Green Synthesis and Characterization of Magnetite Nanoparticles from Margosa Plant *(Azadirachta indica)*

[1]J.O. Olaniyan, [1*]O.I. Sonde, [2]O.R. Adetunji, [1]A.A. Ameye, [3]O.T. Bankole, [1*]E.O. Dare

[1]Department of Chemistry, Federal University of Agriculture, Abeokuta. P.M.B. 2240, Abeokuta, Ogun State, Nigeria

[2]Department of Mechanical Engineering, Federal University of Agriculture, Abeokuta. P.M.B.2240, Abeokuta, Ogun State, Nigeria.

[3]Department of Chemistry, Crawford University, Igbesa, Ogun State, Nigeria

*Corresponding Authors: sonde.idowu@gmail.com & dare3160@hotmail.com

ABSTRACT

Synthesis of nanoparticles has become a matter of great interest in recent times due to their various properties and applications in a variety of fields. The exploitation of different plant materials for the green synthesis of nanoparticles without any harmful chemicals is considered a key issue. This research work aimed at synthesizing Magnetite Nanoparticles (MgNps) from Margosa plant (*Azadirachta indica).* MgNps were synthesized by mixing solution of ferric chloride ($FeCl_3$) and ferrous chloride tetrahydrate ($FeCl_2.4H_2O$) with aqueous *Azadirachta indica* plant extract using convectional heating technique and co-precipitation method. The morphological property of the MgNps was confirmed using UV-Visible spectrophotometer, Scanning Electron Microscope (SEM), and Transmission Electron Microscopy (TEM). SEM and TEM analyses revealed the cap shaped spherical morphology with no agglomeration and uniform size distribution of the nanoparticles within the range of 12.00 nm and 20.00 nm respectively. Capping obtained is an evidence of organic matter in the plant extract. UV-visible analysis showed Plasmon resonance band at 295 nm with no other observable peak confirming total transformation of $FeCl_2.4H_2O$ and $FeCl_3$ to (Fe_3O_4). Therefore, green method of synthesizing MgNps is indeed confirmed to be economical, environmental friendly and resourceful.

Keywords: Green Synthesis, Magnetite Nanoparticles, Margosa Plant.

INTRODUCTION

The need for green synthesis of nanoparticles from ferric chloride ($FeCl_3$) and ferrous chloride tetrahydrate ($FeCl_2.4H_2O$) with aqueous *Azadirachta indica* plant extract using convectional heating technique and co-mixing method arose as the physical and chemical processes were costly [1]. Often, chemical method leads to presence of some toxic materials. MgNps have attracted a great attention due to their unique physical, chemical and structural properties when the particle sizes approach nano-scale. It is these unique features that endow magnetic nanoparticles with wide applications, such as magnetic storage, catalysis, microwave absorption, magnetic resonance contrast, cancer hyperthermia, cell separation and drug delivery [2]. Therefore, more efforts have been devoted to the synthesis of magnetic nanoparticles recently. Many preparation routes, including, sonochemistry, colloidal method, combustion synthesis, solvo-thermal synthesis, hydrothermal method, micro emulsion co-precipitation method and thermal decomposition have been reported [3]. Iron oxides exist in many forms in nature, with magnetite (Fe_3O_4), magnemite (γ-Fe_2O_3) and hematite (α-Fe_2O_3). The stability of magnetite has however been improved by coating with different materials such as *Azadirachta indica* depending on the area of application, which in turn, influences particle size and shape, size distribution, agglomeration, and the low toxicity, biocompatibility, cost-effectiveness and high surface area of the material[4]. The exploitation of different plant materials for the green synthesis of nanoparticles without any harmful chemicals is considered a key issue. This research work aimed at synthesizing Magnetite Nanoparticles (MgNps) from Margosa plant (*Azadirachta indica).*

MATERIALS AND METHODS

Preparation of Plant Extract

Water extraction of the plant leaves was carried out by washing about 5 g of the leaves with distilled water and boiling for 15 min to obtain an aqueous greenish extract, which was then filtered**.**

Procedures

Co-precipitation is a facile and convenient way to synthesize magnetite nanoparticles (metal oxides and ferrites) from aqueous salt solutions. This was carried out with addition of a base under inert atmosphere at room temperatures [5]. $FeCl_3$ (2.4 g) and $FeCl_2.4H_2O$ (1.5 g) was dissolved in 75 mL of distilled water to give a brown colour solution. The solution was magnetically stirred for about 10 min. 7.5 mL of light yellow solution of plant extract was added drop wisely to the prepared solution of iron (ii) and iron (iii). The resulting solution was stirred for 15 min, and then centrifuged. The resulting residue was ashed with distilled water and ethanol. The nanoparticles were dried in an ovem at 50 ^oC .

Synthesis of Magnetite Nanoparticles

2.4 g of $FeCl_3$ + 1.5 g of $FeCl_2.4H_2O$

↓ Dissolve in 75 ml distilled water

Solution

↓ Stir magnetically for 10 minutes

Brown precipitate

↓ Add 7.5 mL of plant extract drop wisely

Deep brownish precipitate

↓ Heat at 80℃ for 30 min

Brown precipitate

↓ Decant and centrifuge

Brown precipitate

↓ Wash with water and ethanol

Brown precipitate

↓ Dry in a vacuum oven at 70℃

Magnetite nanoparticles (Fe_3O_4)

Figure 1: Schematic diagram for the synthesis of magnetite nanoparticles

RESULTS AND DISCUSSION

Characterizations

On the application of leaf extract of *Azadirachta indica* to the solution of $FeCl_3$ and $FeCl_2.4H_2O$, there was an observable colour change in the solution from brown to deep brown. These colour change is an indication of growth and nucleation of magnetite nanoparticles. Also a reduction in size was observed as a result of the interaction of the leaf extract with $FeCl_3$ and $FeCl_2.4H_2O$ [6]. Nanocrystal was obtained after centrifugation. Interestingly, the nanocrystals responded to magnetic field positively which further indicates that the synthesized Fe_3O_4 possesses magnetic properties [7].

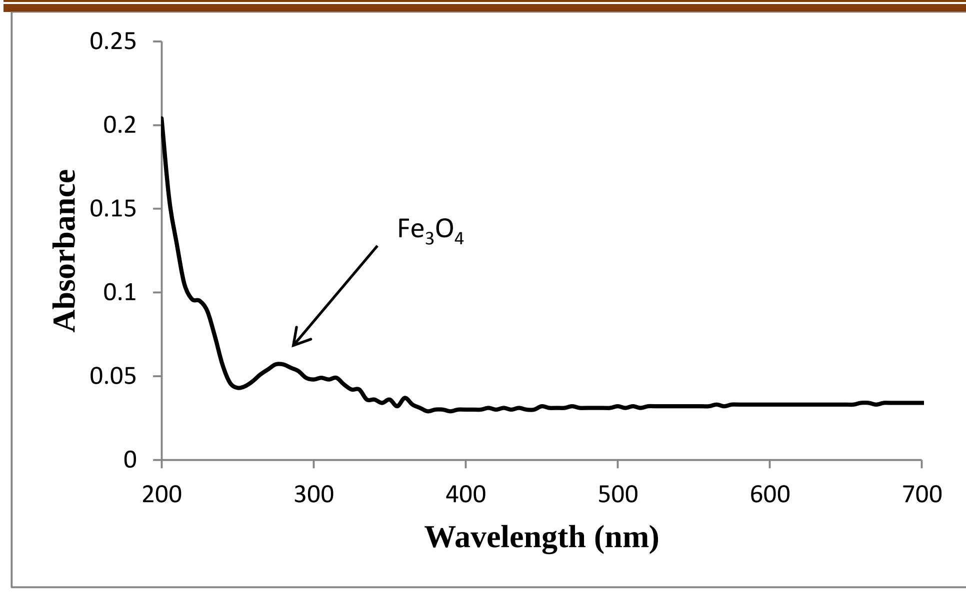

Figure 2: UV-Visible absorption spectrum of magnetite nanoparticles

UV-visible analysis showed Plasmon resonance band at 295 nm with no observable peak confirming total transformation of $FeCl_2.4H_2O$ and $FeCl_3$ to (Fe_3O_4) as could be observed in Figure 1. It also indicates that no impurity was contained in the aqueous solution which reveal indeed that MgNps was formed with an increased intensity leading to bathochromic shift[8,9].

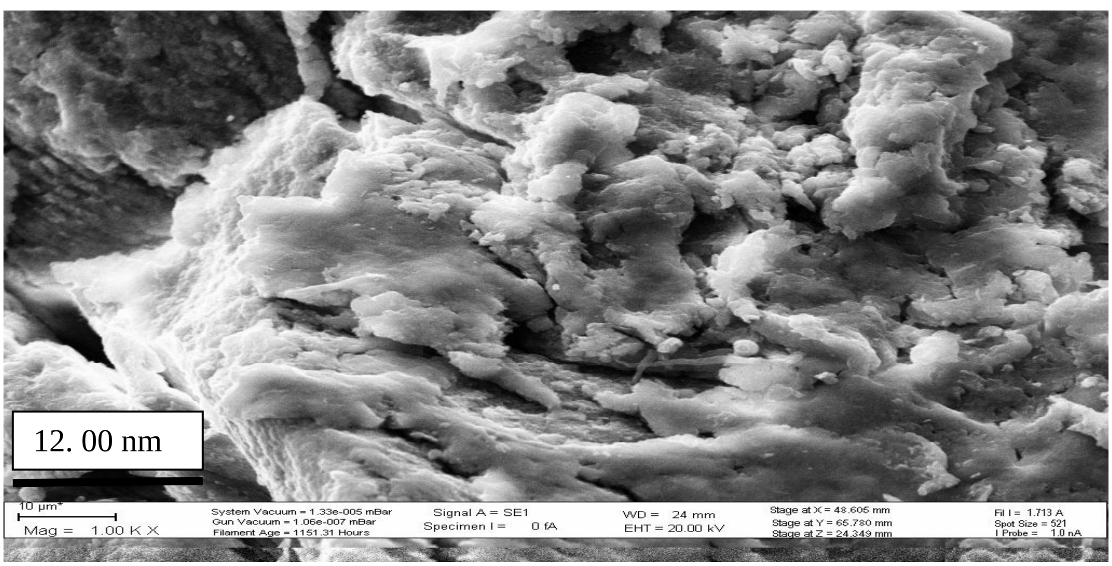

Plate 1: SEM Image of Magnetite Nanoparticles

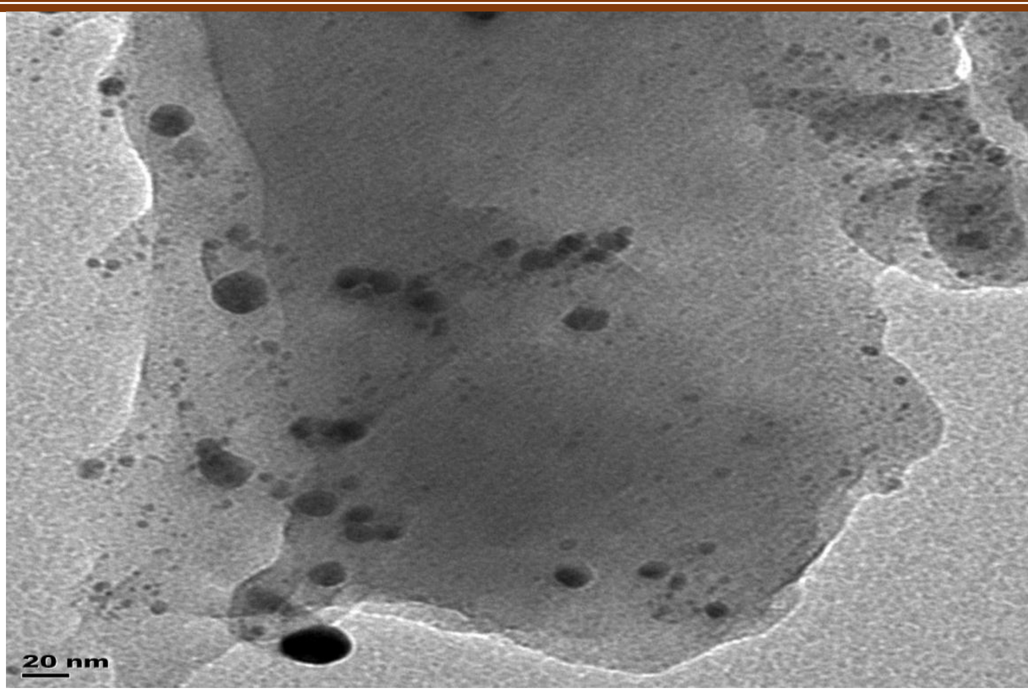

Plate 2: TEM Image of Magnetite Nanoparticles

Transmission Electron Microscope (TEM) Analysis

Plates 1 and 2 showed Scanning Electron Microscope and Transmission Electron Microscopy respectively. SEM and TEM analysis revealed the cap shaped spherical morphology with no agglomeration and uniform size distribution of the nanoparticles within the range of 12.00 nm and 20.00 nm respectively. Capping obtained is an evidence of organic material in the plant extract and it revealed true formation of MgNps (Fe_3O_4) formed [10].

A schematic flowchart showing the preparation of MgNps was provided to show the mixture of iron and water-soluble polymer. According to the scheme, spherical nanoparticles were manufactured (plate 1). The synthesis of metallic nanoparticles from water soluble polymer iron oxide (Fe_2O_3), ferric chloride ($FeCl_3$) and ferrous chloride tetrahydrate ($FeCl_2.4H_2O$) can be removed by centrifugation or filtration and in a drying oven. The solution formed after convectional heating, turned brown, which indicated that the iron and water-soluble polymer were combined via the above reaction, yielding stable MgNps. In the present study, the term ‘‘MgNps’’ (Fe_3O_4) means a composite comprising iron oxide (Fe_2O_3), ferric chloride ($FeCl_3$) and ferrous chloride tetrahydrate ($FeCl_2.4H_2O$) combined with aqueous *Azadirachta indica* plant extract. The synthesized MgNps may be prepared by convectional method. One example of the composite is a structure in which MgNps formed from the combination, surrounded by water-soluble polymer by convectional heating. The MgNps in a colloidal state may be present as nanoparticles separated from each other or separated into loose spherical clusters (plate 1 and 2). Therefore, the clusters are simply separated when the temperature increases. The nanoparticles absorbed UV-Visible light at 295 nm (fig. 2) which is unique wavelength and has a uniform nanoparticles size as shown in plates 1 and 2. The particle size of the nanoparticles ranges from 20 nm and 22 nm respectively. The nano-sized MgNps may be more easily handled, economical and highly resourceful, compared to chemically synthesised MgNps.

CONCLUSION

Magnetite Nanoparticles was synthesized from Margosa plant (*Azadirachta indica).*

Magnetite nanoparticles synthesized by this method employed an environmental friendly solvent, *Azadirachta indica* leaf extract as an alternative to organic solvents and surfactants. The phyto-chemical compound present in the extract acts both as dispersing and stabilizing agents which prevents agglomeration. With these results, green method of synthesizing MgNps (Fe_3O_4) is indeed confirmed to be economical, environmental friendly and highly resourceful [11].

Acknowledgement

The authors wish to acknowledge the Department of Chemistry Federal University of Agriculture Abeokuta, Ogun State, Nigeria.

REFERENCES

1. Grini, G (2006). Non-Conventional Low-Cost Adsorbents for Dye Removal, *A Review Bioresource Technology*, 97(9), 1061-1085.
2. Guptaa, A.K & Guptaa, M. (2005). Synthesis and surface engineering of iron oxide nanoparticles for biomedical applications. *Biomaterials, 26*, 3995–4021.
3. Holm, L.J., Sun, Y.P. & Teja, A.S (2002). Production of Magnetic Oxide Nanoparticles, Supercritical Fluid Technology in Materials Science and Engineering: Synthesis, Properties and Applications, *Elsevier*, 327-349.
4. Gardea-Torresdey, J.L., Parsons, J.G & Peralta-Videa, J.R. (2007). Use of plants in biotechnology: Synthesis of metal nanoparticles by inactivated plant tissues, plant extracts, and living plants. *Develop Environmental Sci*ences, *5,* 463–485.

5. Abhilash, Pandey, B.D. & Revati, K. (2001). Microbial synthesis of iron-based nanomaterials – A review. *Bulletin of Material Science,* 34, 191–198.

6. Badr, W.K., El-Hawary, S.S., El-Tantawy, M.E. & Rabeh, M.A (2013). Chemical composition and biological activities of essential oils of Azadirachta indica A.*International Journal of Applied Research in Natural Products,* 6, 33- 42.

7. Aftabtalab, A., Sadabadi, H. (2014). Application of Magnetite (Fe_3O_4) Nanoparticles in Hexavalent Chromium Adsorption from Aquatic Solutions. *Journal of Petroleum and Environmental Biotechnology 6*: 200.

8. Farideh Namvar, Mahnaz Madavi, Mansor Bin Ahmad & Rosfarizan Mohamad (2013). Green Biosynthesis and Characterization of Magnetic Iron Oxide (Fe3O4) Nanoparticles Using Seaweed (Sargassum muticum) Aqueous extract. *Biocatalysis and Agricultural Biotechnology* 18: 5954 – 5964.

9. Petcharoen, K. & Sirivat, A. (2012). A Synthesis and characterization of magnetite nanoparticles via the chemical co-precipitation method. *Journal of Nanomaterial Bioscience,* 177, 421– 427.

10. Ana, L.D., Ana, M.G., Benilde, F.O.C., Brian, J.G., Rui, N.C. & Trindade, T. (2007). In Situ Synthesis of magnetite nanoparticles in carrageenan gels, *Biomacromolecules* ,8, 2350-2357.

11. Jana, D., Jaromir, H., Oldrich, S., Radim, H., Rene, K. & Vojtech, A. (2009). Preparation and properties of various magnetic nanoparticles, *Sensors*, 9(4), 2352-2362.