Green synthesis of Magnesium oxide nanoparticles with antioxidant potential using leaf extract of Piper nigrum

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Short Report

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Abstract

Green fabrication is an environmental friendly and innovative method and attractive research field for the production of Magnesium oxide nanoparticles (MgO NPs) in clinical and environmental applications. In this current study, a viable green synthetic approach to produce magnesium oxide nanoparticles by using *P. nigrum* leaf aqueous extract as a capping agent. The formation and physicochemical properties of MgO NPs were confirmed through stranded characterization methods. All the analytical data revealed that the formation of pure, rod shaped and crystalline MgO NPs with average size of 20 nm. This study reports that, the synthesized MgO NPs exhibiting good antioxidant activity and it can be used for bio-medical applications.

Introduction

The advances are made in the field of biotechnology and nanotechnology paves the way for the research in the field of nanoparticle synthesis. The nanoparticles are extensively used for the treatments of cancer, diabetes, allergy, infection, inflammation. It is used in various applications in the field of medicine, cosmetics, renewable energies, environmental remediation and biomedical devices (Patil et al., 2015). Magnesium oxide (MgO) is known as a widely used nanoparticles and having unique optical, electronic, thermal, mechanical and chemical properties (Ramanujam and Sundrarajan 2014). MgO NP_S is an important functional metal oxide that has been widely used in various fields, such as catalysis, refractory materials, paints, and superconductors (Salem et al. 2015). Several methods have been used for the synthesis of nano MgO, including the sol–gel method, chemical gas phase deposition, laser vaporization, hydrothermal synthesis, and combustion aerosol synthesis (Mirzaei and Davoodnia 2012). Biological methods for the synthesis of MgO NPs use of plant materials have not been widely exploited (Sushma et al. 2015). Green synthesis of nanoparticles is an eco-friendly approach because the biological components act as reducing and capping agent and no need of high energy, toxic chemicals, high pressures, etc. Biological synthesis can be used at large scale production of nanoparticles. *Piper nigrum* Linn. or Black pepper is a perennial woody climbing liana belonging to the family Piperaceae. It is native to India, Indonesia, Malaysia, South America and West Indies but it is widely cultivated in the tropical regions. It is considered as the 'King of Spices' (Srinivasan, 2007; Mathew et al. 2001). The presence of a pungent alkaloid piperine attributes a spicy taste to the seeds, leaves and other parts of Piper (Tripathi et al. 1996; Khajuria et al. 2002). It contains small amounts of safrole, pinene, sabinene, limonene, caryophyllene and linalool compound. Fruits of *P. nigrum* shows different activities like hepatoprotective, antipyretic, CNS depressant, analgesic, bioavailability enhancer, antioxidant and anti-inflammatory (Majumdar et al. 1990; Khajuria et al. 1997; Zhao et al. 2007; Kasibhatta and Naidu, 2007).

The distinguishing of nanoparticles is decided by the source of plant extracts. Plant extract is act as both reducing and stabilizing agent. Mallikaarujuna et al., (2012) reported the investigation on phytochemical fabrication and characterization of silver nanoparticles by using extract of pepper leaf. The study was performed by Jesi Reeta Thangapandi et al. (2021) on antibacterial and photocatalytic activity for *P.*

nigrum seed extracts mediated zinc oxide nano-rods. They conclude that *P. nigrum* seed extracts mediated zinc oxide nanoparticles shows better antibacterial activity and photocatalytic activity.

Till now there's no study was reported for synthesis of MgO nanoparticles using the leaf extract of *P. nigrum*. In the present study, MgO nanoparticles were synthesized using *P. nigrum* leaves and characterized. In addition, the antioxidant potential of *P. nigrum* leaf mediated MgO nanoparticles was assessed.

Materials And Methodology

Collection of *P. nigrum* and preparation of extract

P. nigrum leaves were collected from pepper garden, Thrissur district, DMS Lat: 10° 31’49.2420” N, DMS Long: 76°12’53.0244” E, Kerala, India. The leaves were washed with running tap water and cut into small pieces. The extract of *P. nigrum* was prepared using 100 ml distilled water and it allow for boiling at 100 C at 30 min. The obtained extract was filtered using Whatmann No1 filter paper and it stored at 4°C for further use (Sivaraj et al., 2014).

Synthesis of MgO NPs

10g of Magnesium nitrate was dissolved in 100 ml distilled water and it was allowed to boil at 60°C for 20 min. Then, the 50 ml of *P. nigrum* extract was added to 100 ml of Magnesium nitrate solution and it was kept at 60°C for 30 min. Then the solution was incubated for 24 hours and dark greenish precipitate was obtained after the incubation period. Next, the obtained precipitate was annealed at 400°C-500°C for 3 hours. After the annealing process fine powder was obtained & stored at 4°C for further studies (Vanathi et al., 2014).

Characterization of MgO NPs

The optical property of MgO NPs was observing the using UV–Visible spectrophotometer, a wavelength ranges of 200–800 nm. The functional groups and chemical residues on surface of MgO nanoparticles were detected using FTIR (Perkin Elmer Spectrum 1000). The crystalline nature of prepared MgO nanoparticles was identify using XRD patterns which was determined by using X-ray diffractometer. The size of MgO NP_S was calculated on the basis of Scherrer’s formula (Dobrucka, 2018). The microstructure, particle distribution, elemental composition and purity of the MgO NPs were determined by scanning electron microscope (SEM) equipped with an energy-dispersive X-ray analyzer (EDX) unit (SEM: JEO LJSM 7660F). The stability of synthesized MgO nanoparticles was analyzed by using zeta potential (ZP) analyzer (Malvern Zetasizer nano-ZS90). Thermogravimetric analysis (TGA) was performed with a TA Instrument TGA-SDT 2960. For this purpose, the samples were heated in flowing air (100 mL/min) from 35 to 900°C at a heating rate of 10°C/min (Karunakaran et al., 2020).

Antioxidant Activity

The antioxidant potential of MgO NPs was determined by DPPH (2,2-diphenyl-1- picrylhydrazyl) method. Different concentration of MgO NPs such as 50, 100, 200 and 500 µg\ml were prepared. Water (0.1 ml) and methanol (0.7 ml) were used as reference. The measurement was made by measuring the absorbance of 0.1 ml of deionized water and 0.7 ml of DPPH solution. The ability to reduce free DPPH radicals was calculated based on the formula: Aa=(Ao−Ai/Ao) ×100%, where Aa means antioxidant activity [%], Ai means average absorbance of the tested solution, and Ao means average absorbance of the DPPH solution.

Result And Discussion

Characterization/ physicochemical properties of MgO NPs

UV-Spectra of MgO nanoparticles was presented in Figure 1 and it shows the peak at 364 -571 nm. The color change depicts the existence of the formation of MgO nanoparticles. The surface Plasmon resonance bands of colloidal Magnesium was observed in the range 364 to 571 nm. Prasanth et al., (2019) reported the formation of MgO NPs by green synthesis and confirmed by UV–Vis spectrophotometry. They obtained peak at 301 nm in UV–Vis spectrum.

FTIR spectrum of MgO NP_S is depicted in Figure 2. The peaks of 3366, 2839, 2672, 1759, 1548, 783 and 470 cm^{-1} were observed in *P. nigrum* mediated MgO NPs. The peaks of 783 and 470 cm^{-1} refer the group of Mg-O (Jeevanandam et al., 2017). A peak at 3366 cm^{-1} corresponding to O–H/N–H vibrations (Abdallah et al., 2019). The peaks at 2839 and 2672 cm^{-1} indicates the presence of C-H asymmetric and symmetric stretching mode. The bands at 1759 and 1548 cm^{-1} which corresponds to O–H stretching vibrations. Nguyen et al. (2021) were produced the MgO nanoparticles from different extracts (flower, bark, leaf) of *Tecoma stans* (L.) by biogenic synthesis and proved the MgO NPs formation by FT-IR analysis through the existence of Mg-O functional groups at 514–658 cm^{-1}.

Figure 3 show the XRD pattern of green synthesized MgO NP_S. It was used for further confirmation of MgO NP formation. The observed intense peaks were representing the (111), (200), (311) and (420). Bragg's reflection angles based on the crystal of magnesium nanoparticles. These phases were indexed to crystalline nature, which was compared to the data from JCPDS card #00–004–0829.

$D=0.9\ \lambda/\beta\cos\ \theta$

Where, D is average particles size, λ is wavelength (1.5418 Å), θ is the Bragg's angle and β is full width half maximum (FWHM) of corresponding peek. The Scherer's formula was used to estimate the particles sizes and was found to be in the range of 15-30 nm (Nguyen et al. (2021). The average size of MgO NPs was calculated and which ranged from 15-30 nm. Srivastava et al., (2015) determine the green synthesis of magnesium oxide nanoflower and they used the XRD for study the crystalline nature of bio-inspired MgO NPs.

Scanning electron microscopy image of MgO nanoparticles were determines that the particles are in rod shape (Figure 4). SEM image showed well-dispersed nano rod of MgO-NPs without any aggregation. Hassan et al. (2021) produce the spherical shape *Rhizopus oryzae*-mediated magnesium oxide nanoparticles and the studied its morphology with help of SEM. The elemental composition of MgO NPs was investigated with help of EDX spectra (Figure 5). The EDX graph showed that the phyto-mediated MgO-NPs are extremely pure. it holds Mg and O ions which refers the formation of MgO through aqueous extract of *P. nigrum*. Fouda et al., (2022) were synthesized the biogenic MgO NPs via *Cystoseira crinite* (Brown Algae) and they revealed the weight percentages of Mg and O by EDX analysis.

Zeta potential is the important analysis for determining the stability of MgO nanoparticles suspension. The zeta potential analysis revealed that MgO NPs holds high negative potential value of −75.1 mV (Figure 6), which indicated the physical stability of synthesized MgO nanoparticles. Ammulu et al., (2021) were investigated on phyto-assisted synthesis of MgO NPs using *Pterocarpus marsupium* rox.b heartwood extract, which have zeta potential of − 2.9 mV. The thermal stability of MgO NPs was analyzed up to 900°C (Figure 7a and b). An initial weight loss of 22.3% was observed up to 300°C due to dehydration. Overall 59% of weight loss was occurred up to 900°C. Figure 7 a represents the exothermic peaks at 300 and 400°C.

Antioxidant activity

DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) assay was used to determine the radical scavenging activity of *P. nigrum* synthesized MgO NPs (Figure 8). The low concentration 50 µg/mL MgO NPs shows 29.99% free radical scavenging whereas the highest concentration of 500 µg/mL MgO NPs exhibits the 91.99% of free radical scavenging compared with ascorbic acid. The IC_{50} of MgO NPs was found to be 89.5 µg/mL. Younis et al., (2021) were produced the MgO NPs using Rosa floribunda charisma extract and they evaluate its antioxidant activities by DPPH method. They conclude that green synthesized MgO NPs have good antioxidant activity. The DPPH assay result suggested that biologically synthesized MgO NPs can act as active antioxidant agents in the field of biomedicine.

Conclusion

In the present study, the aqueous extract of *P. nigrum* was employed as a capping/stabilizing and reducing agent for the biosynthesis of MgO NPs. The obtained MgO nanoparticles were characterized by the FTIR, SEM, EDX, TGA, Zeta Potential. The crystalline and rod MgO NPs with an average size of 26 nm was prepared by biological method. *P. nigrum* assisted d MgO NPs holds good antioxidant properties which proved by DPPH assay. Green synthesized MgO NPs may be used for medical application as nano drug.

Declarations

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The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

-Ethical Approval– Not applicable

-Consent to Participate– Not applicable

-Consent to Publish – Not applicable

-Authors Contributions

PR: Supervision, Funding acquisition and Project administration

NV: Investigation, Methodology, Data Curation and Writing- Original draft preparation

JD: Data Curation and Writing

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-Competing Interests

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-Availability of data and materials– Not applicable

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*** Authors' information (optional).** – Not applicable

Please include the sub-sections below of Compliance with Ethical Standards section. – Not applicable

*** Disclosure of potential conflicts of interest** – Not applicable

*** Research involving Human Participants and/or Animals** – Not applicable

*** Informed consent** – Not applicable

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Figures

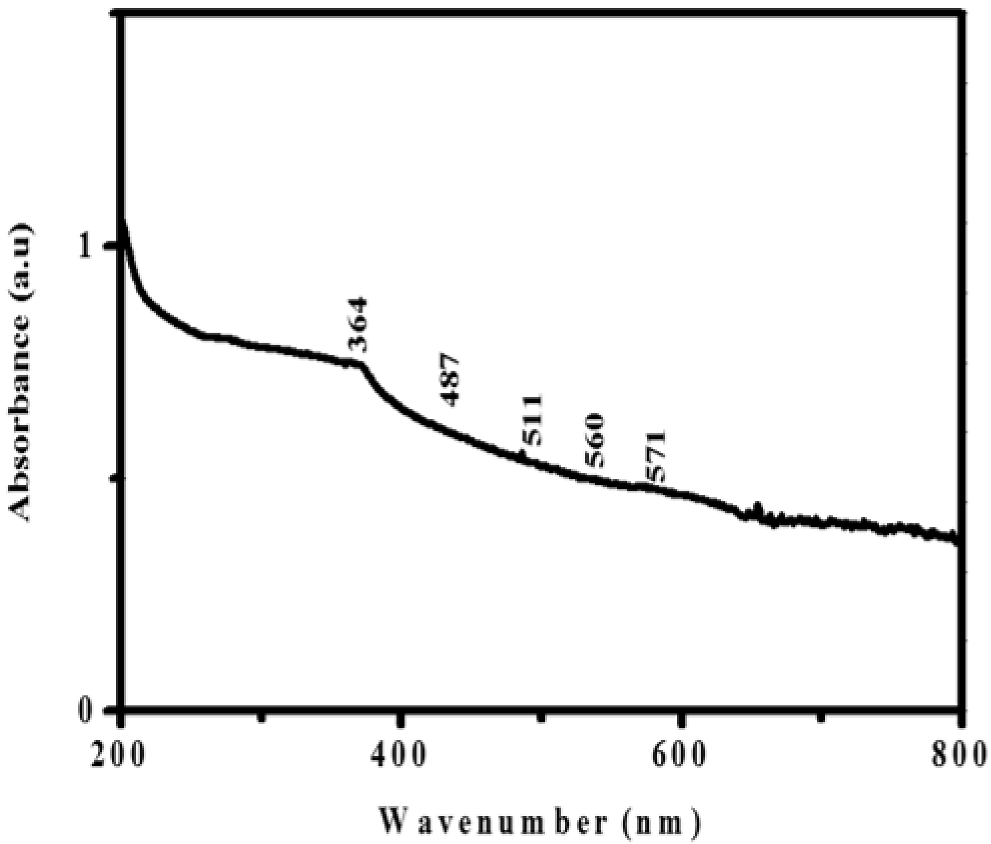

Figure 1

Analysis of optical property for *P. nigrum* mediated MgO NPs.

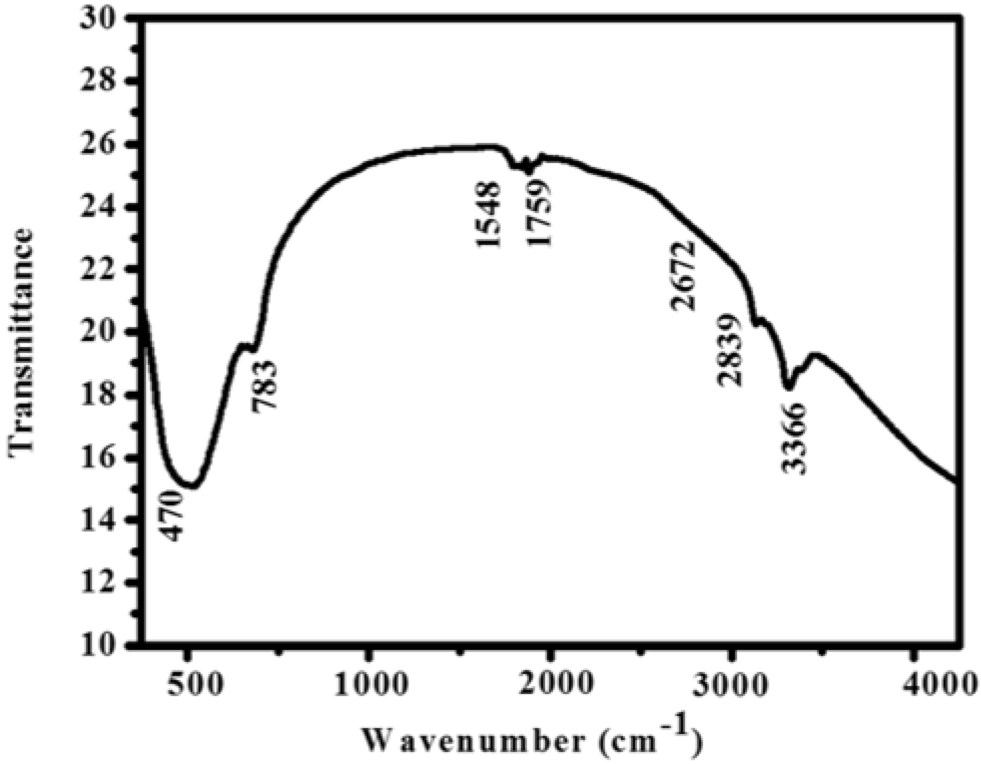

Figure 2

Analysis of functional groups on surface of *P. nigrum* mediated MgO NPs.

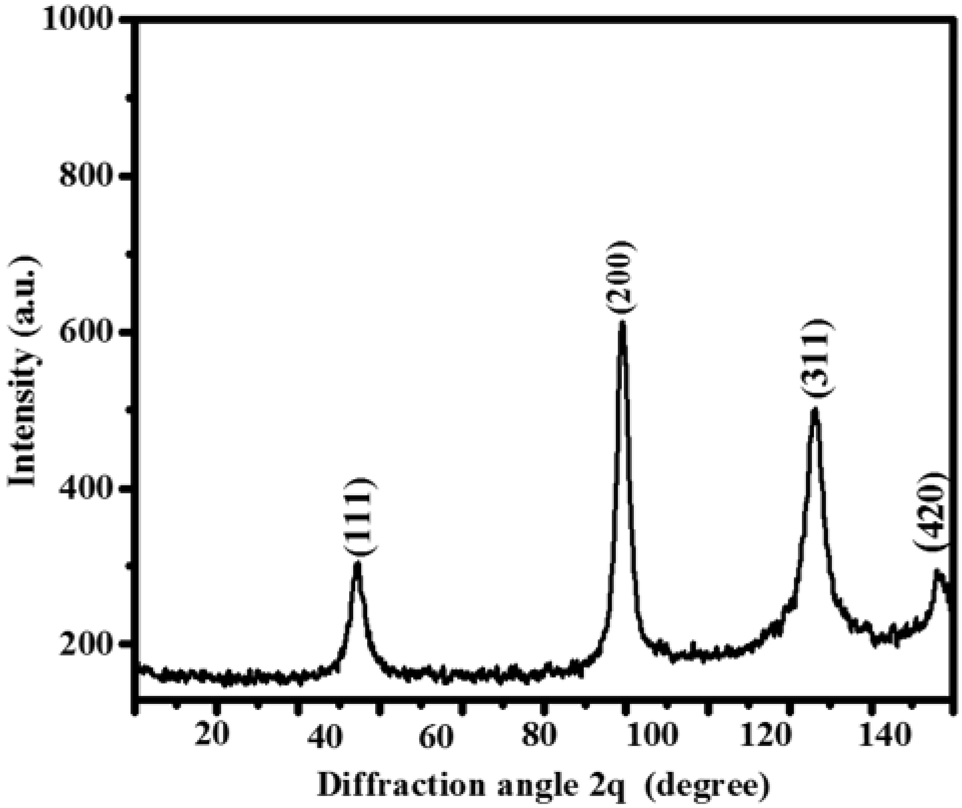

Figure 3

Analysis of crystalline nature for *P.nigrum* mediated MgO NPs by XRD

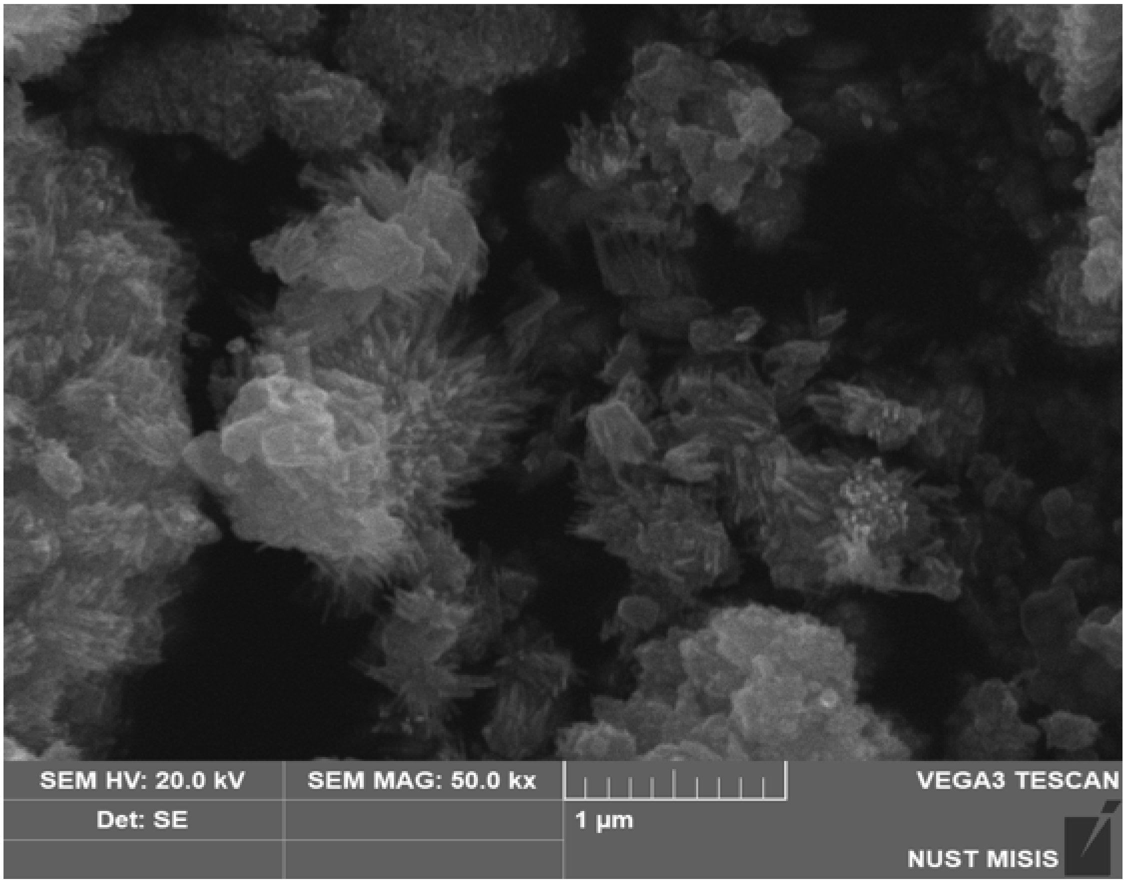

Figure 4

Analysis of shape for *P.nigrum* mediated MgO NPs by SEM

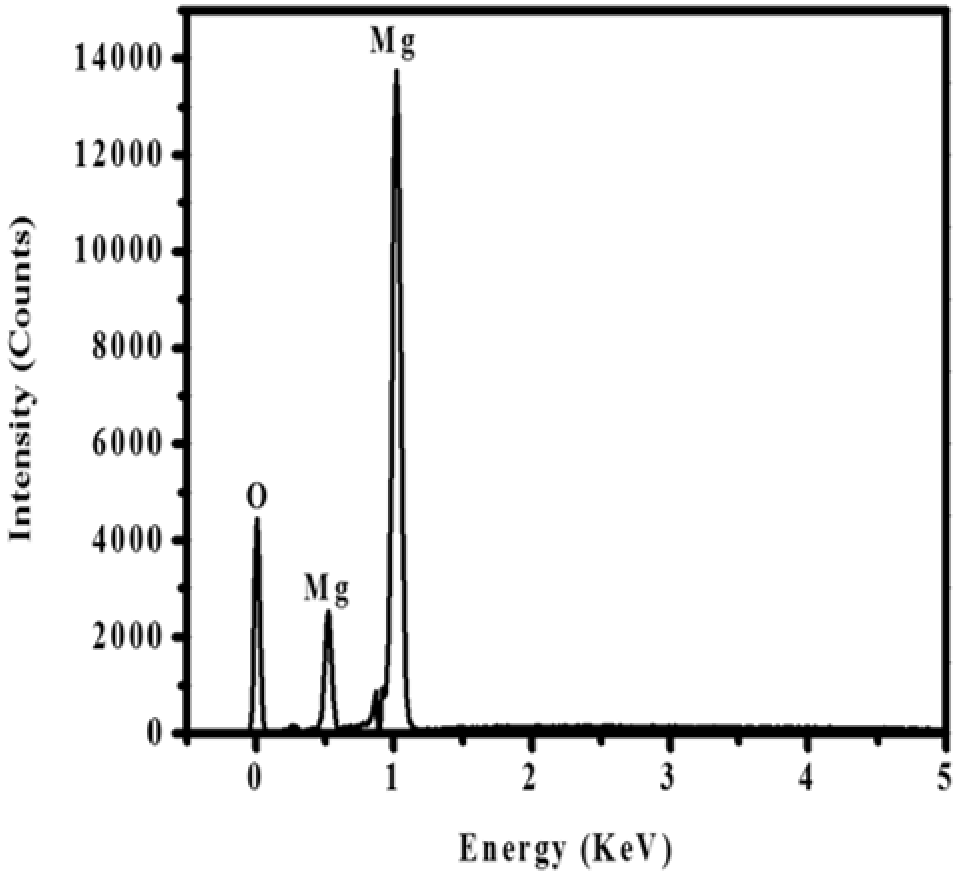

Figure 5

Analysis of elemental composition for *P.nigrum* mediated MgO NPs by EDX

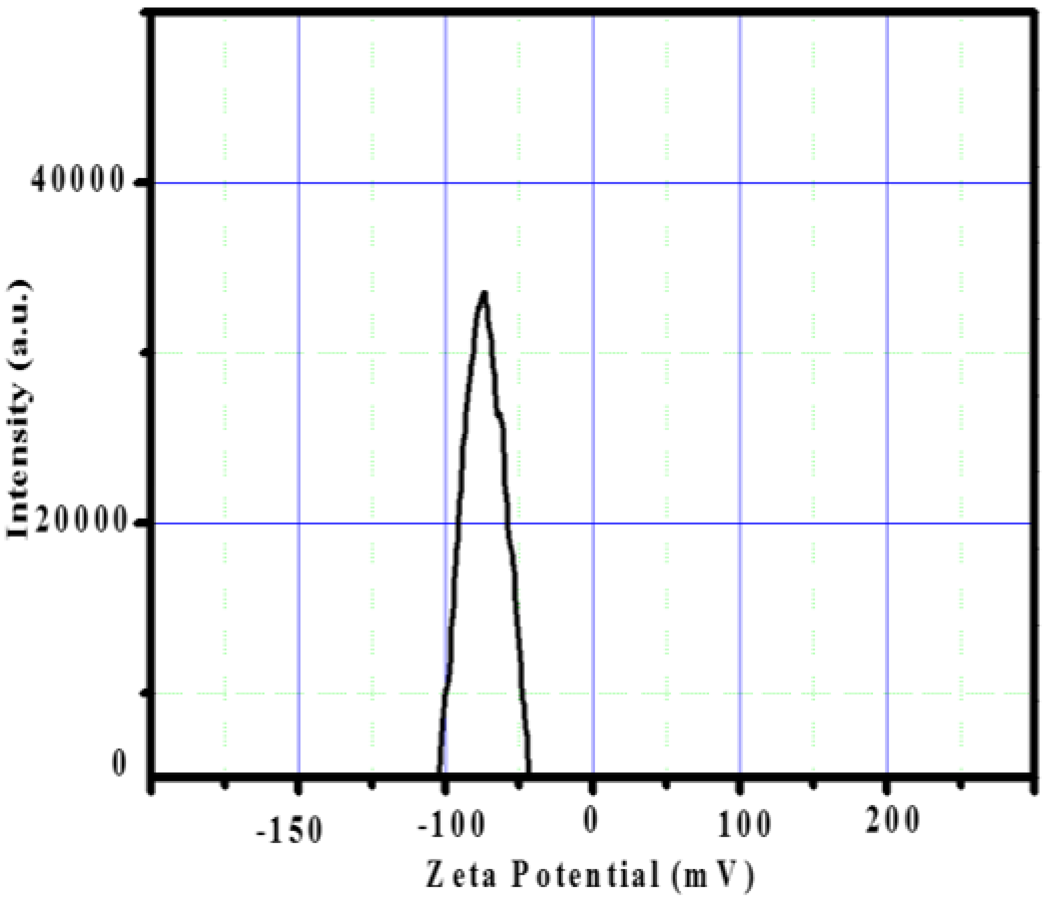

Figure 6

Analysis of zeta potential for *P.nigrum* mediated MgO NPs

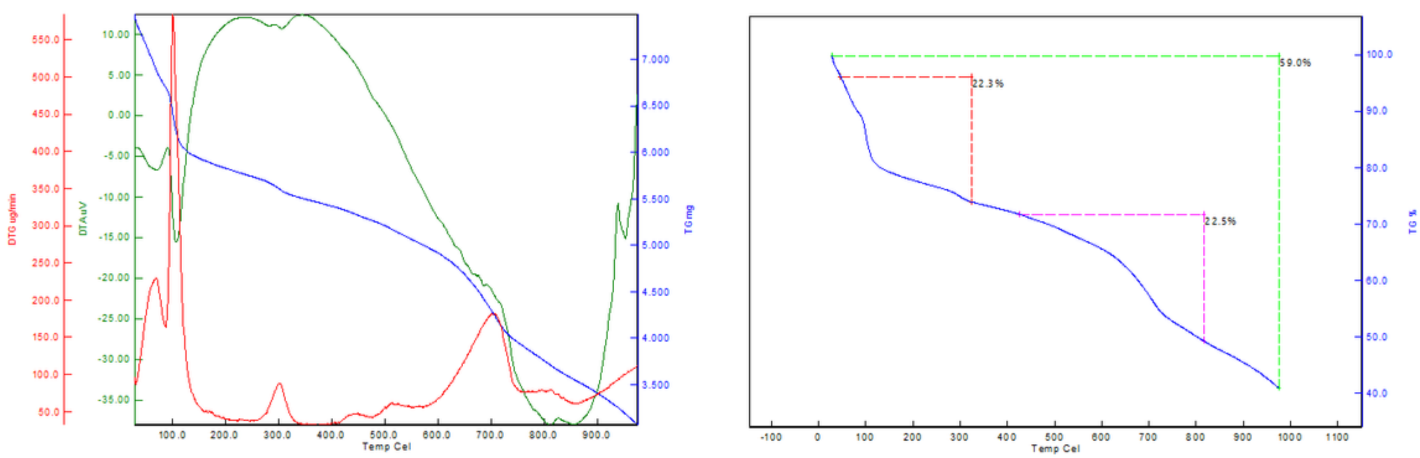

Figure 7

a. Analysis of thermal stability for *P.nigrum* mediated MgO NPs

b. Analysis of thermal stability for *P.nigrum* mediated MgO NPs

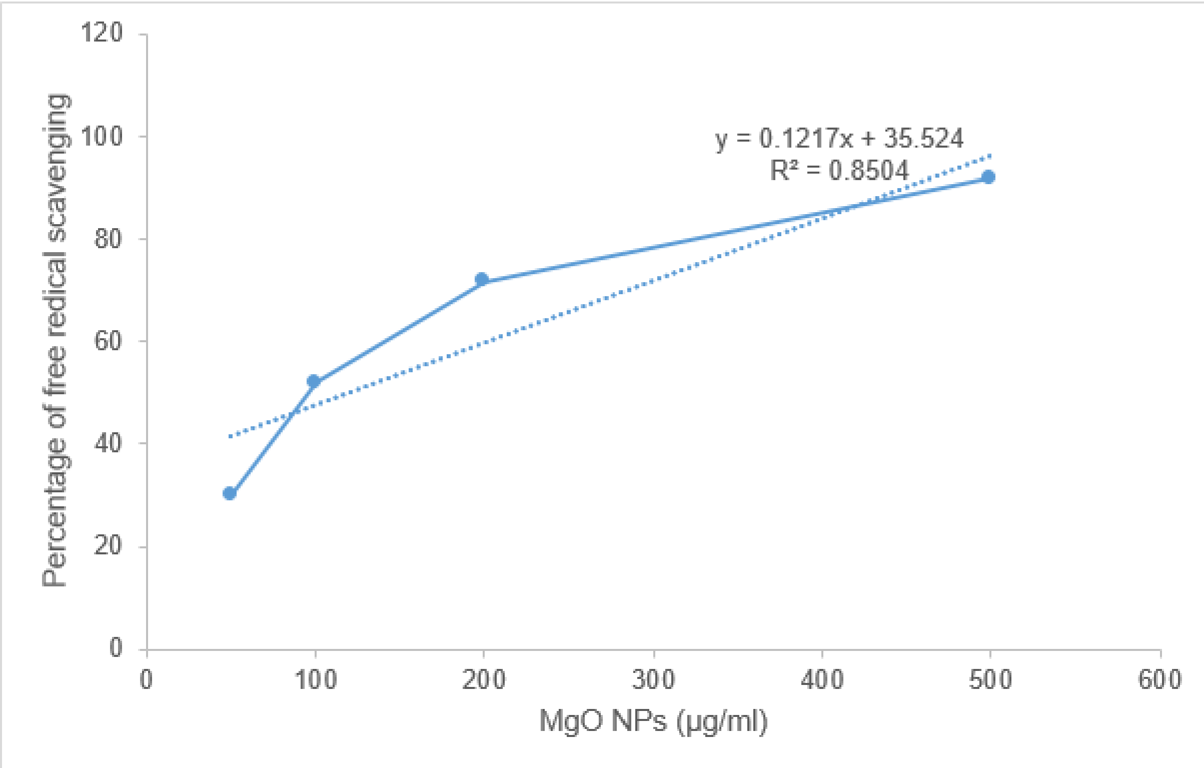

Figure 8

Analysis of antioxidant activity for *P. nigrum* mediated MgO NPs by DPPH method