Preparation of nano material using two different methods

Asif jehan*, shirish joshi ** and M.N. Bapat***

*dept. of physics M.V.M. college Bhopal
**dept. of physics M.V.M. college Bhopal
***dept. of physics regional institute of education Bhopal

ABSTRACT
Zinc aluminate (ZnAl$_2$O$_4$) spinal has drawn considerable attention as a catalytic material. Moreover zinc aluminate tends to prevent sintering of noble materials due to a strong material-support interaction. Nano sized zinc aluminate powder were prepared by heating method at 600 sintering temperatures and by combustion method at 500 temperatures. Zinc sulphate (ZnSO$_4$), alum (Na$_2$SO$_4$-Al$_2$O$_3$-24 H$_2$O) and urea (NH$_2$ CO NH$_2$) were used as materials. Here urea is used as a chelating agent. A method of making zinc aluminate nano-material by Zinc nitrate (ZnNO$_3$), aluminum nitrates (Al$_2$NO$_3$) and urea (NH$_2$ CO NH$_2$) were used as materials in second method. A comparison of the samples made from above two methods is done about their physical properties.

INTRODUCTION

Zinc aluminate (ZnAl$_2$O$_4$), naturally occurring as the mineral, has drawn considerable attention as a catalytic material with improved properties due to high thermal stability, high mechanical resistance, and low surface acidity. Moreover, zinc aluminate tends to prevent sintering of noble metals due to a strong metal-support interaction [1-3]. Nano sized zinc aluminate powders were prepared by sintering method. In high temperature processes sintering resistance and chemical stability are the properties of the most significance for the catalytically active phases. Beside catalytic purposes zinc aluminate has been used as ceramic and electro conductive material because of its high thermal stability, high mechanical resistance and excellent optical properties. Optical properties make it material of interest for ceramic pigments and coatings. [4] Zinc aluminate is normally prepared by a solid state reaction of zinc oxide and aluminum oxide however, for ensuring complete reaction; a temperature above 1,000 _C has to be maintained for several days. [5] In the present paper, the synthesis of ZnAl$_2$O$_4$ nano powders, we use two different methods. The first method is sintering method and 2$^{nd}$ one is combustion method. For achieving combustion at low temp we use a few techniques like use of flux, gelation or sol-gel method because nano sized materials, in comparison with bulk materials, display improved properties viz. lowered sintering temperatures, increased hardness, stability, diffusion etc. which can be overcome by above said special techniques. The disadvantages of solid-state reaction are in homogeneity, high sintering temperature requirement and low surface area.

Sintering method: - Sintering is a method for making objects from powder, by heating the material in a sintering furnace below its melting point (solid state sintering) until its particles attain sufficient energy to adhere to each other. Sintering is traditionally used for manufacturing ceramic objects, and has also found uses in such fields as powder metallurgy.

Advantages and requirements of sintering method
Peculiar advantages of above referred powder technology are:

1. Very high levels of purity and uniformity in starting materials
2. Preservation of impurity, due to the simpler subsequent fabrication process (fewer steps) that it makes possible
3. Stabilization of the details of repetitive operations, by control of grain size during the input stages

4. Absence of binding contact between segregated powder particles – or "inclusions" as often occurs in melt processes

5. No deformation needed to produce directional elongation of grains

6. Capability to produce materials of controlled, uniform porosity.

Sintering is effective when the process reduces the porosity and enhances properties such as strength, translucency and thermal conductivity; yet, in other cases, it may be useful to increase its strength but keep its gas absorbency constant. During the firing process and as it continues; grain size becomes smaller and more spherical because the particle's surface tends to flow into the pores within it based on the difference between vapor-pressure and cross-sectional area of the pore's neck.

**Working:** - a method of making zinc aluminate nano material, the method comprise of following steps. Firstly, providing a growing substrate and the growing device comprising a heating apparatus and a reacting chamber. Secondly, placing the growing substrate and a quantity of reacting materials into the reaction chamber, and the reacting materials comprising zinc and aluminum. Lastly, heating the reaction room to a temp of 600 °C.

**Method**

**Sintering method (A):** - the method of using solid-state reaction for making zinc aluminate nano material includes the following steps.

1. Take a quantity of zinc sulphate (ZnSO₄) powder (700 mg) and alum (Na₂SO₄ Al₂O₃ .24H₂O) powder (800 mg) and urea (NH₂ CO NH₂) powder (500 mg).

2. Grind the zinc sulphate powder, alum powder and urea. Now putting the mixture into a furnace.

3. Heating the mixture to a temp above 600 degree c to obtain a zinc aluminate nano material.

4. Keep the material into the furnace when the temp decreases gradually.

**Combustion method (B):** - the method of using solid-state reaction for zinc aluminate nano material by combustion method includes the following steps.

1. Take a quantity of zinc nitrate (3.986 gm), aluminum nitrate (10 gm) and urea (5.343 gm).

2. Grind the zinc nitrate, aluminum nitrate and urea. Now allow the furnace to attain a temp 500 °C.

3. After that put the mixture into furnace for 5 minutes till the fumes evaporated.

4. Taken out the crucible containing material and placed it on asbestos sheet and cool it down to room temp.

**Result:** the SEM images of material A and material B is shown below the table.
Comparison between heating method and combustion method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Method</th>
<th>temperature</th>
<th>Ignition</th>
<th>Surface</th>
<th>shape</th>
<th>color</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Heating method</td>
<td>600</td>
<td>Not ignite</td>
<td>Pores and upper surface look like moon Internal surface-spinal form</td>
<td>Semi elliptical</td>
<td>White</td>
</tr>
<tr>
<td>B</td>
<td>Combustion method</td>
<td>500</td>
<td>Ignite</td>
<td>Pores and spinal form</td>
<td>Ebullient</td>
<td>White</td>
</tr>
</tbody>
</table>

SEM images of material A (sintering method)

![SEM image 1](image1)

![SEM image 2](image2)

SEM images of material B (combustion method)

![SEM image 3](image3)

![SEM image 4](image4)
References: