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Fabrication and study of Nickel oxide nanoparticles via low combustion synthesis method using different fuels

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Abstract

Nickel oxide nanoparticles have been successfully synthesized by using low combustion synthesis method followed by calcination at 500 ºC for 30 minutes. The calcined Nickel oxide nanoparticles were investigated by various tools such as XRD, FT-IR and FE-SEM. The average crystallite size of the calcined Nickel oxide was determined to be 6.5, 8.2 and 16.7 nm according to various fuels. The characteristic absorption peak at 429 corresponds to Ni-O stretching vibration mode of Nickel oxide. The FE-SEM photograph displayed that the calcined Nickel oxide nanoparticles are agglomerated in network shapes and the average agglomerated particle Nickel in the range 1 µm.

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1. Introduction

Nickel oxide NiO is an important transition metal oxide with cubic lattice structure. It has attracted increasing attention owing to potential use in a variety of applications such as: catalysis [1], battery cathodes[2, 3], gas sensors [4], electrochromic films [5] and magnetic materials [6, 7]. It can also be extensively used in dye sensitized photocathodes [8]. Also for low material cost as an ion storage material, NiO semiconductor becomes a motivating topic in the new area of research .Because of the volume effect, the quantum size effect, the surface effect and the macroscopic quantum tunnel effect, nanocrystalline NiO is expected to possess many improved properties than those of micrometer-sized NiO particles with advancements in all areas of industry and technology , the interest has been focused on nanoscale materials ,which stems from the fact that new properties are required at this length scale and, equally important, that these properties change with their size and morphology [9, 10] . Recently various synthesis methods have been used for fabrication of inorganic material nanoparticles such hydrothermal method [11-13], sol-gel method [14, 15], co-precipitation method [16, 17], combustion synthesis [18-21] and emulsion combustion method [22, 23] .

Low combustion synthesis method is also known as low solution combustion synthesis and it used for the preparation of simple and complex inorganic compounds [24, 25, 18, 26, 27]. We are used low combustion synthesis method due to easy, rapid method and saves both time and energy. It also used to synthesis pure, homogeneous and crystalline materials. In the present work, we aimed to synthesize nickel oxide nanoparticles by using auto-combustion synthesis following by the calcination to improve the crystallinity. The structure, optical and morphology properties of the obtained nickel oxide are well characterized by different tools such as XRD, FT-IR and SEM.

2. Experimental

2.1. Materials and reagents:

All chemicals used in this work were purchased and used as received without any further purification. Nickel nitrate hexahydrate ($Ni(NO_3)_3.6H_2O$; 98%) was purchased from Sigma–Aldrich Chemical Company. Sucrose (Su: $C_{12}H_{22}O_{11}$; 98%) and alanine (Ala: $C_3H_7NO_2$; 99%) were purchased El Nasr pharmaceutical chemicals company.

2.2 Preparation of Nickel oxide nanoparticles via combustion method

0.02 mole of Nickel nitrate and certain amount of fuels were dissolved in 30 mL distilled water in which the fuel to oxidant ratio equal to one and according to the equations (1, 2 and 3). The obtained solutions were heated with stirring at 100 °C for 5 min to complete the solubility. After homogenization, the solutions were heated at 150ºC till to transform into a gel. The viscous gels were ignited on hotplate at 250 ºC until auto-ignition was finished and the ash was obtained. Pure Nickel oxide nanoparticles obtained after the calcination of ashes materials at 500 ºC for 30 minutes. Table (1) shows the ratio between the starting materials and the names of the products after calcination.

$$6\ \mathrm{Ni}(NO_3)_2.6H_2O + 4\ C_3H_7NO_2 \rightarrow 6\ \mathrm{NiO} + 50\ H_2O + 12\ CO_2 + 8\ N_2 \quad (1)$$

$$48\ \mathrm{Ni}(NO_3)_2.6H_2O + 10\ C_{12}H_{22}O_{11} \rightarrow 48\ \mathrm{NiO} + 398\ H_2O + 120\ CO_2 + 48\ N_2 \quad (2)$$

$$27\ \mathrm{Ni}(NO_3)_2.6H_2O + 2\ C_3H_7NO_2 + 5\ C_{12}H_{22}O_{11} \rightarrow 27\ \mathrm{NiO} + 255\ H_2O + 66\ CO_2 + 28\ N_2 \quad (3)$$

Table (1): The sample names, fuels type and the composition of the starting materials.

N	Sample names	Ni^{2+}, mole	Type of fuel	Fuel, mole
1	NA	0.03	Ala	0.02
2	NS	0.03	Su	0.00625
3	NSA	0.03	Ala and Su	0.01+ 0.003125
Where: Ala=alanine and Su= sucrose				

2.3 Characterization of NiO nanoparticles

X-ray diffraction (XRD) of the sample was measured using diffractometer (Bruker; model D8 advance) with monochromnated Cu-Ka radiation, 1.54178 (˚A) in the 2θ range of 15-80º. FT-IR spectra were measured using FT-IR spectrometer (Bomem; model MB 157S) from 4000 to 400 cm^{-1} at room temperature. The morphology and particle size of sample was studied using Field emission scanning electron microscope (FE-SEM, JEOL: model JSM-6500F).

2.3.1X-ray diffraction (XRD)

Figure (1) displays the XRD patterns of the calcined Nickel oxide samples (NA, NS and NSA). The extracted data shows that the pure NiO phase in case of the NA and NSA samples according to standard JCPDS card No. 00-047-1049. NS sample show nickel with high crystallinity. The average crystal sizes of zinc oxide calculated by using the Debye-Scherrer formula.

$$B = 0.9\lambda/D_{1/2}\cos\theta$$

Where, λ is wavelength (1.5406 A for CuKα), θ is the diffraction angle and β is the x-ray full width at half-maximum height of the diffraction peak. The average crystal size of the calcined Nickel oxide is determined to be 6.5, 16.7 and 8.2 nm for NA, NS and NSA samples, respectively.

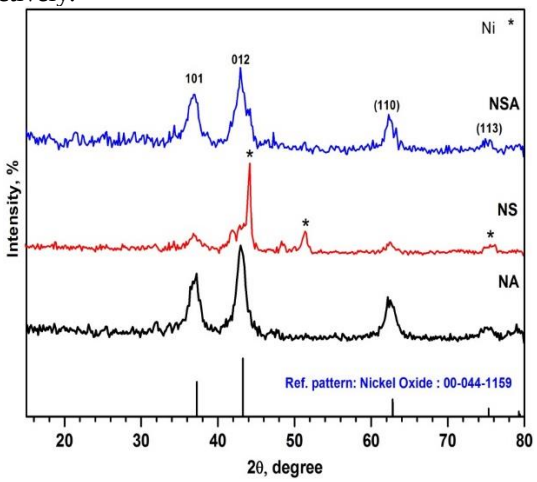

Figure (1). XRD patterns of Nickel oxide after calcination at 500 ºC for 30 minutes

2.3.2 FT-IR analysis

The FT-IR spectra of the calcined Nickel oxide samples in the wavenumber range of 400-4000 cm^{-1} are shown in Figure (2). The peak around at 3451 cm^{-1}, 3428 cm^{-1} and 3421 cm^{-1} are related to O-H vibration of water molecules on the surface of nickel oxide nanoparticles. The absorption at 1650 cm^{-1}, 1643 cm^{-1} and 1673 cm^{-1} correspond to the bending vibration of hydroxyl groups which adsorbed on the surface of nickel oxide. The absorption bonds at 1383 cm^{-1}, 1432-1446 cm^{-1}, 1271-1267 cm^{-1} and 1112(7) cm^{-1} indicates the existence of nitrates, carbon-carbon and carbon-oxygen groups. The absorption bonds at 418-420 cm^{-1} and 440 cm^{-1} are associated to Ni-O vibration bond and absorption bond at 620 cm^{-1} and 640 cm^{-1} is assigned to Ni-O-H stretching bond on the Nickel. The extracted data confirmed formation of the pure NiO nanoparticles [28, 29].

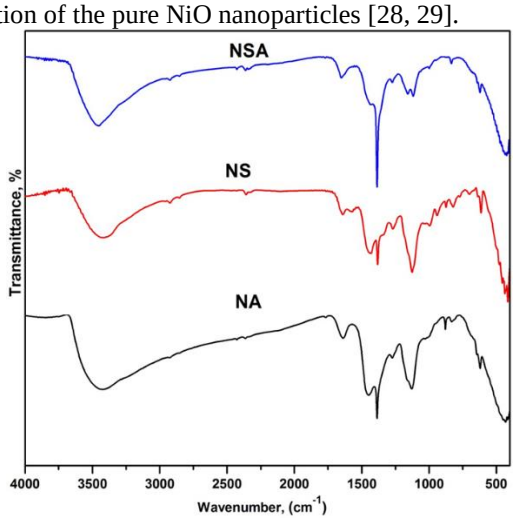

Figure (2). FT-IR spectra of NiO oxide samples synthesized by different fuels.

2.3.3 Morphological studies for Nickel Oxide nanoparticles

FE-SEM images of the synthesized NA sample are shown in Figure (3). FE-SEM micrograph exhibit the high porosity of Nickel oxide nanoparticles with irregular network shap and average particle size in the range of 1 μm.

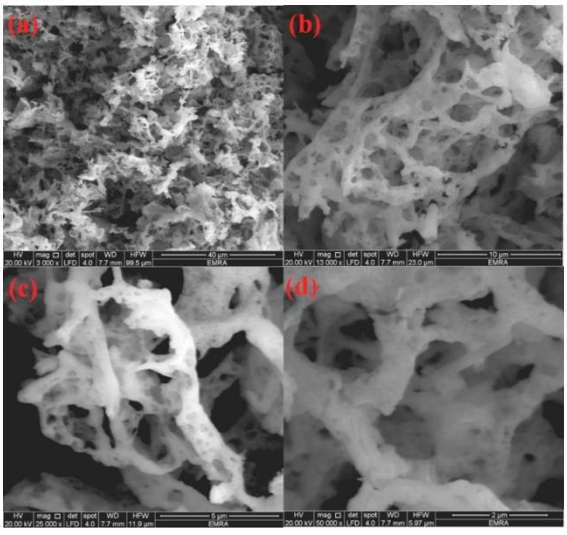

Fig.3. FE-SEM images of nickel oxide (NA) synthesized by using alanine fuel.

4. Conclusion

NiO nanoparticles were prepared by auto-combustion method using nickel nitrate (as an oxidizer), alanine fuel and sucrose fuel with the fuel to oxidant molar ratio is equal to 1. The obtained powder sample was characterized by using various tools such as X-ray powder diffraction (XRD), Fourier transform infrared analysis (FTIR) and Field emission scanning electron microscope (FE.SEM). The XRD pattern of Nickel oxide sample shows the characteristic peaks of after calcination at 500^{o} C with an average crystallite size of 6.5-16.7 nm. The absorption peak at 429 cm^{-1} is attributed to Ni-O stretching of NiO. The FE-SEM photograph displayed that the synthesized nickel oxide nanoparticles show the high porosity material with network shapes and hard agglomeration. Also, the FE-SEM photograph displays the average particle size in the range of 1 μm.

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