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C. Arunkumar and A. Manikandan

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Green Synthesis of Nickel Aluminate ($NiAl_2O_4$) Nanoparticles using the Plant Extract of *Pedalium murex*

C. Arunkumar[1] and A. Manikandan*[2]

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A B S T R A C T

In this present study, spinel nickel aluminate ($NiAl_2O_4$) nanoparticles (NPs) were synthesized using *Pedalium murex Linn* (family: *Pedaliaceae*) extract, aluminium (III) nitrate and nickel (II) nitrate as precursors. Structural and morphological properties of $NiAl_2O_4$ NPs were determined by high resolution scanning electron microscopy (HR-SEM), high resolution transmission electron microscopy (HR-TEM), powder X-ray diffraction (PXRD), vibrating sample magnetometer (VSM), and Fourier transform infra-red (FT-IR) analysis. PXRD analysis confirmed the pure phase formation of $NiAl_2O_4$ NPs. HR-SEM and HR-TEM analysis showed sphere shaped particle like nanostructures. VSM analysis shows a hysteresis loop with superparamagnetic behaviour.

***Key words*:** $NiAl_2O_4$ NPs, *Pedalium murex Linn* extract, Green synthesis, Magnetic properties

Spinel nickel aluminates ($NiAl_2O_4$) NPs have recently been discovered to be ceramic nanoscale materials that can be used in a variety of applications. According to new research, a variety of nanomaterials can cause dye degradation [1]. Because of their nanoscale size, catalytic characteristics, surface plasmonic resonance, magnetic nature, and other qualities, these nanomaterials are used [2-4]. Ferrite magnetic NPs with the chemical formula MAl_2O_4 (M = Ni, Co, Cu, etc.) [5] have piqued the interest of researchers in a variety of biological domains, including photocatalysis, hyperthermia, cell labelling and so on, in recent decades. Magnetic resonance imaging (MRI) contrast agents made of spinel ferrite NPs can be used [6-10]. In this study, $NiAl_2O_4$ NPs were eco-friendly synthesized using *Pedalium murex Linn* extract. The physical and chemical properties of the $NiAl_2O_4$ NPs were determined by X-ray powder diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and vibrating-sample magnetometer (VSM) analyses. The obtained results were discussed herein detail.

MATERIALS AND METHODS

Aluminium (III) nitrate, nickel (II) nitrate (Sigma-Aldrich, 99%), and *Pedalium murex Linn* extract were utilized for the green synthesis of $NiAl_2O_4$ NPs. Deionized (DI) water was utilized in all stages of the synthesis. Aluminium (III) nitrate, nickel (II) nitrate and *Pedalium murex Linn* extract mixture is placed in the microwave oven with the 850W. The mixture was kept in a microwave oven. The final product were washed well with DI water and ethanol twice finally dried at 70°C and used for further characterizations.

Characterization techniques

The structural characterization of $NiAl_2O_4$ NPs were performed using Rigaku Ultima X-ray diffractometer equipped with Cu-Kα radiation (λ =1.5418 Å). The surface functional groups were analyzed by Perkin Elmer FT-IR spectrometer. Morphological studies and energy dispersive X-ray analysis (EDX) of $NiAl_2O_4$ NPs have been performed with a Jeol JSM6360 high resolution scanning electron microscopy (HR-SEM). UV-Visible diffuse reflectance spectrum (DRS) was recorded using Cary100 UV-Visible spectrophotometer to estimate their band gap energy (E_g). Magnetic measurements were carried out at room temperature using a PMC MicroMag 3900 model vibrating sample magnetometer equipped with 1 Tesla magnet.

RESULTS AND DISCUSSION

Powder XRD analysis

Figure 1 shows the powder XRD diagram of $NiAl_2O_4$ NPs. The peaks mentioned in the XRD spectra correspond to the cubic spinel structure of the $NiAl_2O_4$ NPs [11-13]. The crystallization degree of $NiAl_2O_4$ NPs depends on the synthesis procedure, temperature and time. The average crystallite size of $NiAl_2O_4$ NPs was calculated using Debye Scherrer formula given in eqn. 1:

$$L = \frac{0.89\lambda}{\beta \cos\theta} \qquad \text{---- (1)}$$

* **A. Manikandan**
✉ manikandan.research@bharathuniv.ac.in

[1-2] Department of Chemistry, Bharath Institute of Higher Education and Research (BIHER), Chennai - 600 073, Tamil Nadu, India

where L is the crystallite size, λ, the X-ray wavelength, θ, the Bragg diffraction angle and β, the full width at half maximum (FWHM). The average crystallite size 'L' calculated from the diffraction peaks was found to be around 18 nm.

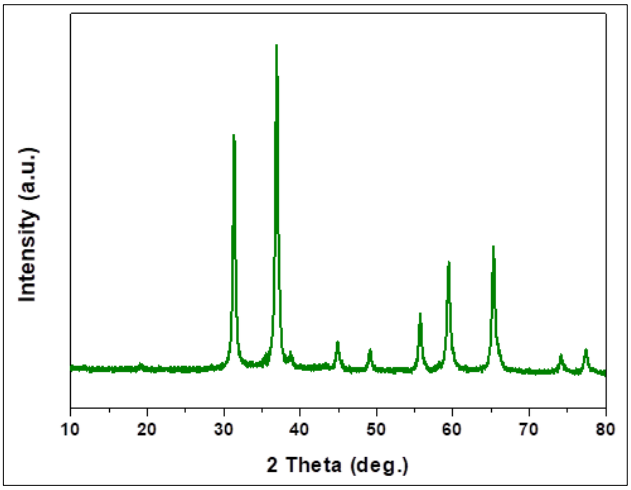

Fig 1 XRD analysis of $NiAl_2O_4$ NPs

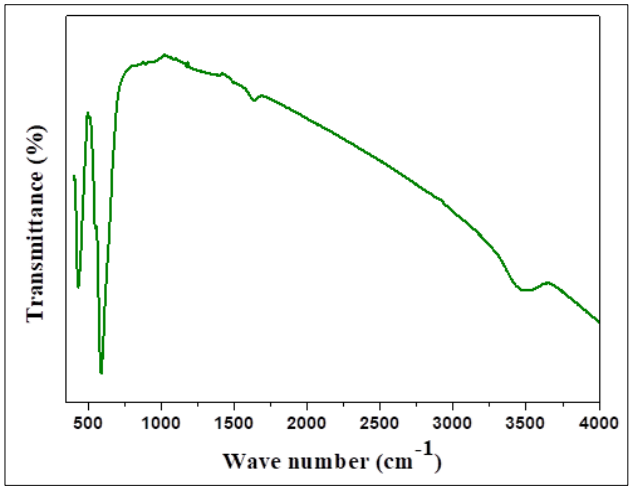

Fig 2 FT-IR spectra of $NiAl_2O_4$ NPs

FT-IR analysis

Fourier change infrared spectra (FTIR) of spinel $NiAl_2O_4$ NPs are shown in Fig. 2. The band around 435 – 750 cm^{-1} shows the formation of spinel $NiAl_2O_4$ NPs. A band at 525 cm^{-1} is distinguished for Al-O and Ni-O. The wave number of 3435 cm^{-1} is more ingestion band is noticed H_2O molecules [22]. The bowing methods of O-H band are seen in roughly 1622 cm^{-1}.

UV-Visible spectral analysis

The band gap energy (E_g) of $NiAl_2O_4$ NPs can be evaluated using the Kubelka - Munk model. UV-Vis. diffuse reflectance (UV-DRS) spectra of $NiAl_2O_4$ NPs were shown in Fig. 3 and the band gap energy E_g 2.47, indicating that $NiAl_2O_4$ NPs exhibited an intense absorption in the visible range [16-18]. DRS analysis was used to study the relation of crystallite size and band gap of the semiconductors. Kubelka-Munk function, $F(R)$ is directly proportional to the absorption coefficient (α) and the value is estimated from the following eqn. 2,

$$(F(R)) = \alpha = \frac{(1-R)^2}{2R} \qquad \text{---- (2)}$$

where, $F(R)$ is Kubelka-Munk function, α, the absorbance, R, the reflectance. A graph is plotted between $[F(R)h\upsilon]^2$ and $h\upsilon$, the obtained intercept value is the E_g of the $NiAl_2O_4$ NPs. The estimated E_g of $NiAl_2O_4$ NPs is 2.47 eV.

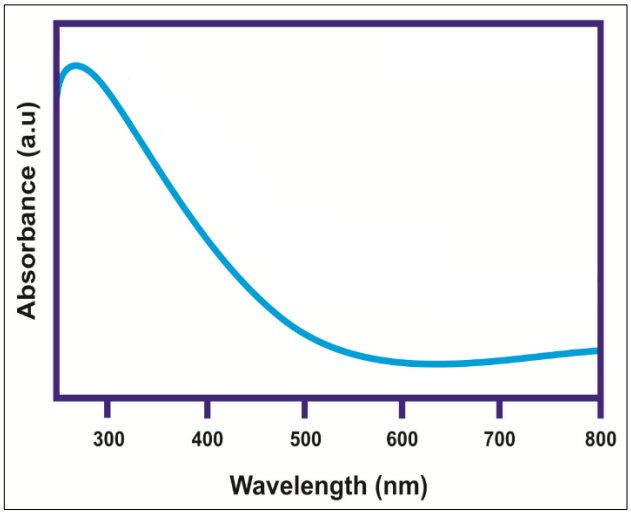

Fig 3 UV-Vis absorption spectra of $NiAl_2O_4$ NPs

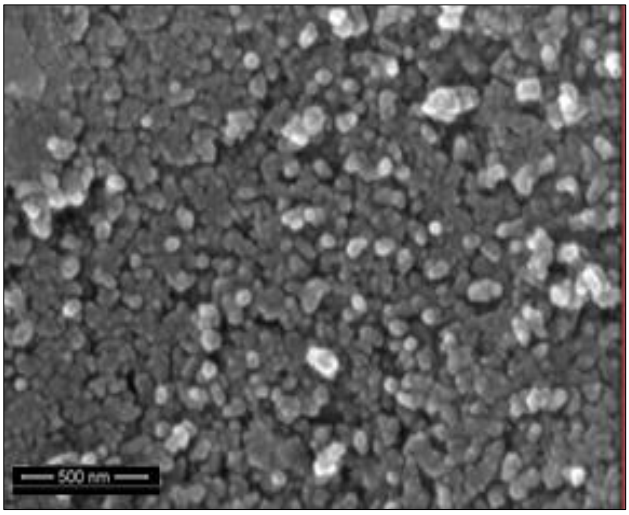

Fig 4 SEM image of $NiAl_2O_4$ NPs

SEM analysis

Figure 4 shows HR-SEM images of $NiFe_2O_4$ NPs prepared by *Pedalium murex Linn* extract assisted microwave combustion method. HR-SEM images of the $NiAl_2O_4$ NPs confirmed the nanostructured spherical shaped surface morphology of the products with 14 to 18 nm sizes of the NPs, which are crystallized well in a short time by the microwave heating route.

TEM analysis

HR-TEM image of $NiAl_2O_4$ NPs are exposed in Fig. 5a. HR-TEM images confirmed the prepared particles are well formed and are in nano regime. The micrographs establish the reasonable uniformity of the particle in size and shape without any agglomeration and particle size around 12-18 nm, which is in close agreement with XRD crystallite size. SAED pattern of of $NiAl_2O_4$ NPs are as shown in Fig. 5b. Several concentric rings in the figure suggest that the as prepared material is well crystalline in nature.

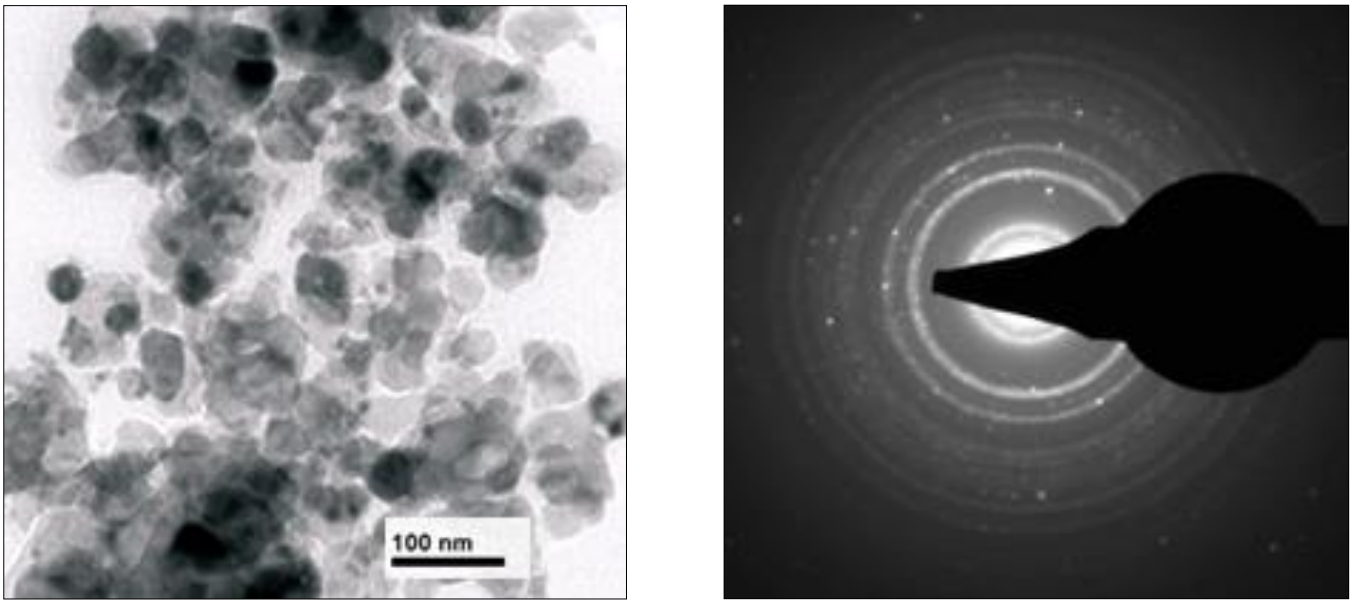

Fig 5 TEM image (a) and SAED pattern (b) of $NiAl_2O_4$ NPs

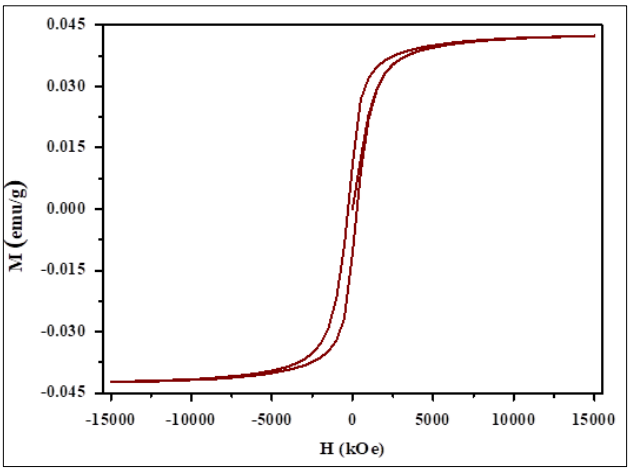

Fig 6 VSM results of $NiAl_2O_4$ NPs

VSM analysis

The magnetic properties of spinel $NiAl_2O_4$ NPs at ±15kOe applied field are shown in Figure 5. The amount of magnetic saturation (M_s) for the synthesized $NiAl_2O_4$ NPs was 4.256×10^{-2} emu/g. The obtained result shows superparamagnetic in nature [19-21]. Additionally, the amount of Ms of $NiAl_2O_4$ NPs depends on their size, crystallinity and structure [22-25]. The spinel structure and superparamagnetic behaviour of spinel $NiAl_2O_4$ NPs were confirmed by XRD and VSM analyses.

CONCLUSION

Spinel nickel aluminate ($NiAl_2O_4$) nanoparticles (NPs) were synthesized using *Pedalium murex Linn* (family: *Pedaliaceae*) extract, aluminium (III) nitrate and nickel (II) nitrate as precursors. Structural and morphological properties of $NiAl_2O_4$ NPs were determined by HR-SEM, HR-TEM, XRD, VSM, and FT-IR analysis. XRD analysis confirmed the pure phase formation of spinel $NiAl_2O_4$ NPs. HR-SEM and HR-TEM analysis showed sphere shaped particle like nanostructures. VSM analysis recorded at room temperature shows a hysteresis loop with superparamagnetic behaviour.

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