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Exploring Reducing Agent Effects in the Synthesis of ZnO Nanoparticles

Avneesh Kumar*

*Department of Physics, SVGC Ghumarwin, (H.P), INDIA

Abstract

In this study ZnO nanoparticles are prepared by using sol gel method. This method is used by many researchers because of its case of use, easily availability of apparatus, low cost and it is easy to perform. The aim of this research is to study the size of ZnO nanoparticles using different reducing agents (NaOH or NH_3OH or Triton X-100). The used chemicals are highly pure with AR grade. The precipitates were formed and annealed at temperature approx.1000^0C.The precipitate was collected and characterized by X-ray diffraction (XRD) to calculate the crystalline size of the nanoparticles. Nanosized ZnO powder was successfully synthesized and XRD analysis revealed that crystalline size is nearly independent of different reducing agents at 1000^0C. Also the results obtained show that sol-gel method can produce good quality ZnO nanoparticles by using any reducing agents.

Keywords: Nanoparticles, Precursor, Reducing agents, XRD, sol-gel

1. Introduction

Nanotechnology is the science that deals with matter at the scale of 1 billionth of a meter and is also the study of manipulating matter at the atomic and molecular scale. A nanoparticle is the most fundamental component in the fabrication of a nanostructure and is far smaller than the world of everyday objects that are described by Newton's laws of motion. Nanoparticles are the particles in the range from 1nm-100 nm [1-2]. These nanoparticles are useful in other fields like electronics, biological, medical etc as they show different properties with different materials [3]. Till date quantum dots, Integrated circuits are the best examples of nanoparticles use. Different methods like physical, chemical, biological are used for construction of nanomaterial [4-7]. Among these, ***sol-gel*** method is used because of low cost, easily availability of tools and many more [8-12]. Much research showed different results and properties of nanoparticles which is useful in different applications and motivating others to invent such nanoparticles which could be a breakthrough [13-18]. Among various nanoparticles, ZnO nanoparticles are promising candidates for various applications, such as nanogenerators [19], gas sensors [20], biosensors [21], solar cells [22], varistors [23], photodetectors [24], and photocatalysts [25]. We report the synthesis of ZnO nanoparticles using different reducing agents in chemical method and the characterization of ZnO nanoparticles using X-ray diffraction.

2. Synthesis

Zinc oxide nanostructure was synthesized by using sol-gel method [8-12]. To prepare a sol, precursor 0.2M of zinc acetate dihydrate and reducing agent (0.2 M of sodium hydroxide or 0.2 M Ammonia Solution (NH_3OH) or 5ml Triton X-100 [13]) were weighted using a weighting balance. Then 50ml of distilled water was measured by measuring cylinder. After that, 0.2 M of zinc acetate dehydrate was dissolved in 50ml of distilled water and reducing agent was dissolved in 50ml of distilled water. The solution was stirred with constant stirring for about 15 minutes each. After well mixed, reducing agent was poured to the solution containing zinc acetate with constant stirring by magnetic stirrer for about 15 minutes. Then burette was filled with 100ml of PVA (Poly vinyl alcohol) and titrate drop wise to the solution containing both reducing agent and zinc acetate [14]. After the reaction, white precipitate was formed. Dried precipitate was calcined at about 1000^{o}C for 24 hours in muffle furnace and sample was prepared.

3. Result and Discussion

3.1 Based on ZnO + NaOH

Based on the experimental work that has been done, there are series of chemical reaction that takes place. The complete reaction is in between transformation of zinc acetate dihydrate into zinc oxide nanoparticles. The addition of sodium hydroxide leading to the formation of zinc hydroxide intermediates in the form of Sol. PVA as a stabilizing agent ensure uniform dispersion of nanoparticles. After Gel formation and calcination, the resulting product comprises stable and dispersed zinc oxide nanoparticles [12].

Following reaction take place:

Precursor Dissolution:

$[Zn(CH_3COO)_2.2H_2O] + H_2O \longrightarrow Zn^{2+} + 2CH_3COO^- + 2H_2O$

Formation of Zinc Hydroxide Sol:

$NaOH + H_2O \longrightarrow Na^+ + OH^-$

$Zn^{2+} + 2OH^- \longrightarrow Zn(OH)_2$

Polyvinyl Alcohol Addition: Gel Formation

$Zn(OH)_2$ + PVA $\longrightarrow$ Stabilized $Zn(OH)_2$-PVA Nanoparticles

Calcination:

Stabilized $Zn(OH)_2$-PVA Nanoparticles $\longrightarrow$ ZnO + Byproducts

These steps convert ***Zinc hydroxide* to *Zinc oxide*** nanoparticles at higher temperature. XRD analysis was carried using X-ray diffractometer with Cu- Kα radiation in the range $10\text{-}80^0$. Fig.1 Illustrate XRD spectrum of ZnO nanoparticles, synthesized using zinc acetate as precursor and NaOH as reducing agent. The sharp peaks of ZnO indicate the crystallinity in nature and also confirm the synthesized nanopowder was free from impurities. The ZnO nanoparticles diameter was calculated for more intense peak corresponding to (101) plane using Debye- Scherrer formula [26].[Table.1]

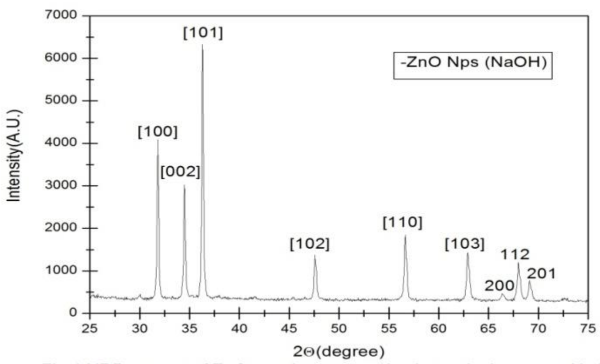

Fig.1 XRD pattern of ZnO powder prepared using reducing agent NaOH

$D = \frac{0.89\lambda}{\beta Cos\theta}$ Where λ is wave length of X-Ray (1.541 A^0), β is FWHM (full width at half maximum) in radians, θ is the diffraction angle and D is particle diameter size.

Table1. Calculation of average size (D) of ZnO nanoparticles

Angle($2\theta^0$)	θ^0	β^0[FWHM]	D(nm)
36.31	18.155	0.2155	38.36

3.2 Based on ZnO + Triton x-100

Following reaction take place:

[Zn ($CH_3COO)_2$ + Triton X-100 + PVA +H_2O → ZnO nanoparticles + Byproducts

Fig.2 Illustrate XRD spectrum of ZnO nanoparticles, synthesized using zinc acetate as precursor and Triton X-100 as reducing agent. Here Triton x-100 used as reducing agent leads to prevent agglomeration of nanoparticles and control their growth. The sharp peaks of ZnO indicate the crystallinity in nature and also confirm the synthesized nanopowder was free from impurities. The ZnO nanoparticles diameter was calculated for more intense peak corresponding to (101) plane using Debye- Scherrer formula [26]. [Table.2]

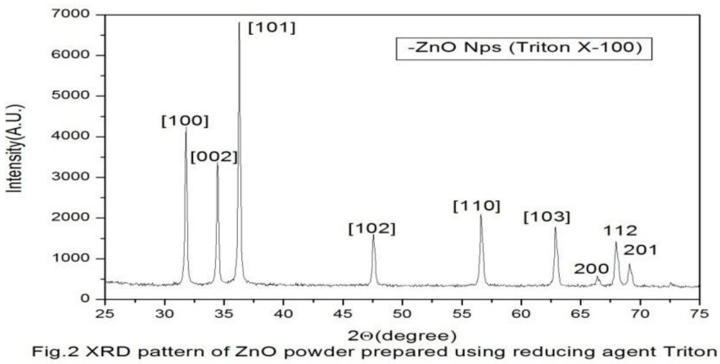

Fig.2 XRD pattern of ZnO powder prepared using reducing agent Triton X-100

Table2. Calculation of average size (D) of ZnO nanoparticles

Angle($2\theta^0$)	θ^0	β^0[FWHM]	D(nm)
36.28	18.14	0.2264	36.51

3.3 Based on $ZnO + NH_3OH$

Following reaction take place:

Precursor Dissolution:

$$[Zn(CH_3COO)_2.2H_2O] + H_2O \longrightarrow Zn^{2+} + 2CH_3COO^- + 2H_2O$$

Formation of Zinc Hydroxide Sol:

$$NH_3OH + H_2O \longrightarrow NH_4^- + OH^-$$

$$Zn^{2+} + 2OH^- \longrightarrow Zn(OH)_2$$

Polyvinyl Alcohol Addition: Gel Formation

$$Zn(OH)_2 + PVA \longrightarrow \text{Stabilized } Zn(OH)_2\text{-PVA Nanoparticles}$$

Calcination:

$$\text{Stabilized } Zn(OH)_2\text{-PVA Nanoparticles} \longrightarrow ZnO + \text{Byproducts}$$

Fig.3 Illustrate XRD spectrum of ZnO nanoparticles, synthesized using zinc acetate as precursor and as NH_3OH as reducing agent. The sharp peaks of ZnO confirm the crystallinity in nature and also confirm that synthesized nanopowder was free from impurities. The ZnO nanoparticles diameter was calculated for more intense peak corresponding to (101) plane using Debye- Scherrer formula [26].[Table.3]

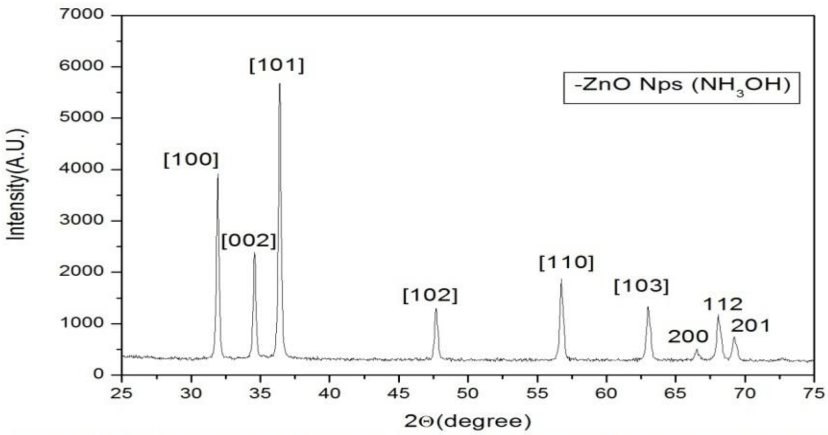

Fig.3 XRD pattern of ZnO powder prepared using reducing agent NH_3OH

Table3. Calculation of average size (D) of ZnO nanoparticles

Angle($2\theta^0$)	θ^0	β^0[FWHM]	D(nm)
36.4	18.2	0.24153	34.26

4. Conclusion

Using different reducing agents, we have synthesized fine powder of ZnO nanoparticles. Also, XRD data produce the well-defined peaks of ZnO nanoparticles which confirm the crystalinity of nanoparticles. We have found that the average size of ZnO nanoparticles is independent of reducing agents at 1000^{o}C temperature. So, we may use any of the reducing agent to produce good quality of ZnO nanoparticles.

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