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Characterization and Photocatalytic activity of Nickel oxide nanoparticles

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Abstract: Nickel oxide (NiO) nanoparticles were prepared by simple thermal decomposition method. The XRD pattern reveals the crystallinity and crystal structure of NiO. In addition, the average grain size, lattice parameter values were also calculated using XRD pattern. Moreover, the diffraction peaks were broadened due to the smaller size of the particle. The FT-IR analysis confirms the formation of the NiO by showing bands of Ni-O. The morphology of nickel oxide was analyzed using SEM analysis. Further, the photocatalytic degradation of Rhodamine B was investigated by UV-Vis spectrophotometer, which indicates NiO nanoparticles will be a potential material in the field of photocatalysis.

Keywords: Nickel oxide, crystal structure, Rhodamine B, photocatalysis.

Introduction

Nanostructured materials have been extensively explored for the fundamental scientific and technological interests in accessing new classes of functional materials with unprecedented properties and applications [1-2]. NiO is a very important material extensively used in catalysis, gas sensors, battery cathodes, heterogeneous catalytic materials and magnetic materials [3]. NiO nanoparticles have been prepared by various methods like sol-gel, surfactant-mediated synthesis, thermal decomposition, polymer-matrix assisted synthesis and spray-pyrolysis [4]. Some of the above methods suffer from the difficulty in size-homogeneity and well dispersion of NiO nanoparticles. In this study, we have reported the synthesis of NiO nanoparticles using thermal decomposition method and characterized its structural, optical, and photochemical properties. Additionally we have performed the phototocatalytic activity of the synthesized NiO towards the degradation of rhodamine B.

Experimental

Materials

Nickel chloride hexahydrate (NiCl₂·6H₂O), dimethyl glyoxime (DMG), ethanol, rhodamine B (RhB) and glycerol were purchased from Qualigens

Characterization Methods

The crystal structure of NiO nanoparticles was analyzed by a Rich Siefert 3000 diffractometer with Cu- $K\alpha_1$ radiation ($\lambda = 1.5406 \text{ \AA}$). FT-IR spectrum of the NiO was recorded on Shimadzu FT-IR 8300 series instrument by using potassium bromide pellets. The morphology of the materials was analyzed by SEM HITACHI SU6600 scanning electron microscopy respectively. The UV-Vis absorbance spectrum was recorded using Perkin- Elmer lambda650 spectrophotometer.

Synthesis of NiO Nanoparticles

NiO was prepared by thermal decomposition of nickel DMG. The nickel DMG complex was prepared by reacting aqueous solutions of 0.01 M nickel chloride and 0.01 M alcoholic solution of dimethyl glyoxime. The 0.01 M nickel chloride was dissolved in 500 ml distilled water and 10% of glycerol. This suspension was stirred for 20 min at 50 °C in magnetic stirrer. After 20 min, the dissolved alcoholic solution of dimethyl glyoxime solution (50 ml) was slowly added to control the agglomeration. The obtained nickel DMG is calcined in air for 3 h at 600 °C to form NiO nanoparticles.

Photocatalytic activity

Proper amount of Rhodamine B ($2 \times 10^{-5} \text{ M}$) was added to a beaker and then the beaker was placed in stirrer for 30 min. For the accuracy of data, all samples were prepared as triplicate. The photocatalytic degradation of the dyes was carried out in a photocatalytic chamber containing halogen visible lamp. The samples were collected at the time interval of 15 min and their absorption maxima was monitored by UV-Visible spectrophotometer.

Results and Discussion

Structure and Morphology

XRD patterns of the NiO nanoparticles calcined at 600 °C, is shown in Figure 1, which indicates the nickel oxide has monoclinic phase structure. The peak positions ($2\theta = 37.28^\circ$ and 43.23° and 63.20°) and intensities obtained for the NiO matched with JCPDS No: 89-7131 file. There were no characteristic peaks of impurity observed. The average crystallite size of NiO is determined using Scherrer relation, and it was found to be around 49 nm.

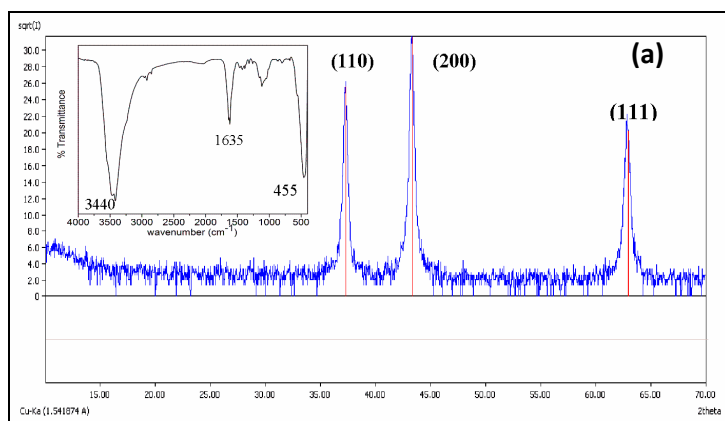


Figure 1. XRD pattern (inset: FTIR spectrum) of NiO.

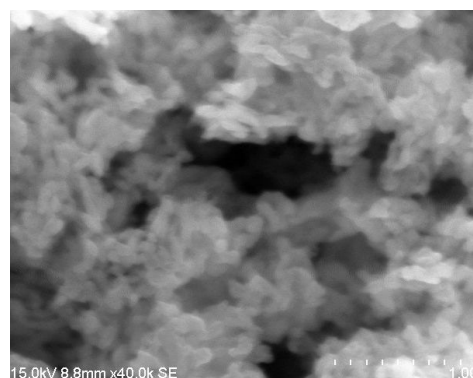


Figure 2. SEM image of NiO

FTIR spectrum of NiO nanoparticles (inset) showed significant peaks at 3440, 1635 and 455 cm^{-1} . The band at 455 cm^{-1} was assigned to Ni–O stretching vibration mode. The broad band at 3440 cm^{-1} was assigned to the O–H stretching vibration and the weak band near 1635 cm^{-1} was assigned to H–O–H bending vibration mode were also presented due to the adsorption of moisture. The SEM micrograph of the NiO calcined at 600 °C is shown in Figure 2. The particles adopt irregular morphology with different sized particle due to aggregation. It clearly indicates the agglomerated rod-like particles with nanoscale. It shows the rod-like agglomerate was due to the calcination temperature [5].

Optical Studies

Figure 3i shows the optical absorption spectrum of nickel oxide sample calcined at 450 and 600 °C were measured using UV-Visible spectrophotometer in the range of 200 to 800 nm. The absorption band of nickel oxide sample shows the absorption bands at wavelength around 320, 380, 435 and 730 nm. The slight variation in the absorption spectrum was due to the calcination temperature.

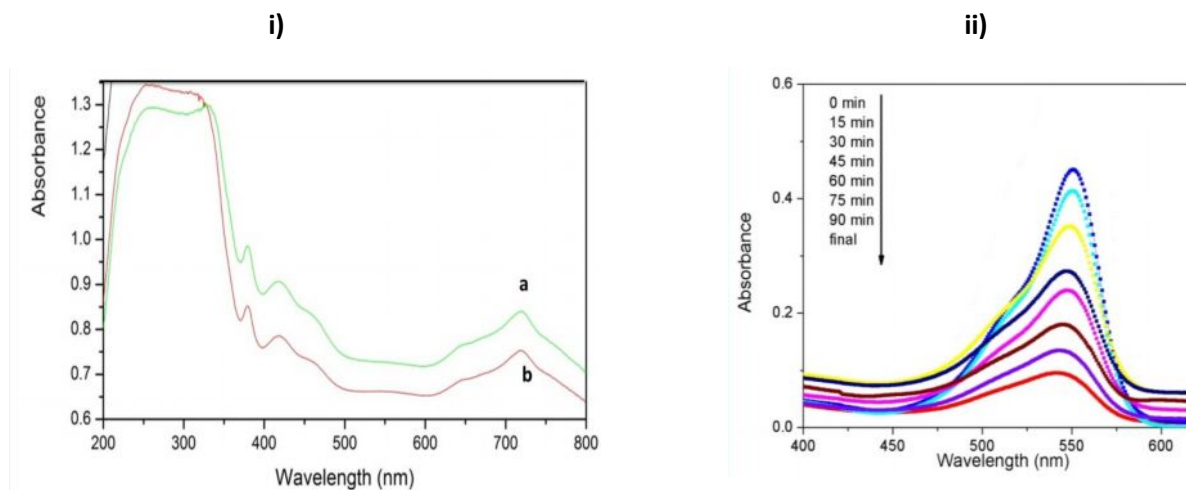


Figure 3: (i) UV- Vis absorption spectrum of NiO at a) 450, b) 600 °C ; (ii) UV–Vis spectra of RhB degradation under visible light irradiation.

Photocatalytic Property

The photocatalytic degradation of the RhB aqueous solution can be regarded as a pseudo first-order reaction. The reaction were performed by adding 0.02 g of photocatalyst into a 100 ml of dye with light irradiation. The photooxidation of colored dye solution to colorless solution in presence of NiO under visible light (300-W Halogen lamp; $\lambda > 420$ nm) irradiation. Fig. 3ii shows the UV–Vis absorption spectra of RhB, which were taken after the irradiation of visible light with 15 min time intervals and absorbance of dye were found to be 546 nm. Time required for 88% degradation of dye is about 90 min for RhB.

Conclusions

The NiO nanoparticles synthesized from Ni-DMG by thermal decomposition method were characterized using FT-IR analysis, XRD and SEM. The optical absorption spectrum of nickel oxide sample was studied by DRS-UV-Visible spectroscopy. The kinetics of dye degradation was examined by UV-Vis spectrophotometer. The results conclude that the nickel oxide nanoparticles have higher activity for the degradation of rhodamine B.

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