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Research Article

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The Structural Properties of Iron Oxides using Raman Spectroscopy

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Abstract The Raman spectroscopy was used to study the structural properties of five samples of iron oxide (magnetite, hematite, akaganeite, ferrihydrite and goethite), firstly the five samples were prepared through chemical methods, then samples irradiated with doubled Nd-YAG laser (532 nm) at room temperature. The Spectra database was used for the spectral analysis of the Raman shift of these samples. The obtained results showed that the Characteristic bands of magnetite, hematite, akaganeite, ferrihydrite and goethite. Other materials characteristic bands were appeared in spectra of the five samples, like silicate, disulfide, alkyl disulfide, chlorolkanes and aliphatic fluoro. Raman spectroscopy is good and fast methods to characterizations magnetite, hematite, akaganeite, ferrihydrite and, goethite, could be used for other materials.

Keywords iron oxides nanoparticles; Structural Properties; Raman Spectroscopy

1. Introduction

Raman spectroscopy technique was discovered by Raman and Krishnan. The function of this technique to examine molecular vibrational and rotational modes within a system using in elastically scattered visible or near-infrared (NIR) radiation [1]. Raman spectroscopy occurs when a molecule is irradiated by a light with frequency (v), a small portion of it is scattered in elastically but most part is scattered elastically. The elastically scattered light with the same frequency(**v**) of the incident light is called Rayleigh scattering, and the in elastically scattered light with frequencies($\mathbf{v}\pm\mathbf{v}_1$) is called Raman scattering named stoke and un tistoke [2] .

2. Materials and Methods

Samples from magnetite, hematite, akaganeite, 2 line Ferrihydrite and goethite were investigated. These samples were prepared through the chemical methods firstly, the magnetite (Fe_3O_4), sample can be obtained, using Fe^{11} Sulphate in air at 500 ºC through below equation

$$FeS_{2_+} + 5O_2 = Fe_3O_4 + 3S + 3SO_2 \quad (1)\ [3]$$

Secondly, thirdly the hematite (Fe_2O_3), was obtained using 40 gram of ferric nitrate ($Fe\ (NO_3)_3.9\ H_2O$) was dissolved in 500 ml of twice distilled water in polyethylene flask. Then, 300 ml of one molar (1M) potassium hydroxide (KOH), was added to the flaked followed by 50 ml of one molar (1M) $NaHCO_3$. The mixtures were heated to 90 ºC, until formation of 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 [3]. Thirdly, Akaganeite (FeOOH), 0.1 molar (0.1M) of $FeCl_3$ solutions was hold in to 2 liter in closed vessel at 70 ºC for 48h. During this time the pH of the system drops from 1.7 to 1.2 and compact yellow precipitate of akaganeite was formed [4]. Fourthly, 2Line Ferrihydrite 40 gm of Ferric nitrate ($Fe\ (N0_3)_3.9H_2O$) was dissolved in 500 ml distilled water and added with stirring, 330 ml 1molar KOH to bring the pH to 7-8, centrifuge , then dialye rapidly as possible to remove electrolytes and freeze dry [4]. Finally, Goethite (αFeOOH) sample, was prepared using100 ml of one molar (1M) Ferric nitrate ($Fe(NO_3)_3$ solution was added into 2 liter polyethylene flask, then 180 ml of five molar (5M) potassium hydroxide (KOH) was added rapidly with stirring, till formation of Red brown precipitates of ferrihydrite at once. The suspension was diluted with twice stilled water and holed in a closed polyethylene flask maintained at 70 ºC in an oven for 60h. At the end of this period of time the red-brown suspension of ferrihydrite transformed in to a compact yellow precipitate of goethite [3].

2.1. Instruments

Laser Raman spectra were recorded at room temperature using barker sentrra spectrometer show in figure (1) at 532 nm from Nd-YAG laser was used to excite the samples. The laser power was justify at 5mW. The Raman shift in wave number, and the change in intensities of the scattered light were compared with data in the references and previous studies.

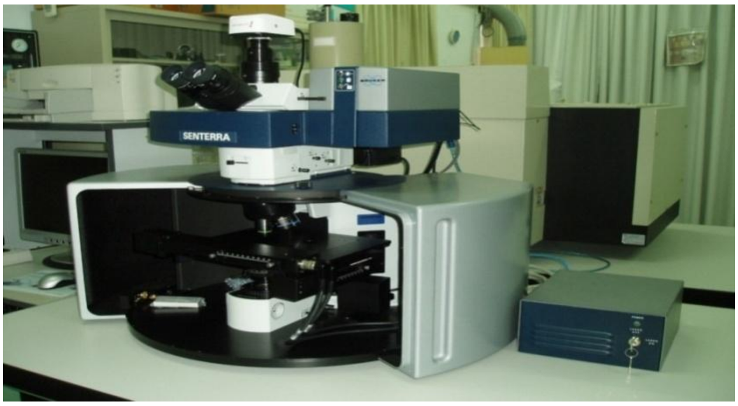

Figure 1: The parts of burker Sentrra laser Raman microscope spectrometer

3. Result and Discussion

The Raman spectroscopic analysis great the phase of this result to be magnetite, Table 1 and Fig. 2, as shown by the presence of the characteristic bands of magnetite Raman bands occurring at 272, 319, 543 and 668 cm^{-1} consistent with references [5], and magnetite band also appeared at 616 and 362 cm^{-1} [6,7]. Bands of other material are noticed, peaks observed at 702 cm^{-1} provide evidence due to the presence of hematite in agreement of mentioned in the literatures [8], also bands at 458 ,511 , 591 and 764 cm^{-1} and assigned to silicate, dialkyl disulfide, alkyl sulfides and Aliphatic Fluoro respectively mentioned in references [9,10] .

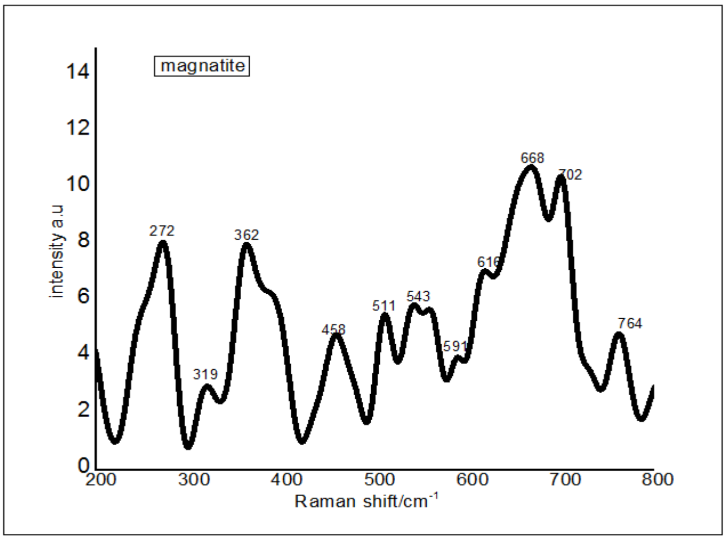

Figure 2: Raman spectrum of magnetite in the range (200 cm^{-1} to 800 cm^{-1})

Table 1: Raman shift and assignments of iron oxides

Compounds	Raman shift (cm^{-1})	Assignment	References
Magnetite	272, 319, 362, 543, 616, 668	FeIII-O	[5-7]
Hematite	220, 252, 288, 407, 491, 610	FeII-O	[8, 11,16]
Goethite	247,299, 387, 475, 549, 689	α FeOOH	[14, 16]
Akaganeite	302, 385, 708, 490, 533, 612, 670	βFeOOH	[13]
2-Line frryhidrite	707, 511 , 607, 389		[14-15]

Hematite

Table 1 and fig. 3 shows the Raman spectrum of hematite. The characteristic bands of hematite appeared at 288, 220, 407, 491, 610 and 252 cm^{-1} [8,11,16]. Additionally characteristic bands of other materials are appeared, magnetite noticed at 660, 323, 362, 713 cm^{-1} [5,6]. Aliphatic Fluoro at 764 and 784 cm^{-1} agreed with the results of other research [9,10], silicate, alkyl sulfides and dialkyl disulfide at 464, 685 and 514 cm^{-1} respectively mentioned in references [12].

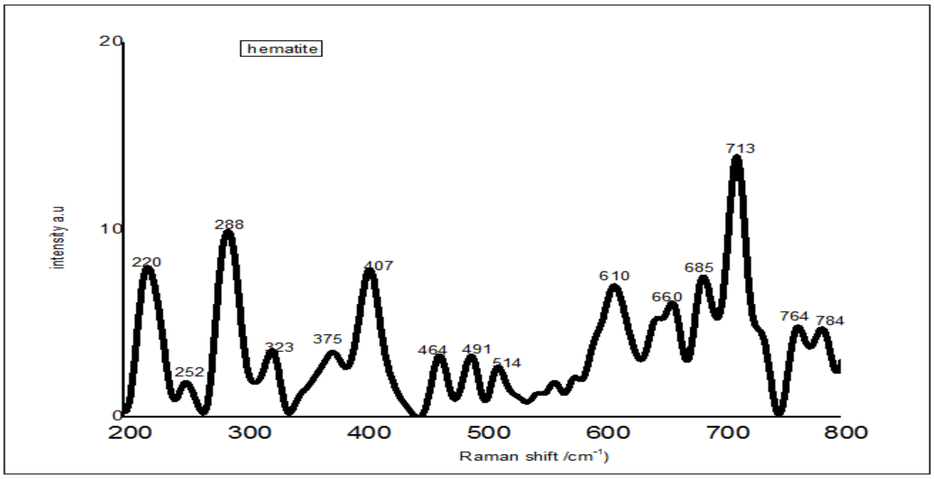

Figure 3: Raman spectrum of hematite in the range (200 to 800) cm^{-1})

Akaganeite

characteristic bands of akaganeite appeared at 302, 385, 708, 490, 533, 612 and 670 cm^{-1} [13] see in fig 4 and table 1, beside characteristic bands of other materials like hematite noticed at 215 and 258 cm^{-1} [8], dialkyl disulfide, alkyl disulfide and e aliphatic fluoen according at 448, 579 and 773 cm^{-1} [12].

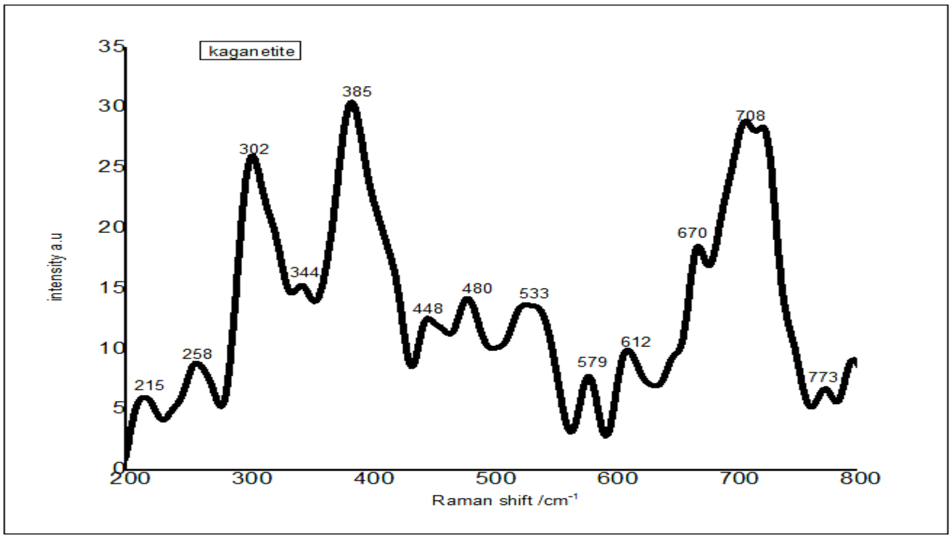

Figure 4: Raman spectrum of akaganeite in the range from 200 -800 cm^{-1}

Ferrihydrite

For ferrihydrite, fig 5 and table 1, the characteristic bands appeared at 707, 511, 607 and 389 cm^{-1} [14,15] with appeared other materials, hematite at 282, 650, 218 and 488 cm^{-1} and mentioned by De Faria, et al [11]. Silicate, alkyl sulfides, dialkyl disulfide, primary chlorolkanes and aliphatic fluorine at 458, 679, 430, 576 cm^{-1} and (763, 729) cm^{-1} [9,12].

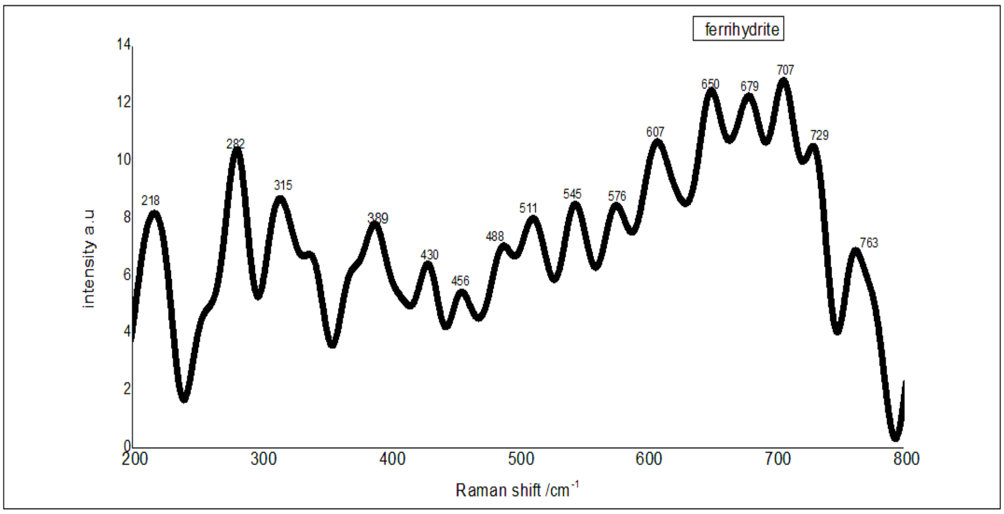

Figure 5: Raman spectrum of ferrhdrite in the range (200 -800) cm^{-1}

Goethite

As shown in table 1 and figure 5, the goethite samples the characteristic Raman features are located at 247, 299, 387, 476, 689, 585 and 549 cm^{-1} [14,15]. Characteristic bands of other materials are found, hematite bands noticed at 624 cm^{-1} [8] , dialkyldisulfide at 514 and 431 cm^{-1} and aliphatic fluoro appeared at 729 and 756 cm^{-1} [9,10].

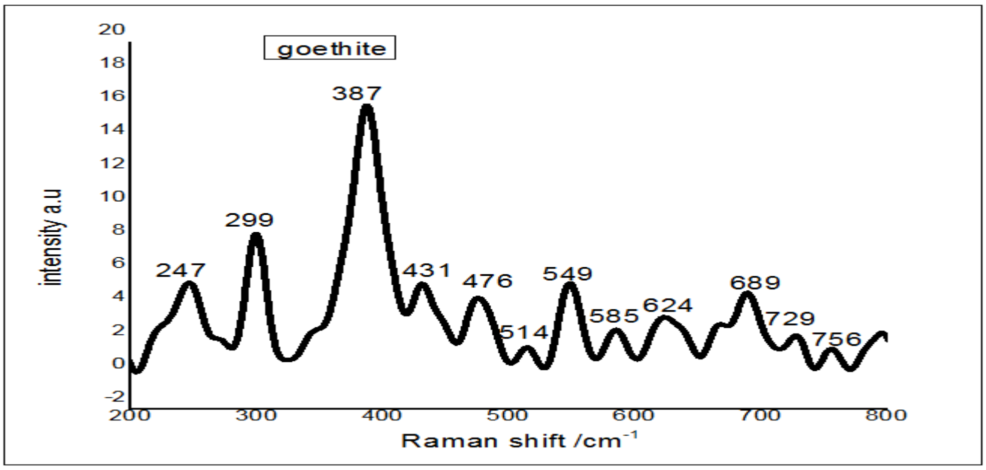

Figure 6: Raman spectrum of goethite in the range from (200 -800) cm^{-1}

4. Conclusions

The Raman spectroscopy was useful tools for analyses iron oxides that provided valuable and the most cases detailed information, moreover, generally gave better results for, both organic and inorganic materials. Also can offers higher resolution and the possibility for confocal measurements**.** Furthermore, Laser Raman spectroscopy technique is easy, the results are obtained in a short period time and little sample preparation is required for Raman experiments. Raman spectroscopy can distinguish between the magnetite of hematite, akaganeite, 2line ferrihydrite efficiently and goethite**.** Specific information about other materials can be obtained from the five samples.

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