

Original Article

Solid State Synthesis of LiNiO₂

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E-mail: arun_lagashetty@yahoo.com**Keywords:**Bimetallic oxide,
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Bonding,
Structure,
Morphology**Abstract**

Bimetallic oxide nanomaterials are synthesized by solid state combustion route integrates the synthetic chemistry. Simply, burning of two metal oxides in presence of polymer fuel for its bimetallic oxide nanomaterials. Nanosized lithium niobate material is prepared by self-propagating combustion methods using polymer as a fuel. Lithium oxide and nickel oxide with polyvinyl alcohol was ignited in an open atmosphere for few minutes to form lithium niobate residue. As prepared lithium niobate sample was well characterized for its structure by employing powder X-ray diffraction (XRD) tool. The morphology of as prepared lithium niobate material was studied by Scanning Electron Micrograph (SEM) tool. Fourier Transform infrared (FTIR) spectral study was undertaken to know the bonding in the prepared bimetallic oxide sample.

1. Introduction

Synthesis of nano sized bimetallic oxide materials by combustion method is attracted much to the researchers because of its simplicity. Lithium nickelate is a cheaper and exhibit a higher specific capacity [1-2]. It exhibits electrochemical features as cathode in lithium batteries. LiNiO₂ is a transition metal ion surrounded by oxygen atom shows octahedral sites. LiNiO₂ phase has rhombohedral structures and the parameters of the unit cell are usually defined in terms of the hexagonal setting [3-4]. The way of preparation counts lot to obtain stoichiometric layer of lithium nickelate [5-6]. The structures and electrochemical properties of LiNiO₂ strongly depend upon the conditions of synthesis. Proper processing methods are necessary to obtain LiNiO₂ at high temperature. Solid state reaction by grinding and calcinations produces the perfect phase using polymer fuel [7-8].

Lithium nickelate nanomaterials is prepared by using lithium oxide and nickel oxide employing solid state combustion route is reported in the present work. Polyvinyl alcohol is used as a fuel for the conversion of nickel and lithium oxides in to its lithium nickelate nanomaterials. Combustion treatment is given for the complete conversion of metal oxides in to bimetallic oxide nanomaterials. The prepared sample is well characterized for its structure by X-ray diffraction (XRD), morphology by Scanning Electron Microscope (SEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques.

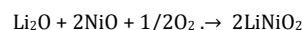
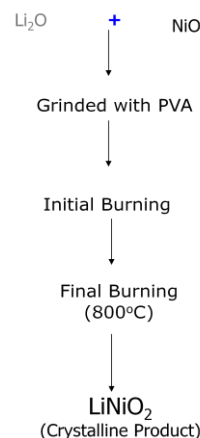
2. Experimental**2.1. Synthesis of Lithium nickelate****2.1.1 Materials and methods**

Lithium oxide, Nickel oxide and polyvinyl alcohol chemicals are used in the present study were of AR (Analytical Reagent) grade. Polyvinyl alcohol is used as fuel for the reaction and solid state combustion method is adopted for the synthesis of lithium nickelate nanomaterials

2.1.2. Preparation of Lithium nickelate nanomaterials

The lithium nickelate is prepared by igniting lithium oxide and nickel oxide with a polymer fuel. Equimolar quantity of lithium oxide and nickel oxide was grinded well with polyvinyl alcohol in the ratio of 1:1:5 in a pestle and mortar [9-10]. The resultant mixture was transferred into a crucible and ignited in an electrical oven to get the

desired product. Initially reaction mixture was frothed and completes the evolution of large volume of gases. Continue the heat treatment till the crystalline product. During burning the approximate temperature of the reaction may be around 800°C, the reaction mixture burns and leaving behind a solid lithium niobate sample. Following is the possible reaction taking place in the above process and the synthetic scheme is given in scheme-1

**Scheme-1****Synthesis of LiNiO₂****2.1.3. Characterization**

The structures of as prepared lithium niobate were studied by X-ray diffraction using Phillips X-ray diffractometer (PW3710) with Cu K α as source of radiation. Morphology and bonding of the above oxide was studied by Phillips XL 30 ESEM and Perkin-Elmer 1600 spectrophotometer in KBr medium tools respectively.

3. Results and Discussion

3.1. X-ray diffraction

Figure-1 shows indexed XRD pattern of combustion derived LiNiO₂ sample. The pattern shows the presence of some Bragg's reflections confirm the formation of crystalline product. The d-spacing value of the sample matches well with standard data [11]. Unit cell parameters were obtained by least square refinement of the powder XRD data. This study reveals that the sample is monophasic with cubic spinal structure having nanosized particles.

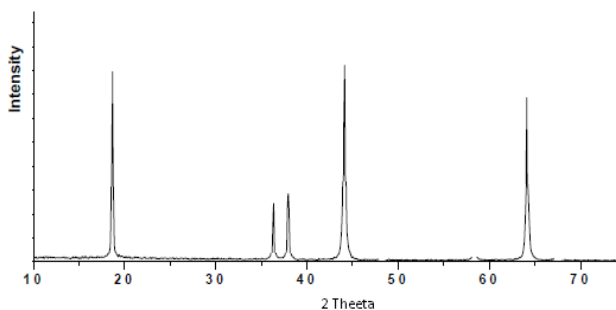


Figure-1: XRD pattern of LiNiO₂ sample

3.2. Scanning Electron Microscopy (SEM)

Figure-2 shows SEM image of as prepared lithium nickelate sample. This image shows, the most of the particles are spherical in shape and self assembled compact structure. In addition to this some particles are irregular shape with a self assembled arrangement due to crystalline behavior.

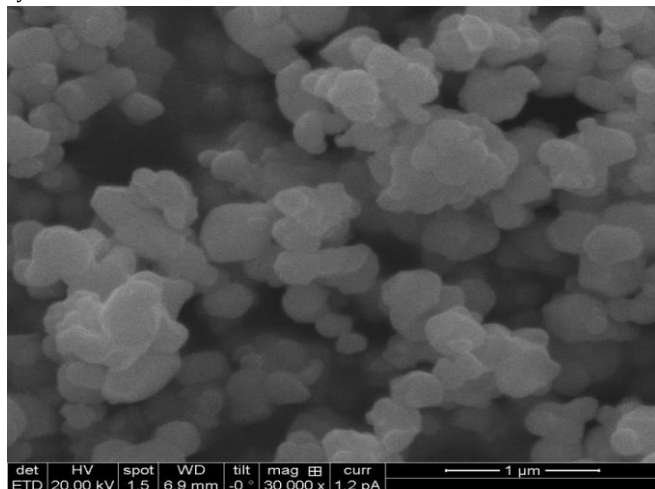


Figure-2: SEM image of LiNiO₂ sample

3.3. Infrared Study

Figure 3 shows FT-IR spectrum of as prepared lithium nickelate sample. The metal-oxygen bonding and nature of the synthesized lithium nickelate sample was carried out by infrared study. Metal oxides generally give absorption bands below 1000cm⁻¹ arising from inter-atomic vibrations [12]. The peak 3400 cm⁻¹ corresponds to water of absorption and the peak at 1700 cm⁻¹ may be the presence of carbon dioxide. Vibrational frequency at 1150 cm⁻¹ due to the presence of some overtones. Peaks below 1000 cm⁻¹ corresponds to Metal-oxygen vibrational modes of the sample conform the formation of lithium nickelate.

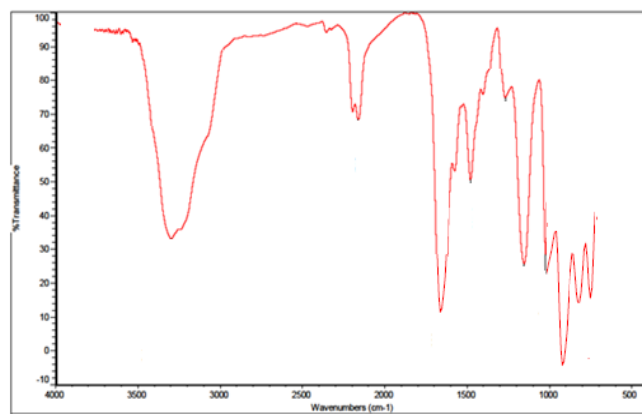


Figure-3: FT-IR spectrum of LiNiO₂ sample

4. Conclusions

The lithium nickelate is prepared successfully by combustion method using polyvinyl alcohol as a fuel. This method finds its importance because of its simplicity. Hence, this method can also adopt for the synthesis of other metal oxide materials at nano dimensions. Single metal oxides are converted in to bimetallic oxide materials with applicable morphology.

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