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Research Article

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Green synthesis and characterization of iron oxide magnetic nanoparticles using Shanghai White tea (*Camelia sinensis*) aqueous extract

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ABSTRACT

The green synthesis of metallic nanoparticles (NPs) has attracted much attention in recent years because these protocols are low cost and more environmentally friendly than standard methods of synthesis. The green synthesis of iron oxide magnetic nanoparticles (Fe_3O_4-MNPs) using the aqueous extract of White tea (Camelia sinensis) as a reducing and capping agent has been done in this article. The formation of Fe_3O_4-MNPs was observed by the change of colour from colourless to dark brown by the addition the leaf extract. To the best of our knowledge, it is for the first time this plant is used for synthesizing nanoparticles. Evaluating its properties and morphology of the Fe_3O_4-MNPs is done using FT-IR, X-ray and SEM. The average particle diameter as determined by SEM was found to be 35 nm. X-ray diffraction demonstrated that the nanoparticles are crystalline in nature, with spinel shape. These Fe_3O_4-MNPs fabricated through biosynthesis method, are promising candidate in various applications like biomedical and utilizing as recyclable magnetic nano-catalyst for organic reactions.

Keywords: green synthesis; White tea; Fe_3O_4 magnetic nanoparticles; Polyphenols

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INTRODUCTION

Nanostructured materials are attracting increasing attention in recent years because they exhibit useful and unique properties compared to conventional polycrystalline materials. And also, much attention has been paid to metal oxide nanoparticles for their potential applications and physicochemical, thermal and mechanical properties, which are strongly influenced by their specific sizes, morphology and structure. These nano-crystalline particles have a high surface/volume ratio leading to magnetic properties different from those of materials [1]. Among all the metal oxides, iron oxide magnetic nanomaterials are very interested due to their unique properties. In particular, magnetite (Fe_3O_4) is a common ferritic material having a cubic inverse spinel structure. The compound exhibits amazing electric and magnetic properties based on the transfer of electrons between Fe^{2+} and Fe^{3+} in the octahedral sites [2].

Metallic nanoparticles of specific sizes and morphologies have been produced via chemical and physical methods. However, such methods are toxic chemicals as reducing agents, organic solvents, not easily disposable due to environmental issues, have low production rate and are expensive [3]. For this reason, it is very desirable to devise alternative, 'green' methods of nanomaterial preparation that reduce or eliminate toxic substances to restore the environment. Green synthesis of nanoparticles by inactivated plant tissue and plant extracts makes use of environmental friendly, non-toxic and safe reagents [4].

Interest in the magnetite has centered on applications such as magnetic storage media [5], bio sensors [6], separation process and environmental remediation [7], besides, many biomedical usages like cellular therapy, tissue repair, magnetic resonance imaging (MRI), hyperthermia [2], catalysis [8] and drug delivery [9].

Historically, including plants, tea and the potable from *Camellia sinensis* (L), is by far one of the world's most greatly beloved beverages [10-11] which its reputation stands for medicinal attributes that have been widely explored [12]. In addition, its health promoting properties have been known from the early periods of the Chinese civilization, going back almost 5000 years [13]. White tea that is the least processed tea and is exclusively prepared from shootings and buds, contains the major polyphenols like the flavan-3-ols and the flavonols. The flavan-3-ols are characterized by (-) epicatechin and its galloylated derivatives. The flavonols are principally derivatives of quercetin and kaempferol [14], but there are smaller amounts of tannins and hydroxyl cinnamate derivatives. Of course, tea also contain substantial and physiological relevant levels of caffeine and theobromine [15], Epigallocatechin gallate, EGCG is the most abundant and powerful antioxidant in white tea for cancer chemoprevention [16].

Considering, white tea contains the majority of polyphenols and this causes white tea to be a good candidate for synthesizing nanoparticles and applying the phenolic compounds as reducing also capping agent.

In this current research paper we attempted to fabricate Fe_3O_4-MNPs using ferric chloride as iron precursor and white tea aqueous extract as reducing agent and stabilizer. Further, Fe_3O_4-MNPs were characterized by Fourier transform infrared (FT-IR), scanning electron microscope (SEM) and powder X-ray diffraction (XRD).

EXPERIMENTAL SECTION

Material: The whole plant White tea (*Camelia sinensis*) was collected from Shanghai, and was purchased from a local herb shop in Iran. Ferric chloride hexahydrate ($FeCl_3.6H_2O$, 97%) and ferrous chloride tetra hydrate ($FeCl_2.4H_2O$, 99%) were purchased from Merck (Darmstadt, Germany). All reagents in this research were analytical grade and were used as received without further purification. All aqueous solutions were freshly prepared using distilled deionized water (DDW).

Preparation of plant extract: White tea (*Camelia sinensis*) plant is shown in Figure 1A. White tea leafs were washed to remove the possible impurities, and then dried in sunlight to completely remove the moisture. After that ground into powder, and kept at 4 °C until further analyses. The resultant ground white tea sample (about 4 g, Figure 1B) were boiled with DDW (100 mL) in an Erlenmeyer flask while being continuously stirred for 30 min. After filtration with filter paper using a vacuum pump, the residue was cooled to room temperature, and stored at 4 °C until used.

Figure 1. White tea (*Camelia sinensis*) (A), White tea (*Camelia sinensis*) powder (B)

Preparation of Fe_3O_4 nanoparticles: 2 mole $FeCl_3.6H_2O$ and 1 mole $FeCl_2.4H_2O$ was dissolved in 100 ml DDW and stirred. After that, 20 ml of the White tea aqueous extract was added and stirred for 1 hour. After complete bio reduction of iron ions, the white tea extract was centrifuged for 2 min to isolate the Fe_3O_4-NPs from the compounds present in the solution and dried in an oven for 24 hours.

RESULTS AND DISCUSSION

Mechanism of the Fe_3O_4 -NPs formation in white tea Extract: The present work focused on the development of a biosynthetic method for the production of Fe_3O_4-MNPs using White tea (*Camelia sinensis*) extract. The reduction potential of caffeine and other polyphenols in white tea (Figure 2) is sufficient to reduce metals. Decreasing in pH during the formation of Fe_3O_4-NPs signifies the involvement of the OH group in the reduction process. The formation of Fe_3O_4-NPs with caffeine/polyphenols occurs via the following steps: (1) complexation with Fe salts, (2) simultaneous reduction of Fe (III) capping with oxidized polyphenols/caffeine. As shown in Figure 3, the colour of the iron solution/white tea extract solutions at room temperature rapidly changed from yellow to dark brown, indicating the formation of Fe_3O_4-MNPs in the White tea (*Camelia sinensis*) extract. The proposed green synthesis method for Fe_3O_4-NPs was found to be constructive and extremely reproducible. [17-19]

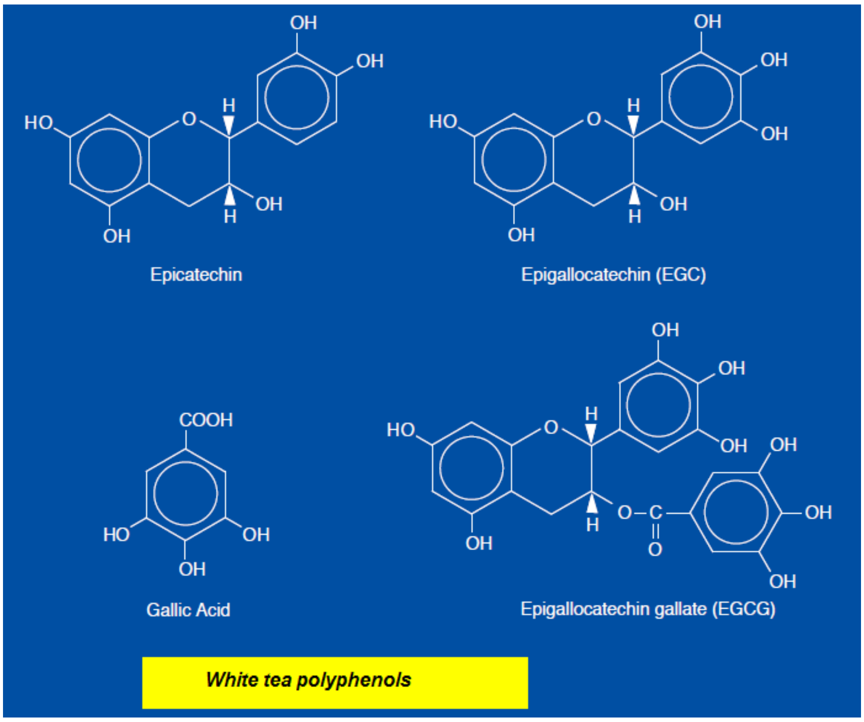

Figure 2. Chemical structures of tested tea polyphenols

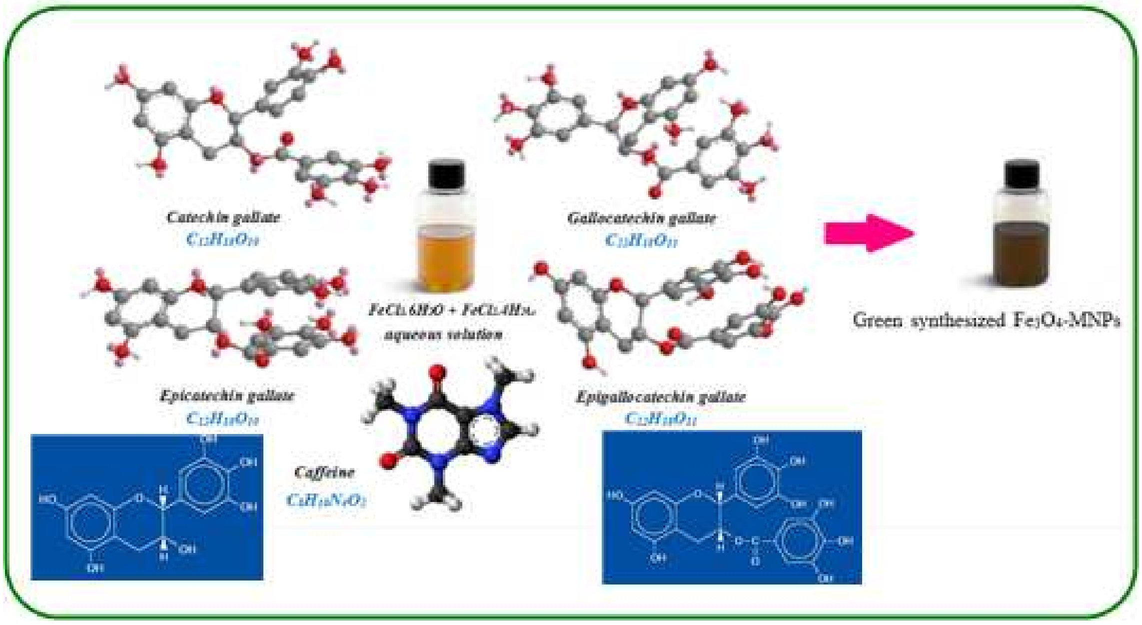

Figure 3. Schematic illustration of synthesized Fe_3O_4-MNPs in White tea(*Camelia sinensis*) extract

Fourier Transform Infrared Spectroscopy (FT-IR): FT-IR spectra were obtained on a PerkinElmer 1000 FT-IR spectrometer. FT-IR analysis was carried out to determine the functional groups present in White tea extract and their possible involvement in the synthesis of Fe_3O_4-MNPs. The dual role of the plant extract as a bio reductant and capping agent was confirmed by FT-IR analysis of the prepared Fe_3O_4 NPs. The FT-IR analysis of white tea plant (Figure 4) shows vibration bands at 3417 Cm^{-1} stretch of O-H in polyphenols; a peak at 2918 Cm^{-1} indicated stretching C-H bonds; 1618 and 1639 shows C=C aromatic bonds, and 1035 also 1241 Cm^{-1} for C-O stretching bonds in polyphenol compound. The absorption peaks at 1372 and 1541 Cm^{-1} corresponding to the CH_3 and CH_2, while 1514 Cm^{-1} shows N-H bonds. The spectrum of green synthesized Fe_3O_4-MNPs (Figure 5) showed, two absorption bands at 556 and 479 Cm^{-1} which correspond to the Fe-O bond in magnetite [17-19]. On the other hand, absorption in 3446 and 1639 Cm^{-1} declines, which shows that hydroxyl and carbonyl groups play a key role in the formation of the final structures of the Fe_3O_4 NPs since polyphenols are important for reducing and capping behaviours.

Detailed analysis of the plant extract spectra strongly suggested the presence of flavonoids and polyphenols, apart from other phytochemicals, which were mainly responsible for the formation of the Fe_3O_4-MNPs.

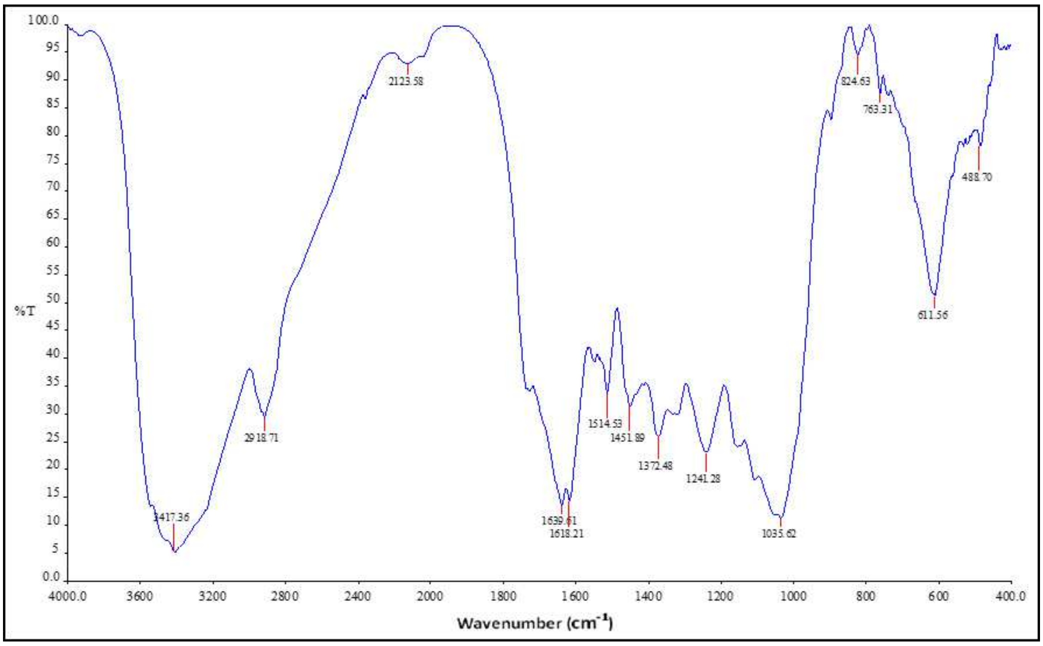

Figure 4: Fourier transform infrared spectra for the White tea powder

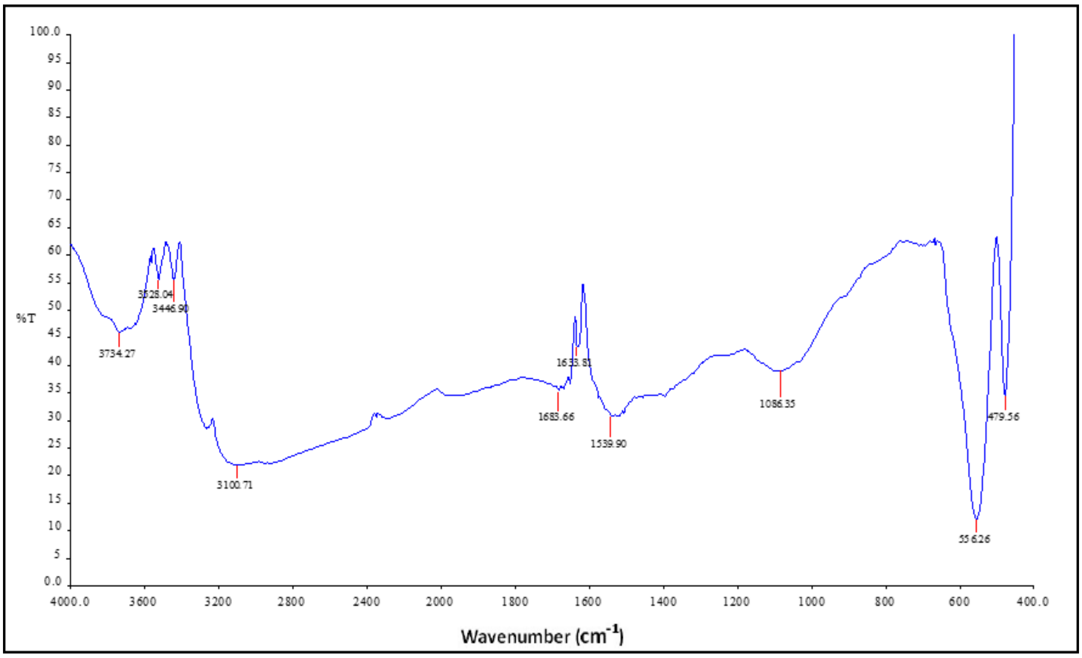

Figure 5: Fourier transform infrared spectra for the green synthesized Fe_3O_4-MNPs

Powder X-ray Diffraction (XRD): The phase identification and crystalline structures of the nanoparticles was characterized by X-ray powder diffraction as it is demonstrated in figure 6. From the X-ray diffraction patterns obtained for the Fe_3O_4-MNPs synthesized using White tea found that there exist strong diffraction peaks with 2θ values of 30°, 35.5°, 43°, 57° and 63° that corresponds Fe_3O_4-MNPs. [17-19].

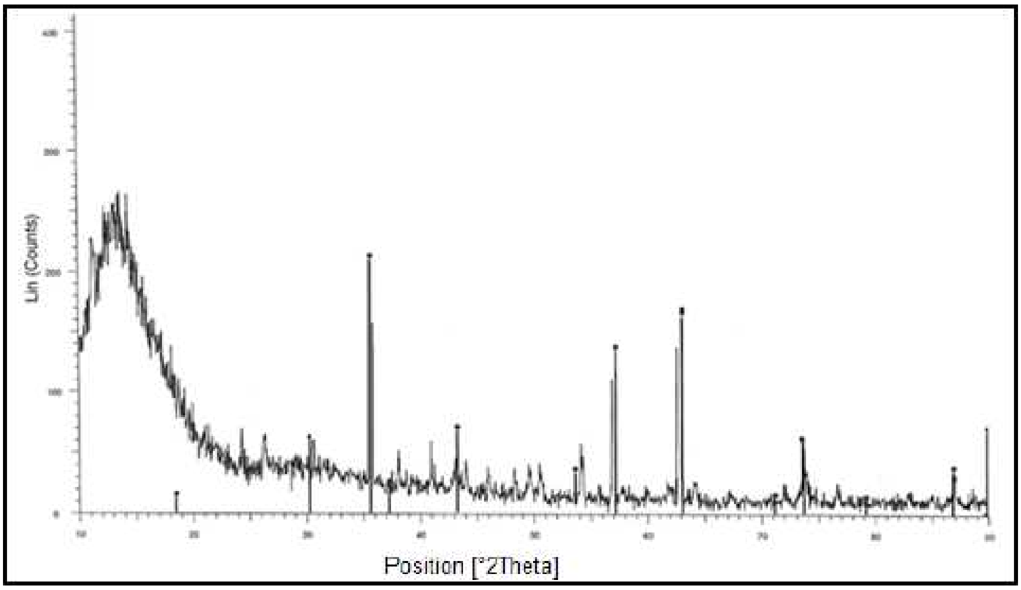

Figure 6: The X-ray Diffraction pattern of the green synthesized Fe_3O_4-MNPs

Scanning Electron Microscopy (SEM): To further confirm the role of tea extracts in synthesizing iron oxide NPs, we used SEM microscope to show the size and morphology of Fe_3O_4-NPs using White tea extract as can be seen in figure 7. This figure clearly show the cubic morphology of the MNPs, with an average particle size of 35 nm, in which few NPs were agglomerated. In comparison to chemical synthesis of Fe_3O_4-NPs and other plant extracts slight aggregation of Fe_3O_4-NPs was observed. This outcome can be explained by the fact that the polyphenols/caffeine concentrations in White tea extract plays a key role in the formation of the final structures and size of these green synthesized Fe_3O_4-NPs.

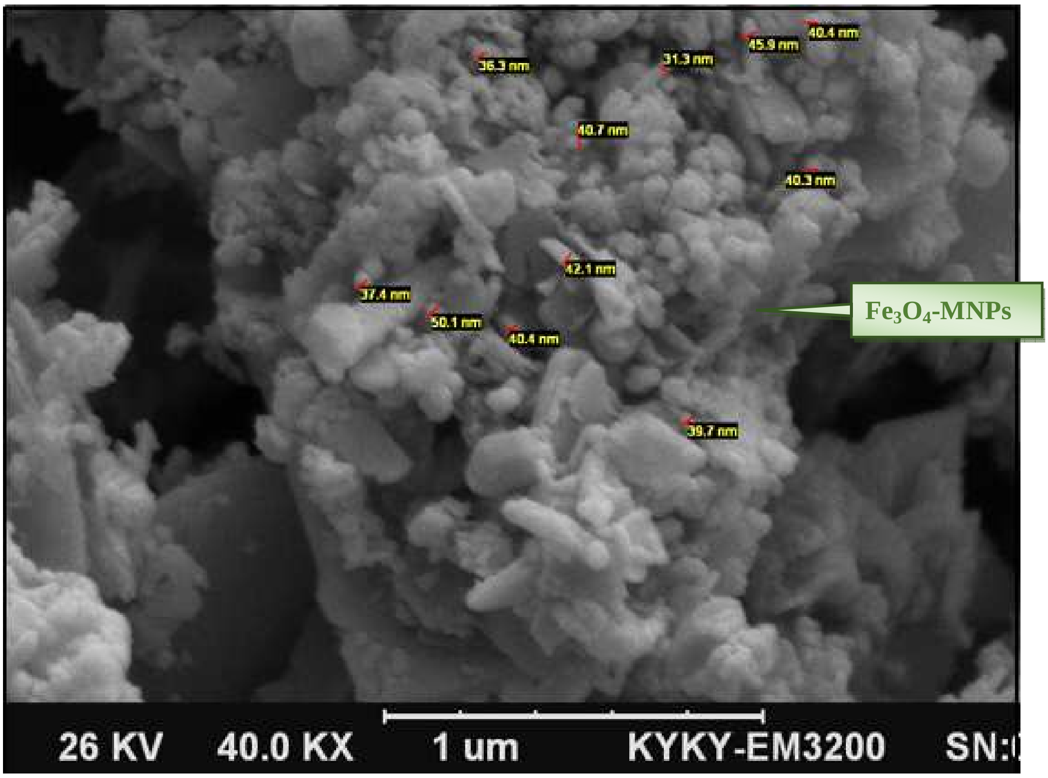

Figure 7. The SEM image of Fe_3O_4-MNPs green synthesized using white tea extract

CONCLUSION

It is been a demand in nanotechnology to improve credible and eco-friendly procedures for synthesis of various metal oxide nanoparticles. In this project, a completely green single-step synthesis of Fe_3O_4-MNPs using White tea polyphenols is reported which utilizes no additional surfactants/polymers as capping or reducing agents. The reaction between White tea polyphenols and iron salt solution occurs within a few minutes at room temperature. In this reaction, White tea polyphenols perform as reducing agent and capping agent, these compounds played a major role in decreasing the aggregation of Fe_3O_4-NPs, and improved the stability and the consequence is green synthesized Fe_3O_4-MNPs with spinel shape and the mean size of 35 nm. The participation of polyphenol groups and caffeine in green synthesis of Fe_3O_4-NPs is exhibited by FT-IR analysis. The characteristics of the obtained Fe_3O_4-NPs were studied using FT-IR, XRD, and SEM techniques. Green synthesis of Fe_3O_4-NPs using plant extract is a simple, environmentally friendly, pollutant-free and low-cost approach that could also be extended to fabricate other, industrially important metal NPs.

REFERENCES

[1] DA Bazylinski; RB Frankel; KO Konhauser, *Geomicrobiol J.*, **2007**, 24, 465–475.
[2] Abhilash; K Revati; BD Pandey, *Bull. Mater. Sci.*, **2011**, 34, 191–198.
[3] G Liangpeng; L Qingtao; JO Meng Wang; L Xiaojian; X Malcolm, *Int. J. Nanomedicine.,* **2014**, 9, 2399–2407.
[4] M Mahdavi; F Namvar; MB Ahmad; R Mohamad, *Molecules*, **2013**, 8(5), 5954-596.
[5] SH Sun; CB Murray; D WelleR; L Folks; A Moser, *Science,* **2000**, 287, 1989–1992.
[6] MM Miller; GA Prinz; SF Cheng; S Bounnak, *Appl. Phys. Lett.,* **2002**, 81, 2211–2213.
[7] M Mahdavi; MB Ahmad; MJ Haron; Y Gharayebi; K Shameli; B Nadi, *J. Inorg. Organomet. Polym. Mater*, **2013**, 23, 599–607.
[8] JL Zhang; Y Wang; H Ji; YJ Wei; NZ Wu; BJ Zuo; QL Wang, *J. Catal.*, **2005**, 229, 114–118.
[9] AK Guptaa; M Gupta. M, *Biomaterials,* **2005**, 26, 3995–4021.
[10] JM Hamilton miller, *Antimicrob Agents Chemother.*, **1995**, 39(11), 2375–2377.
[11] S Hilal; U Engelhardt, *Journal of Consumer Protection and Food Safety*, **2007**, 2, 414 – 421.
[12] S Alexander, *Yale global online; a publication of Macmillan center,* **2004.**
[13] CM Hall; R Mitchell; L Sharples, *Elsevier*, **2003**.
[14] D Del Rio; AJ Stewart; W Mullen; J Burns; EJ Lean; F Brighenti, *J. Agric. Food Chem.,* **2004**, 52 (10), 2807–2815.
[15] M Monagas; M Urpi-Sarda; F Sánchez-Patán; RI Llorach; Garrido; C Gómez; CA Lacueva; B Bartolomé, *Food Funct.* **2010,** 1, 233-253.
[16] GJ Du; ZH Zhang; DX Wen; CH Yu; T Calway; CH Yuan; CH Wang, *Nutrients*, **2012**, 4(11), 1679–1691.
[17] T Shahwan; SA Sirriah; M Nairat; E Boyacı; AE Eroglu; TB Scott; KR Hallam, *Chemical Engineering Journal*, **2011**, 172, 258–266.
[18] L Huang; X Weng; Z Chen; M Megharaj; R Naidu, *Molecular and Biomolecular Spectroscopy*, **2014**, 130, 295–301.
[19] GE Hoag; JB Collins; JL Holcomb; JR Hoag; MN Nadagoudab; RS Varma, *J. Mater. Chem.,* **2009**, 19, 8671–8677.