

The Effect of Replacement of Zn²⁺ Cation with Ni²⁺ Cation on the Structural Properties of Ba₂Zn_{1-x}Ni_xWO₆ Double Perovskite Oxides (X = 0, 0.25, 0.50, 0.75, 1)

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Abstract

The Ba₂Zn_{1-x}Ni_xWO₆ double perovskite oxides were synthesized using solid state reaction method. The effect of replacement of Zn²⁺ with Ni²⁺ cation on the structural properties was investigated by X-ray diffraction (XRD) at room temperature. From the X-ray diffraction and by means of standard Rietiveld method, the samples showed the same cubic crystal structure with (Fm-3m) space group and the crystallite size ranging from 71.91 nm to 148.71 nm. The unit cell volume was found to decrease as a result of the replacement, while there was no significant difference in the value of tolerance factor of the samples. This is may be due to the convergence of ionic radii of Ni²⁺ and Zn²⁺ cations. The Fourier Transform Infrared Spectroscopy (FTIR) was performed for the samples and the resultant characteristic absorption bands confirmed the double perovskite structure.

Keywords

Double Perovskite, XRD, FTIR, Solid State Reaction Method, Tolerance Factor

1. Introduction

Many authors were interested in the study and preparation of the new double perovskite materials. The general chemical formula of double perovskite oxides was expressed as $A_2BB'O_6$, where A is occupied by an element of group one or group two such as Ca, Sr or Ba and B, B' site is occupied by a translational element, while the O

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atom is located in between, forming the alternate BO_6 octahedral and B-O-B bonds. The wide variety of double perovskite materials is due to the alternation of the magnetic and non-magnetic B and B' elements [1] [2]. In particular, the compositions A_2BMOO_6 , where B = Fe, Mn or Cr are magnetic ions, are currently studied for their potentiality as magnetoresistive and thermoelectric systems [3].

Synthesize of $Sr_2CaW_xMo_{1-x}O_6$ double perovskite series by solid state reaction was carried out by Zhou *et al.* [4]. The self-activated host material emits strong blue lights at round 435 nm and 468 nm, while the excitation emits at or near UV at about 290 nm due to W-O charge transfer transition. Tian et al. [5] studied the crystal structure and magnetic properties of Sr_2MWO_6 (M = Co or Ni), using sol-gel method, showing tetragonal (I4/m) structure and homogeneous grain size. The magnetic parameters of the material refer to paramagnetic behavior. The magnetic momentum originates mainly from the interaction between the Ni²⁺ ions and Co²⁺ ions in Sr₂NiWO₆, Sr₂CoWO₆ respectively. Manoun et al. [6] investigated the effect of temperature in the structure of Sr₂ZnWO₆ and Sr₂CoWO₆ double perovskite oxides when characterized by Raman spectroscopy. The first phase transition from monoclinic (P21/n) crystal structure to tetragonal (I4/m) crystal structure occurred at 80°C, while the second transition to cubic (Fm-3m) occurred at 480°C. The effect of the preparation methods on the catalytic activity of the LaSrFeMo_{0.9}Co_{0.1}O₆ methane combustion double perovskite oxide was studied by Zheng *et al.* [7] using two methods; co-precipitation and sol-gel. The sol-gel method had greater impacts on the catalytic activity than co-precipita- tion method. The Ca_2NiWO_6 double perovskite oxide structure and magnetic behavior at 1150°C with P21/n space group monoclinic crystal structure was investigated by Lopez et al. [8]. Ba₂MgWO₆ and Ba_2ZnWO_6 double perovskite oxides were synthesized using the solid state method by Bugaris *et al.* [9]. They characterized the samples by single-crystal X-ray diffraction, Neutron diffraction, UV-visible spectrometry and LS 55 Fluorescence Spectrometer. The cubic (Fm-3m) crystal structure was obtained.

In this work, the $Ba_2Zn_{x-1}Ni_xWO_6$ double perovskite oxides were synthesized using solid state reaction method. The aim was to investigate the effect of replacement of Zn^{2+} cation with Ni^{2+} cation. The X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) were used in order to explore the effect of replacement on the structural properties of $Ba_2Zn_{x-1}Ni_xWO_6$ double perovskite oxides.

2. Experimental

The new samples of double perovskite oxides were synthesized using solid state interaction method and many different treatments were applied in order to get single phase for the samples. The samples were prepared by mixing stoichiometric amounts of ZnO, NiO, WO₃ and BaCO₃ and these chemicals were purchased from Alfa Acer of purity 99.9% in order to prepare the $Ba_2Zn_{1-x}Ni_xWO_6$ double perovskite oxides. The mixtures of compounds grinded in agate mortar with the addition of acetone and then kept in crucibles and heated in air at 800°C for 12 hours. The samples pellet in around shape and heated in at 1000°C and 1200°C, respectively. Every step of heating treatment was repeated twice with the rate of 10°C per minute during the heating process and coaling as well. The compound permeated every time grinding with the addition of acetone. The ratio of the amounts calculated was by the following equation:

$$2\text{BaCO}_{3} + (1-x)\text{ZnO} + (x)\text{NiO} + \text{WO}_{3} \rightarrow \text{Ba}_{2}\text{Zn}_{1-x}\text{Ni}_{x}\text{WO}_{6} + 2\text{CO}_{2} \uparrow$$
(1)

The X-ray diffractometer (Model Bruker D-8) using Cu-K_{α} radiation ($\lambda = 1.5406$ Å) and a nickel filter operating at 40 KV and 40 mA at scanning the samples was used at room temperature. The data were collected for the 2 θ range 20° - 80° at step size of 0.02 and count time of 5 s. The collected data were then fed to full Prof suite [10] for determination of the lattice parameters, space group, atom's positions and crystalline size (*D*) using Scherer equation [11].

$$D = \frac{0.94\lambda}{\beta_{1/2}\cos^{(\theta)}} \tag{2}$$

where D is crystallite size, λ is the wavelength of X-ray and $\beta_{1/2}$ is the half full width of the peaks.

The tolerance factor [12] [13] for the samples calculated by

$$t = \frac{(r_A + r_o)}{\sqrt{2} \left(\frac{(1-x)}{2} r_B + \left(\frac{x}{2}\right) r_B' + \frac{1}{2} r_B'' + r_o \right)}$$
(3)

A Fourier transform infrared spectroscopy (FTIR) spectrum was recorded in transmittance mode at room temperature using KBr pellet method, the samples were diluted in KBr of ratio 1:100 for FTIR measurement between 400 and 2000 cm⁻¹ the FTIR of samples were carried out with a (Satellite FTIR 5000 of the wavelength range of 400 to 4000 cm⁻¹) [14] where the characteristic bands and peaks of perovskite structure can be assigned.

3. Results and Discussion

3.1. The XRD Results

The study and determination of the structural properties of perovskite oxide compounds are very important; such as lattice parameter, space group, type of crystal, atomic position, and molecular binding because they predict and expect the electrical, optical and magnetic properties of materials [15]. The X-ray powder diffraction obtained for $Ba_2Zn_{1-x}Ni_xWO_6$ where (x = 0, 0.25, 0.50, 0.75, 1) double perovskite series prepared by the solid state reaction is shown in **Figure 1**. The difference in lattice parameters for each sample is shown by the different peaks position in **Figure 2**. The shift in reflection positions is referred to the differences in lattice parameters of unit cells of the samples. The peaks appeared at low intensity in the XRD pattern shown in **Figure 1** are attributed to (Ba_2WO_5/Ba_2WO_4) impurities following Manoun *et al.* [16].

3.2. XRD Refined Result

The XRD data of each sample of series are refined by Retiveld method. All samples obtained are showed cubic structure with (Fm-3m) space group. **Table 1** shows the atom positions, the space groups and the lattice parameters of the samples. **Figures 3(a)-(e)** show the refined XRD patterns of the Ba₂Zn_{1-x}Ni_xWO₆ double perovskite series where x = 0, 0.25, 0.50, 0.75, 1. In the case of **Figure 3(c)**, the XRD refinement of Ba₂Zn_{0.50}Ni_{0.50}WO₆ is shown, which represents (Fm-3m) cubic structure with a = b = c8.097554 Å, $\alpha = \beta = \gamma$ 90 lattice parameters with x = 0.50. It is clear that the shift is dependent on the ratio of replacement. Bugaris *et al.* [9] found similar results



Figure 1. X-ray powder diffraction of $Ba_2Zn_{1-x}Ni_xWO_6$ double perovskite oxides series.



Table 1. The atom positions, the space groups, the lattice parameter and crystallite size of the $Ba_2Zn_{1-x}Ni_xWO_6$ double perovskite series where (x = 0, 0.25, 0.50, 0.75, 1).

Element	Coordinates	Ba ₂ ZnWO ₆	$Ba_2Zn_{0.75}Ni_{0.25}WO_6$	$Ba_2Zn_{0.50}Ni_{0.50}WO_6$	$Ba_2Zn_{0.25}Ni_{0.75}WO_6$	Ba ₂ NiWO ₆
		Fm-3m	Fm-3m	Fm-3m	Fm-3m	Fm-3m
Ba_1^{+2}/Ba_2^{+2}	Х	0.2500	0.2500	0.2500	0.2500	0.2500
	Y	0.2500	0.2500	0.2500	0.2500	0.2500
	Z	0.2500	0.2500	0.2500	0.2500	0.2500
Zn^{+2}	Х	0.5000	0.5000	0.5000	0.5000	
	Y	0.5000	0.5000	0.5000	0.5000	
	Z	0.5000	0.5000	0.5000	0.5000	
Ni ⁺²	Х		0.5000	0.5000	0.5000	0.5000
	Y		0.5000	0.5000	0.5000	0.5000
	Z		0.5000	0.5000	0.5000	0.5000
W^{+6}	Х	0.0000	0.0000	0.0000	0.0000	0.0000
	Y	0.0000	0.0000	0.0000	0.0000	0.0000
	Z	0.0000	0.0000	0.0000	0.0000	0.0000
O_1^{-2} / O_2^{-2}	Х	0.2399	0.23697	0.21735	0.23495	0.23317
	Y	0.0000	0.0000	0.0000	0.0000	0.0000
	Z	0.0000	0.0000	0.0000	0.0000	0.0000
a/b/c (nm)		8.118340	8.108754	8.097554	8.082387	8.080683
$\alpha/\beta/\gamma$		90	90	90	90	90
V (nm ³)		535.0590	533.166	530.960	527.982	527.648
Crystallite size (nm)		105.09	126.77	110.53	71.91	148.71
Tolerance factor		1.008	1.01	1.014	1.017	1.02







Figure 3. (a) Refined XRD patterns of the Ba₂ZnWO₆ sample; (b) Refined XRD patterns of the Ba₂Zn_{0.75}Ni_{0.25} WO₆ sample; (c) Refined XRD patterns of the Ba₂Zn_{0.50}Ni_{0.50} WO₆ sample; (d) Refined XRD patterns of the Ba₂Zn_{0.25}Ni_{0.75} WO₆ sample; (e) Refined XRD pattern of the Ba₂NiWO₆ sample.

for the Ba₂MWO₆ double perovskite samples where (M = Zn, Mg) using the Single-crystal X-ray diffraction and Neutron diffraction. Furthermore, Y. Tamraoui *et al.* [17] found the similar structure (Fm-3m) cubic structure for Ba_{2-x}Sr_xMgTeO₆ series where x lies between 0 and 1.5.

From X-ray result, the cubic structure is confirmed for all samples so the Ba-cation is surrounding by 12 oxygen anions in a regular dodecahedral environment and the Zn-cation, Ni-cation, W-cation is octahedral coordinated by six oxygen ions, giving rise of 180° B-O-B bond angles [18] [19]. The crystallite size for $Ba_2Zn_{1-x}Ni_xWO_6$ double perovskite series is calculated from full-width at half- maximum (FWHM) using Scherer formula for the major peaks at (220) that observed to vary between 71.91 to 148.71 nm for the samples. The tolerance factor is calculated and is found to be between (1.008 - 1.020) as shown in **Table 1**. This is confirmed the (Fm-3m) cubic structure according to the criteria adopted by Correa *et al.* and Popov *et al.* [12] [13]. **Figure 4(a)** and **Figure 4(b)** show the relation between the tolerance factor and volume of unit cell respectively with ratio of replacement. The tolerance factor of the samples is found to take almost the same value. This may be attributed to the same values of ionic raduii of Ni²⁺ and Zn²⁺ cations. D. Serrate *et al.* [20] verified the rule of double perovskite tolerance factor for the (Fm-3m) cubic structure to be between 1.05 - 1.00. The volume of unit cells is found to decrease with the increasing ratio of replacement ofZn²⁺ cations by Ni²⁺ cations. This is may be due to the size of Zn²⁺ cations being larger than the size of Ni²⁺ cations.

3.3. The IR Spectroscopy Results

The FTIR spectra of the perovskite structure have three characteristic absorption bands between 850 - 400 cm⁻¹, respective to composition and these are usually used to identify the perovskite phase formation [21]. It is based on infrared spectrum can be used for molecules such as fingerprint for humans [22]. The strong high-energy band centered at about 620 cm⁻¹ can be assigned to the anti-symmetric stretching mode of the (Zn-O₆), (Ni-O₆) and (W-O₆) octahedral is due to the higher charge of this cation. Another interesting point appear by this spectra

is the presence of the high intensity band at about 825 cm⁻¹ which can be assigned to the symmetric stretching vibration of these octahedral as explain. The positions of these bands suggests relatively long (Zn-O₆), (Ni-O₆) and (W-O₆) bands [23] [24]. The bands appeared around 420 cm⁻¹ are may be due to deformational modes of theZnO₆, NiO₆ and WO₆ octahedrals. From **Figure 5** that shows the transmittance of Ba₂Zn_{1-x}Ni_xWO₆ double perovskite series versus wave number; all the samples confirmed the molecular bands on the form perovskite oxide [24]. The peak between 1450 - 1400 cm⁻¹ is likely corresponds to overtones of the fundamental vibrations of carbonate [25] [26].







Figure 5. The IR spectra of theBa₂Zn_{1-x}Ni_xWO₆ double perovskite oxides.

4. Conclusion

The $Ba_2Zn_{1-x}Ni_xWO_6$ double perovskite series, where x = 0, 0.25, 0.50, 0.75, 1, were synthesized using solid state method. The X-ray diffraction (XRD) and the Fourier Transform Infrared Spectroscopy (FTIR) were used as analytical tools. The XRD patterns were measured at room temperature for the series and showed the same cubic structure before and after the replacement of Zn^{2+} cation by Ni²⁺. As a result of a convergence of the ionic radius of the Zn^{2+} cation and Ni²⁺ cation, the unit cell volume was found to decrease from 535.059 nm³ to 527.648 nm³, while the values of tolerance factor were found to slightly increase from 1.008 to 1.02. FTIR absorption bands were found to be characteristic of perovskite structure.

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