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Synthesis and characterization of biogenic iron oxide nanoparticles using green chemistry approach and evaluating their biological activities

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ABSTRACT

The present research work determines the synthesis and characterization of iron oxide nanoparticles from *Lantana camera* by green technology through the reduction of ferrous sulphate. The main objective of the research work is, synthesize of iron oxide nanoparticles using cost effective and environment friendly method. The biosynthesized iron oxide nanoparticles were characterized by XRD (X-ray diffractometer), FT-IR (Fourier transform infrared spectroscopy), SEM (scanning electron microscope), EDX (Energy Dispersive X-Ray spectrometer) and UV absorption spectroscopy. The average particle size ranged from 10 to 20 nm and shapes were nanorods, crystalline and highly stable. The antibacterial activity was analyzed for synthesized iron oxide nanoparticles. The biological activities of iron oxide nanoparticles such as antibacterial activity and seed germination studies were also analyzed. The germination and growth of *Vigna mungo* was done with different concentration of iron oxide nanoparticles. In antibacterial studies, the highest zone of inhibition was observed in Pseudomonas (20.10 ± 1 mm) at 100 µg/ml of iron oxide nanoparticles. The maximal seed germination was observed in 200 ppm concentration of nanoparticles treated seeds. The shoot and root length were low when the concentration of the nanoparticles increased. This green synthesized iron oxide nanoparticle was proved to have good biological activities and can be used for various applications.

1. Introduction

Nanotechnology is becoming a fast emerging field which focuses on synthesis and using of particles at 1–100 nm size. These nanoparticles have drawn more attention from researchers worldwide because of their specific characteristics (size, shape and distribution) which could be used in different fields of applications (Zargar et al., 2011). Various physical and chemical methods are developed for the nanoparticles production. However, these methods have certain drawbacks such as they leave deadly residues and contaminate the environment. Hence, biosynthesis of nanoparticles by bacteria, algae, fungi and higher plants has emerged as cost effective and environment friendly alternative technology (Herlekar et al., 2014; Husen and Siddiqi, 2014; Makarov et al., 2014; Siddiqi and Husen, 2016). These metal oxide nanoparticles are broadly exploited in various fields such as agriculture, cosmetics, paints, catalysis, food coatings, and textiles. In industries, these magnetic nanoparticles cover a broad spectrum of biomedical applications (Rai and Ingle, 2012). Each nanoparticle needs a specific property in specific field to give its full potential. The conventional nanoparticles synthesis methods make use of harmful chemicals and solvents in synthesis process with the addition of artificial preservatives or capping agents as stabilizers thus preventing the applications in biomedical and clinical fields. Hence, there is an emergent need for the improvement of environmental friendly, biocompatible, dependable and synthetic methods to evade some undesired ecological and health effects (Mrinmoy et al., 2008). The biosynthesis method, involve the reduction in the precursor to metal oxide nanoparticles by the phytochemicals (Jain et al., 2005). There is no need of additional capping agent needed for the synthesis of metal oxide nanoparticles due to the presence of phytochemicals in the plant extracts. This method is beneficial over the other synthetic methods. Fe_2O_3 nanoparticles synthesis from the extracts of *Camellia sinensis, Melaleuca nesophila, Eucalyptus globules, Tridax procumbens, Terminalia chebula*, etc. has been reported recently (Hoag et al., 2009; Kumar et al., 2013; Madhavi et al., 2013; Senthil and Ramesh, 2012; Wang et al., 2014). *Lantana camara* Linn (*L. camara*) belongs to the Verbaceae family. It is grown as popular ornamental and garden plant. The leaves of the plant are utilized in the healing of malaria, rheumatism and tumors etc., and its antibacterial properties have been reported (Bag et al., 2013; Mittal et al., 2013). The present research work focuses on an environmental friendly method for the synthesis of iron oxide nanoparticles from the *L. camara* leaf extract without the addition of stabilizing agents.

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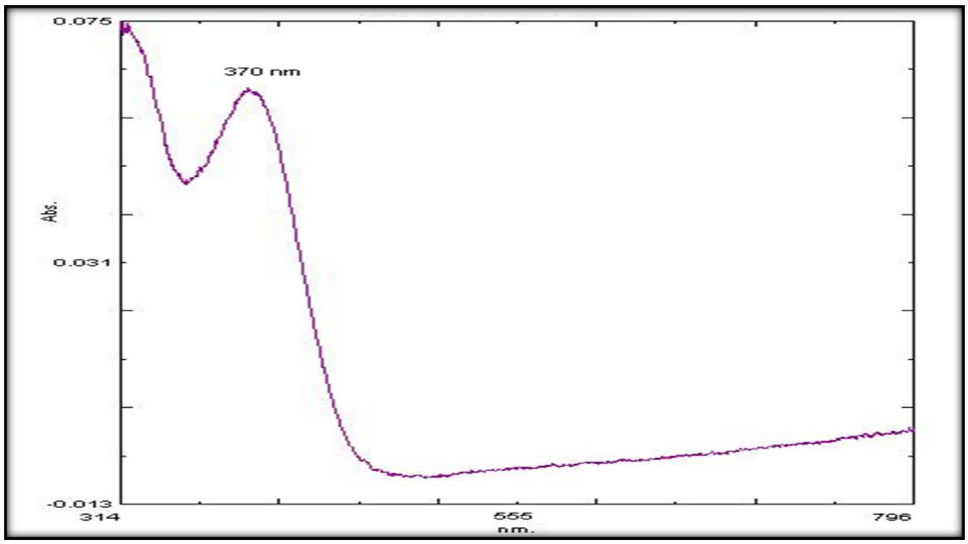

Fig. 1. UV spectra of *Lantana* mediated iron oxide nanoparticles.

2. Materials and method

2.1. Materials

Lantana camara were collected at Karpagam University, Coimbatore, Tamil Nadu, India (10°90′N, 77°01′E). All the chemicals used in the experiments were purchased from Sigma-Aldrich Chemicals, India. Laboratory glass wares were immersed overnight in acid solutions and rinsed in tap water and distilled water. Bacterial pathogens (*Pseudomonas sp., Klebsiella sp., Salmonella sp*. and *Staphylococcus sp*.) were acquired from the Department of Microbiology, Karpagam University, Coimbatore, India. *Vigna mungo* seeds were collected from Department of cereals and millets, Tamil Nadu Agricultural University (TNAU), Coimbatore, Tamil Nadu, India.

2.2. Preparation of Lantana camara leaf extract

5 g of fresh *L. camara* leaves were weighed up and rinsed thoroughly in tap water and distilled water. Samples were crushed well using mortar and pestle and boiled with 100 ml of distilled water for 15 min. Then the extract was filtered using filter paper and stored in refrigerator for further investigation (Mrinmoy et al., 2008).

2.3. Synthesis of iron oxide nanoparticles

Ferrous sulphate (1 M) was used as precursor and ferrous sulphate solution was prepared by means of deionized water. 50% of leaf extract was prepared and made up to 250 ml with deionized water. The plant extract (50 ml) and sodium hydroxide pellets (1 g) were mixed to the 50 ml of ferrous sulphate solution. The mixture was centrifuged and supernatant was discarded. The pellet was dried using hot air oven. A brownish black color powder was obtained and it was stored in the air tight containers for further studies (Madhavi et al., 2013).

2.4. Characterization of Iron oxide nanoparticles

The optical property of iron oxide nanoparticles were examined by UV absorption spectroscopy (Shimadzu). The phase purity of the synthesized *L. camara* mediated iron oxide nanoparticles were analyzed using an X-ray diffractometer (XRD). Fourier transform infrared (FT-IR) spectroscopy was done to categorize the functional groups present in synthesized iron oxide nanoparticles. Morphological and size distributions of the particles were analyzed using scanning electron microscope (SEM) images. Elemental analysis of particles were observed using Energy Dispersive X-Ray spectrometer (EDX) (Rajeshwari et al., 2014).

2.5. Evaluation of biological activities using iron oxide nanoparticles

2.5.1. Determination of Antibacterial activity of L. camara mediated iron oxide nanoparticles

Antibacterial activity of *L. camara* mediated iron oxide nanoparticles were determined by well diffusion method (Bauer et al., 1966). The bacterial cultures were maintained in nutrient broth. The 100 µl of bacterial cultures were spread over Muller Hinton agar plates using sterile swap. The wells of 5 mm size were punctured on plates using sterile gel puncture and different concentrations (25, 50, 75 and 100 µg/ml) of the green synthesized iron oxide nanoparticles were added into the well. Tetracycline was used as a positive control. The swabbed plates were incubated at 37 °C for 24hrs. After incubation, the zone of inhibition was measured in millimeter. Each test was performed with three replicates and the mean value was recorded.

2.5.2. Assessing the effect of iron oxide nanoparticles on seed germination, shoot and root development in Vigna mungo

Vigna mungo seeds were washed using 2.5% of sodium hypochloride solution for 15 min. After washing the seeds they were rinsed in deionized water three times and were soaked in various concentrations (Distilled water, 100, 200, 300 and 400 ppm) of aqueous iron oxide nanoparticles for soaking period of 24 h at laboratory condition (30 ± 1 °C, 63% RH) in an incubator (dark condition). A thin layer of cotton was placed on each petri dish (90 mm × 15 mm), 3 ml of iron oxide nanoparticles suspensions or distilled water were added. 30 seeds were transferred to each petri dish and then it was covered with parafilm and kept for incubation at room temperature. After 3 days of incubation, the seed germination was recorded by counting the germinated seeds and the root and shoot length by measuring. Each treatment has three replicates and the mean values were recorded (Boonyanitipong et al., 2011).

3. Result and discussion

3.1. Characterization of iron oxide nanoparticles

UV–Vis absorption spectrum reveals that synthesized iron oxide nanoparticles were mono dispersed and showed a wide absorption peak at 370 nm (Fig. 1). The synthesized iron oxide nanoparticles have continuous absorption in the visible range of 200–800 nm. Basavegowda *et al.* reported that the optical properties of iron oxide