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Biosynthesis and Characterization Studies of NiO Nanoparticles Using Cactus Plant Extract

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A B S T R A C T

Using biosynthesis process, nickel oxide (NiO) nanoparticles were prepared using cactus plant extract – assisted microwave combustion method. For NiO nanoparticles synthesis, materials such as nickel nitrate and cactus plant extract are used as precursors. NiO nanoparticles products were characterized by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and high-resolution scanning electron microscopy (HR-SEM) and photocatalytic studies. The XRD results showed NiO NPs with a crystallite size of 15 nm was face-centered cubic (FCC) with crystalline structure. HR-SEM images have shown the formation and agglomeration of spherical-sized nanostructured crystallites. The FTIR spectrum reveals that at 684 cm^{-1} is characteristic of Ni-O. NiO nanoparticles are applied to the 100 ml of Rhodamine B (RhB) dye and stored under visible light in the photoreactor to study the efficiency of dye degradation. The PCD studies showed that, under visible light irradiation, the as-prepared NiO nanoparticles degrade the RhB dye effectively.

***Key words*:** NiO nanoparticles, Biosynthesis process, Microwave combustion, Rhodamine B, Dye degradation

Nickel-based nanomaterials are widely used in military and civil field as to the outstanding high temperature (T) properties, such as longer creep rupture life, higher corrosion resistance, and better high T microstructure stability and so on [1-3]. Nowadays, more and more nanomaterials are used in civil field, especially nuclear or fossil power plants. These materials are designed to serve in high temperature environment for long-term and sometimes endure impact loadings from foreign components, during which impact toughness deterioration and embrittlement can occur [4-8]. Thus, high temperature impact toughness is required to prevent catastrophic failure. It can be seen that impact toughness is one of the major mechanical properties for these alloys and high impact toughness is extremely important to guarantee sufficiently safe operation [9-12].

NiO has been recognised in switching, magnetics, supercapacitors, catalysis, etc., and promising LIB anode materials are expected to be 718 mA h g-1 due to their low cost, natural abundance, safety, and high theoretical specific ability [13-15]. However, NiO 's application in LIBs was limited by its poor electrochemical efficiency. Due to their relevant biological and therapeutic properties, which may include their unique surface region, metal ion releasing and adsorbing capability, cytotoxic effects, NiO NPs have also been widely used in biomedicine. In different fields, including electronics, magnetism, energy technology, and biomedicine, Nickel NPs discover possible applications [16-20]. Due to their high reactivity, operational simplicity and eco-friendly properties, they are used to catalyse various organic reactions, including chemo-selective oxidative thiol coupling, reduction of aldehydes and ketones, olefin hydrogenation, synthesis of stilbenes from alcohol by Wittig-type olefination, and al-alkylation of methyl ketone. Some inorganic reactions are also catalysed by them, such as ammonia decomposition [21-25]. Their position in manufacturing carbon nanotubes (CNTs) is one of their recent applications. Environmental applications in the field of hazardous dye adsorption and inorganic contaminants are also recognised and thus play a key role in environmental cleanliness [26-30]. They are used in the biomedical sector due to their strong antibacterial and anti-inflammatory behaviour. As is evident from the distortion of the morphology of these cells following their treatment with NiO NPs, they also exhibit cytotoxicity towards cancerous cells. The biocompatibility of NiO NPs with biomolecules such as glucose is greatly improved and they are used for cancer hyperthermia as biosensors and heat non-mediators.

MATERIALS AND METHODS

In the NiO material synthesis, materials such as nickel chloride and ammonium hydroxide are used. The precursor materials are of AR grade and, without further purification, are used directly. Nickel chloride was mixed with 30 ml of de-ionized water and 8 ml of ammonium hydroxide solution was poured into the solution at a steady stirring at 75°C and a

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temperature of 75 ° C for 30 minutes. The solution is then cooled for 2 hours at room temperature. Using Whatman philtre paper, the resulting green colour solution is filtered. Then the resulting solution is washed with DI water 3 times and ethanol 3 times.

Characterization techniques

X-ray diffractometer with CuKa radiation (1.5418 Å) is used to analyze the structure of the sample. For chemical analysis, the FTIR spectrometer, (Spectrum Two FTIR spectrometer) in the 400-4000 cm^{-1} wavenumber range with a resolution of 0.5 cm^{-1}. The sample was absorbed using a double beam spectrophotometer (UV-1800, Shimadzu) with a wavelength range of 200-1000 nm. High resolution scanning electron microscope (HR-SEM) (Carl Zeiss microscopy ltd, UK & SIGMA) was used to analyze the morphology and composition. NiO nanoparticles are used as a catalyst and its photocatalytic activity is studied using the photoreactor (HEBER, MODELHVAR-MP400) with visible light and 300 Watts. The degradation was carried out for 2 hrs after the dark condition

RESULTS AND DISCUSSION

XRD analysis

XRD pattern reveals the peaks at angles of 37.35, 43.33, 62.92, 75.47 and 79.42°, confirming face-centered cubic (FCC) crystalline structure of NiO. In all these diffraction peaks, the direction of the peaks and their relative strength correspond to the regular JCPDS NO. 04-0835. From XRD data, the lattice parameter of NiO nanoparticles is 3.98 Å, which is in good agreement with the data published [31-33].

FTIR spectrum

Figure 2 shows the FTIR spectrum of the NiO NPs and shows the bands ~3460, 2885, 1624, 676 and 650 cm-1. The band at 3460 cm-1, attributed to the hydroxyl group [34]. The weak band is assigned to the symmetric stretching vibration of C-H at 2980 cm-1. In the study, these peaks suggest the presence of water [35]. The existence of CH_2 bending vibration [36] is confirmed by the band at 1385 cm-1. The spectrum contains one 676 cm-1 broadband, which is characteristic of the hydroxyl group of stretching vibration to Ni-O [37].

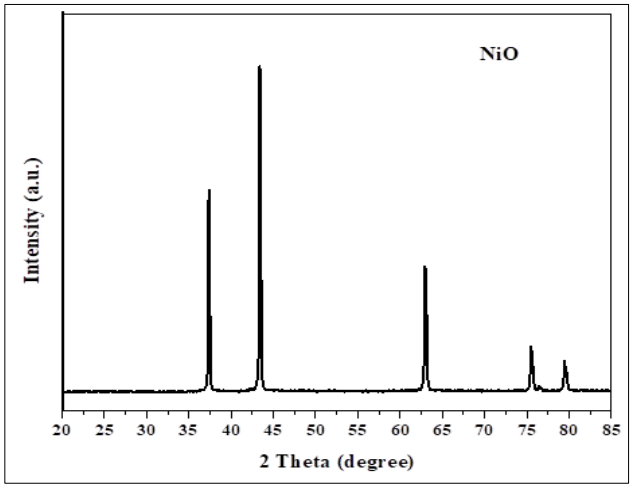

Fig 1 XRD pattern of NiO NPs

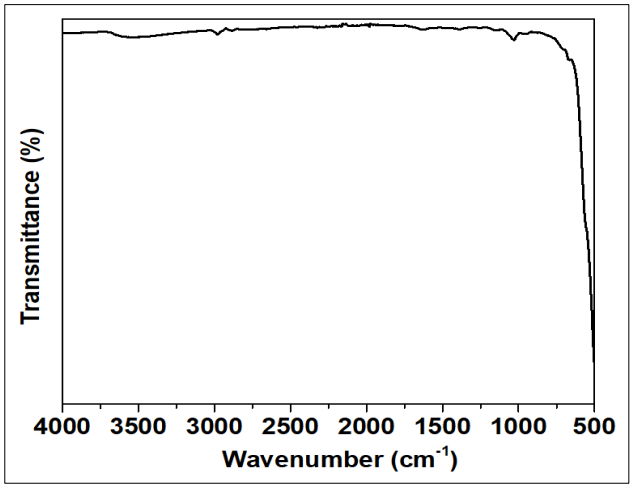

Fig 2 FTIR pattern NiO NPs

HR-SEM and HR-TEM studies

Using HR-SEM and HR-TEM, the surface morphology and particle size of the NiO nanoparticles was analyzed. The SEM morphology of the NiO crystallites is shown in Fig. 3. It is noted that some crystallites are spherical in nature and some of the particles are agglomerated plate-like structures with less than 20 nm in size (Fig. 4). All the crystallites are distributed evenly and are thick and equal in size.

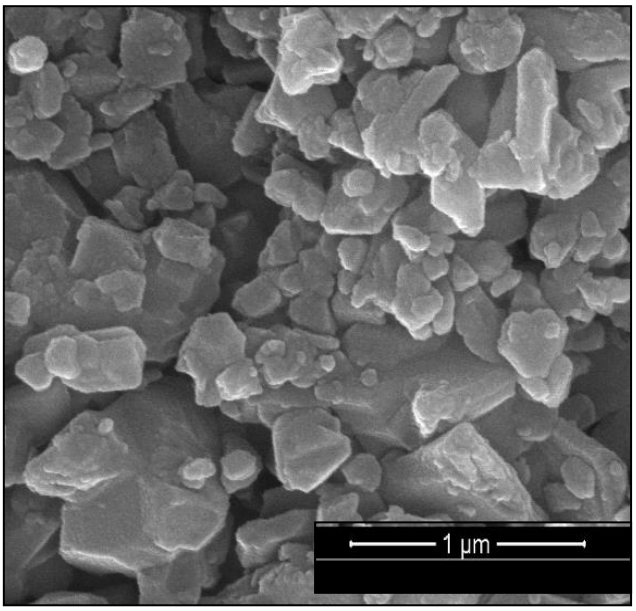

Fig 3 HR-SEM images of NiO NPs

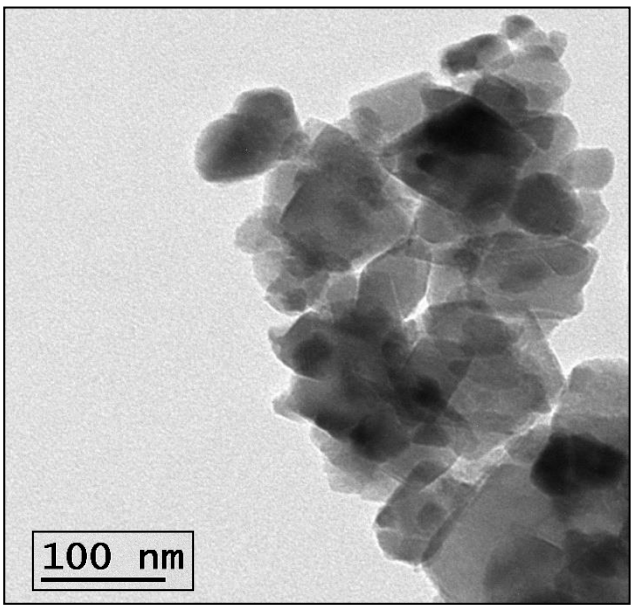

Fig 4 HR-TEM images of NiO NPs

Optical studies

Figure 5 shows the DRS spectra of the sample indicating the absorbance of materials near the visible region. A strong absorption is noticed for the NiO nanoparticles. The optical bandgap is calculated from the reflectance using the Kubelka-Munk (K-M) function as follows,

$$F(R) = (1-R)^2/2R$$

R- Reflectance of the sample. The wavelength is converted to energy and plotting the graph $[F(R)\ h\gamma]^2$ vs $h\gamma$ and the tangent is drawn to the linear region and the intercept at X-axis denote the bandgap of the material. The bandgap of the sample is calculated and found to be 2.85 eV (Fig 5).

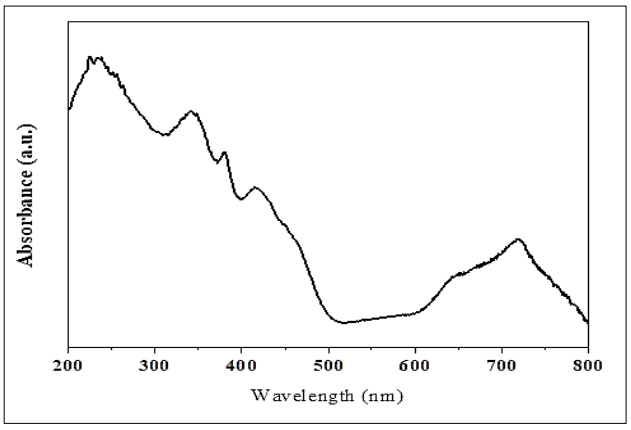

Fig 5 UV-Visible diffuse absorbance spectrum of NiO nanoparticles

Photocatalytic studies

Photocatalytic dye degradation behavior of NiO nanoparticles under the photo reactor was tested for the degradation of RhB dye. 100 mg of the photocatalyst NiO nanoparticles is extracted and dispersed under a magnetic stirrer in 100 ml of RhB solution. This test is carried out over various periods of time. This procedure is carried out for two hours. Using the UV Visible Spectrometer, RhB dye degradation is reported. More active absorption capacity can be generated by NiO NPs having a high surface area. By absorbing water in the air, Ni ions create hydroxyl groups. The electrons shift from the valence band to the conduction band when light falls on the sample, and the formation of holes and electron pairs will occur [38-40]. The time required for 75% dye degradation is around 150 minutes.

CONCLUSION

NiO nanoparticles are synthesized successfully using nickel nitrate by biosynthesis process, using cactus plant extract – assisted microwave combustion method. XRD, FTIR, HR-SEM, HR-TEM, UV-Visible spectroscopy and photochemical reactors are used to research structural, optical and photocatalytic properties. XRD results indicate that NiO NPs are well crystalline in nature with the FCC phase. The presence of Ni-O bonds is recommended by the FTIR. HR-SEM demonstrates the formation of agglomerated NiO nanoparticles. The UV-Visible DRS spectrum indicates that the highest absorption edge of NiO nanoparticles occurs at 2.85 eV bandgap for the NiO nanoparticles was shown in UV-Visible studies. The photocatalytic studies showed a RhB dye degradation efficiency of 76% using NiO nanoparticles.

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