the dispersion of active phases due to its high specific surface area. Similarly, in recent years, Fe₂O₃ nanomaterials have been studied, including their shape-specific properties, synthesis methods, and potential applications in catalysis, adsorption and field emission etc. There are different synthetic methods to prepare nanostructured materials, however in the present study we have on hydrothermal and combustion synthesis to prepare alumina / iron oxide nanocomposite materials. In case of hydrothermal method nanomaterials can be prepared by creating a high pressure at elevated temperature (> 100 °C) and pressure (> a few atmosphere) in presence of water solvent. The apparatus used in such method is a Teflon lined pressure pot so called autoclave. Advantages of the hydrothermal method include the ability to create crystalline phases which are not stable at the melting point. Likewise, Combustion synthesis is characterised by highly exothermic reaction with temperature ranging from 400-600 °C. Due to strong heating, a pallet of ignited material will exhibit combustion waves which propagate through the material in self-sustained manner. Hence it is called as self-propagation because the process no longer requires the input energy. The most important advantages of this process are: low energy requirements, time saving and environmentally friendly (the combustion reaction by-products are N₂, CO₂ and H₂O).
OBJECTIVE OF THE STUDY

- To synthesize the nano-sized Iron oxides and alumina-Iron oxide mixed composite using hydrothermal and combustion synthetic method
- To study the feasibility, formation and structure of mixed oxide by varying the weight percentage of aluminium and iron salt precursors.
- To characterize the formation of alumina-iron mixed oxides such as their structure, surface morphology, particle size, surface area etc by using experimental techniques like XRD, SEM, TEM, BET etc

EXPERIMENTAL SECTION

We have used the combustion synthesis and hydrothermal synthetic method to prepare the mixed alumina/iron oxide nanocomposite materials.

Combustion synthesis

Mixed iron/alumina oxide nanocomposite was synthesized by combustion synthesis keeping the oxidizers (Fe(NO$_3$)$_3$.9H$_2$O; Al(NO$_3$)$_3$.9H$_2$O) and fuel (urea) ratio at 1:1. However we have varied the ratio of Fe(NO$_3$)$_3$. to Al(NO$_3$)$_3$ at 1:1, 1:4 & 4:1. For this synthesis, the aqueous solution of Fe(NO$_3$)$_3$.9H$_2$O and Al(NO$_3$)$_3$.9H$_2$O were prepared by mixed required amounts salt precursors. Then this solution is mixed with aqueous urea (NH$_2$CONH$_2$) fuel solution with vigorous stirring and the ratio of oxidizer to fuel was maintained at 1:1. After mixing a brown gel precipitated appeared. To this, 5 ml distilled water was added and stirred until complete transparent solution appears. Then it was heated in a preheated furnace for half an hour at 400 °C to obtain the desired mixed nanocomposite sample.

Hydrothermal synthesis:

In this experiment work, to prepare mixed alumina/iron oxide nanocomposites, 2.42 g Fe(NO$_3$)$_3$.9H$_2$O; 2.42 gm of Al(NO$_3$)$_3$.9H$_2$O were dissolved in 10 ml distilled water and 2.73 g KOH were dissolved separately in 10 ml distilled water and then, the latter solution was added slowly in drop wise manner into the iron-aluminium nitrate salt solution with vigorous stirring. The resulting solution mixture was loaded into 100 ml teflon–lined autoclave, which was then...
filled with distilled water upto 80 % of the total volume. The autoclave was sealed and maintained at 100 °C for 6 h. After the reaction was completed, the autoclave was cooled to room temperature, and the resulting solid products were filtered off, washed several times with absolute ethanol and distilled water and then dried at 60 °C for 12 h to obtain mixed alumina/iron oxide composites. We have also prepared different mixed composite of alumina and iron oxide by varying the weight percentage of aluminium to iron nitrate.

RESULT AND DISCUSSION

FT-IR ANALYSIS: The FT-IR analysis of mixed alumina/iron oxide composite oxides prepared by hydrothermal method is presented in figure 1. The peak at 808 cm\(^{-1}\) corresponds to the asymmetric stretching of the Al=O bond present in the AlO(OH). The peak around 1028 cm\(^{-1}\) corresponds to the Fe-O stretching vibration of Fe\(_2\)O\(_3\) phase. The peak at 3433 cm\(^{-1}\) corresponds to OH stretching frequency of AlO(OH) phase. From FT-IR analysis it is observed that the sample is a mixed composite of Alumina/iron oxide.

![FT-IR Analysis](image)

**Fig-1** FT-IR ANALYSIS of mixed alumina/iron oxide nanocomposite by hydrothermal synthesis

XRD ANALYSIS: The X-ray diffraction pattern of the alumina/iron oxide nanocomposites prepared by hydrothermal synthesis is presented in Figure 2 A. The crystal structures and phases of the alumina were ascertained from the XRD study. Fig 2 A shows the XRD patterns of Al\(_2\)O\(_3\) (10 wt%)-Fe\(_2\)O\(_3\), Al\(_2\)O\(_3\) (20 wt%)-Fe\(_2\)O\(_3\) and Al\(_2\)O\(_3\) (50 wt%)- Fe\(_2\)O\(_3\) composite oxides along
with pure iron oxide. Pure iron oxide synthesized using hydrothermal method shows well defined diffraction peaks at 2θ values of 33.2 and 36.7 degrees corresponding to the reflections from the (104) and (110) planes. These peaks are characteristics of the α-FeO(OH) phase. After loading of alumina into the Fe₂O₃ matrix, new XRD peaks are observed at 2θ values of 24.3, 33.5, 35.66, 54.03, 41.09 and 64.37. The peaks at 2θ values of 24.3, 33.35, 35.66 and 54.03 corresponds to the presence of α-Fe₂O₃ phase with rhombohedral structure (JCPDS FILE NO-(79-1741)) whereas the peaks at 41.09 and 64.37 corresponding to the presence of Al₂O(OH) phase having orthorhombic structure. The composite oxide synthesized by hydrothermal process contains a mixture of α-Fe₂O₃ and Al₂O(OH) phases (JCPDS FILE NO-(81-0464)). Whereas the XRD patterns of the combustion synthesized Alumina/iron oxide composite oxides using urea as a fuel is presented in Fig 2 B. The diffraction peaks at 2θ values 30.72 and 46.16 degrees corresponds to the presence of α-Al₂O₃ (JCPDS FILE NO-(42-1468) and the intense peak at 2θ values of 36.10 corresponds to the presence of α-Fe₂O₃ (JCPDS FILE NO (79-1741)).

**Fig-2** XRD pattern of mixed oxide nanocomposite of Alumina/Iron by (A) Hydrothermal and (B) Combustion synthetic method respectively.

**SEM ANALYSIS**

Fig 3 A represents the SEM micrographs of different weight percentage of alumina loaded iron mixed oxide nanocomposite. The SEM images suggest that the morphology of the composites
synthesized by hydrothermal method is needle shaped. The particles are uniformly distributed and attached to each other through the grain boundary to form agglomerated particles. Fig 3 B and Fig 3 C represents the SEM analysis of composite oxides prepared by combustion synthesis. Pure iron oxide synthesized by this method shows porous spherical grains. When alumina is doped into the iron oxide, the morphology was found to change. It was observed that, with increase in the Al content in the composite oxide shows flake like particles

Fig 3 A represents the SEM micrograph of hydrothermal synthesis & B, C represents the SEM micrographs of the 1:4 and 4:1 of Alumina/iron oxide composite respectively.

CONCLUSION

The studies as part of this project have established the precise protocol for the synthesis of mixed Alumina/Iron oxide nanocomposite.

- We have synthesized mixed nanocomposite of alumina/iron oxide by hydrothermal and combustion method.
- FT-IR studies showing the presence of both alumina and iron oxide in sample
- XRD studies suggest the formation of boehmite (AlO(OH)) and Fe₂O₃ in mixed composite prepared by hydrothermal method. However formation of α-Al₂O₃ and α-Fe₂O₃ mixed composite in combustion method.
- The surface area is found to be 35.06 m²/gm and nonporous in nature as observed by BET analysis in case of hydrothermal synthesis.
SEM studies suggest that in combustion synthesis, porous structures are found as compared to hydrothermal synthesis. Flake like morphology is also observed for composite prepared by combustion method, whereas needle shaped particles are observed for composites prepared by hydrothermal method.

SEM EDAX clearly suggests the presence of Al, Fe and O elements in the composite powder.

REFERENCE


