Synthesis and Characterization of Alumina/Iron Oxide Mixed Nanocomposite

Project Report

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

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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 — Supervisor

05 May'10 — **Dr.Garudadhwaj Hota**

DECLARATION

I, **B**ibhu chandan **P**attanayak 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**

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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 α-Fe_2O_3 and y-AlO(OH) crystalline phase where as α-Fe_2O_3 and α–Al_2O_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 α-Fe_2O_3 and y-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 m^2/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, Al_2O_3) 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, Fe_2O_3 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 N_2, CO_2 and H_2O).

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 ($Fe(NO_3)_3.9H_2O$; $Al(NO_3)_3.9H_2O$) 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 of $Fe(NO_3)_3.9H_2O$ and $Al(NO_3)_3.9H_2O$ were prepared by mixed required amounts salt precursors. Then this solution is mixed with aqueous urea (NH_2CONH_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 $Fe(NO_3)_3·9H_2O$; 2.42 gm of $Al(NO_3)_3.9H_2O$ 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_2O_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 Alumna/iron oxide.

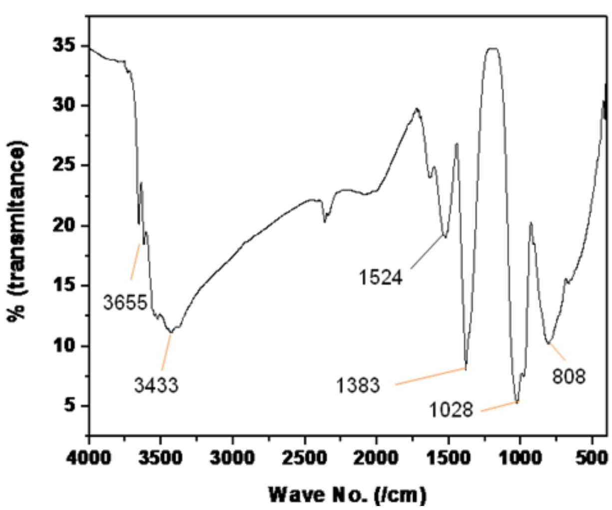

Fig-1 FT-IR ANALYSIS of mixed alumina/iron oxide nanocomposite by hydrothermal synthesis

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_2O_3 (10 wt%)-Fe_2O_3, Al_2O_3 (20 wt%)-Fe_2O_3 and Al_2O_3 (50 wt%)- Fe_2O_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 Fe_2O_3 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-Fe_2O_3 phase with rhombohedral structure (JCPDS FILE NO-(79-1741)) whereas the peaks at 41.09 and 64.37 corresponding to the presence of AlO(OH) phase having orthorhombic structure. The composite oxide synthesized by hydrothermal process contains a mixture of α-Fe_2O_3 and AlO(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-Al_2O_3 (JCPDS FILE NO-(42-1468) and the intense peak at 2θ values of 36.10 corresponds to the presence of α-Fe_2O_3 (JCPDS FILE NO (79-1741).

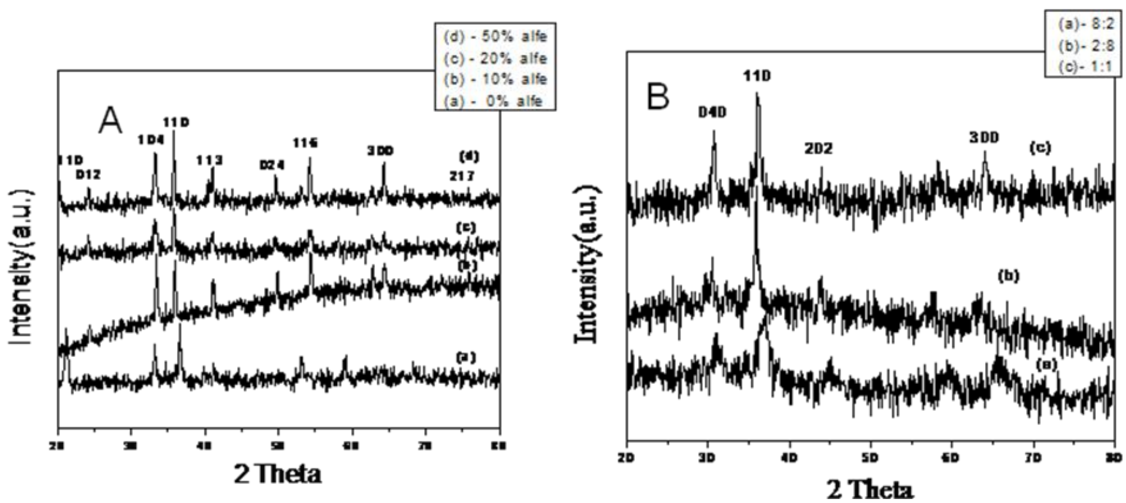

Fig-2 XRD pattern of mixed oxide nanocomposite of Alumina/Iron by (A) Hydrothermal and (B) Combustion synthetic method respectively.

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

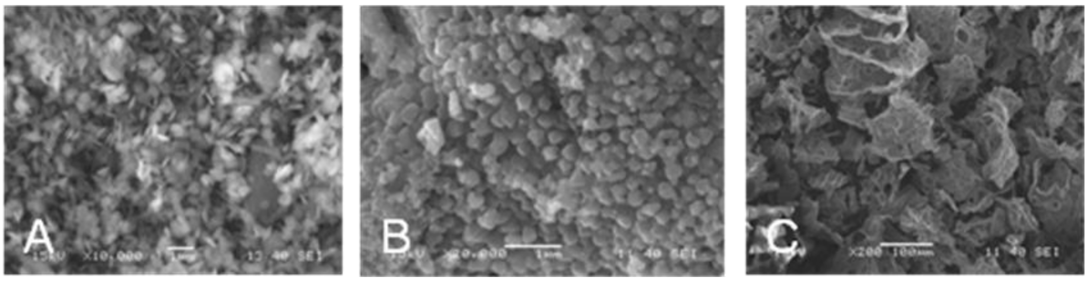

Fig 3 A represents the SEM micrograph of hydrothermal synthesis & B, C represents the SEM micrographs of the 1:4 and 4:1 of Alumina/iron oxide composite respectively.

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 Fe_2O_3 in mixed composite prepared by hydrothermal method. However formation of α-Al_2O_3 and α-Fe_2O_3 mixed composite in combustion method.
- The surface area is found to be 35.06 m^2/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, where as 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.

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