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The comparison synthesis of $CuNiO_2$ nanoparticles prepared by sol-gel auto-combustion, microwave and co-precipitation techniques

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ABSTRACT

The $CuNiO_2$ nanoparticles have been synthesized by sol-gel auto-combustion, microwave and co-precipitation techniques. Then, the results of the three techniques were compared. In the sol-gel auto-combustion synthesis was used glycine as a fuel. At first, sol was prepared. Then, gel was achieved from drying sol. The prepared gel was burnt at 350°C. In the microwave technique, gel was prepared. Then, the final nanoparticles were produced on influence of microwave irradiation. The $CuNiO_2$ nanoparticles were prepared by co-precipitation method invalved metal sulfates. The temperature of calcinations was 620°C. These nanoparticles were investigated by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques. The XRD results show that the nanocrystal contains various phases, structures and the average size of nanostructured $CuNiO_2$ crystallities was calculated by Scherrer equation. Scanning Electron Microscopy was also used to characterize the microstructure and morphology.

Keywords: Sol-gel auto-combustioa; Co-precipitation; Microwave; SEM; XRD

INTRODUCTION

Nanocrystalline semicooductor particles have interested a great deal of attention because of their properties and applications in electronics, magnetic and catalysis [1, 2]. Among the oxides of transition metals, CuO has attracted much attention because it is the basis of several high-T_c superconductors. CuO is a semiconducting compound with a narrow band gap and used far photoconductive and photothermal applications [3] Several methods are conventionally used for the synthesis of nanoparticles such as sol-gel [4], co-precipitation [5], hydrothermal [6] aod microwave [7, 8]. Sol-gel auto-combustion is a unique combination of the combustion and the chemical gelation processes This method exploits the advantages of cheap precursors, simple preporation and a resulting ultra fioe and homogeneous powder [9, 10].

Combustion synthesis is a particularly simple, safe and rapid process where in the main advantages are energy and time savings [11].

Ni and NiO composite prepared and hinary oxide nanoparticles by using microwave irradiotion were also reported [12, 13]. The microwave synthesis, which is generally quite fast, simple and efficient in energy, has been developed and is widely used in various fields such as molecular sieve preparation, the preparation of inorganic complexes and oxide, organic reactions, plasma chemistry, analytical chemistry and catalysis [14]. Besides its applications in chemical analysis and in radiochemistry, co-precipitation is also potentially important to many environmental issues closely related to water resources, including

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acid mine drainage, radionuclide migration in fouled waste repositories, metal contaminant transport at industrial and defense sites, metal concentrations in aquatic systems, and wastewater treatment technology [15].

Co-precipitation is also used as a method of magnetic nanoparticle synthesis [16].

In this paper, $CuNiO_2$ nanoparticles were compared with three methods includes sol-gel, microwave and co-precipitation.

EXPERIMENTAL

In sol-gel auto-combustion method, copper nitrate (Cu $(NO_3)_2.3H_2O$), nickel nitrate (Ni $(NO_3)_2.6H_2O$), glycine ($C_2H_5NO_2$), ammonia 25% (NH_3) and deionized water was purchased for preparing $CuNiO_2$ nanoparticles. In microwave method, copper nitrate (Cu $(NO_3)_2.3H_2O$), nickel nitrate (Ni $(NO_3)_2.6H_2O$) ammonia 25% (NH_3), deionized water and tert-butyl alcohol was used. In co-precipitation method, copper sulfate ($CuSO_4.5H_2O$), nickel sulfate ($NiSO_4.6H_2O$), NaOH and deionized water was produced. All the reagents were used without further purification.

For three methods were used, X-ray diffraction (XRD) made in Netherland, Philips Xpert MPO model Scanning Electron Microscopy instrument (SEM) was used to characterize morphology and particle size. The model of this instrument was XL30 Philips.

For synthesis of $CuNiO_2$ nanoparticle by sol-gel auto-combustion method was used appropriate amount of Cu $(NO_3)_2.3H_2O$ and Ni $(NO_3)_2.6H_2O$ powders. The molar ratio Cu/Ni was 1:1. These materials were dissolved in deionized water. Then, glycine was added with the two moles equal to those materials. The aqueous solution was neutralized at pH=7 by adding liquor ammonia 25%. During this procedure, the sol was continuously stirred by mechanical stirrer in bath water at 65°C for 4h. When ignited at any point of the gel, the dried gel burnt in a self-propagating combustion manner until all gels were completely burnt out to form a fluffy loose powder. The as-burnt ash was calcined at 620°C for better crystallization and homogeneous cation distribution in the spinel.

For synthesis of $CuNiO_2$ nanoparticle by microwave method, deionized water and tert-butyl alcohol were mixed with 5:1 molar ratio and Cu $(NO_3)_2.3H_2O$ and Ni $(NO_3)_2.6H_2O$ were poured continuously in deionized water and alcohol solution. For adjusting pH=7 was used ammonia 25%. A round bottom glass vessel (total volume≈150 mL) was used for the microwave irradiation which was carried out under ambient air. The solution was placed in a microwave refluxing (300W) system for 3-5 min. At the end of the reaction, the precipitate was centrifuged 4000 rpm for 10 min and washed repeatedly with deionized water annealed 620°C for 3h.

For synthesis of $CuNiO_2$ nanoparticle by co-precipitation, copper and nickel sulfate were dissolved in 500 ml deionized water. Then, pH of this solution was adjusted to 7 using NaOH solution. The solution was stirred at 60 °C for 12h. Then, used filter for separating precipitation from solvent. Then, washed it with distilled water. Precipitation was heated at for 1 hour. Finally powders were calcined at 620 for 2h.

RESULTS AND DISCUSSION

The structural characterization of $CuNiO_2$ powders was performed by X-ray diffraction analysis. The XRD pattern of $CuNiO_2$ prepared by sol-gel auto-combustion, microwave and co-precipitation methods was shown in Figure 1a, b and c, respectively.

The average crystallite size was calculated using the Scherrer relationship. $d = 0.9 \lambda / \beta \cos\theta$, where d is the crystallite size, β is the half maximum line width of peak and λ is the wavelength of X-rays.

The average size of $CuNiO_2$ nanoparticles were produced 30, 20 and 45 nm by sol-gel autocombustion, microwave and co-precipitation techniques, respectively. The crystallite percents were calculated for every technique. The crystallite percent of $CuNiO_2$ nanoparticles by using sol-gel, microwave and co-precipitation were 30, 64 and 16 nm, respectively.

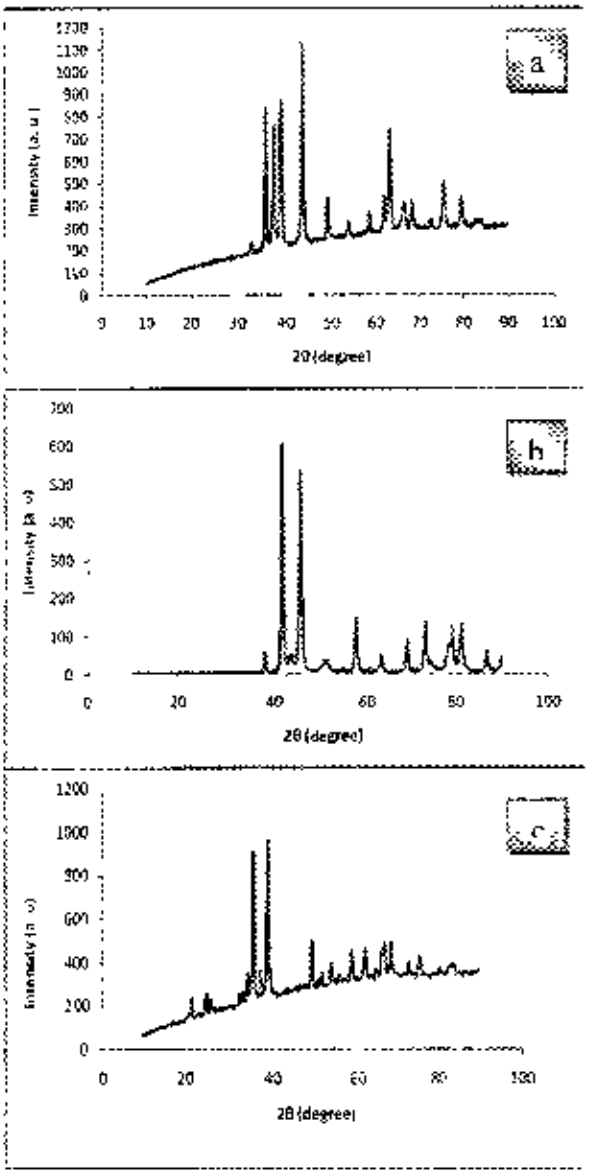

Fig.1. XRD patterns of $CuNiO_2$ nanoparticles prepared by (a) Sol-gel, (b) microwave and (c) co-precipitation techniques.

In sol-gel auto-combustion process, the XRD patterns verified that specimen concluded CuO and NiO phases with monoclinic and cubic structures, respectively.

In microwave method, specimen consisted of crystalline CuO, $Cu(OH)_2.H_2O$, $CuNiO_2$ and NH_4NO_3 phases with monoclinic, triclinic, tetragonal and orthorhombic structures, respectively.

In co-precipitation method, the XRD pattern of $CuNiO_2$ nanoparticles is shown monoclinic and cubic structures of CuO and NiO phases, respectively.

The SEM device was utilized for characterizing of morphology. The homogeneous structures were observed by this instrument.

SEM micrographs reveals changes in microstructure, grain size and structure morphology. The SEM micrographs of as-prepared powders processed with glycine are shown in Fig. 2.1a. The particle size was confirmed by this method. The microwave and co-precipitation SEM micrographs were shown in Fig. 1b and c, respectively.

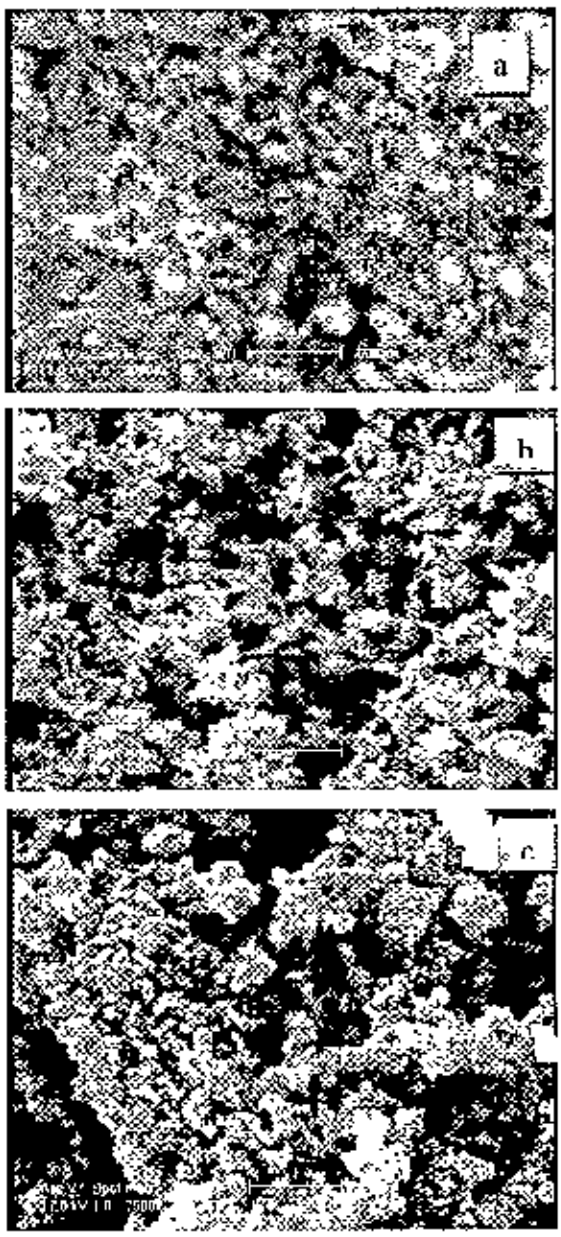

Fig. 2. The SEM micrographs of $CuNiO_2$ nanoparticles prepared by (a) sol-gel, (b) microwave and (c) co-precipitation techniques.

CONCLUSION

The $CuNiO_2$ nanoparticles were synthesized by sol-gel auto-combustion, microwave and co-precipitation methods. In so-gel and microwave methods metal nitrates were used but in the co-precipitation method metal sulfats was used. The XRD patterns was considered on three specimens. The average size nf nanoparticles were calculated by Scherrer equation. The average size of $CuNiO_2$ nanoparticles was produced 30, 20 and 45 nm by sol-gel autocombustion, microwave and co-precipitation techniques, respectively.

The similar phases and lattice structure were exibited in sol-gel and co-precipitation methods, respectively. The smallest particle with highest crystallite percent belonged to microwave technique. In three methods, morphology and structural properties were investigated with Scanning Electron Microscopy (SEM). The results of sol-gel and microwave techniques shown that average size of nanoparticles are the same. The considered co-precipitation method leads to production of $CuNiO_2$ nanoparticles. The achieved size of $CuNiO_2$ nanoparticle corresponded by average size of the XRD results.

REFERENCES

[1] A. S. Waingankar, S. G. Kulkarni. M. S. Sagare, J. Phys., IV (1997) 155-156.

[2] O. H. Kwon, Y. Fukushima, M. Sugimoto, N. Hiratsuka, J. Phys., IV (1997) 165-166.

[3] E. P. Wolhfarth, Ferromagnetic Materials, Vol. II, North Holland, Amsterdam, New York, Oxford, Tokyo, 1980, p. 405

[4] H. Tang, Y. W. Du, Z. Q. Qiu, J. C. Walkar, J. Appl. Phys., 63 (1988) 4105.

[5] L. L. Hench, J. K. West, Chem. Rev. 90 (1990) 33.

[6] X. Jiao, D. Chen, Y. Hu, Mater. Res. Bull. 37 (2002) 1583.

[7] S. Komarneni, M. C. D'Arrigo, C. Leonelli, G. C. Pellacani, H. Katsuki, J. Am. Ceram. Soc. 81 (1998) 3041.

[8] S. Verma, P. A. Joy, Y. B. Khollam, H. S. Potdar, S. B. Deshpande, Mater. Lett. 58 (2004) 1092.

[9] W. Wang, Y. Qi, Y. Ma, X. Li, X. Guo, J Gao, M. Chen, Mater. Chem. Phys., 36 (2004) 192.

[10] J. Azadmanjiri, S. A. Seyyed Ebrahimi, Phys. Status Solidi (c) 1 (2004) 3414.

[11] D. A. Fumo, M. R. Morelli, A. M. Segadaes, Mater. Res. Bull. 31 (1996) 1243.

[12] O. Palchik, J. J Zhu, A. Gedanken, J. Mater. Chem. (2000) 1251.

[13] O. Palchik, S. Avivi, D. Pinkert, A. Gedanken, Nanostruct. Mater., 11 (1999) 415.

[14] A. G. Saskia, Chem. Soc. Rev. 26 (1997)233 and the refereneos eited therein.

[15] P. Patnaik, Dean's Analytical Chemistry Handbook, 2nd ed. McGraw-Hill, 2004.

[16] A. H. Lu, E. L. Salabas and F. Schüth, Angew. Chem., Int. Ed, 46(2007) 1222–1244.