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Compositional, Structural, Surface Characterizations of Natural Magnetite from Air Massif (Niger) in Relation to Its Catalytic Activity

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Abstract

Ferrimagnetic materials such as natural magnetite are used for practical applications because of their electronic, magnetic and catalytic properties in the degradation of organic compounds. In order to determine its physicochemical properties in relation to its catalytic activity, the natural magnetite of Ofoud Mount (Niger) is characterized by X-ray florescence (XRF), X-ray diffraction (DRX), specific surface area (BET) and Fourier transformed infrared (FTIR). The result shows an iron content of 97.09% and a specific surface area of 69.742 m^2/g. The crystal structure of magnetite is cubic with lattice parameters $\alpha = \beta = \gamma = 90^\circ$, a (Å) = b (Å) = c (Å) = 8.3740. The results of this study suggest that the natural magnetite of Ofoud Mount can be used as iron source in various fields of science despite the presence of a few impurities that can improve its catalytic activity.

Keywords

Magnetic, Mineral Composition, Structural, Surface Area

1. Introduction

Ferrimagnetic materials such as natural magnetite are used widely in various fields of science because of their electronic, magnetic and catalytic properties [1] [2] [3]. Characterized by a magnetic susceptibility which allows their recovery after use, natural magnetite, magnetite fixed on a support and ferromagnetic nanoparticles are used as catalysts in environmental remediations because of their stability [4] [5] [6]. According to H. He *et al*. [7], natural magnetite exhibits catalytic performance superior to the pure synthetic magnetite. It also contains ferrous and ferric ions which improve its catalytic activity more than other ferromagnetic materials such as hematite and goethite which contain only ferric ion [8]. Due to its redox properties, magnetite offers high activity in the oxidation process of many non-biodegradable organic compounds (pentachorophenol, phenol, polycyclic aromatic compounds PAHs) [9] [10]. Magnetics are ideal for treating soil contaminating with hydrocarbons through the heterogeneous Fenton reaction [11]. Many studies have demonstrated that the capacity of raw rocks rich in magnetite to adsorb and degrade pollutants is largely dependent on their mineral structure and composition [12]. It specifically depends on parameters such as the presence of impurities, the specific surface area, the oxidation state. The chemical composition of magnetite also varies from one iron ore to another, which gives it variable chemical properties [13]. The characterization of each raw rock is therefore essential for a better understanding of its properties and its use. Previous studies on general characterization have been carried out on raw rocks rich in magnetite [14], on purified materials [15] and synthesized magnetite [6]. The aim of this study was to characterize the iron mineral rich in magnetite collected from the Air massif at Ofoud Mount in relation to its catalytic activities.

2. Material and Methods

The natural materials rich in magnetite used in this study was collected from the Air massif at Ofoud Mount [5]. These materials have been characterized after grinding and sieved through a mesh to obtain an average size of 0.05 mm [16]. The mineral composition of the raw magnetite was generated by X-ray fluorescence (XRF), the mineralogical analysis was done with the X-ray diffraction method (XRD) and the specific surface area by the standard BET method.

3. Result and Discussion

3.1. Mineral Composition

The raw rock content (**Table 1**) indicated that iron is dominated by 97.09% (considered as Fe_3O_4) indicating a probable high content of magnetite and a low content of impurities. It is thus mainly composed of iron with trace elements (Ti, Ni, Co, Mn, Al and Mg). These elements had already been found in natural magnetite by Razjigaeva *et al*., 1992 [9]. Previous studies on synthetic magnetite have shown that the incorporation of Mn, Co, V, and Cr enhances the heterogeneous catalytic activity in the degradation of organic pollutants by hydrogen peroxide [3] [10] [17] [18]. The chemical composition of the raw rock proves that it can be used as iron source in various fields of science. Some studies have shown that Ni has an inhibitory effect in the catalytic activity of the magnetite [19]. In the magnetite characterized in this study Ni content is very low.

3.2. Structural Properties

The physical and chemical properties of natural magnetite depend also on its structure. The crystal structure and unit parameters of magnetite were studied at room temperature using an Empyreal X-ray diffractometer. The Mount Ofoud magnetite diffractogram is shown in **Figure 1**.

The XRD pattern is characterized by several reflections (**Figure 1**), the most intense occurred at the Position [˚2Th.] = 43.1763. **Figure 1** showed intense and sharp diffraction peaks attesting a high degree of crystallization [20]. The XRD measurements indicated that magnetite was the only crystalline phase detected despite the presence of the minor elements obtained by XRF analysis. Its structural formula is $Fe_{24\cdot00}$ $O_{32.00}$ with scale fac of 0.773. The presence of a single phase can be explained by the fact that in natural magnetite, divalent cations (Co, Ni, Zn, Cu, Mn, etc.), trivalent cations (Al, V, Cr, etc.) and tetravalents (Ti) can substitute isomorphically for iron cations without modifying the reverse

Table 1. Chemical composition of the raw rock.

Elément	Raw Rock	
	Ppm	Percent (%)
Co	1331.53	0.207631848
Ni	2007.27	0.313003221
Fe	622,636.38	97.09067165
Mn	168.84	0.02632803
Ti	4154.81	0.647879415
Mg	2177.01	0.339471592
TOTAL	632,475.84	100

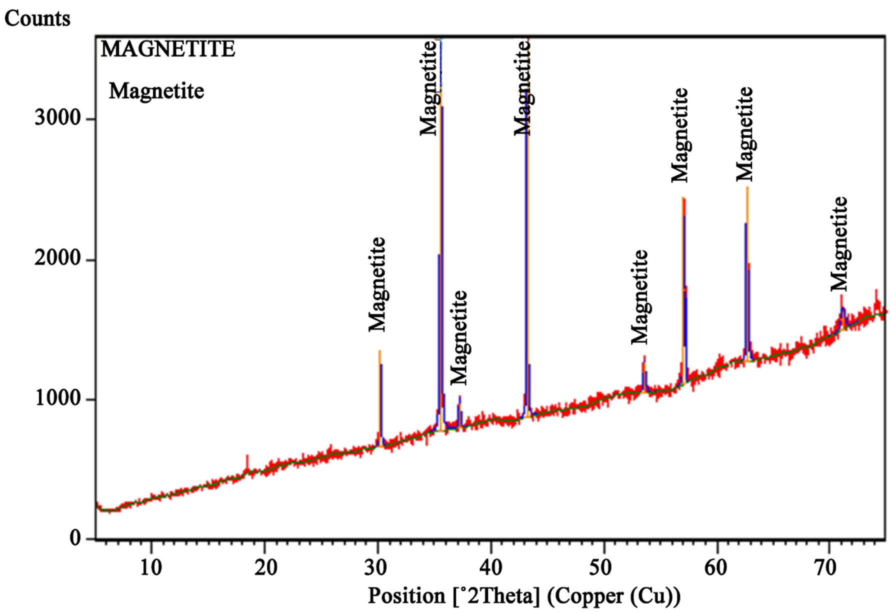

Figure 1. Magnetite diffractogram.

spinel structure [3] [17]. According to Zhong *et al.* [21] with the increase of titanium content in the magnetite, the average particle size decreases and the size distribution becomes narrow. The low intensity peak at the position [˚2Th.] = 18.336 can thus be linked to the presence of titanium in the magnetite. The crystal structure of magnetite is cubic with lattice parameters $\alpha = \beta = \gamma = 90˚$, a (Å) = b (Å) = c (Å) = 8.3740. The only magnetite phase detected crystalize in the Fd-3m with space number 227 [22]. The fit was satisfactory for the Fe_3O_4 phase after introducing the atom coordinates (**Table 1**). Its density of 5.24 g/cm^3 was appropriate with previous research [23]. The result of the DRX analysis shows the chemical purity of this natural magnetite.

3.3. Specific Surface

In the heterogeneous Fenton process, the reaction between ferrous or ferric ions and hydrogen peroxide the active sites are located on the surface of the catalyst [24] [25]. The specific surface of the magnetite was determined by N2-BET multipoint analysis using a Novantin kantachrome type surface analyzer.

As can be seen in **Table 3**, eight methods were used to determine the surface area of the magnetite. Among these analyses, BET is the most popular which has been successfully proved in evaluating the specific area of materials. The main surface area was determined to be 69.742 m^2/g with Multi Points BET (**Figure 2**). The relatively high value obtained by adsorption of nitrogen on the magnetite powder is similar to other researches Gorski *et al.* [22] for a synthetic magnetite. Compared to the specific surface of pure magnetite obtained by several authors, this surface is relatively high [26] [27]. The high value may be related to the presence of titanium in the magnetite because it is approximately equal to the titanomagnetite surface area obtained by Zhong *et al.* [21]. During the heteroge-

Table 2. Atom coordinates of Fe_3O_4.

NO.	Name	Element	X	Y	Z	Biso	Sof	Wyck
1	FE1	Fe	0.37500	0.37500	0.37500	0.8504	1.0000	8b
2	FE2	Fe	0.00000	0.00000	0.00000	1.0399	1.0000	16c
3	O	O	0.24600	0.24600	0.24600	0.7604	1.0000	32e

Table 3. Comparison on results of surface area by different methods.

Method	S (m^2/g)
Single point BET	4.320e+01
Multi Points BET	6.974e+01
Langmuir surface area	3.644e+02
BJH method cumulative adsorption surface area	7.990e+01
DH method cumulative adsorption surface area	8.512e+01
T method external surface area	6.974e+01

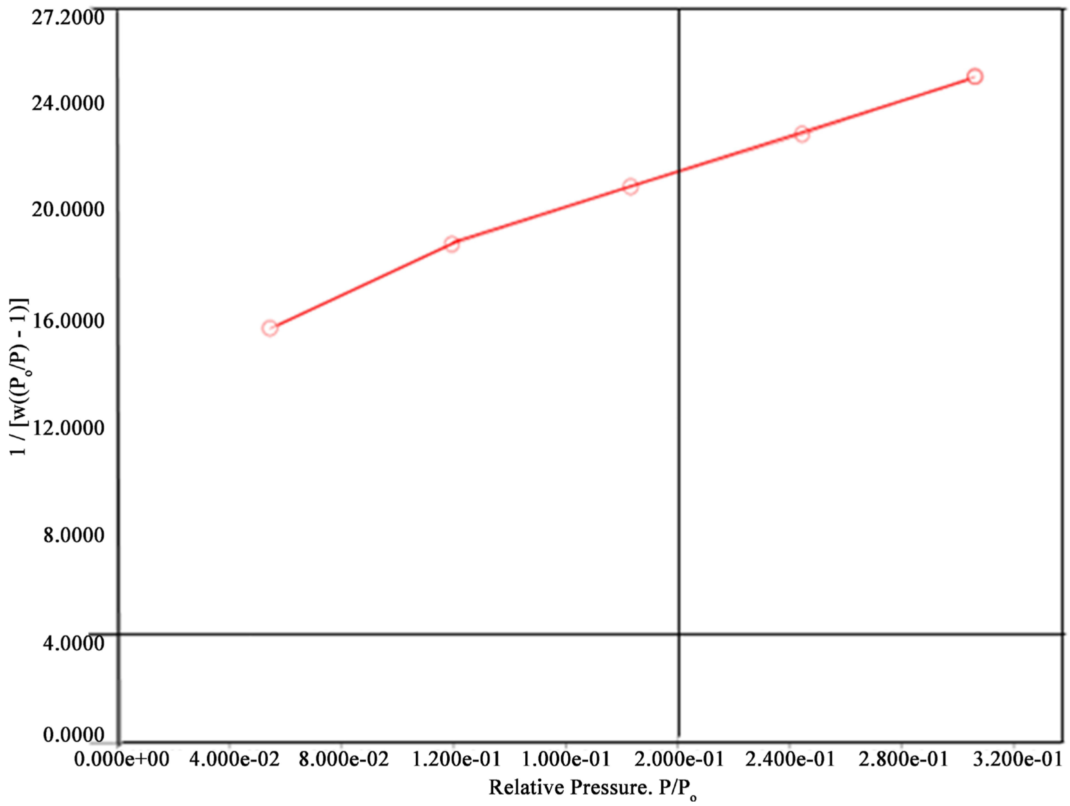

Figure 2. Multipoint BET curve on natural magnetic powders where P is the measured pressure, P˚ is the saturated vapor pressure of the adsorbate at the temperature of 77.350˚ K.

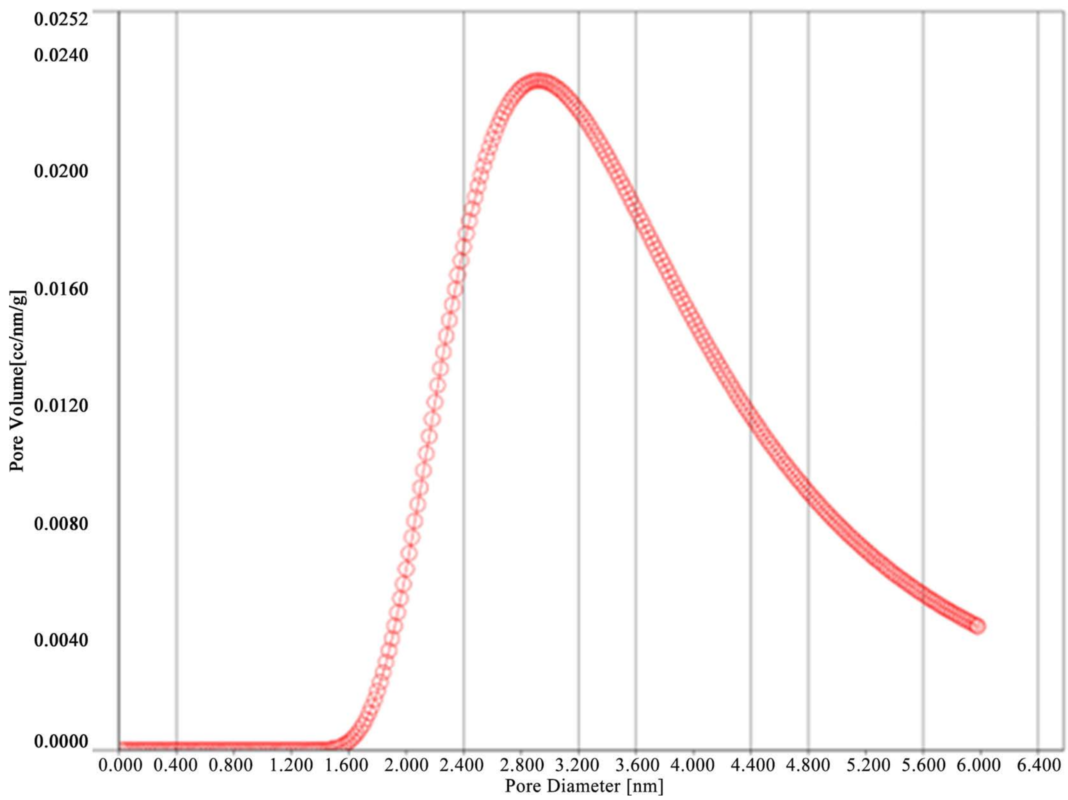

Figure 3. Pore diameter (nm).

neous Fenton process, the reaction between ferrous and ferric ions with hydrogen peroxide occurs on the surface of the catalyst [25]. The high specific surface of the crushed magnetite can thus favor the oxidation of the adsorbed compounds. The cumulative pore size distribution for magnetite is shown in **Figure 3**. It shows that the micropores vary from 1.6 to 6.0 nm. The average pore diameter estimated from the peak position is around 2.8 nm [8]. The most of the pore volume is in the mesoporous range (2 to 10 nm), showing the strong porous structure of the magnetite. The BET surface area and the average pores diameter values show that it can be used for pollutants adsorption.

4. Conclusion

This study demonstrates that the natural magnetite from Ofoud mount is highly pure materials which can be used in various fields of science. The XRD analysis showed that magnetite is the only phase detected despite the presence of traces elements (generated by XRF) that can improve its catalytic activities.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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