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Synthesis and characterization of double phase metal nickelates/borates

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ABSTRACT

Purpose: Purpose of this research is obtaining of metal nickelates and borates as double phase. These types of compounds display extraordinary structural diversity caused by boron and nickel which applicable in flame retardant, detergent, ceramic and such industries.

Design/methodology/approach: Double phase metal nickelates and borates were weighed in an appropriate molar ratio and homogenized in an agate mortar. The mixture placed into a porcelain crucible to heat in conventional high temperature furnace. After the material was exposed to heat treatment at 900°C for 4 hours, and cooled down to room temperature with many grindings. Then, final products were analyzed by powder X-ray Diffractometer (XRD) using PANanalytical X' Pert PRO Diffractometer (XRD) with Cu Kα (1.5406 Å, 45 kV and 30 mA) radiation. Fourier transform infrared spectroscopy (FTIR) was achieved on a Perkin Elmer Spectrum 100 FTIR Spectrometer from 4000 to 650 cm^{-1}. Scanning electron microscopy was achieved in SEM JEOL 6390-LV. Luminescence properties were performed by Andor Solis Sr 500i spectrophotometer. Conventional solid state syntheses were done in Protherm furnace.

Findings: $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds as double phase were identified by powder XRD patterns and phase analysis of these compounds were completed by High Score Plus program. Vibrations of B-O and Ni-O bonds of functional groups were determined in FTIR spectrum benefiting from literature.

Research limitations/implications: Implication of the synthesis method has some disadvantages such as low homogeneity, non-uniform product etc. We tried to minimize these negative aspects in our research and succeeded.

Practical implications: Double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds were synthesized by high temperature solid state synthesis route. Structural properties and phase composition analysis were realized using powder X-ray diffraction patterns.

Originality/value: Value of the paper is first time conventional synthesis of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds, characterization of the structures, and investigation of morphological and luminescent properties.

Keywords: Nickelates; Borates; X-ray diffraction; Solid state chemistry; High score plus program

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MATERIALS

1. Introduction

After the investigation of high superconductivity of the systems containing d and f transition metal compounds have drawn much attention with high critical temperature (T_c) [1,2]. The mixed metal oxides in a perovskite related structure with high stability have been investigated in detail to applicable in various industrial areas such as electrically, magnetically, and catalytically. Thus, these types of compounds are good for their application in new technologies [3-7]. The methods which reduce the diffusion distances for the incorporation of cations in the polymeric matrix is preferred to obtain such properties, in the meantime there are several methods not to reach these properties. Pechini method [8], sol-gel process [7], and the polymeric precursor method [9] are some of the methods used to obtain powders with high homogeneity, purity, uniform distribution of phases, lower processing temperature and possibility of rolling particle size [10]. Conversely, microwave method, hydrothermal method and chemical vapor deposition methods are inadequate to obtain good features [11].

Structural chemistry of metal borates is commonly studied because of their stoichiometry, phase relations geochemical complexity and technological importance. It is known that there is mononuclear, bi-, tri-, tetra- or pentanuclear multidimensional network included glasses in the structural unit of borates. $M^{III}BO_3$, $CaSn(BO_3)_2$ and $Mg_3(BO_3)_2$ compounds and minerals are rare examples containing monomeric triangle BO_3 units. Binuclear plane triangle presents in $Mg_2B_2O_5$, $Co_2B_2O_5$ and $Fe_2B_2O_5$ polyborates. Monomeric tetrahedral BO_4 unit exist in $TaPO_4$ zirconia type compound, $TaNbBO_4$ and $Ca_2H_4BaSO_8$ minerals. Tetrahedral $[B(OH)_4]^-$ unit is formed at $Na_2[B(OH)_4]Cl$ and $Cu_2[B(OH)_4]Cl$. Binuclear tetrahedral units exist at $Mg[B_2O(OH)_6]$, cyclic binuclear structure at $NaBO_3.4H_2O$. A complex polynuclear structure, which is formed by compressing of layers of $BO_3(OH)$ units coordinated as tetrahedral, exists at $CaB(OH)SiO_4$ [12].

In this paper $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ were prepared by standard solid-state reaction and phase compositions these compounds were analyzed by Rietveld Refinement Method by using X-ray powder diffraction data. Morphological and thermal properties support the formation of double phases and stability.

2. Material and methods

Double phase metal nickelates and borates were weighed in an appropriate molar ratio and homogenized in an agate mortar. The mixture placed into a porcelain crucible to heat in conventional high temperature furnace. After the material was exposed to heat treatment at 900°C for 4 hours, and cooled down to room temperature with many grindings. Then, final products were analyzed by powder X-ray Diffractometer (XRD) using PANanalytical X' Pert PRO Diffractometer (XRD) with Cu Kα (1.5406 Å, 45 kV and 30 mA) radiation. Scanning electron microscopy were achieved in SEM JEOL 6390-LV. Perkin Elmer thermogravimetric analyzer was used to determine thermal behaviour of the compound.

3. Results and discussion

In Figure 1, the XRD patterns of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds are displayed. The comparison results of the XRD patterns with database, they are coupling with $Ni_3B_2O_6$ (ICSD: 00-2016)/YBO_3 (ICSD: 10-9264) and $MoNiO_4$ (ICSD: 08-1059)/$Ni_3B_2O_6$ (ICSD: 00-2016) compounds as double phases. While $Ni_3B_2O_6$ has been marked as '‖' in both samples; YBO_3 has been marked as '*' and $MoNiO_4$ has been displayed as 'x'. The phase compositions ratios of $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds were calculated by Rietveld Refinement Program as %78.5/21.5 and %89.7/10.3, respectively.

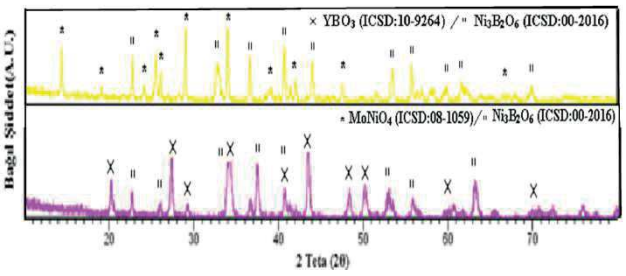

Fig. 1. The XRD patterns of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds

Figure 2 exhibits SEM micrographs of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds. The images are displayed heterogeneous distributions with average particle size 2-5 μm in both samples because of double phases.

The thermograms of the results of thermogravimetric analysis of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds are given in Figure 3. In the thermogram of $Ni_3B_2O_6/YBO_3$ double phase only one mass loss about 900°C shows the high stability of both compounds. On the other hand, in the thermogram of $MoNiO_4/Ni_3B_2O_6$ double phase only one mass loss about 1000°C shows the high stability of both compounds.

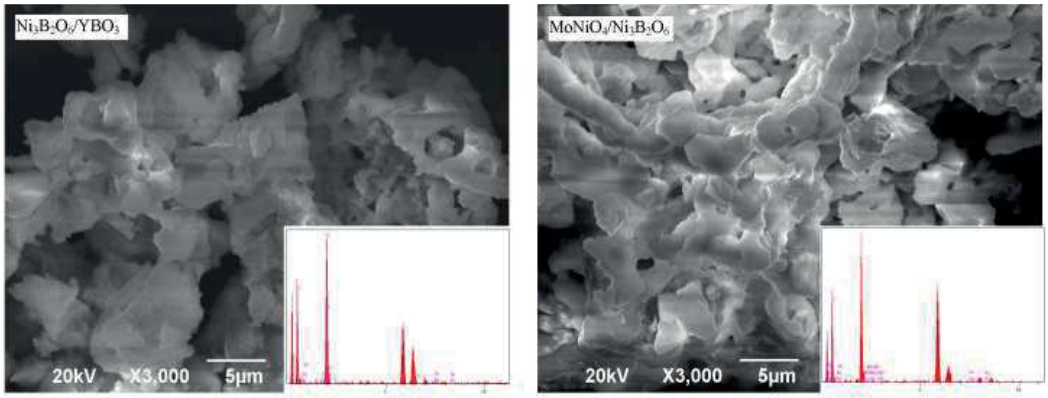

Fig. 2. SEM micrographs of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds

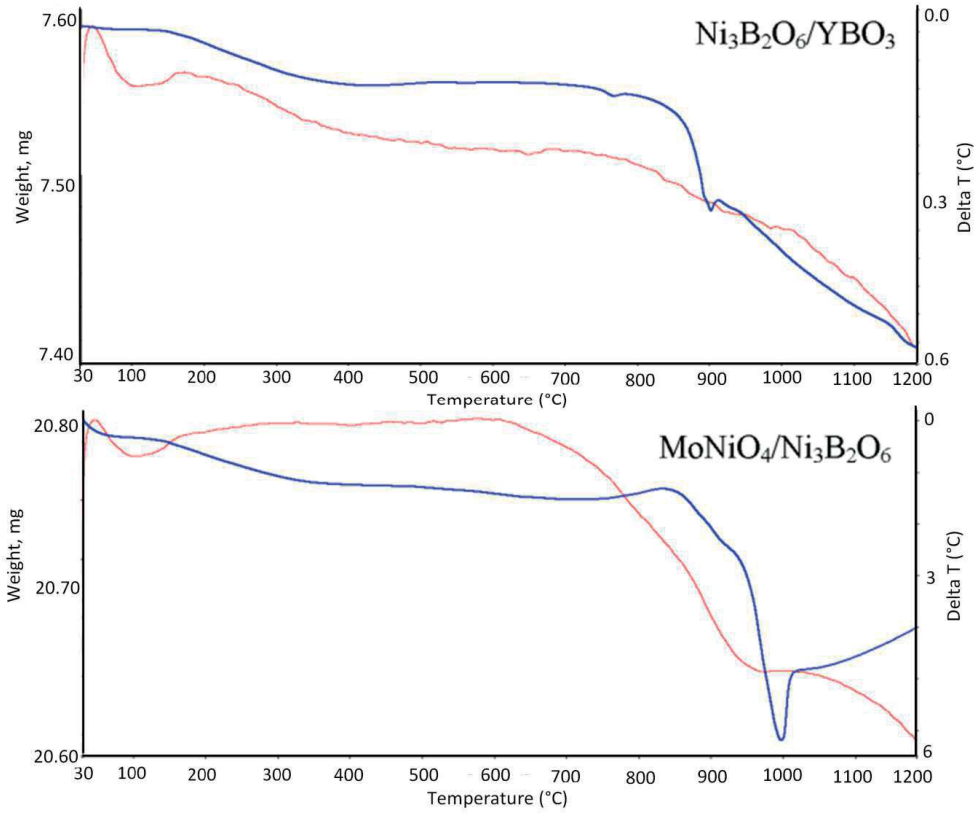

Fig. 3. Thermograms of double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds

4. Conclusions

Double phase $Ni_3B_2O_6/YBO_3$ and $MoNiO_4/Ni_3B_2O_6$ compounds were synthesized by high temperature solid state synthesis route. Structural properties and phase composition analysis were realized using powder X-ray diffraction patterns.

Acknowledgements

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