



The effect of parameters on the formation of ZnO nanoparticles by statistical experimental design method in vibrating milling process

Ki Do Kim, Dae Woo Choi, Yong-Ho Choa, Hee Taik Kim*

Department of Chemical Engineering, Hanyang University, 1271 Sa 3-dong, Sangnok-gu, Ansan-si, Gyeonggi-do 426-791, Republic of Korea

ARTICLE INFO

Article history:

Received 9 June 2007

Received in revised form

30 July 2007

Accepted 27 September 2007

Keywords:

ZnO

Vibrating milling process

Statistical experimental design

Taguchi method

ABSTRACT

Zinc oxide (ZnO) nanoparticles were synthesized by vibrating milling process. Amorphous ZnO powders prepared by the milling process were washed with water to remove NaCl, and then the powders were sintered at 300–500 °C for 2 h. In this work, statistical experimental design method was used and it was found that the particle size and size distribution of ZnO nanoparticles were dependent on the milling time and mass ratio of ball to powder. Therefore, ZnO nanoparticles with about 100 nm of mean crystallite size were obtained.

© 2007 Elsevier B.V. All rights reserved.

1. Introduction

Zinc oxide (ZnO) nanopowders have been widely used in many fields such as barristers, dyes, and cosmetic materials due to its unique electrical and optical properties (Yin et al., 2005). It is also chemically stable and has superior electrical conductivity and transmissivity (Wang, 2004). Moreover, nanosized ZnO particles have highly conductive band gap energy (3.27 eV) and binding energy (60 meV). So, it can be cut-off ultraviolet rays and is used as photocatalyst (Hong et al., 2006). Because of its photocatalytic characteristics, it can be applied in high-quality paint, cosmetic as well as coating materials.

The synthesis of the above ZnO nanoparticles can be accomplished by precipitation method (Kim et al., 2006), sol-gel method (Kim and Kim, 2002), solvent evaporation method, hydrothermal process, ultrasonic spray pyrolysis

(Kim et al., 2005), and solid-state reaction method. Of these methods, the solid-state reaction method has some disadvantages of high-reaction temperature, large particle size, and limited degree of chemical homogeneity, but it is simple to operate and uses the cheap and easily available oxides as starting materials. In general, vibrating milling process applied as a process of solid-state reaction method has not been mainly used in the synthesis of nanosized powders. However, so many works have been achieved to improve the milling system, because it has the advantages of simple, low cost, and large-scale production (Takuya et al., 2001). In recent, a wide variety of nanoparticles including SeO₂, SnO₂, ZnO, CdS, ZrO₂, CeO₂, LiMn₂-xCo_xO₄, and LiB₂O₇ (Cukrov et al., 2001; Liu and Shen, 2003; Balaz et al., 2003; Dodd and McCormick, 2002; Li et al., 2000; Soiron et al., 2001) have been prepared by vibrating milling process.

* Corresponding author. Tel.: +82 31 400 5274; fax: +82 31 419 7203.

E-mail address: khtaik@hanyang.ac.kr (H.T. Kim).

0924-0136/\$ – see front matter © 2007 Elsevier B.V. All rights reserved.

doi:10.1016/j.jmatprotec.2007.09.053

Table 1 – Parameters and levels used in this experiment

		Levels		
		1	2	3
A	Milling time (h)	1	4	7
B	Amount of inert diluent (g)	0	0.5	1
C	Mass ratio of ball to powder (–)	4	7	10

Properties of ZnO nanoparticles obtained by vibrating milling method are affected by various parameters such as milling time, amount of inert diluent, ball to powder mass ratio, molar ratio of ZnCl_2 – Na_2CO_3 , and so on. The interrelationships between the above parameters are complex, and the analysis of this vibrating milling system to optimize the factors is a time and labor consuming work. Hence, the analyses using conventional experimental methods are inefficient. The efficient analyses of the complex system using statistical experimental design and the Taguchi method (Roy, 1990) have been performed recently. The statistical experimental design can determine the effect of the factors on characteristic properties and the optimal conditions of factors. It uses the tables of orthogonal arrays and analysis of variance (ANOVA) as the tools of analysis. ANOVA can estimate the effect of a factor on the characteristic properties, and an experiment can be performed with the minimum replication using the table of orthogonal array.

The objectives of this work are: (1) to suggest a method for the synthesis of ZnO nanoparticles by using vibrating milling process and to characterize the ZnO nanoparticles, (2) to evaluate the effect of several parameters on the particle size and particle size distribution of ZnO nanoparticles, and (3) to apply the statistical experimental design on the optimization of properties and to obtain the monodispersed ZnO nanoparticles by using optimal synthesis conditions.

2. Experimental procedure

2.1. Starting materials and preparation of ZnO nanoparticles

In this work, anhydrous zinc chloride (ZnCl_2 , DAEJUNG), anhydrous sodium carbonate (Na_2CO_3 , DAEJUNG), and sodium

Table 3 – The ANOVA table of particle size

Factor	Sum of square (S)	Degree of freedom (φ)	Mean square (V)	F
A	35.3	2	17.65	2.60
B	10.0	2	5.00	0.74
C	35.5	2	17.74	2.62
Error	13.6	2	2.62	
Total	94.3	8		

chloride (NaCl , DAEJUNG) were used as a starting material. Zirconia balls (6.0 and 9.5 mm, DAIHAN) were also used to grind the powders. Na_2CO_3 and NaCl were used as a catalyst to promote the reaction and diluent, respectively. The milling time, amount of inert diluent (NaCl), and mass ratio of ball to powder were varied in the range of 1–7 h, 0–1 g, and 4–10, respectively. The molar ratio of ZnCl_2 – Na_2CO_3 was fixed at 1:1 and 2.3 g of ZnCl_2 was used in this work. Reactant mixtures were milled within a hardened steel vial, using a vibratory SPEX 8000 mixer/mill with zirconia balls. The vibrating milling method with chemical reaction can provide products of fine and homogeneous particles with high-specific surface area and it enables us to synthesize many materials. The reaction mechanism of this method is as follows:



As shown in the above reaction mechanism, the reaction is progressed by three steps. First, ball mill acts as a low temperature chemical reactor and reaction process results from focal heat and pressure at contact surface. Secondly, chemical reactions of particles are kept apart by salt matrix and low temperature enables controlled particle formation. And finally, reaction product is heat-treated and solid-phase chemistry prevents particles from agglomeration. To remove the NaCl obtained during reaction, the milled powder was washed with de-ionized water, followed by centrifugal separation. The washed powders were dried at 100°C for 24 h, and then the powders were sintered at 300 – 500°C for 2 h under a sealed N_2 atmosphere. Particle size and size distribution of ZnO pow-

Table 2 – Experimental measured values and S/N ratio for particle size and standard deviation (S.D.) of ZnO nanoparticles (Taguchi orthogonal array table of $L_9(3^4)$)

Experimental number	A	B	C	Error	Average particle size		Standard deviation	
					Raw data (nm)	S/N ratio (dB)	Raw data (%)	S/N ratio (dB)
1	1	1	1	1	376.8	–51.5	218.4	–46.8
2	1	2	2	2	295.7	–49.4	390.1	–51.8
3	1	3	3	3	211.1	–46.5	59.5	–35.5
4	2	1	2	3	141.4	–43.0	48.8	–33.7
5	2	2	3	1	169.8	–44.6	99.6	–39.9
6	2	3	1	2	345.3	–50.7	327.2	–50.3
7	3	1	3	2	120.0	–41.6	36.4	–31.2
8	3	2	1	3	176.4	–44.9	48.2	–33.6
9	3	3	2	1	211.9	–46.5	55.5	–34.9

Download English Version:

<https://daneshyari.com/en/article/795481>

Download Persian Version:

<https://daneshyari.com/article/795481>

[Daneshyari.com](https://daneshyari.com)