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Production of Zinc Oxide (ZnO) Nano Material

Babli Varsha[1] and Anamika Paul[2,*]

[1,2]Galgotia University, School of Chemical Engineering. Greater Noida, U.P.
E-mail: [1]bablivarsha68@gmail.com, [2]anamika.sikdar@gmail.com

Abstract—*Nano materials possess enhanced catalytic abilities due to their highly stressed surface atoms which are very reactive e.g. ZnO, TiO_2. Nano particles have been known to be used in cosmetic, electronics, paints, etc. In the present paper, the laboratory study of synthesis of ZnO nanoparticles by means of a rapid combustion synthesis method using citric acid as fuel and zinc nitrate as oxidant is presented. A gel combustion method was used to prepare ZnO nano powder. Nitrate, nitric acid and different fuels of glycine, urea, and citric acid were used with various ratios of fuel to salt. The ratio of fuel to metallic nitrate was selected as 0.5, 1 and 2. Tests were also conducted with varying the pH values and the calcinations temperatures was varied between 400-600^oC. After mixing and homogenizing metallic nitrate with fuel, while heating the solution, the gel combustion process was carried out and ZnO nano powder was prepared. The ZnO nanoparticles later have been characterized by FTIR (Fourier Transform Infrared Specstroscopy). The results showed that for the synthesis of ZnO nano particles, the optimum conditions was using citric acid as fuel, fuel to raw material ratio of 0.5, a neutral pH and a calcination temperature of 500^oC. The above optimum conditions facilitates the production of ZnO nano powder of about 30 nm in size.*

Keywords*: Nano particles, Zinc oxide, combustion synthesis, FTIR, calcination, catalysts*

1. INTRODUCTION

Zinc oxide is an important metal oxide that has attractive optical, electrical, magnetic and catalytic properties [1]. These remarkable properties have made ZnO a multifunctional material that finds applications in field effect transistors, light emitting diodes, diluted and ferro magnetic material for spintronics, solar cells, gas sensors and photocatalysts . ZnO is also an oxide semiconductor with a large direct band gap (~3.3eV) [2].

Control of size, shape and orientations of ZnO crystallites is the general prerequisite for the creation of high surface area materials for use in many applications. Diverse methods of synthesis like physical vapour deposition, chemical vapour deposition, Spray Pyrolysis, hydrothermal method, Coprecipitation method, Sol-gel method and Combustion methods have been used for architecturing a variety of morphologies in the nano dimensions of ZnO. Among the various synthesizing techniques, combustion method is characterized by the use of relatively simple equipment, formation of high purity products, stabilization of metastable phases and ability to yield desirable size and shapes of the products [3, 4, 5].

The scope of the present project was to test the formation of ZnO nano particles at different calcinations temperatures using varying pH and fuel to metallic nitrate ratios [6].

2. METHODOLOGY

In the gel combustion method, the raw materials, which are usually a nitrate compound and a fuel, are dissolved in water. After controlling the pH, by a weak base such as ammonia, the mixed solution is heated to change the sol to a high viscosity gel [3]. Increasing temperature, causes an exothermic combustion process and both organic materials as a reducing agent and nitrates as an oxidation agent change the gel to a very grey fine and intensively porous substance which later with the help of calcinations a final product will be prepared. Zinc nitrate and citric acid were used with various ratios of fuel to salt and different primary pH values, followed by calcinations at temperatures of 400-600^oC [7]. The results showed that using citric acid, fuel to the raw material ratio of 0.5, a neutral pH, and calcination temperature of 500^oC were the optimum conditions to prepare ZnO nano powder.

3. EXPERIMENTAL PROCEDURES

For all the experiments, material such as zinc nitrate and citric acid were used. The ratio of the fuel to metallic nitrate was selected as 0.5 .The material were weighed, dissolved in distilled water and homogenized on a magnetic stirrer. After mixing and homogenizing mettalic nitrate with fuel, while heating the solution, the gel combustion method process carrired out and ZnO nano powder was prepared [8, 9]. The powder were calcined at 500 ^oC. Subsequently, FTIR (Fourier Transform Infrared Spectroscopy) was used and the result were analyzed.

4. PROCEDURAL DETAILS

The method followed for the formation of the nano particles is known as GEL COMBUSTION METHOD. The steps involved in the process are as follows:

Step-1: Nitrate compound, in this case, Zinc Nitrate, $ZnNO_3$ and a fuel, in this case Citric acid, $C_6H_8O_7.H_2O$) are weighed and dissolved in water. One gram of Zinc Nitrate (1gm) and half a gram of Citric acid (0.5 gm) were taken.

Step-2: A homogenized solution was formed by the use of a magnetic stirrer. Also, the pH was controlled using a weak base such as ammonia.

Step-3: After maintain the pH of the solution, the solution was heated. This was done to increase the viscosity of the solution and obtain a high viscous gel through gel combustion process.

Step-4: After the high viscous gel solution is formed by the combustion method carried out, ZnO nano powder was prepared.

Step-5: The ZnO nano powder were calcind at 500 ^oC [10]. The calcinations process was done in the absence of O_2.

Step-6: CO_2 is required for calcination. For this purpose, CO_2 was produced by using calcium carbonate ($CaCO_3$) and concentrated hydrochloric acid (HCl) as per the reaction [11]:

$$CaCO_3 + 2HCl \Rightarrow CO_2 + CaCl_2 + H_2O$$

Step-7: Subsequently, after the procedure is complete, particle size analysis was conducted using FTIR (Fourier Transform Infrared Spectroscopy).

Step-8: Finally, the formation of nanoparticles was analyzed.

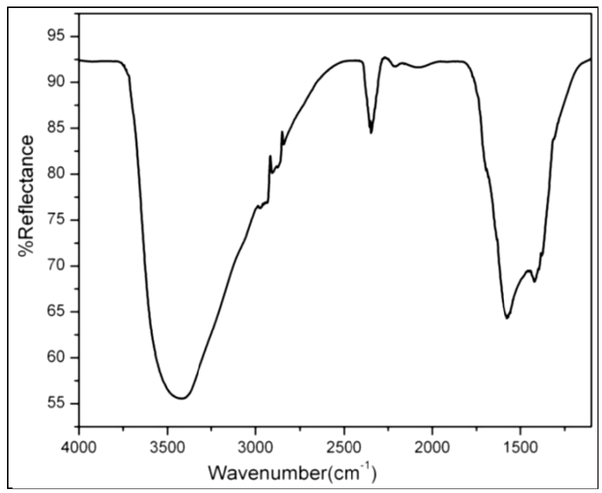
5. FTIR ANALYSIS AND RESULTS

FTIR analysis was conducted to understand the materials and products formed. Analytical testing sample screens, profiles and data interpretation are available on a global basis. FTIR testing identifies chemical compounds in consumer products, paints, polymers, coatings, pharmaceuticals, foods and other products. Laboratories with FTIR expertise are located throughout the Intertek global laboratory network.

FTIR offers quantitative and qualitative analysis for organic and inorganic samples. Fourier Transform Infrared Spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The spectra produce a profile of the sample, a distinctive molecular fingerprint that can be used to screen and scan samples for many different components. FTIR is an effective analytical instrument for detecting functional groups and characterizing covalent bonding information.

Principle of FTIR Spectroscopy: When infrared radiation passes through a sample (solid, liquid or gas), certain frequencies of the radiation are absorbed by the molecules of the substance leading to the molecular vibrations. The frequencies of absorbed radiation are unique for each molecule which provides the characteristics of a substance. Since, the strength of the absorption is proportional to the concentration, FTIR can also be used for quantitative analysis.

FTIR analysis shows the complexation of the precursor metal nitrates. The broad stretching bands at 3356, 3436 and 3449 cm^{-1} in the as-prepared varistor grade precursor gel and the samples calcined at 500°C respectively, indicate the presence of bonded and free hydroxyl group. The series of transmittance peaks at 1653 cm^{-1}, 1630 cm^{-1} and 1593 cm^{-1} are indicating the bending modes of –OH groups. The precursor gel exhibits the characteristic stretching bands of nitrate group at 1394 cm^{-1}. The nitrates decompose during calcinations and therefore these peaks are not visible in the calcined samples. The following figure shows a typical FTIR graph.



6. CONCLUSION

In the present project, the following conclusions were obtained:

- It is possible to prepare nano powder using a gel combustion method.
- Citric acid used in a gel combustion method acts both as a gel agent and a fuel, which makes a homogeneous suitable nano powder.
- The optimmum fuel to raw materials ratio is 0.5 when citric acid is used.
- A neutral pH, in the gel combustion process creates a fine and homogeneous ZnO nano powder.
- An optimum calcination temperature of 500^{o}C is required to change amorphous particles to a crystalline phase.

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