Journal of Engineering Sciences
& Information Technology
Issue (1), Volume (2)
March 2018
ISSN: 2522-3321

المجلة العربية للعلوم و نشر الأبحاث
Arab Journal of Sciences & Research Publishing
AJSRP

# Characterization of Magnetite and Hematite Using Infrared Spectroscopy

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**Abstract:** In this work, Infrared spectroscopy has been carried out to characterize the composition of the as-prepared samples in magnetite and hematite.

The infrared spectra presented in this work were recorded in the range from 400 to 4000 $cm^{-1}$ with FT-IR spectrometer. Infrared light is guided through an interferometer and then through the sample (or vice versa). A moving mirror inside the apparatus alters the distribution of infrared light that passes through the interferometer. The results obtained showed that the characteristic bands of magnetite and hematite are appeared in the spectra of two samples. Characteristic bands of hematite appeared in spectra of magnetite. Besides other materials appear in spectra of the two samples as, sulphate, water, silica and carbonate. Infrared spectroscopy proved to be easy and fast methods for the identification of characterization composition in hematite, magnetite samples and could be used for other materials.

**Keyword**: Magnetite and Hematite, Characterization, Infrared

## 1. Introduction

Infrared (IR) spectroscopy is one of the most common spectroscopic techniques used by organic and inorganic chemists. Simply, it is the absorption measurement of different IR frequencies by a sample positioned in the path of an IR beam. The main goal of IR spectroscopic analysis is to determine the chemical functional groups in the sample. Different functional groups absorb characteristic frequencies of IR radiation. Using various sampling accessories, IR spectrometers can accept a wide range of sample types such as gases, liquids, and solids. Thus, IR spectroscopy is an important and popular tool for structural elucidation and compound identification (Steele, D., 2006)

When electromagnetic radiation interacts with matter, energy is absorbed. If the energy of incident photon corresponds to the energy gap between ground state and excited state of atoms or molecules the energy absorbed and atom or molecule excited where an electron promoted from it's to higher energy state (Le Ru, E. and Etchegoin, P., 2008). Absorption spectroscopy is based on the Lambert-Beer law. Let $I_0$ is the intensity incident light on the sample and I is the intensity of the transmitted light. If the sample absorbs a part

of the light then the law states that fraction of light **Log** $\frac{I_o}{I}$ absorbed by the sample is the sample dI so that, proportional to the sample concentration (c) and the length of so that

$$\text{Log } I_o / I = \varepsilon \, c \, dI \qquad 1$$

Where ε is the extinction coefficients. The probability per second that atom or molecule will absorb a photon, $dP_{12}/dt$, is proportional to the number of photons of energy hν per unit volume ρ(ν) and is usually expressed as:

$$\frac{d}{dt}\rho_{12} = B_{12}\,\rho(\nu) \qquad 2$$

Where the constant $B_{12}$ is the Einstein coefficient of induced absorption (units $J^{-1}\ m^3\ s^{-2}$). It depends on the electronic structure of the atom, i.e. on its electronic wave functions in the two levels 1 and 2. Each absorbed photon of energy hν decreases the number of photons in one mode of the radiation field by one (Berman, P.R. and Malinovsky, V.S., 2010)

## 2. Experimental part

### 2.1 Sample preparation

Two sample were prepared through chemical methods in the lab

The Firstly, the magnetite ($Fe_3O_4$), a sample can be obtained, using$Fe^{11}$ Sulphate in the air at $500^0$ C through equations:

$3FeS_2 + 5O_2 = Fe_3O_4 + 3S + 3SO_2$ (Schwartzman, U. and Cornell, R.M., 2008).

Secondly, the hematite ($Fe_2O_3$) was obtained using 40 gram of Ferric nitrate (Fe $(N0_3)_3$. 9 $H_20$) was dissolved in 500 ml of twice distilled water in polyethene flask. Then, 300 ml of one molar (1M) potassium hydroxide (KOH) was added to flasked followed by 50 ml of one molar (1M) $NaHCO_3$. The mixtures were heated to $90C^0$, till the formation change to red- brown precipitates of ferrihydrite. The flask and the content were allowed to stand for 48 hours. During this time the red brown suspension of ferrihydrite was transformed to hematite with pH of 8 to 8.5 (Schwertmann, U. and Cornell, R.M., 2008).

### 2.2 Equipments

Figure (1) shows schematic structure diagram of FTIR spectrometer which was used in this work. FTIR spectrometer is better speed and sensitivity. A complete spectrum can be obtained during a single scan of the moving mirror, while the detector observes all frequencies simultaneously, simpler mechanical design. There is only one moving part, the moving mirror, resulting in less wear and better reliability, elimination of stray light and emission contributions. ,

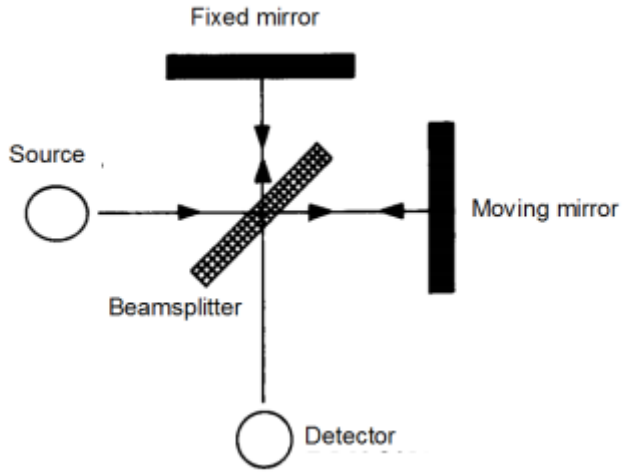


**Figure1; schematic structure diagram of FTIR spectrometer**

## 3. Result and discussion

FTIR spectrum of magnetite ($Fe_3O_4$) samples were collected and shown in Figure 2, the spectrum shows clear peaks and by comparison with the vibrations recorded in the some references we these vibrations are attributed in table (1)

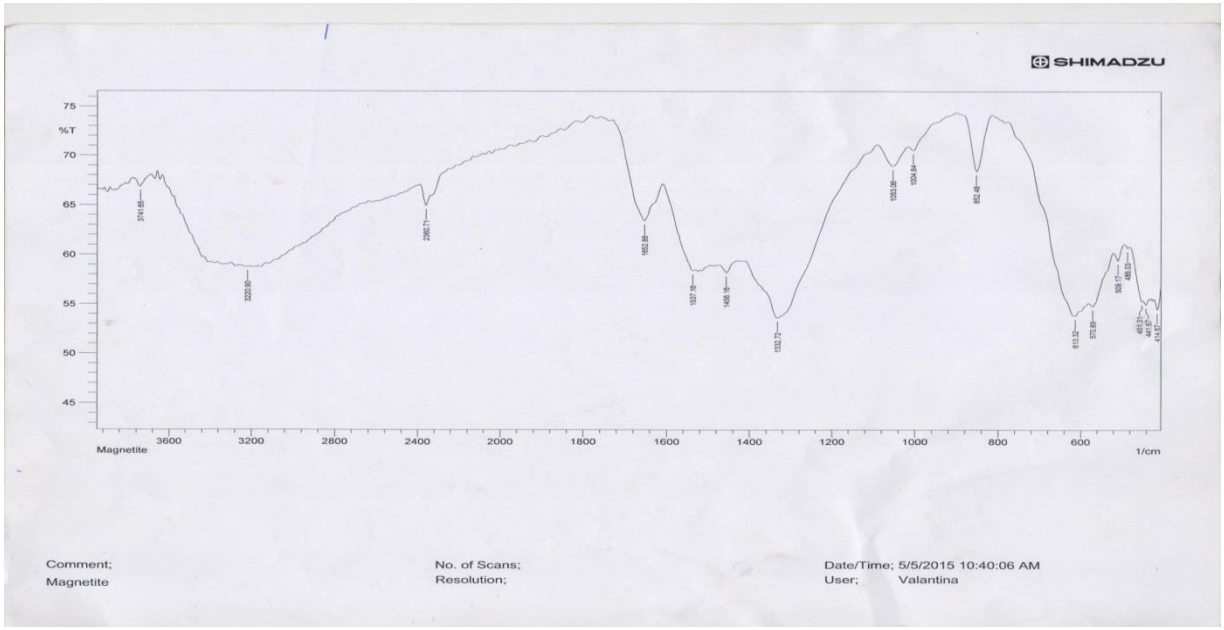


**Figure 2: Raman spectrum of magnetite in the range (200 $cm^{-1}$ to 800 $cm^{-1}$).**

Table1: The analyzed data of infrared spectrum of the magnetite

| Compounds | Wane number/$^{cm-1}$ | Functional groups |
|---|---|---|
| Magnetite | 414. 76 | Fe111-O |
| | 441.76 | Fe111-O |
| | 451.13 | Fe11I-O |
| | 486.3 | Fe11I-O |
| | 509.17 | Fe11-O |
| | 570.89 | Fe111-O |
| | 613.32 | Fe11-O |
| | 852.48 | O-H |
| | 1004.84 | S=O |
| | 1053.0 | S=O |
| | 1323.72 | C-O |
| | 1465 .16 | C-O |
| | 1537.16 | C-O |
| | 1652.88 | O-H |
| | 2360.71 | O-H |
| | 3220.90 | O-H |
| | 3741.65 | O-H |

Table (1) illustrates that most of the infrared spectroscopy for vibrations mode of materials were found between 400-4000 cm-1 . Two vibrational modes of FeIII -O at 570.89 $cm^{-1}$ and 414. 76 $cm^{-1}$ are assigned to magnetite and mentioned in the literature (Schwertmann, U. and Cornell, R.M., 2003, A. L. Andrade et al., 2009).

Vibration mode of FeII-O appeared at 613.32 $cm^{-1}$, 509.17 $cm^{-1}$ are assigned to maghemite and mentioned in the literature (Jacintho, G.V., et al 2009) . Vibration mode of FeII1-O noticed at 486.3 $cm^{-1}$, 451.13 $cm^{-1}$ and 441.76 $cm^{-1}$ are assigned to magnetite and this agreed with the results of other research (Perla E, et al 2012, Sivakumar, D.,et al 2017 ) . Other materials vibration modes are appeared at 3741.65 $cm^{-1}$ , 2360.71 $cm^{-1}$, 3220.90 cm-1 and 1652.88 $cm^{-1}$ are assigned to the vibrations of hydrogen-bonded water molecules adsorbed on the surface and O-H bending vibration (Gotić, M. and Musić, S., 2007, Holland, H. and Yamaura, M., 2009.) , and additional band were found at 1053.0 cm-1 and 1004.84 $cm^{-1}$ are assigned to specifically adsorbed sulphate groups according to old reference (Schwertmann, U. and Cornell, R.M., 2003) ,also the vibration mode of Si O-Si appeared at 852.48 cm-1 is assigned to silicate, additionally C-O vibration modes showed at 1323.72 $cm^{-1}$ , 1465 .16 $cm^{-1}$ and 1537.16 are assigned to carbonate mention in reference (Sivakumar, D et al2017).

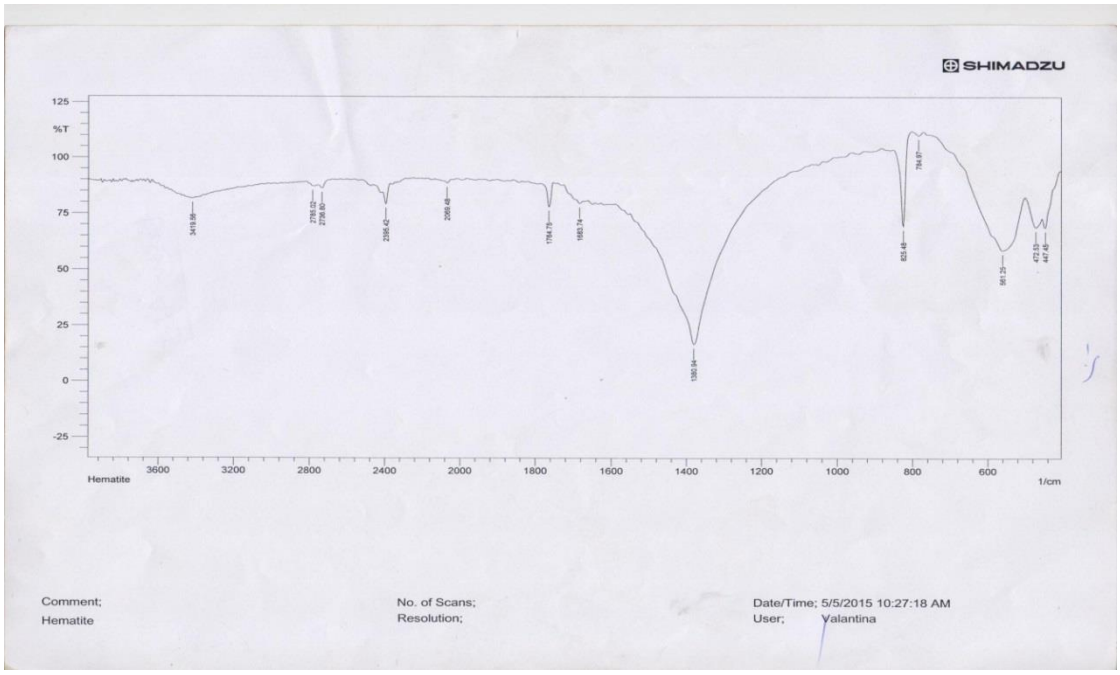


**Figure 3: Raman spectrum of hematite in the range (200 $cm^{-1}$ to 800 $cm^{-1}$).**

**Table2: The analyzed data of infrared spectrum of the hematite**

| Compounds | Wane number/$cm^{-1}$ | Functional groups |
|---|---|---|
| hematite | 447.45 | FeII-O |
| | 472.53 | FeII-O |
| | 561.25 | FeII-O |
| | 784.97 | Si-O - Si |
| | 825.48 | O-H stretching |
| | 1380.94 | C—O stretching |
| | 1683.74 | H—O—H bending |
| | 1764.75 | C-O stretching |
| | 2069.48 | O-H stretching |
| | 2395.42 | O-H stretching |
| | 2736.80 | C-H |
| | 2785.2 | C-H |
| | 3419.56 | O-H stretching |

For the peaks of hematite listed in the table (2) three vibration mode of FeII-O at 561.25 $cm^{-1}$ ,472.53 $cm^{-1}$ and 447.45 $cm^{-1}$ are assigned to hematite a corroding to old references (Schwertmann, U. and Cornell, R.M., 2003). Bands of Other material vibration mode have appeared in spectrum at 3419.56 $cm^{-1}$ , 1683.74$cm^{-1}$ and 825.48$cm^{-1}$ are assigned to adsorbed water and vibration of the OH groups agreed with the results of other research (Lu Baiqing.,2014, Srivastava, S., 2012) . additionally, C- H vibration

mode are noticed at 2785.2 $cm^{-1}$ and 2736.80 $cm^{-1}$ are is attributed to hydrocarbons impurities introduced during the preparation of the sample according to references (Maya Shopska et al 2012). And Vibration mode of C-O appeared in spectrum at 1764.75 $cm^{-1}$ and 1380.94 $cm^{-1}$ are assigned $CO_3$ 2and $HCO_3$-, which indicates that samples contained carbonate contaminants to carbonate according to reference (Lu Baiqing.,2014,Tharani, K. and Nehru, L.C., 2015 ) .

## Conclusions

Magnetite and hematite nanoparticles were successfully synthesized using Thermal decommission $Fe^{11}$ sulphite and precipitation of ferrihydrite respectively , through chemical methods in the lab , by using FTIR spectrometer The results presented show that IR spectroscopy technique is an efficient and fast method to determine compositions in hematite and magnetite.

## Acknowledgment

I wish to thank Prof. Nafie A. Almusle for his diligently guide. I really appreciate that you can give me a chance to write my PHD thesis. I will also thank to university of Khartoum department of chemistry for help me to-do the practical part and great thank to the ministry of high education for finical support.

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## توصيف المجناتيت والهماتيت باستخدام مطيافية الأشعة تحت الحمراء

**الملخص**: في هذا البحث استخدمت مطيافية الأشعة الحمراء لدراسة الخصائص والمكونات في كل من عينة الهماتيت، المجناتيت تم تشعيع هذه العينات باستخدام مطياية فورير الانتقالية في مدى الطول الموجي 400 إلى 4000 سم-1 ووجهة الضوء إلى مقياس التداخل ثم إلى العينة توجد مرآة متحركة في مطياف فورير تعمل على توزيع الضوء المار الى مقياس التداخل . أوضحت النتائج المتحصل عليها ظهور حزم الهماتيت، المجناتيت في أطياف كل من العينتين حيث ظهرت حزم الهماتيت في عينة المجناتيت مع ظهور بعض المواد الأخرى في طيف كل من العينتين مثل السلفيت، الماء، السليكا والكربون، برهنت النتائج أن مطيافية الأشعة الحمراء طريقة سهلة وسريعة لدراسة الخصائص المكونات في كل من عينة الهماتيت، المجناتيت ويمكن استخدامها أيضاً في معرفة المركبات في المواد الأخرى.

**الكلمات المفتاحية**: الهماتيت، المجناتيت، توصيف، الأشعة تحت الحمراء.