

## GREEN SYNTHESIS OF $\alpha$ - $\text{Fe}_2\text{O}_3$ NANOPARTICLES USING ROMAN NETTLE

N. BADNI<sup>1,3</sup>, F. Z. BENHERAOUA<sup>1,3</sup>, B. TADJER<sup>1,3</sup>, A. BOUDJEMAA<sup>3</sup>, H. EL HAMEUR<sup>2</sup>,  
K. BACHARI<sup>3</sup>

<sup>1</sup>Centre de Recherche Scientifique et Technique en Analyses Physico-Chimiques. Plateau technique d'analyses physico-chimiques Chlef, Hay Salem, route nationale N° 19 02000 Chlef, Algeria  
nadiabadni@gmail.com, benheraoua.fz@hotmail.com & tadjer16@yahoo.fr

<sup>2</sup>Laboratoire de biologie moléculaire, Génomique et bioinformatique, université de chlef, Hay Salem, route nationale N° 19 02000 Chlef, Algeria. hacelhameur@yahoo.com

<sup>3</sup>Centre de Recherche Scientifique et Technique en Analyses Physico-Chimiques, BP 384, Siège ex-Pasna Zone Industrielle, Bou-Ismaïl CP 42004, Tipaza, Algeria.  
amel\_boudjemaa@yahoo.fr & kbachari2000@yahoo.fr

### ABSTRACT

The environmental protection and remediation of environmental problems became major issues of the 21<sup>st</sup> century. However, nanotechnology is now regarded as a distinct field of research in modern science and technology. Among the various approaches currently available for the generation of metallic nanoparticles, plant materials are the most readily available. In this review, we have provided information on the method and agents used for the green synthesis of hematite  $\text{Fe}_2\text{O}_3$  nanoparticles, the hematite was synthesized using extract of Roman Nettle (RN) and iron nitrate. Structural, morphological and optical proprieties were characterized by TEM, DRX, SEM, BET, RD and FTR. The results show a formation of nanoparticles with a diameter around 61nm with a specific surface area of 96.2 m<sup>2</sup>/g.

**Mots Clés:** *Green Synthesis, Roman Nettle, Algeria, Hematite, Nanoparticles.*

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### NOMENCLATURE

**Symbols :**

Eg band gap eV

**Subscripts :**

RN Roman nettle

FeNPs iron nanoparticles

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### 1. INTRODUCTION

The green synthesis method utilizing plant extracts has been used as an environmental-friendly approach for the synthesis of metallic nanoparticles. Among several nanoparticles, recently iron oxide ( $\alpha$ - $\text{Fe}_2\text{O}_3$ ) nanoparticles FeNPs have been extensively used in sol remediation, engineering, cosmetics, water treatment [2, 3]. Therefore, designing and developing a simple, one-step, reliable, low-cost, non-toxic and eco-friendly method for the fabrication of multifunctional nanoparticles is of great importance. In this context, synthesis of FeNPs using green chemistry approach has several advantages over conventional chemical methods for being (i) simple, fast,

inexpensive and most reliable method, (ii) environmentally friendly due to the avoidance of many toxic chemicals, (iii) based on the bio-resources such as plants, (iv) universally acceptable solvent like water etc..

Plant extracts are capable of reducing a variety of iron salts. Roman Nettle (*Urtica pilulifera*; Family: Urticaceae) is an annual herb with stinging foliage and small clusters of green flowers. The leaves of the plants have stinging hairs, causing irritation to the skin. This action is neutralized by heat. Fig. 1 illustrates the formation of FeNPs synthesized from the aqueous extract of Roman nettle leaves.

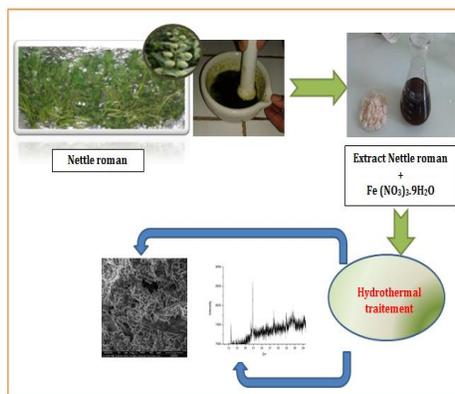


Figure 1. Schematic illustration of the green synthesis of FeNPs using leaves of Roman Nettle as aqueous extract.

## 2. EXPERIMENTALE METHODE

### 2.1 GREEN SYNTHESIS OF $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> SAMPLES

Romaine Nettle (RN) leaves were collected from a field, in Chlef, Algeria, April, 2016. The samples were washed thoroughly to remove dirt and other contaminants, and dried at room temperature for five days. After that an extract was prepared by heating dry RN in deionized water at 80 °C for 20 min. After, the extract solution was filtered and stocked at 10 °C for further use. Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O was dissolved in the RN-extract and then adjusted with distilled water. The solution was transferred to hydrothermal reactor which was heated at 180°C overnight. Finally, the product was washed with distilled water and centrifuged several times. The clean product was then subjected to drying at 80 °C under normal atmospheric pressure Fig. 2.

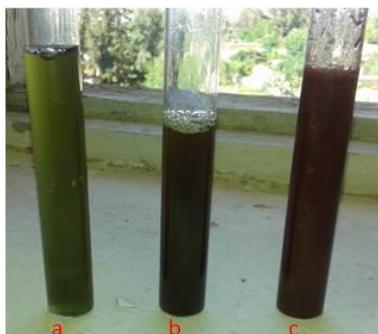


Figure 2. Illustration of the three main steps in the green synthesis of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles.

- a. Extract of NR
- b. Addition of the iron solution
- c. formation of FeNPs in suspension after treatment at 180 °C for 13h

## 2.2 CHARACTERIZATION OF Fe-NPs NANOPARTICLES

BET analysis was carried out with a Micromeritics ASAP2020 M volumetric adsorption analyzer at 77 K. Before measurement, the powder was degassed over night under vacuum at 180 °C. The Fourier Transform InfraRed (FTIR) spectrum was recorded with a Perkin-Elmer FTIR 1000 spectrometer, 32. Scans were taken to improve the signal-to-noise ratio in the wavelength range (400 - 4000  $\text{cm}^{-1}$ ). UV-Vis diffuse reflectance (DR) spectrum was obtained with a Shimadzu model UV-2100 spectrophotometer, equipped with an integrating sphere accessory.

## 3. RESULTS

UV-Vis diffuse reflectance (DR)

The absorption spectra of  $\alpha$ -FeNPs are presented in Fig. 3. The strong absorption band centered at 730 nm gave a band gap ( $E_g$ ) which was performed according to the equation Tauc and kabulka munk. The gap  $E_g$  was equal to 1.89 eV, which is the same as the iron's  $E_g$  [4].

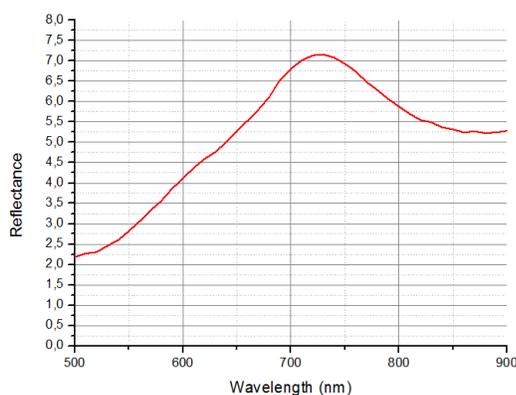


FIGURE 2 . UV-visible spectra of the synthesized powder

SEM and EDX analysis

RN synthesized nanoparticles revealed by SEM images were aggregated as irregular spherical shapes with rough surfaces (Fig. 4.). The polyphenols concentrations in RN leaves extracts play a key role in the formation of the final structures and size of the  $\alpha$ -FeNPs.

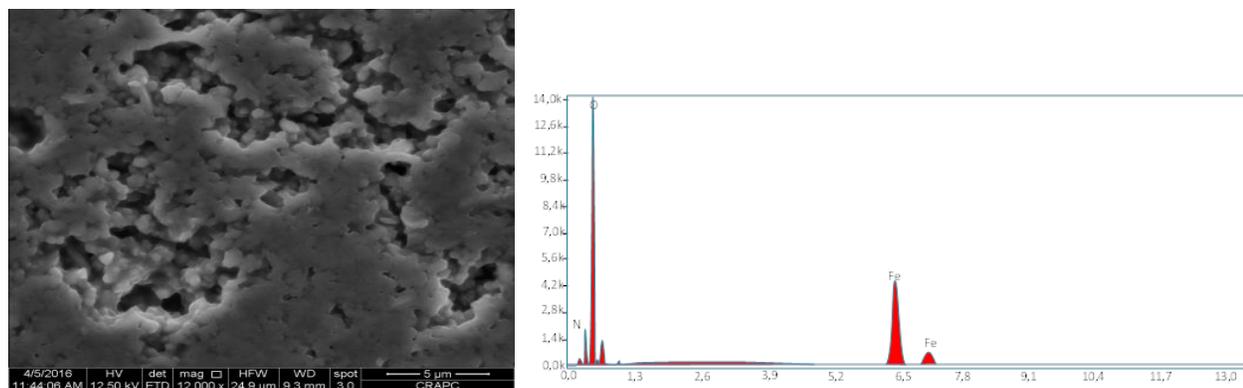


FIGURE 4: SEM image and EDX spectrum of synthesized hematite

The EDX spectrum for  $\alpha$ -FeNPs gives the information of the surface elements. The atomic percentages of different elements are represented in Table 1.

Elements	% mass	% atomic
Fe	<b>42.80</b>	<b>17.20</b>
O	<b>43.67</b>	<b>61.25</b>
N	<b>9.87</b>	<b>15.82</b>
other	<b>3.66</b>	<b>5.73</b>

**TABEL 1 .** The percentage of various elements

The obtained results showed in Table 1 and Fig. 4 revealed a high Fe content followed by a higher level in Oxygen (O peak) in the  $\alpha$ -FeNPs. These data clearly demonstrates and confirm the formation of iron oxide. Furthermore, N and O signals were originated predominantly from polyphenolic substrates [5].

#### FTIR and BET analysis

FTIR spectrum (Fig.5.) shows a broad band in the region  $3500 - 2900 \text{ cm}^{-1}$  assigned to the stretching vibration of hydroxyl O–H. Several peaks in the spectral range  $800\text{--}1800 \text{ cm}^{-1}$  were attributed to the polyphenols that were presumably present at the surface of FeNPs. A very strong peak at  $1034 \text{ cm}^{-1}$  was attributed to the stretching vibration of C–O–C. A peak located at  $1621$  and  $1600 \text{ cm}^{-1}$  characterize by the in-plane bending vibration of –OH in phenol and C=C ring stretching in polyphenols, respectively. The band positioned around  $525$  and  $458 \text{ cm}^{-1}$  attributed to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>.

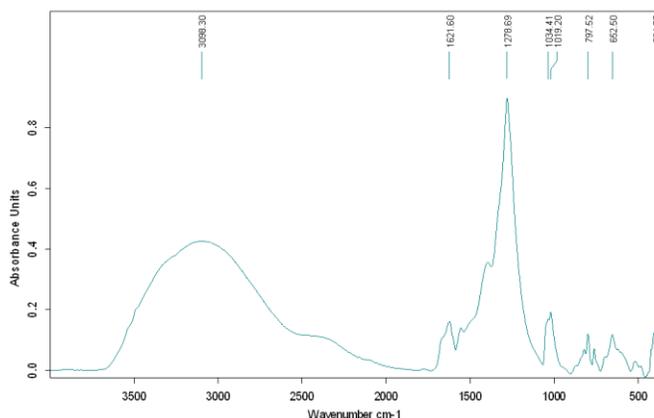


FIGURE 5: FTIR spectrum of FeNPs

The surface specific area and pore volume of FeNPs play an important role via the catalytic activities in several applications such adsorption, storage, catalysis...etc. The BET (Brunauer-Emmett-Teller isotherm) method is used to provide this information for a particular nanomaterial. The BET surface areas of FeNPs was found around  $96.2 \text{ m}^2 \text{ g}^{-1}$

#### 4. CONCLUSIONS

Obviously, the  $\alpha\text{-Fe}_2\text{O}_3$  nanoparticles were successfully prepared by hydrothermal green synthesis of Roman Nettle leaves. We find this simple method appropriate to synthesize a high quality of nanoparticles using nitrates iron and nettle leaves. The obtained  $\alpha\text{-Fe}_2\text{O}_3$  NPs were well characterized using UV-vis spectroscopy, FTIR and SEM analysis. FTIR analysis revealed the formation of hematite at  $525 \text{ cm}^{-1}$ . However, Polyphenolics compounds of the nettle leaves have improved characteristics of nanoparticles which have at the end presented a specific surface area of about  $96.2 \text{ m}^2 \text{ g}^{-1}$ .

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