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Original Research Paper

Rapid synthesis of nickel oxide nanorods and its applications in catalysis

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ABSTRACT

This work reports a rapid, efficient and economical synthesis of nickel oxide nanorods *via* microwave assisted method. The synthesis of nickel oxide nanorods has been achieved by using nickel (II) acetate as a precursor in 1,4-butanediol as a solvent under microwave irradiation within few minutes eliminating the need of conventionally used fuels and capping agents. It was observed that 1,4-butanediol plays multiple roles in the synthesis such as solvent, reactant, promoter and capping agent. The prepared nickel oxide nanorods were characterized using different techniques like XRD, FEG-SEM, EDS, TEM and FT-IR. Furthermore the application of synthesized NiO nanorods as a catalyst for the synthesis of substituted benzimidazole, benzoxazole and benzothiazole has been studied. Excellent yields of respective products under mild reaction conditions suggest a big potential for the synthesized NiO nanorods.

1. Introduction

Metal and metal oxide nanomaterials have received considerable attention due to their interesting size dependent optical, electronic, magnetic, and catalytic properties as compared to their bulk counterparts. During the past decade, several efforts have been made towards the preparation of nanomaterials with an objective of size and shape selectivity, elimination of hazardous chemicals in the synthetic protocols with scalability and simple workup procedures [1].

Nickel oxide (NiO) nanoparticles are important materials which are extensively used for various applications such as electrochemical, antibacterial [2], magnetic [3], antioxidant in the biosystem [4] and catalytic decomposer [5]. These nanoparticles also show a broad range of potential applications in the field of nanoscience e.g. as anode material for lithium-ion batteries, in electrochemical supercapacitors, in dye-sensitized photocathode and in catalytic reaction [6]. Additionally this material is also useful in waste–water treatment to remove the various pollutants [7]. NiO nanoparticles show p-type semiconductor activity because of the wide band gap i.e. 3.6–4.0 eV [8]. There are various reports on the synthesis of NiO nanomaterials with different size and morphology using microemulsion technique [9,10], thermal decomposition [11,12], wet chemical route [13], hydrothermal method [14], sol–gel route [15], ultrasound method [8,16], direct calcination method [17], spray pyrolysis [18] reverse micellar route [19], template-assisted method [20], complexation–precipitation method [21], etc. Most of these methods have disadvantages like requirement of high temperatures and pressures, utilization of toxic reagents, specialized instruments and long reaction times. In these reported techniques there is always a requirement of external additives like stabilizers, bases, promoters and reducing agents. Therefore, there is a necessity to develop a simple, rapid, one step, economic, ecological and additive free protocol in the nanostructures synthesis.

Wang and co-workers [6] have demonstrated the synthesis of NiO nanoplatelets by hydrothermal treatment using Teflon-lined stainless steel autoclave but the methodology has one or more limitations such as higher temperature (120 °C) and long reaction time (12 h). Tao and co-workers [7] have reported the fabrication of flower-like NiO hierarchical nanostructures *via* a wet-chemical method combined with thermodecomposition technology. This method has also some disadvantages like use of many chemicals for synthesis of NiO, higher temperature (140 °C) and longer reaction time (8 h).

Currently, the synthesis of nanoparticles by using microwave assisted technique is gaining interest due to the several advantages associated with microwave irradiation [22–27]. Microwave provides high frequency electromagnetic radiations and this technique requires less energy, fast reaction kinetics, high selectivity, less time, homogeneous mixing, and convenience along with compactness of equipments [28]. The efficiency of the microwave heating is given by following equation:

$$P = cE^2 f \varepsilon''$$

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0921-8831/

Scheme 1. Synthesis of NiO nanorods by microwave irradiation.

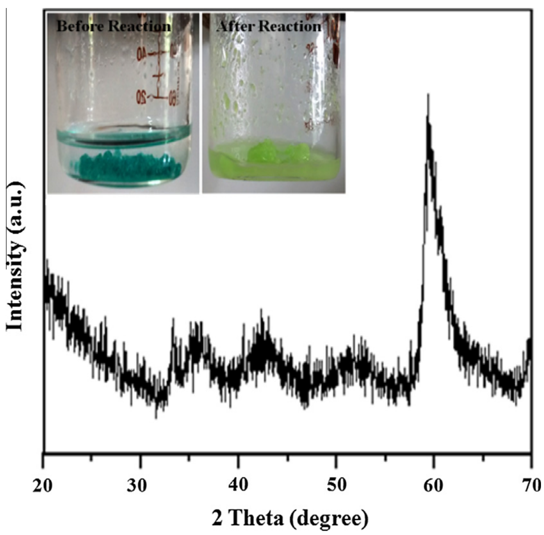

Fig. 1. XRD pattern of as prepared $Ni(OH)_2$ powder with reaction progress observed before and after by colour change (inset). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

wherein P is microwave power dissipation per unit volume in solvent, c is radiation velocity, E is electric field in the material, f is radiation frequency and ε'' is the dielectric loss constant. ε'' is the most significant parameter that determines the ability of the a material to heat in the microwave field. 1,4-Butanediol has a high value of ε'' and it having high boiling point of 235 °C. In our recent works, we have demonstrated the importance of microwave assisted nanoparticles synthesis for the ZnO, Cu_2O, MgO and Cu/Cu_2O nanomaterials [29–32].

In this study, we report two step, additive free, rapid technique for synthesis of NiO nanorods *via* microwave irradiation followed by calcination using nickel acetate as starting material and 1,4-butanediol as solvent. The morphological result obtained from FEG-SEM and TEM reveals the formation of the tips of the NiO nanorods with spherical as well as cubic shape. We further investigated the catalytic activity of synthesized nano NiO for the synthesis of substituted benzimidazole, benzoxazole and benzothiazole.

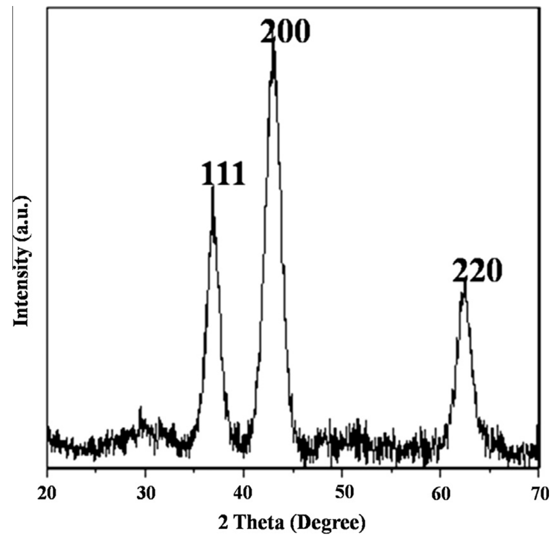

Fig. 2. XRD pattern of NiO nanorods.

2. Experimental procedure

2.1. Materials

Nickel acetate ($Ni(CH_3COO)_2 \cdot 4H_2O$) was procured from Spectrochem Pvt. Ltd. India and 1,4-butanediol was procured from S.D. Fine Chemicals Ltd. India. All the chemicals used are highly pure and were used without further purification.

2.2. Synthesis of NiO nanorods

In a typical experiment for the synthesis of NiO nanorods, a mixture of 1 g $Ni(CH_3COO)_2$ dissolved in 10 mL 1,4-butanediol in a 50 mL glass beaker and kept inside the domestic microwave oven (LG intellowave, operating at 100% power of 800 Watt (W) and a frequency of 2.45 GHz) for 2 min at 360 W using on/off mode with

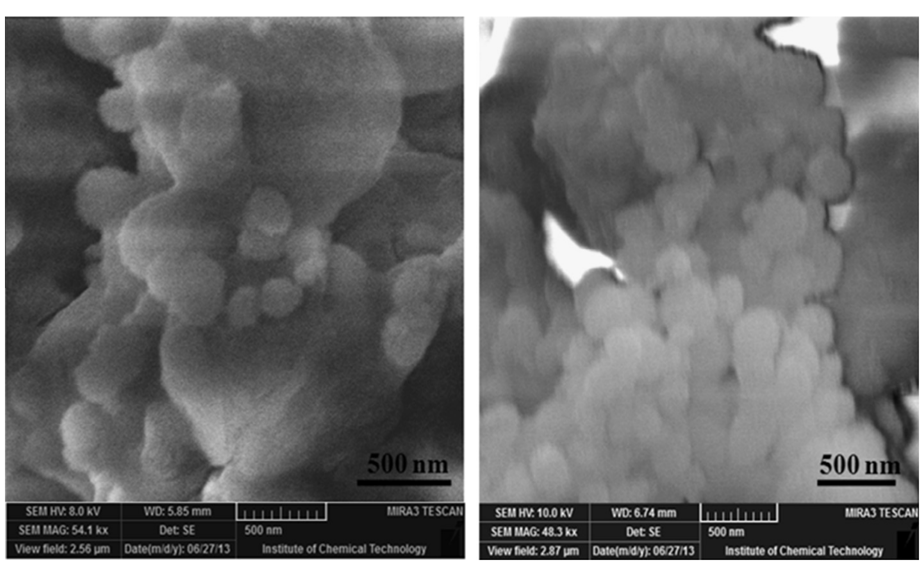

Fig. 3. FEG-SEM images of as prepared $Ni(OH)_2$.