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Photocatalytic activity of NiO nanoparticles stabilised with Polyethylene Glycol

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ABSTRACT

NiO nanoparticles stabilized with polyethylene glycol (PEG) polymer were synthesized and characterized using FESEM, EDAX, XRD, UV and FT-IR techniques. FESEM image shows that the prepared nanoparticles exhibits leaf structure. From XRD analysis the average size of the NiO nanoparticles were found to be 9 ± 0.5 nm. The as prepared NiO nanoleaves were used as photocatalyst in the degradation of bromophenol blue under uv light irradiation in aqueous solution. UV – VIS spectroscopy was used to follow the reaction kinetics. Degradation of the dye followed pseudo first order kinetics. Photocatalytic activity of NiO-PEG nanoparticles were compared with NiO nanoparticles without stabilizer and P25 TiO₂ powder.

Keywords: NiO nanoparticles, photocatalysis, polyethylene glycol, dye degradation

ARTICLE INFO

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1. Introduction

For the past two decades, there has been increasing interest in developing nanostructured metal and metal oxide materials because of their wide applications in various fields. Metal oxide nanomaterials have a large surface area.

This gives nanomaterials great advantages over conventional materials in many applications. NiO being one of the p type semiconducting material drawn much attention because of its wide band gap energy in the range of 3.5 to 4

eV. NiO has found number of applications in different fields such as catalysts, lithium ion batteries, fuel cells, electrochromic films, electrochemical supercapacitors, magnetic materials, and gas sensors.

Among the different methods available from the literature to synthesize nanostructured nickel oxide, such as evaporation [1], sputtering [2–4], electrodeposition [5], and sol-gel techniques [6], thermal decomposition of nickelous precursors, especially Ni(OH)₂, was the most commonly used. There are only very few reports on different morphologies and sizes of NiO nanoparticles. In the present work, NiO nanoparticles stabilized with poly ethylene glycol (PEG) have been synthesized. The morphology of the synthesized nanoparticles was found to be leaf structured.

Waste water containing organic pollutants, especially dyes from textile industries are of more concern because of its adverse effects on the environment. Therefore, their presence even at low concentrations has been the driving force for different water treatment techniques [7-9]. In this work, the photocatalytic activity of the NiO nanoparticles have been tested taking bromophenol blue (BPB) as the model dye. Structure of the dye bromophenol blue is shown in Fig. 1 and the dye has λ_{max} at 589nm. Degradation studies were made using UV VIS spectroscopy by following the peak at 589nm tracing baseline. Kinetic plots were made and rate coefficient values are determined.

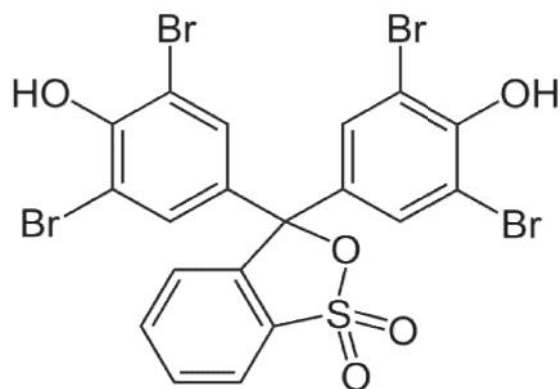


Figure 1: Structure of Bromophenol dye

2. Experimental

BPB dye was purchased from Aldrich and was used without further purification. NiCl₂·6H₂O and PEG from Fischer Chemicals were used. The molecular weight of PEG used was 57 ~ 63KD, 90KD and purchased from SRL, India. All reagents used were of analytical grade and were directly used without purification.

Synthesis of NiO nanoleaves

In a typical procedure, 0.237g of NiCl₂·6H₂O was dissolved in 1:1 volume ratio of ethanol water mixture. After stirring for 5 minutes, 50ml of 1mM solution of PEG and 100ml 1mM NaOH were added to the beaker. Stirring was continued for 30minutes by maintaining the temperature at 50°C. Green precipitate formed were filtered and washed several times with water and finally with ethanol. The precipitate was heated to 400°C for 1 hour in an oven.

Black powder was obtained. Using the above procedure NiO nanoparticles without PEG stabiliser were prepared. The synthesized nanoparticles with and without stabilizer were subjected to FESEM, XRD, EDAX and FT-IR measurements.

Characterization of NiO nanoleaves

The metal oxide nanoparticles prepared are size characterized using FESEM SU6600, HITACHI model operating at an accelerating voltage of 100 kV and Bruker tensor 27 instrument was used for FT-IR measurements. A Bruker D8 advance diffractometer was used for XRD data. Also powder X-ray diffraction (XRD) patterns of NiO nanoparticles are given. Applying the Scherrer formula, the size values of the nanoparticles are calculated to be 9±0.5nm and 30±2nm with and without PEG stabilizer respectively.

Photocatalytic measurements

The photocatalytic property of the products was determined by measuring the decoloration of BPB aqueous solution, which was selected as a test compound. 5 mg of the as-prepared NiO nps were added to 10 mL of 20 mg/L BPB solution. Prior to irradiation, the suspensions were oscillated for 5 min to establish the adsorption/desorption equilibrium between the dye and the photocatalysts. The mixed solution was then irradiated with ultraviolet (UV) lamps (365 nm) at a distance of about 10 cm. At given irradiation time interval, sample was withdrawn from the test tube for analysis. The absorption spectra of these solutions were measured.

3. Results and discussion

NiO nps morphology and size analysis

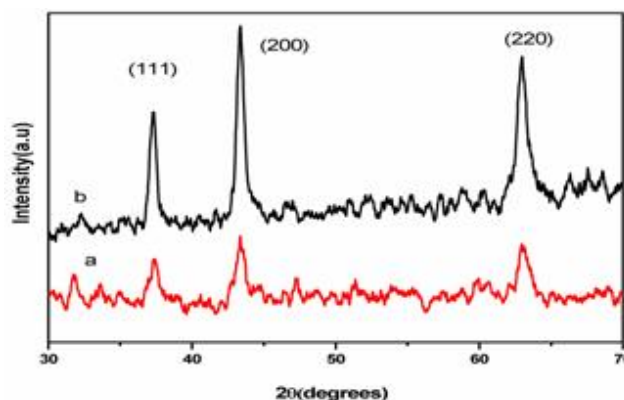


Figure 2: XRD pattern of a. NiO-PEG nps and b. NiO nps

Fig. 2 shows the XRD patterns of the as-calcined NiO nanoparticles at 400°C. All diffraction peaks can be perfectly indexed to the cubic structure crystalline NiO. XRD pattern indicates that there is no impurity in the powder [10,11]. XRD peaks of NiO-PEG nanoleaves are markedly broader than the NiO nanoparticles without stabilizer and the peaks can be readily indexed as (111), (200) and (220) crystal planes. The peak patterns are assigned to JCPDS data file 47-1049. The detailed morphology of the nanoleaves was observed by FESEM. The typical images are shown in Fig. 3. An elemental composition of the composite was elucidated with EDAX

shown in Fig. 4 and confirmed the presence of nickel and oxygen. Presence of trace amounts of carbon may be due to the stabilizer.

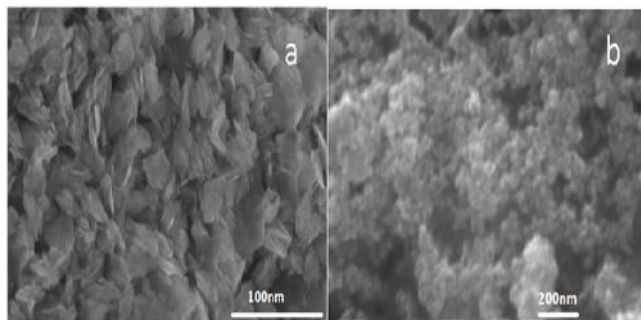


Figure 3: FESEM images of a. NiO-PEG nps & b. NiO nps

In Fig.5, the FT-IR spectra in KBr pellets of the pure sample of PEG along with the NiOnps counterparts are shown. The characteristic peaks at 2874cm^{-1} and 3379cm^{-1} corresponding to CH_2 and O-H groups in the pure PEG are shifted and merged in the nanoparticles spectra. The peak of $-\text{C-O}-$ group at 1959cm^{-1} is shifted in the nanoparticles spectra. The fingerprint region peaks are completely diminished from 1462 to 1000cm^{-1} region, there is a prominent peak at 500cm^{-1} for NiO nps. In the NiOnp surface, strong H-bonding interactions hold the OH groups of the stabilizing agent PEG.

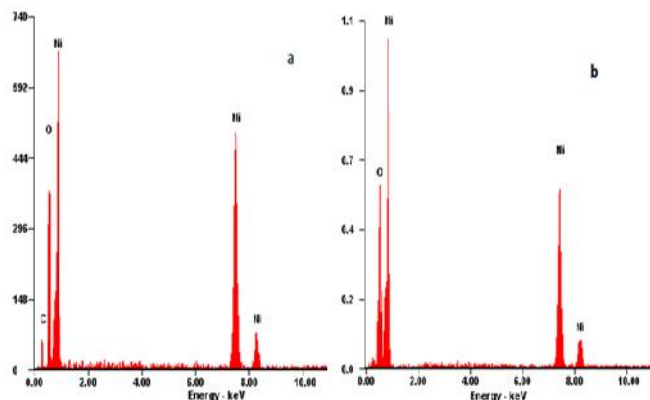


Figure 4: EDAX of a. NiO-PEG nps and b. NiO nps

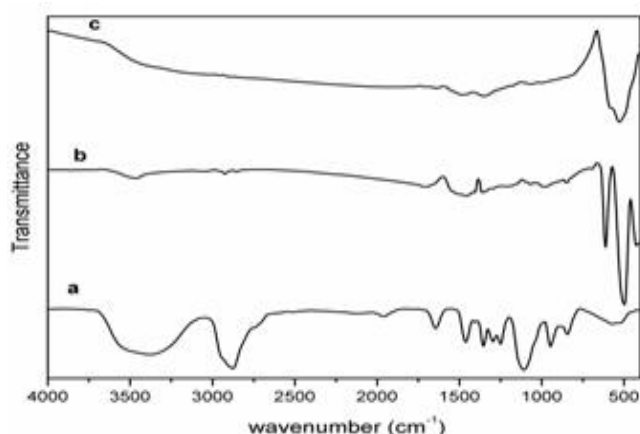


Figure 5: FT-IR spectra of a. PEG b. NiO-PEG nps and c. NiO nps

Decolourisation kinetic studies

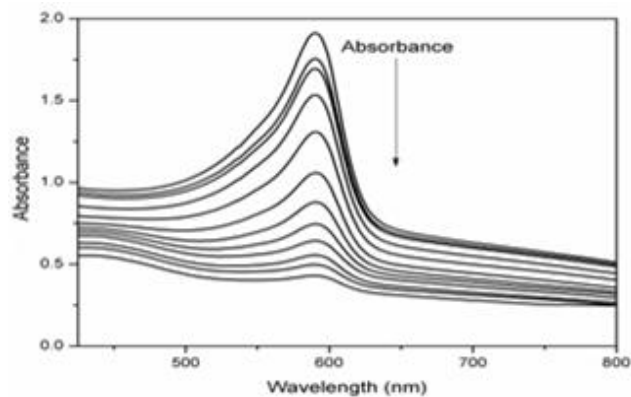


Fig.6: Absorbance time variance visible spectra of BPB dye

The absorbance time variance UV-VIS spectra of the dye system in the presence of NiO-PEG nps as catalyst is shown in Figure.6. It is well known that commercial P25 TiO_2 powders are one of the most active photocatalysts [12], and a comparison of photocatalytic activity of the NiO nanoparticles with that of P25 TiO_2 powders were carried out. The absorbance versus time plots determined from the spectra are shown in Figure. 7a. It may be seen that a gradual exponential decrease in the absorbance values with time are found out.

The pseudo first order rate coefficient values for the decolourisation are determined from the kinetic plots which are obtained by plotting $\log(\text{OD}_0/\text{OD}_t)$ versus time. The kinetic plots are shown in Figure. 7b. Rate coefficients can be obtained by multiplying slope with 2.303 and the k values for different catalysts are given in Table 1. From the k values, it is inferred that the decomposition rate of the BPB dye over the polycrystalline NiO-PEG nanoleaves is faster than that over the NiO nanoparticles, but slower than that over the commercial P25 TiO_2 powders. The results indicate that the as-prepared polycrystalline NiO nanoleaves stabilized with PEG exhibit an enhanced photocatalytic ability to degrade BPB dye in comparison with the NiO nanoparticles without PEG stabilizer, although its photocatalytic ability is weaker than that of the commercial P25 TiO_2 powders.

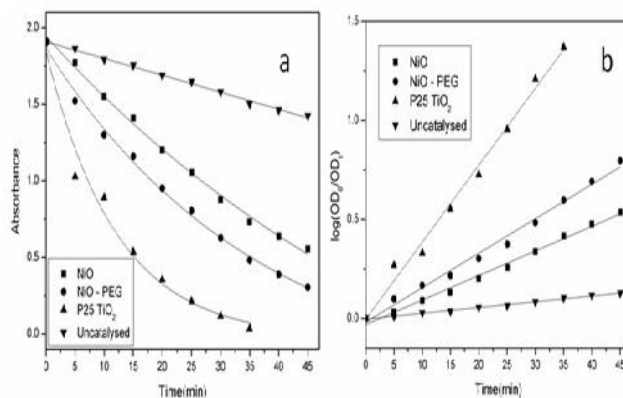
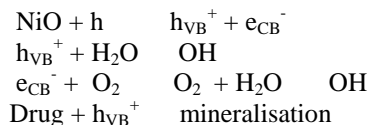


Figure 7a. Absorbance Vs Time plots and **b.** Kinetic plots for the decolourisation of BPB dye in presence of various catalysts under uv light irradiation.

Table 1: Rate coefficient values for the decolouration of BPB dye in the presence of various catalysts

Entry	Catalyst	k (10 ⁻⁴ sec ⁻¹)
1	NiO	4.76
2	NiO-PEG	6.68
3	P25-TiO ₂	14.93
4	Uncatalyzed	1.11

Based on the experimental results and from the earlier reports on photocatalytic degradation of organic molecules, we assumed that on irradiation with UV light, the NiO nanoparticles generate an excitation of electrons in valence band into the conduction band (electron-hole separation). The generated electron (hole) transfer to the adsorbed drug on the nanoparticle surface has always been considered as the first step of the photocatalytic action of semiconductor nanoparticles. Photo-generated holes as well as hydroxyl radicals oxidize the drug adsorbed at NiO surface, as follows:



4. Conclusion

NiO nanoparticles were synthesized with and without polymeric stabilizer PEG. The prepared nanoparticles are characterized using FESEM, XRD, EDAX and FT-IR. From FESEM images, the morphology of the NiO-PEG nanoparticles were found to be leaf shaped. Photocatalytic activity of the prepared nanoparticles are tested with decolourisation of BPB dye. NiO nanoparticles stabilized with PEG was found to be more efficient photocatalyst in the decolourisation of the BPB dye than NiO nanoparticles without stabilizer, which are expected to be useful in waste water treatment.

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