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Synthesis of NiO nanoparticles via green route using *Ageratum conyzoides* L. leaf extract and their catalytic activity

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Abstract. Nickel Oxide nanoparticles (NiO-NP) have been successfully synthesized using *Ageratum conyzoides* L. leaf extract via green method. The optical, structural, and morphology of NiO-NP were characterized. The characterization UV-Vis spectrophotometer showed a typical peak of NiO-NP at maximum wavelength of 324 nm. FT-IR characterization confirms that the stretching vibration of Ni-O occurs at wavenumber of 434 cm^{-1}. The particle size distribution of NiO-NP was 50.70 – 91.3 nm characterized by PSA. The XRD pattern showed the crystallinity of NiO-NP with cubic structure. TEM image showed the particle size of NiO-NP was 8-15 nm. The catalytic activity percentage of NiO-NP shown 83% in the reduction of methylene blue compared to catalytic activity without catalyst was 28% for 18 minutes.
Keywords: *Ageratum conyzoides* L.; catalyst; green route; NiO nanoparticle

1. Introduction

NiO-NP shows several unique characteristics such as optical, electrical, magnetic and chemical properties [1]. NiO-NP is widely used as magnetic material, electrochromic film, battery electrode, and heterogeneous catalyst material [2]. Synthesis of NiO-NP has been carried out using several methods such as sol gel [3], co-precipitation, and thermal decomposition [4].

In other side, the phenomenon of nanoparticles green synthesis still attracts many researchers' interests. The selection of this method is mostly performed because it is more economical, easy to obtain, and environmentally friendly [5]. In green route, the secondary metabolite act as a base source and capping agent like chemical substitute [6]. The various sources of raw material for green synthesis of nanoparticle have been widely published such as plant extracts, enzymes, fungi, and microorganisms. Green synthesis of NiO-NP had been reported using *Physalis angulata*, *Moringa oleifera*, and *Calotropis gigantea* leaf extract [7-9].

Ageratum conyzoides L. or bandotan, has many benefits. *Ageratum conyzoides* L. leaf had been previously reported which contained secondary metabolites compounds such as alkaloids, tannins, phenols, saponin, and flavonoids [10]. Previous research using alkaloid and flavonoid in leaf extract for the synthesis of metal oxide nanoparticles [6, 11, 12]. The secondary metabolite has been reported not only act as ingredients but also as capping agents [8, 13, 14]. Herein, we report a green route to synthesis NiO-NP using *Ageratum conyzoides* L. leaf extract, for the first time, and their catalytic activity for the reduction of methylene blue dye.

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2. Materials and methods

2.1. Material
Nickel nitrate hexahydrate, $Ni(NO_3)_2.6H_2O$, $NaBH_4$, and methylene blue were purchased from Merck (Germany) with analytical grade. *Ageratum conyzoides* L. leaf was collected from conservation unit of biopharmaca.

2.2. Ageratum conyzoides L. leaf extract preparation
Ageratum conyzoides L. leaf were washed, dried, and grinded to get green powder. The powder was macerated into methanol and stirred continuously every day for nine days. The result was separated by n-hexane to obtain methanol and hexane fraction. Methanol fraction was evaporated and dissolved into water to obtain the aqueous fraction of *Ageratum conyzoides* L. leaf extract [7, 15]. The aqueous fraction of *Ageratum conyzoides* L. leaf extract was used in phytochemically tested to identify the secondary metabolite of alkaloids, flavonoids, tannins, saponins, and polyphenols [15].

2.3. Synthesis of NiO-NP using Ageratum conyzoides L. leaf extract
Ageratum conyzoides L. leaf extract was added drop wise into $Ni(NO_3)_2$ by stirring at 80°C for 6 hours. The colloid was heated at 120°C and centrifuged to form the gel. The gel was calcined at 450°C for 2 hours, to obtain the blackish grey powder.

2.4. Characterization of NiO-NP
The absorption spectra of NiO-NP were measured using Shimadzu 2600 UV-Visible spectrophotometer. The identification of functional groups of *Ageratum conyzoides* L. leaf extract and NiO-NP were confirmed using FTIR spectroscopy (IR Prestige-21Shimadzu). The particle size of NiO-NP was measured by particle size analyzer (PSA) Malvent Zetasizer. The crystallinity of NiO-NP was examined by X-ray diffraction (XRD) PANalitycal Empyrean instrument. The morphology and size of NiO-NP was characterized by TEM (JEOL JEM 1400).

2.5. Catalytic activity of NiO-NP
3×10^{-3} g of NiO-NP was added into 2×10^{-5} M methylene blue and 0.1 M $NaBH_4$ as a model reaction. The decrease of absorbance by time was recorded to investigate the catalytic activity of NiO-NP in the reduction of methylene blue.

3. Results and discussion
Ageratum conyzoides L. leaf extract contains alkaloids, flavonoids, tannins, saponins, and polyphenols from the result of phytochemical test. The secondary metabolites of leaf extract act as a base source and a capping agent in nanoparticle synthesis [6]. NiO-NP has been synthesized using *Ageratum conyzoides* L. leaf extract via green method. The maximum wavelength of NiO-NP was observed using UV-Visible spectrophotometer as shown at Fig. 1. The absorption spectrum of the NiO-NP shows a broadening spectrum at the wavelength of 324 nm. It is a good agreement in literature [16]. Moreover, this absorbance was different with $Ni(NO_3)_2$ as precursor and *Ageratum conyzoides* L. leaf extract. $Ni(NO_3)_2$ as precursor has the absorption spectra at maximum wavelength 302 and 391 nm. NiO-NP has an unique optical properties due to the property of surface plasmon resonance [17, 18].

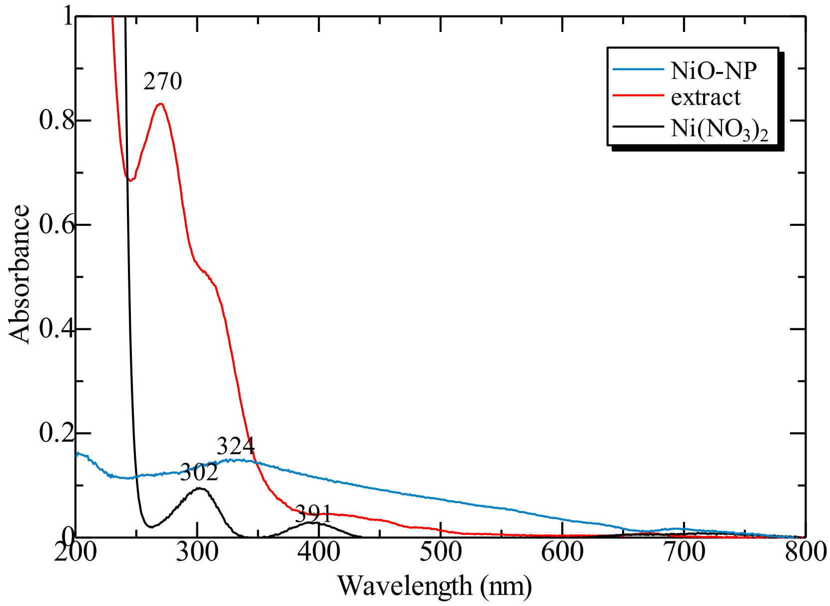

Figure 1. Absorption spectra of NiO-NP, precursor, and extract

Identification of functional group of NiO-NP was recorded using FT-IR spectroscopy. Fig. 2 shows the wavenumber shifting of *Ageratum conyzoides* L. leaf extract and NiO-NP. *Ageratum conyzoides* L. leaf extract shows FT-IR spectra at the wavenumber 1077 cm^{-1} as C-N stretching indicating that leaf extract contain alkaloid [19]. FT-IR spectra of NiO-NP shows the absorption peak at the wavenumber of 434 cm^{-1} as Ni-O stretching vibration mode. This result is related to the previous study [7].

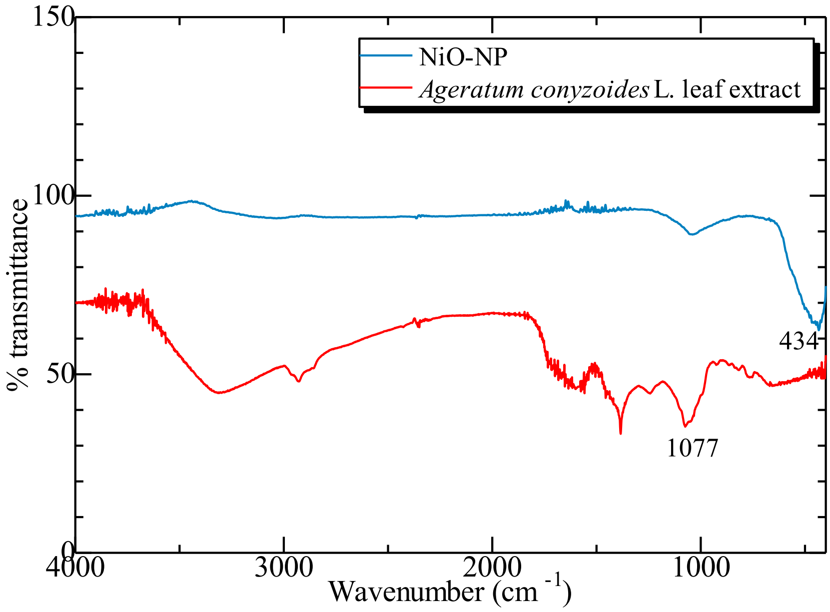

Figure 2. FTIR spectra of NiO-NP and extract.

The particle size distribution of NiO-NP was analysed by PSA as shown in Fig. 3. PSA result show the particle size distribution of NiO-NP was at 50.70 - 91.3 nm. This size proved that the synthesized NiO using *Ageratum conyzoides* L. leaf extract was in nanoparticles form.

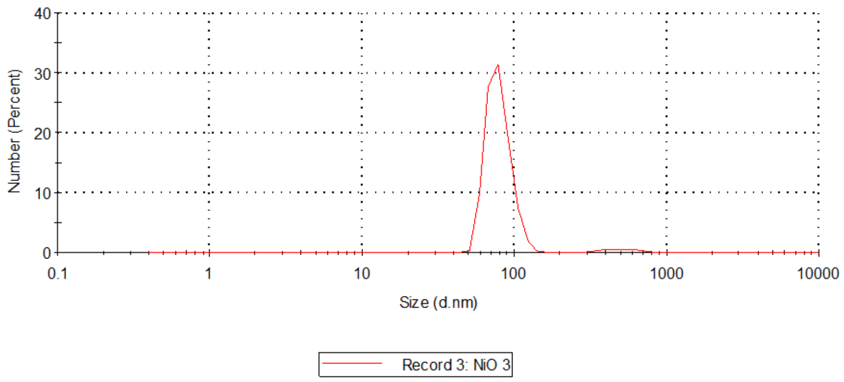

Figure 3. Particle size by number of NiO-NP.

The structural information and crystalline size of NiO-NP were studied by XRD. Fig. 4 shows the strong intense peak of NiO-NP. XRD pattern shows five diffraction peaks at 2θ of 37.18°, 43.12°, 62.88°, 75.24°, and 79.23° associated with COD data of NiO 96-101-0096. According to this data, the miller indices of NiO correspond to (111), (200), (220), (311), and (222). The crystalline structural of NiO-NP was cubic with the space group Fm-3m (COD 96-101-0096). The average crystalline size of NiO-NP was 11.5 nm which was calculated using Debye Scherrer equation.

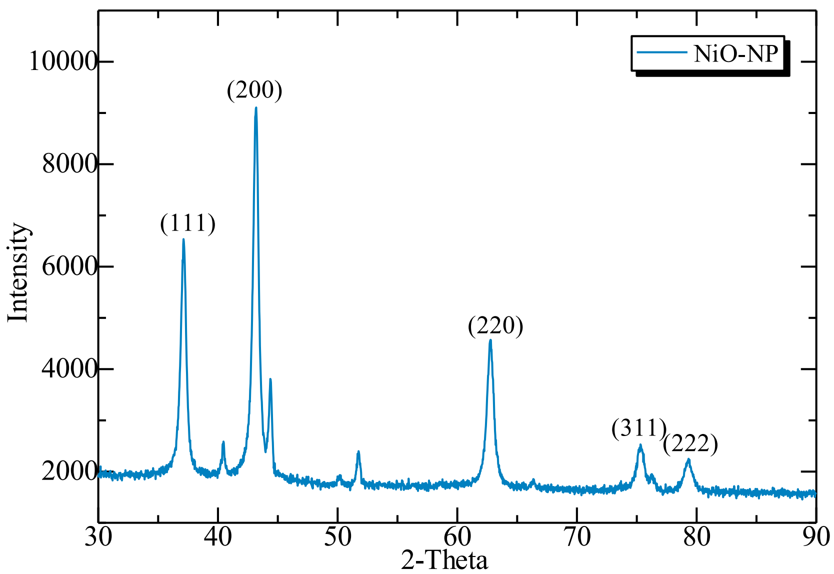

Figure 4. XRD pattern of NiO-NP.

The morphology and particle size of NiO-NP were examined using TEM characterization. Fig. 5 (a) and (b) shows the TEM image of NiO-NP with scale bar 20 nm and 50 nm. The image showed that NiO-NP had agglomeration with cubic shape morphology. The particle size of NiO-NP was about 8-15 nm.

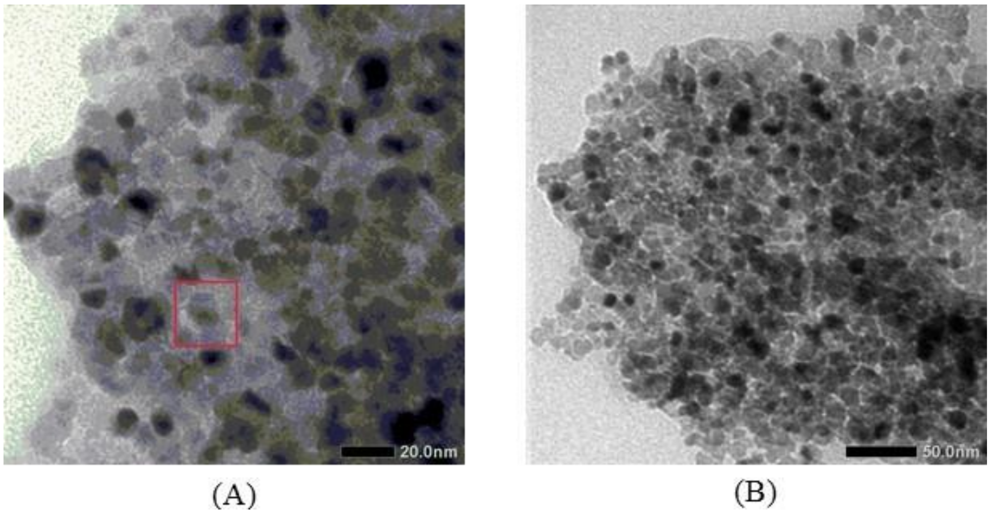

Figure 5. TEM image of NiO-NP.

The reduction of methylene blue as a dyestuff model was used to investigate the catalytic activity of NiO-NP. The reduction of methylene blue to leucomethylene blue was carried out using $NaBH_4$ as a reducing agent and monitored by UV-Vis spectrophotometer. Methylene blue shows the absorption typical peak at the wavenumber of 664 nm. As a comparison, methylene blue was reduced without NiO-NP catalyst. The complete reduction takes place for 18 minutes with the reduction percentage of 83% as shown in Fig. 6. However, the catalytic activity without catalyst was 28%. This result demonstrated that NiO-NP acts as an effective catalyst in the reduction of methylene blue. The accelerated reduction of methylene blue in the presence NiO-NP indicate that NiO-NP help in the electron relay from BH_4^- (donor) to methylene blue (acceptor) [20].

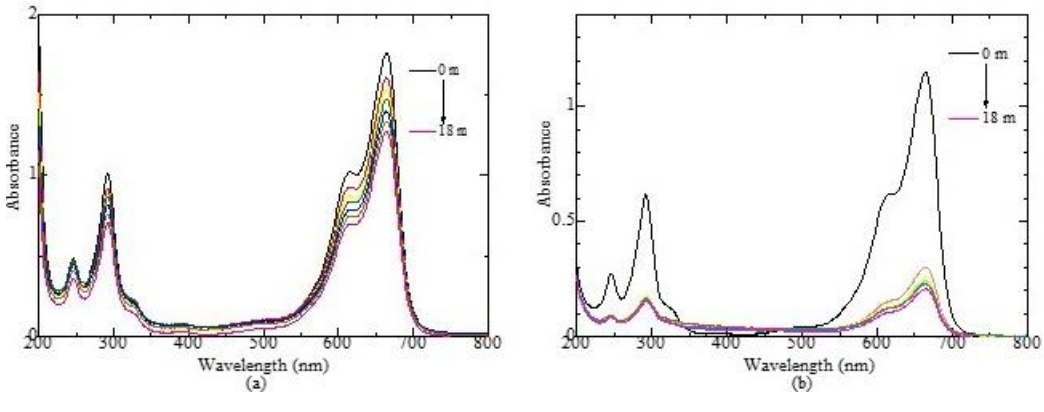

Figure 6. The reduction process of methylene blue (a) without (b) with NiO-NP catalyst

4. Conclusions

NiO-NP was successfully synthesized using *Ageratum conyzoides* L. leaf extract via green method. The absorption spectra of NiO-NP by UV-Vis spectrophotometer was observed at the maximum wavelength of 324 nm. FT-IR result showed that there was Ni-O stretching vibration at the wavenumber of 434 cm^{-1}. The particle size distribution of NiO-NP was at 50.70 – 91.3 nm. XRD pattern showed the cubic crystalline phase with the average crystalline size of 11,5 nm. TEM characterization showed the particle size of NiO-NP was at 8-15 nm. NiO-NP was proven as an efficient catalyst with the reduction of methylene blue with percentage value of 83% for 18 minutes.

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