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# Synthesis, structural and morphological characteristics of NiO nanoparticles Co-doped with boron and nitrogen

Ali Fakhri<sup>a,\*</sup>, Sajjad Behrouz<sup>b</sup>, Mohammad Asif<sup>c</sup>, Inderjeet Tyagi<sup>d</sup>, Shilpi Agarwal<sup>d,f</sup>, Vinod Kumar Gupta<sup>d,e,f,\*\*</sup>

<sup>a</sup> Young Researchers and Elites Club, Science and Research Branch, Islamic Azad University, Tehran, Iran

<sup>b</sup> Department of Biology, Central Tehran Branch, Islamic Azad University, Tehran, Iran

<sup>c</sup> Department of Chemical Engineering, King Saud University, Riyadh, Saudi Arabia

<sup>d</sup> Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee 247667, India

e Center for Environment and Water, The Research Institute, King Fahd University of Petroleum & Minerals, Dhahran 31261, Saudi Arabia

<sup>f</sup> Department of Applied Chemistry, University of Johannesburg, Johannesburg, South Africa

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#### ABSTRACT

To improve the performance of NiO nanoparticles under source light, a nitrogen and boron co-doped nano powder photocatalyst was collected. The UV–Vis diffusive reflectance spectroscopy (DRS) and photoluminescence (PL) spectroscopy were used to measure photoabsorbance. The morphology of NiO nanophotocatalyst was characterized using Brunauer–Emmett–Teller (BET), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy equipped with energy dispersive X-ray spectroscopy (SEM-EDS), and X-ray diffraction (XRD). The particle size was in correlation with the calcination temperature. It is found that the average crystallite size and specific surface area of the three co-doped NiO catalysts are near 9.57–10.21 nm, and 132.7–151.9 m<sup>2</sup>/g, respectively. Degradation of 1-chloro-4-nitrobenzene under UV and visible light illumination was applied to appraise the photocatalytic efficiency. The results indicated that the co-doping of nitrogen and boron performance is a major designation in the band gap decrease, which led to the enhancement of the activity of photocatalytic reaction. The binding energies of O 1s, Ni 2p<sub>1/2</sub>, Ni 2p<sub>3/2</sub>, N 1s and B 1s ready a justly complete image of the synthesis sample. The peak of Ni 2p<sub>3/2</sub> that emerges at 855.6 eV indicated the detection for NiO. The peak of O 1s at 530.9 eV is ascribed to the O<sup>2-</sup> in the NiO. The particles exhibited excellent antimicrobial property.

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#### 1. Introduction

The organic pollutants are one of the original sources of water contaminations extent in our natural surroundings. For water filtration several technologies are applied [1]. There are two major instruments for water contamination removal. The first instrument focuses on the use of inexpensive adsorbents [2,3] and the second one focuses on those of occupation photocatalysts like metal oxide [4]. Several other previously developed adsorbents such as carbon nanotubes [5–12], MWCNTs [13,14], nanoparticles and nanocomposites [15–19], rubber tire [20,21], and other low cost adsorbents [22–39] are extensively used for the rapid removal of noxious impurities from the aqueous solution.

Photocatalysis using metal oxide as catalysts has been widely studied for promoting degradation of organic pollutants. The present process, photon with energies corresponding to or exceeding the energy

E-mail addresses: ali.fakhri88@yahoo.com (A. Fakhri), vinodfcy@iitr.ac.in, vinodfcy@gmail.com (V.K. Gupta).

http://dx.doi.org/10.1016/j.molliq.2015.09.004 0167-7322/© 2015 Elsevier B.V. All rights reserved. band gap of the semiconductor catalyst can excite electrons from the valence band (VB) to the conduction band (CB), producing high-energy electron-hole pairs. They can react with water and dissolving oxygen to produce •OH free radicals with high chemical activity and with the pollutant molecules adsorbed on the surface of metal oxide photocatalyst. However, a high recombination rate between photogenerated electrons and holes is a major factor to reduce the photocatalytic efficiency.

NiO is as a photocatalyst to decompose organic contamination. Some studies have assayed to use the NiO for the ability of efficiently using visible light or solar light [40–42]. Other investigations tried to outstretch the contingency of electron-hole separation to increase the activity of photocatalytic by constructing a p–n junction heterogeneous NiO catalyst [43–45].

In the study the functional and structural evolution with B–N codoping NiO, and the possible enhancement in photocatalytic activity were presented. Agents of B–N co-doping on the NiO properties have never been the topic of an earlier research. Also, the effects of B–N codoping on photocatalytic activity and particle morphology were investigated and compared with commercial NiO. Then, these co-doping nanoparticles were applied as nano-photocatalysts under UV and visible

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<sup>\*</sup> Corresponding author.

<sup>\*\*</sup> Correspondence to: V. K. Gupta, Department of Chemistry, Indian Institute of Technology Roorkee 247667, India.

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light irradiation in the decomposition of 1-chloro-4-nitrobenzene (4NCB) aqueous.

#### 2. Material and methods

#### 2.1. Preparation of nanosized photocatalysts

In this study, raw materials were procured from Sigma-Aldrich Ltd. The boron and nitrogen co-doped NiO nanoparticles were synthesized by the following procedure. A certain amount of boric acid and ammonium nitrate was dissolved in the mixture of 10 ml of deionized water, 10 ml of glacial acetic acid, and 80 ml of ethanol at room temperature to gain solution A. Nickel nitrate (28.6 g, 0.1 Mol) was dissolved in 100 ml of absolute ethanol to form solution B. Then, the solution B was added drop-wise into the solution A within 60 min under vigorous stirring, followed by stirring for 2 h. The resulting sediment was aged for 48 h at room temperature and was dried for 12 h at 80 °C. Thus, the product was to be obtained. The resultant product was milled and annealed at different temperatures for 3 h to remove the residual organic compounds to prepare the boron and nitrogen co-doped photocatalyst. The sample was labeled as B(x)N(y)NiO-t, where x and *v* represented the mole ratios of boric acid to nickel nitrate and ammonium nitrate to nickel nitrate, respectively, and t denoted the corresponding temperature of calcination (°C).

#### 2.2. Characterization and analytic technique

The XRD patterns were recorded using an X-ray diffractometer (type HZG41B-PC). The XPS spectra were recorded by a Kratos Axis Ultra DLD XPS system. UV–Vis DRS mensuration was performed in a double beam spectrophotometer (JASCO V-550). The scanning electron microscope (SEM) Leo Stereoscan, model-1400 equipped with an energy dispersive X-ray microsound (EDS) was used for testing the surface morphology. The specific surface area of NiO sample was measured by the accelerated surface area porosimetry instrument (ASAP2010, Micromerities Company, USA). Photoluminescence studies were performed using a TEC Avaspec 2048 spectrophotometer (excitation source Xenon arc lamp 450 W).

#### 2.3. Photocatalytic experiments

Photocatalytic activity was led in a reactor with a 500 ml aptitude and appraised using the decomposition of 4NCB under the under UV (125 W UV lamp at 365 nm) and visible (1000 W halogen lamp) light illumination. The pH of the solution was corrected by diluting NaOH and HCl solutions. Stimulation was constructed by a magnetic stirrer rotating fast enough. The resulting solution was shaking constantly in the dark for 20 min to ensure that the suspension is uniform. Samples of solution were regulars, centrifuged at 6000 rpm for 5 min, and then filtered before being assayed on a UV–vis spectrophotometer with Hitachi, U-3010 model. Photocatalytic activity of nano powder was appraised according to the photoabsorbance of 4NCB at 279 nm. The percentage degradation of 4NCB was calculated by using the following equation,

$$\% \text{ Degradation} = \frac{(A_C - A_T)}{A_C} \times 100 \tag{1}$$

where  $A_c$  is the absorbance of control that contains only 4NCB solution and exposed to light and  $A_T$  is the absorbance of test that contain 4NCB and NPs, exposed to light.

#### 2.4. Antimicrobial effect of boron and nitrogen co-doped NiO nanoparticles

The antibacterial effect of B-N co-doped NiO NPs was tested by interacting several concentrations of B-N co-doped NiO NPs with

Escherichia coli (ATCC 25922), and Enterococcus faecalis (ATCC 29212) bacterial cells. 10 ml of log phase cultures was centrifuged at 5000 × g for 10 min and the pellet was suspended in sterile saline (0.75 mM NaCl) containing different concentrations of B–N co-doped NiO NPs (0.2, 0.6 and 1.0 µg/ml) and adjusts the cell number to  $1 \times 10^8$  CFU/ml. After 4 h of interaction, the cultures were plated on nutrient agar plates. The number of colony forming units was examined after 24 h of incubation. The same procedure was adopted in the control experiment (without B–N co-doped NiO NPs). Six replicates were kept for each concentration.

#### 3. Results and discussion

#### 3.1. Analysis of X-ray diffraction spectra

Fig. 1 illustrates the typical XRD pattern of boron and nitrogen codoped NiO nanoparticles. The peaks of prepared mono and co doped NiO nanoparticles are compatible with cubic structures of NiO (JCPDS Card No. 78-0423). It could be realized that all those boron and nitrogen co-doped NiO powders were present in the similar phases irrespective of the calcination temperature. This may be also observed that the plane diffraction peak ( $2\theta = 43.60$ ) became narrower for the boron and nitrogen co-doped NiO with the annealing temperature increasing. The calculated values of crystallite size are presented in Table 1 for all samples of NiO nanoparticles.

#### 3.1.1. Scanning electron microscope photograph of photocatalyst

Fig. 2 reveals an SEM image of the synthesis sample. As can be seen, the sample B–N co-doped NiO extended almost in the spherical particle form and has porous structures. Pursuant to the estimation of statistical data, the average particle size was near 20.3 nm, which was coincident



**Fig. 1.** XRD patterns of (A) NiO-500, (B) B(0.1)NiO-500, (C) N(0.1)NiO-500, (D) B(0.1)N(0.1)NiO-400, (E) B(0.1)N(0.1)NiO-500, and (F) B(0.1)N(0.1)NiO-600.

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