



## Physicochemical characterization of an industrial waste: A case study of the pyrrhotite ash from south west of Morocco.

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### Abstract

This work concerned the physicochemical characterization of an industrial waste which is the pyrrhotite ash from south west of Morocco. A physical and hydrodynamic analyzes were carried out, namely loss on ignition, moisture content, pH, solubility, density and granulometry, as well as chemical, morphological and mineralogical analyzes by SEM, XRF, XRD, FTIR and TGA/DTA. The results show that the pyrrhotite ash is a non hygroscopic waste with a moisture content equal to 3.4%. This weak hygroscopic character is mainly attributed to the spherical morphology of the waste particles. The distribution of the particle size varies from less than 1  $\mu\text{m}$  to more than 125  $\mu\text{m}$  and more than three quarter of particles have size less than 20  $\mu\text{m}$ . So, pyrrhotite ash could be useful to produce materials with high density for heat accumulation materials, construction materials...

Furthermore, the chemical composition reveals that the pyrrhotite ash is an iron rich waste with 64.9% of hematite  $\text{Fe}_2\text{O}_3$ . It contains 81.91% of  $\text{Fe}_2\text{O}_3 + \text{SiO}_2 + \text{Al}_2\text{O}_3$  and 0.526% of CaO. One the other hand, it is shown that pyrrhotite ash is a stable material at high temperatures until 1000°C and also at neutral pH.

At the end, all the cited results highlight the possibility of making the pyrrhotite ash very useful in various fields as building construction, heat accumulation materials, waste water treatment...

## 1. Introduction

Nowadays, the reuse of industrial solid wastes is a major challenge for scientists and industrialists. Recovery of these materials generates economic and environmental benefits. In fact, the reuse of industrial wastes allows developing resource-saving and environmentally friendly technologies, decreasing adverse impact on the environment and the landfilling of wastes in large disposal areas and reducing the cost of producing new products. Therefore, industrial wastes are widely studied in many areas of research. However, valorization of those wastes depends on their chemical composition, microstructure and physical and hydrodynamic properties. Industrial wastes containing iron oxide, silicon oxide, aluminum oxide - as major oxides at various content- such as fly ash ( with major oxides:  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  ), pyrite ash (major oxide:  $\text{Fe}_2\text{O}_3$ ), red mud (major oxides:  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ), were studied and reused in various fields. There are many applications of these wastes. The following are some examples:

- The fly ash is mostly used in concrete production, road basement material, waste stabilization/solidification, cement clinkers, and more recently geopolymer concrete [1, 2];
- The red mud is used to produce heat accumulator ceramic blocks [3, 4]. It is also used in plastic industry and that for production of pigments and bricks for the building industry, road construction and in agriculture [5];
- The pyrite ash is used to produce materials with high density for heat accumulation materials and road construction materials [6].

Pyrrhotite ash is an industrial solid waste, containing high concentration of hematite  $\text{Fe}_2\text{O}_3$  and traces of other oxides [7, 8], which could be considered as belonging to the group of wastes cited above, but which didn't receive the attention of scientists in order to explore the areas of its application in industry. This waste was generated during the period between 1964 and 1982 by the process of sulfuric acid manufacture from roasted pyrrhotite ore in south west of Morocco [9]. This process was proved to be unprofitable, and then it wasn't used anymore [8]. Thus, the unit of pyrrhotite roasting was replaced by another unit using the process of burning of the native sulfur. The pyrrhotite ash is currently stored in a large open space and any estimation of the stored quantity is available in literature. However, it is well known that during the above-mentioned period, 8 million tons of pyrrhotite ore were extracted and used by the manufacture of sulfuric acid [10]; and that 429000 tons of sulfuric acid were produced annually [11]. These numbers expect that a huge quantity of pyrrhotite ash is stored. To the best of our knowledge only 150000 tons were recovered by Moroccan cement industries until 2011 [7]. Definitely, there is an information gap regarding this waste in literature. In fact, any scientific study was published neither on the characterization of the pyrrhotite ash nor on the impact of this waste on the environment. So, the aim of the present research work is to study physico-chemical, mineralogical and morphological characteristics of the pyrrhotite ash, so that relevant and useful information on basic characteristics can be generated and may help carrying out further research: (i) to assess the impact of this waste on the environment and (ii) to suggest utilization of this waste as potential substitutes in some industries.

## 2. Materials and methods

### 2.1. Materials

Pyrrhotite ash samples were collected from an ash monofill located in south west of Morocco. This monofill was the sole disposal area of residue from roasting pyrrhotite for the period between 1965 and 1982.

Three samples were retrieved from one slide slope in the north of the disposal area. The first one was collected on the surface and the two others from the depths of 0.5 m and 1m respectively.

Pyrrhotite ash is a solid waste powder consisting of fine and coarse grains without odor at room temperature. Samples were collected and stored in plastic bottles before analysis.

### 2.2. Physical and hydrodynamic analysis

- Determination of the average of moisture content consists on weighing specimens before and after drying at  $120^\circ\text{C}$ . The loss of weight makes possible to calculate the average of the moisture content;
- To measure the average of the loss on ignition, samples were crushed to fine particles with a small size in the micrometer range and then calcined at  $1000^\circ\text{C}$  for 6 hours;  
For determination of both moisture content and loss on ignition a Wise Therm type oven was used;
- The average of the Hydrogen potential of the pyrrhotite ash was measured on a water leach with a ratio of 10 L/kg of dry matter. The mixtures were heated with refluxing at  $100^\circ\text{C}$  for 16 hours. Then the mixtures were filtered and the pH of the filtrates were measured [12].
- The solubility of the ash was tested in various acids of 2M concentration, using the reflux heating under magnetic stirring for 2 hours at  $100^\circ\text{C}$ .

The analyses cited above were performed in the Condensed Matter Chemistry Laboratory of University of Sidi Mohamed Ben Abdellah of Fez;

- Particle size distribution was conducted by the Sifting machine DIGITAL FI-FTL0150 & FI-FTL0200. To achieve this analysis, sample was grinded with a wooden roller. The median diameter of particles was determined using the SediGraph 5100 instrument. This technique was used only for the fraction of ash with size smaller than  $60\mu\text{m}$  and resulting from a previous granulometric separation realized by column of sieves.
- Density was measured by a Capillary cap pycnometer.

The latter analyses were performed in the Galician Institute of Ceramics, University of Santiago de Compostela, Spain.

### 2.3. Chemical, mineralogical and morphological characteristics

- The mineralogical characteristics were determined by X-ray diffraction using Siemens D500 Diffractometer. The XRD patterns were performed in the range of  $2\theta$  between  $2^\circ$  and  $60^\circ$  with operating conditions of 40 kV and 50 mA;
- The presence of functional groups on the ash was investigated using Fourier transform infrared spectroscopy (JASCO 4000 Fourier Transform Spectrometer) in wavenumber range of  $4000-400\text{ cm}^{-1}$ ;
- The thermogravimetric analysis (TGA–DTA) has been performed with a TA Instruments balance model STA-1640. It allows obtaining simultaneous DTA and TGA diagrams under similar experimental conditions. Experiments were performed under an  $\text{N}_2$  flow, from 298 K to 1073 K at a heating rate of  $5^\circ\text{C}/\text{min}$ ;

These three analyses were performed in the Galician Institute of Ceramics, University of Santiago de Compostela, Spain.

- The morphological characteristics were determined for a calcined pellet of pyrrhotite ash at  $900^\circ\text{C}$  by Scanning Electron Microscopy (SEM). This was done using SEMHitch S-2500 microscope - operating with an acceleration voltage of 16kv - of the Innovation Center of Sidi Mohamed Ben Abdellah University in Fez, Morocco;
- Major and minor element analysis was conducted using X-ray fluorescence. The equipment used was the Wavelength dispersion spectrometer - Type Axios of the National Center for Scientific and Technical Research in Rabat, Morocco.

## 3. Results and discussion

### 3.1. Physical and hydrodynamic analysis

The table 1 summarizes the average values of physical and hydrodynamic parameters

**Table 1:** Physical and hydrodynamic parameters of the pyrrhotite ash

Settings	H%	L.O.I %	pH	D
values	3.4	7.7	2.92	4.33

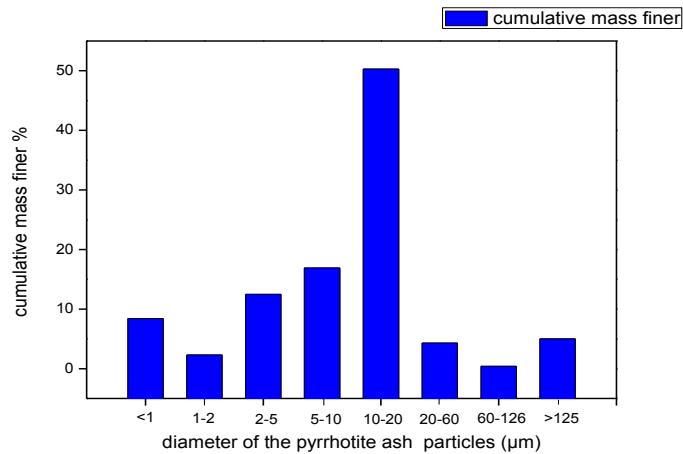
With: H: Moisture content; L.O.I: Loss On Ignition at  $1000^\circ\text{C}$ ; D: Density.

The results show that the pyrrhotite density is around 4.33. The waste is non-hygroscopic with a low loss on ignition that could be mainly caused by the presence of elements as carbon and sulfur which are not burned during the process leading to this waste [13].

There were no discernable trends regarding the variation of physical and hydrodynamic parameters with depth. The results presented below are relative to the sample retrieved from the depth of 0.5 m.

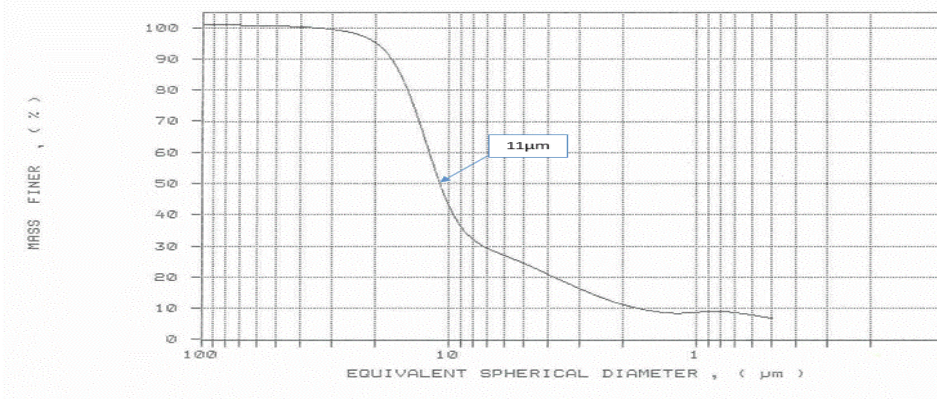
#### 3.1.1. Particle size distribution

The particle size distribution was determined for the dry ash using a vibrating sieve column. The results are shown in Figure 1. Particle size distribution of the pyrrhotite ash is continuous and contains fractions of grains from a diameter  $1\ \mu\text{m}$  to  $125\ \mu\text{m}$ . And it is known that the mixing of different fractions generally results in a compact material because the relatively small particles can be accommodated in the interstices between the larger ones. So, the material fills the volume more compactly [14].



**Figure 1:** Histogram of grain size distribution of the pyrrhotite ash

A fraction of pyrrhotite ash which has a diameter less than 60 μm was analyzed by SediGraph 5100 instrument (this instrument analyses only samples that have particles less than 100 μm). The results show that the median diameter ( $D_{0.5}$ ) is 11 μm.



**Figure 2:** Cumulative particle size distribution of pyrrhotite ash (fraction less than 60 μm)

### 3.1.2. Solubility

The solubility rate of the pyrrhotite ash in various acids has been determined by means of the following relationship:

$$\frac{(w_i - w_f) * 100}{w_i}$$

$w_i$ : is the initial weight of the pyrrhotite ash

$w_f$ : is the final weight of the pyrrhotite ash

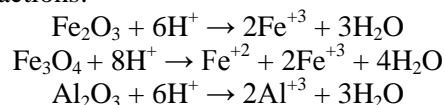
The obtained results are given in Table 2. The ash is more soluble in the hydrochloric acid than the other acids.

**Table 2:** Solubility rate of the pyrrhotite ash in various acids of 2M concentration

Acid	HNO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	H <sub>3</sub> PO <sub>4</sub>	HCl
<b>Solubility rate of the ash</b>	40%	54%	60%	64%

The 100% solubility rate was obtained for the concentration of 8M according to the following protocol: Mix 0.5 g of the ash with 100 ml of HCl (8M) and then make the mixture in a refluxing heater at 120°C for 22 hours with a magnetic stirring model “C-MAG HS 7”.

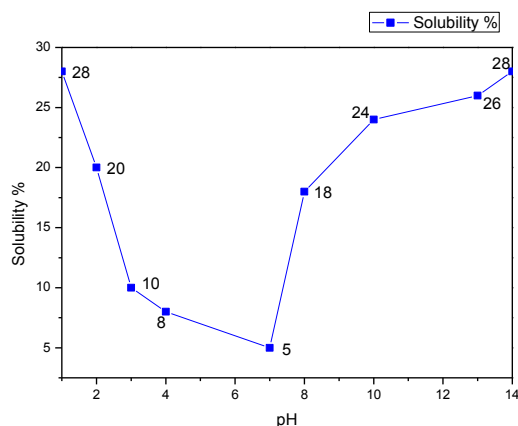
This result is in accordance with the previous work [15] related to the study of the fly ash leachability under acidic conditions for the extraction of metal elements. It has been reported that when the fly ash -that is rich in aluminum and iron oxides - contacts with hydrochloric acid, most of the metals such as Fe and Al can leach into the solution through the following reactions:



### 3.1.3. Solubility rate of the pyrrhotite ash versus pH

0.5 g of ash was mixed with 100 ml of solvent (HCl for acid pH, NaOH for basic pH) at various concentrations. For the neutral pH, distilled water was used as solvent. Hydrochloric acid was chosen since the pyrrhotite ash had a high solubilization rate (64%) as stated in the table 2.

The mixtures were heated at reflux for 3 hours and then filtered. The collected solid is weighed after drying.



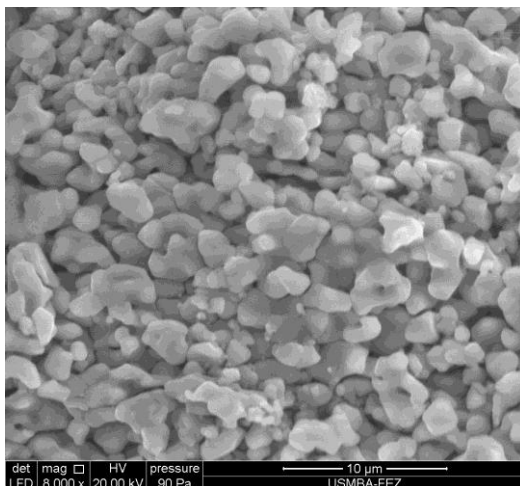
**Figure3:** Solubility rate vs the pH

It is noted that the solubility rate of the pyrrhotite ash increases with increasing of acidity or basicity of the solution. The lowest solubility rate of pyrrhotite ash is obtained at neutral pH.

## 3.2. Chemical, mineralogical and morphological analysis

### 3.2.1. Scanning electron microscopy (SEM)

The morphology of the ash as observed from SEM measurements is shown in Figure 4. The particle shape is reported to be spherical with some of the particles seen to be tightly attached to each other, forming agglomerates. This morphological analysis highlights also the sintering effect between grains and confirms the variation in particle size of the spheres, as observed by particle size determination.



**Figure 4:** SEM micrograph of pyrrhotite ash



Figure 4 shows also that the spheres have a relatively smooth surface and it is obvious that particles having a spherical shape with a smooth surface don't retain water. This could confer to the pyrrhotite ash an anhygroscopic character as indicated above in the section 3.1.

A similar morphology was seen for the fly ash for which it has been reported in literature that the spherical shape of its particles leads to a positive effect on the workability and cohesiveness when they are used as cements replacement material in concretes and mortars [16, 17].

### 3.2.2. X-Ray Fluorescence spectrometry (XRF)

The chemical composition of the pyrrhotite ash provided by fluorescence X is given in Table 3. It is shown that iron oxide (hematite  $\text{Fe}_2\text{O}_3$ ) and silicon dioxide (quartz  $\text{SiO}_2$ ) are the major constituents of the pyrrhotite ash with concentrations of 64.9% and 13% respectively. Minor proportions of  $\text{Al}_2\text{O}_3$ ,  $\text{SO}_3$ ,  $\text{MgO}$ ,  $\text{P}_2\text{O}_5$  and  $\text{Cr}_2\text{O}_3$  are also present with total concentration of 15.63%. Traces of  $\text{CaO}$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{MnO}_2$ ,  $\text{NiO}$ ,  $\text{TiO}_2$ ,  $\text{CoO}$ ,  $\text{ZnO}$  are also present.

**Table 3:** Chemical analysis data for pyrrhotite ash.

Oxides	$\text{Fe}_2\text{O}_3$	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{SO}_3$	$\text{MgO}$	$\text{P}_2\text{O}_5$	$\text{Cr}_2\text{O}_3$	$\text{Na}_2\text{O}$	$\text{NiO}$	$\text{CaO}$	$\text{MnO}_2$	$\text{K}_2\text{O}$	$\text{TiO}_2$	$\text{CoO}$	$\text{ZnO}$	$\text{MoO}_3$
Wt %	64.9	13	4.01	3.96	2.73	2.7	2.23	0.662	0.626	0.526	0.336	0.139	0.111	0.0764	0,0402	0,0274
Elements	Fe	O	Si	Al	Mg	S	Cr	P	C	Ni	Na	Ca	Mn	K	Cl	Ti
Wt %	45.4	37.2	6.1	2.12	1.65	1.59	1.52	1.18	0.917	0.492	0.491	0.376	0.212	0.115	0.111	0.0668

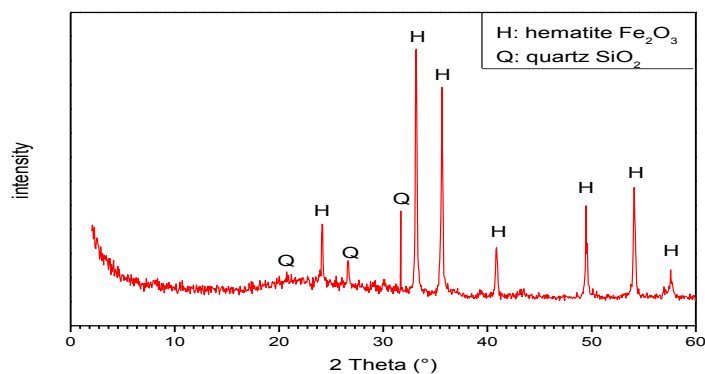
The obtained results confirm the acidity of pyrrhotite ash as the  $\text{Ca} / \text{S}$  ratio =  $0.376 / 1.59 = 0.24 < 2.5$  [18].

Generally, the reuse of ash depends mainly on its chemical composition. It has been reported in literature [16, 19] that fly ash containing more than 70% of  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  with a low content of calcium exhibits pozzolanic properties but possesses little or no self hardening property. Thus, it is expected that pyrrhotite ash which contains 81.91% of  $\text{Fe}_2\text{O}_3 + \text{SiO}_2 + \text{Al}_2\text{O}_3$  and a low percentage of calcium could exhibit a pozzolanic property and this explains the use of the pyrrhotite ash by some cement industries [20]. It is also noticed that pyrrhotite ash contains a very high percentage of  $\text{Fe}_2\text{O}_3$  (64.9%) than the fly ash [2] which could improve mechanical properties of construction materials [21].

On the other hand, the XRF analysis reveals the presence of  $\text{SO}_3$  with concentration of 3.96% (equivalent to 1.59% S) in the pyrrhotite ash. This indicates that, despite its iron rich composition, the pyrrhotite ash can't be reused in the metallurgical industry [22]. It is well known that sulfur is a weakening element as soon as its content exceeds 0.08% [23].

### 3.2.3. X-Ray Diffraction (XRD)

Figure 5 illustrates the X-ray diffraction pattern of the pyrrhotite ash.



**Figure 5:** Diffractogram X of the pyrrhotite ash.

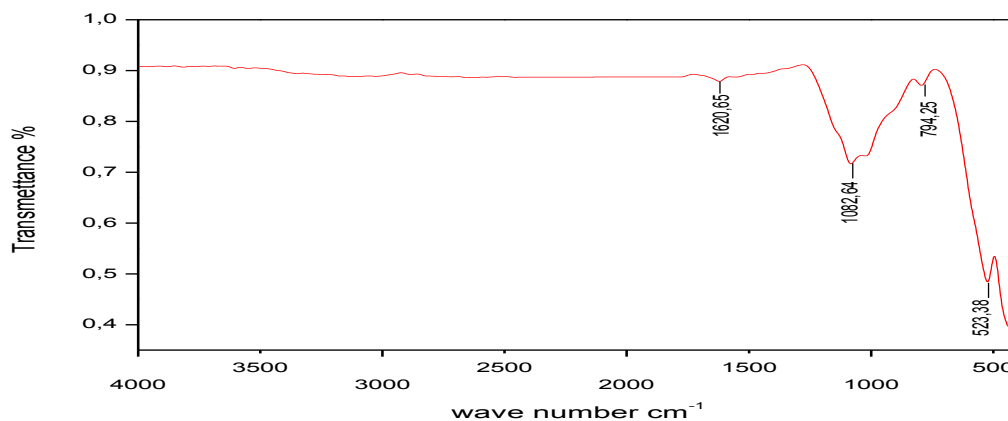
The X-ray diffraction diagram shows the presence of the following peaks:

$2\theta = 24,3^\circ ; 33,2^\circ ; 35,6^\circ ; 41^\circ ; 49,5^\circ ; 54^\circ ; 57,5^\circ$  characteristic peaks of the hematite phase ( $\text{Fe}_2\text{O}_3$ ) [24];

2 theta = 21°; 26,5°; 31,9° characteristic peaks of the quartz (SiO<sub>2</sub>) phase [25]. The main diffraction peaks are assigned to hematite (Fe<sub>2</sub>O<sub>3</sub>) and quartz phases (SiO<sub>2</sub>). Aluminum mineralogical phase is not observed in the diffractogram as the content of Al<sub>2</sub>O<sub>3</sub> is approximately 4% as shown by X-ray fluorescence (Table 3). Thus, hematite is denoted as the main crystalline phase along with minor content of quartz confirming the results obtained by X-ray fluorescence.

### 3.2.4. Fourier Transform Infrared Spectrometry (FTIR)

The infrared absorption spectrum obtained for the pyrrhotite ash is illustrated in Figure 6.



**Figure 6:** Fourier Transform Infrared spectrum of the pyrrhotite ash

The main absorption bands found on the spectrum are given in Table 4. Bands characterizing oxides of metals lies between 400-1500 cm<sup>-1</sup> [26]. It is noted a very small band related to traces of water molecules that confirms the character non hygroscopic of the ash as stated above.

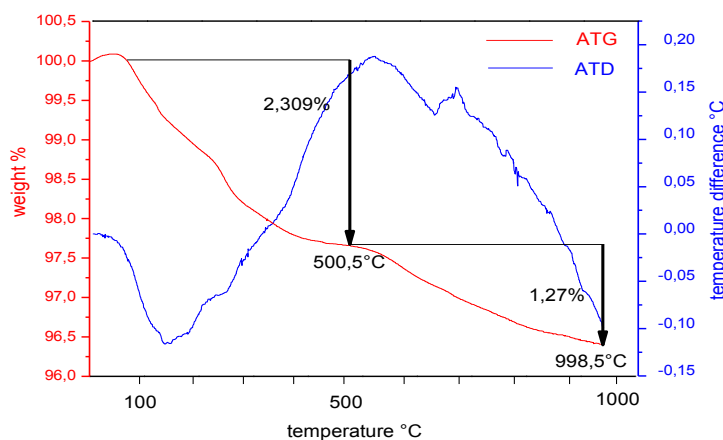
**Table 4:** Main absorption bands of pyrrhotite ash

Frequency in cm <sup>-1</sup>	Attribution
1620,65	Deformation of water molecules OH-H
1082,64	Bond elongation $\nu_{\text{Si-O}}$
794,25	Link elongation vibration $\nu_{\text{Si-O}}$
523,38	Bond elongation $\nu_{\text{Fe-O}}$

The results obtained in the analysis of infrared spectrometry are compatible with those presented by X-ray diffraction and X-ray fluorescence.

### 3.2.5 Thermogravimetric and Differential thermal analysis (TGA/DTA)

The TGA / DTA curves of pyrrhotite ash are shown in Figure 7.



**Figure 7:** TGA/DTA analysis of the pyrrhotite ash

The TGA spectrum shows a weight loss of 3%. This is associated initially with water loss. The broad band between 300–1000 °C is related to the elimination of SO<sub>3</sub>/SO<sub>2</sub>. There is no thermic effect in the analysis. The pyrrhotite ash is mainly composed by hematite Fe<sub>2</sub>O<sub>3</sub>, small amount of quartz SiO<sub>2</sub> and traces of other oxides. The hematite Fe<sub>2</sub>O<sub>3</sub> is inert to the thermic analysis and no thermic effects are expected in the range of temperatures less than 1000°C.

### 3.3. Discussion

Pyrrhotite ash is a solid waste containing a very high percentage of hematite (64.9% of Fe<sub>2</sub>O<sub>3</sub>), small amount of quartz (16% of SiO<sub>2</sub>) and traces of other oxides. It contains 81.91% of Fe<sub>2</sub>O<sub>3</sub>+ SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> and 0.526% of CaO. This chemical composition could confer to the pyrrhotite ash an interesting pozzolanic property which is appreciated in the field of construction materials. Further, pyrrhotite ash physical properties, especially the size and the spherical shape of particles could play an important role on the reuse of this waste. As shown in the case of fly ash, the spherical shape increases the workability and consistency of mortars and concretes [27].

Moreover, the small size of the pyrrhotite ash particles- almost three quarters of particles have size less than 20 µm- will allow the reuse of the waste without providing much effort for grinding. Also, it could allow the reuse of the waste to produce materials with high density for application in the building industry, road construction and heat accumulation materials, as it was shown for the pyrite ash and red mud which are also hematite rich wastes [3, 4, 6].

Furthermore, the stability of the pyrrhotite ash at neutral pH suggests studying the possibility of its use in the treatment of wastewater. On the other hand, its thermal stability will make possible to broaden the field of pyrrhotite ash application to high temperatures.

As it is stated above, the presence of sulfur (3.96% of SO<sub>3</sub>) doesn't allow the reuse of the pyrrhotite ash in metallurgical industry, but it could be a good reason to carry out research works in order to reuse it in agriculture.

Moreover, the pyrrhotite ash is a rich hematite waste that could present interesting magnetic properties. Also, it could be recovered in fields based on iron compounds (iron oxide, iron salts, iron complexes, etc.), since such compounds have a wide spectrum of use in the fields of dyes, waste water treatment, agro-food industry, etc.

Finally, the pyrrhotite ash is an acid waste and it is known from literature review that the maximum amount of heavy metals gets released under acidic environment [28]. And therefore, the study of the leaching of this waste is crucial for predicting environmental risks.

### Conclusion

In the present work, the characterization of the pyrrhotite ash which is an industrial waste from south west of Morocco has been investigated. The obtained results reveal that the pyrrhotite ash is an iron rich waste that is acid, non-hygroscopic and completely soluble in hydrochloric acid (8M). Moreover, this waste is stable at neutral pH and at High temperature. Its chemical composition and morphological and physical properties are promising in terms of the reuse of this waste- as whole or partial replacement- in construction materials, highways construction, production of dense materials for heat accumulation, agriculture, wastewater treatment. In order to better exploit this waste, it is suitable to carry out some research works on the mechanical, thermal, magnetic and electrical properties of the pyrrhotite ash.

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