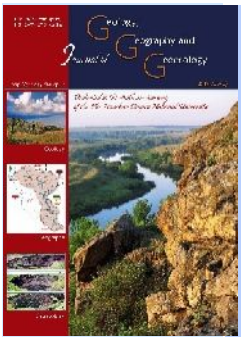

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Mineralogical characterization of limonitic iron ore from the Rouina mine, Ain Defla (Algeria).

Ali Messai[1], Abdelaziz Idres[1], Aissa Benselhoub[2]

[1]*Laboratory°Mineral Resources Valorization and Environment, Badji Mokhtar University, Annaba, Algeria, E-mail: benselhoub@yahoo.fr*
[2]*Laboratory of Valorization of Mining Resources and Environment,Badji Mokhtar University, Annaba, Algeria*

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Abstract. The Rouina mine is one of the oldest operated mines of iron ore in Algeria, its product is used like an adjuvant in the cement industry because the extracted raw material is considered as a low-grade ore. The present paper investigates on the one hand its mineralogical composition with the aim of understanding the morphology, texture, phase identification and iron properties**;** and on the other hand studying the influence of washing on its quality. For characterization, X-Ray Diffractions (XRD) of collected samples, analysis of thin sections with scanning electron microscope (SEM), and a sieve analysis followed by washing of each size fraction using a sieve mesh 0.074 µm were adopted. The obtained results revealed that the raw material of the Rouina mine is clayey low-grade iron ore and it is possible to obtain a pre-concentrate through the washing method. This article suggests in addition conducting deep studies of Rouina iron ore with physico-chemical characterization in order to confirm the prior results (mineralogical characterization) and then to permit a suitable enrichment method to be applied with the aim of obtaining a high-grade iron ore acceptable for the metallurgical industry.

Keywords: the Rouinamine, iron ore, cement, mineralogical, washing, enrichment.

Мінералогічна характеристика лімонітової залізної руди з шахти Руйна, Айн-Дефла (Алжир).

Алі Месса, Абделазіз Ідре, Айса Бенсельхуб

[1]*Лабораторія санітарії та навколишнього середовища мінеральних ресурсів, Університет Бадзі Мохтар, Аннаба, Алжир,E-mail: benselhoub@yahoo.fr*
[2]*Лабораторія орієнтації гірничих ресурсів та навколишнього середовища, Університет Бадзі Мохтар, Аннаба, Алжир*

Анотація.Шахта Роуїна - одна з найстаріших експлуатованих шахт залізної руди в Алжирі, її продукція використовується як допоміжний компонент в цементній промисловості, оскільки вилучена сировина вважається низькосортною рудою. Дана робота досліджує з обного боку мінералогічний склад сировини з метою розуміння морфології, текстури, фазової ідентифікації та властивостей заліза; а з іншого боку - вивчення впливу промивання руди на її якість. Для характеристики рентгенівських дифракцій (XRD) зібраних зразків, аналізувалися тонкізрізи із скануванням електронним мікроскопом (SEM) та проводився ситовий аналіз з подальшою промиванням кожної розмірної фракції з використаннямсита 0,074 мкм. Отримані результати показали, що сировина шахти Роуїна - глиниста низькосортна залізна руда, і вона придатна для отримання попереднього концентрату методом промивання. Це дослідження передбачає, крім того, проведення глибоких досліджень залізної руди Руїни з фізико-хімічною характеристикою для підтвердження попередніх результатів (мінералогічна характеристика) і дозволяє у подальшому застосувати відповідний метод збагачення з метою отримання повноцінної залізної руди, придатної для металургійної промисловості.

***Ключові слова**: шахта Руїна, залізна руда, цемент, мінералогічний, промивання, збагачення.*

Introduction. Problem setting. Iron is the main component of the steel industry, that why it plays a significant role in the evolution of the global economy (R.J. Holmes, L. Lu 2015). The growing demand for iron as a raw material coupled with the deterioration and exhaustion of high-grade iron ore deposits is a serious problem for the steel industry on a global scale (Matis, K. A et al 1993). The en-

rich-ment of low-grade iron ores, that are used like an adjuvant in the cement and ceramic industries, repre-sents an alternative solution for the future which would ensure the continued availability of raw materials (Li Ch et al 2010, Da Silva F.L et al 2014, Liu, S et al 2014, Osinubi, K.J et al 2015, Singh, S et al 2015).

Kesearch on valorisation is commonly related to the physicochemical and mineralogical composition of minerals and their liberation size (Rath, S. S et al 2016) where low-grade iron ores are capable of being enriched by primary mechanical preparation (crushing and grinding), magnetic, gravimetric separation and the flotation method, (A. Jankovic 2015, D. Xiong, L et al 2015)

Several deposits are located in the North-West of Algeria at Rouina, Zaccar and Beni Saf (Popov 1976). These deposits were identified as metasomatic carbonate replacement deposits that were formed through the process of epigenetic replacement of limestone by siderite followed by supergene enrichment by hematite. (Chaa, H., & Boutaleb, A. 2016)

The Rouina mine is one of the oldest operated mines in Algeria and its production is designed for the cement industry because it is considered as low-grade iron ore that contains a high percentage of clay materials .However, most previous studies are not detailed enough to assess the possibility of its enrichment for obtaining a high tenor concentrate for use in other field industries such as the steel industry and, the pigments manufacture.

Because of the lack of real mineralogical ore characterisation , this paper presents the X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) results. The (XRD) and (SEM) were used on collected samples and thin sections; the quality and the quantity of minerals contained in Rouina deposit were investigated .Then, a washing test of different particle sizes was carried out, which permitted us to estimate the liberation where clay materials were removed from useful minerals.

Study area description. *Geographic situation.*TheRouina iron ore deposit is situated in the town of Rouina, the state of Ain Defla in the north west of Algeria. The national road N° 04 linking Algiers with Oran passes 3 kilometres from the deposit; the geographic situation is illustrated in Figure 1.

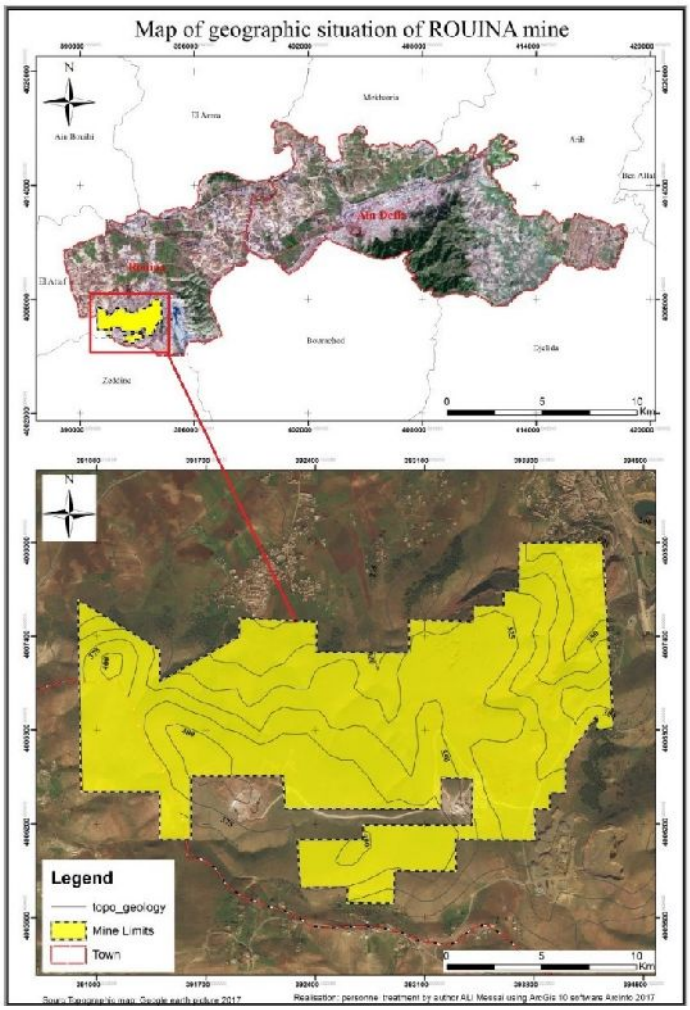


Fig.1. Geographic situation of Rouina mine –Ain Defla

Local geology. The Rouina iron deposit is a part of the Rouina massif. This massif originated after the Alpine orogeny at the borders of the mega geosyncline, in the form of a directional anticlinal 30°- 40° NE emerging in the middle of the alluvial deposits of the Chlef Valley. Figure 2 illustrates the re-gional geology and the location of the Rouina deposit.

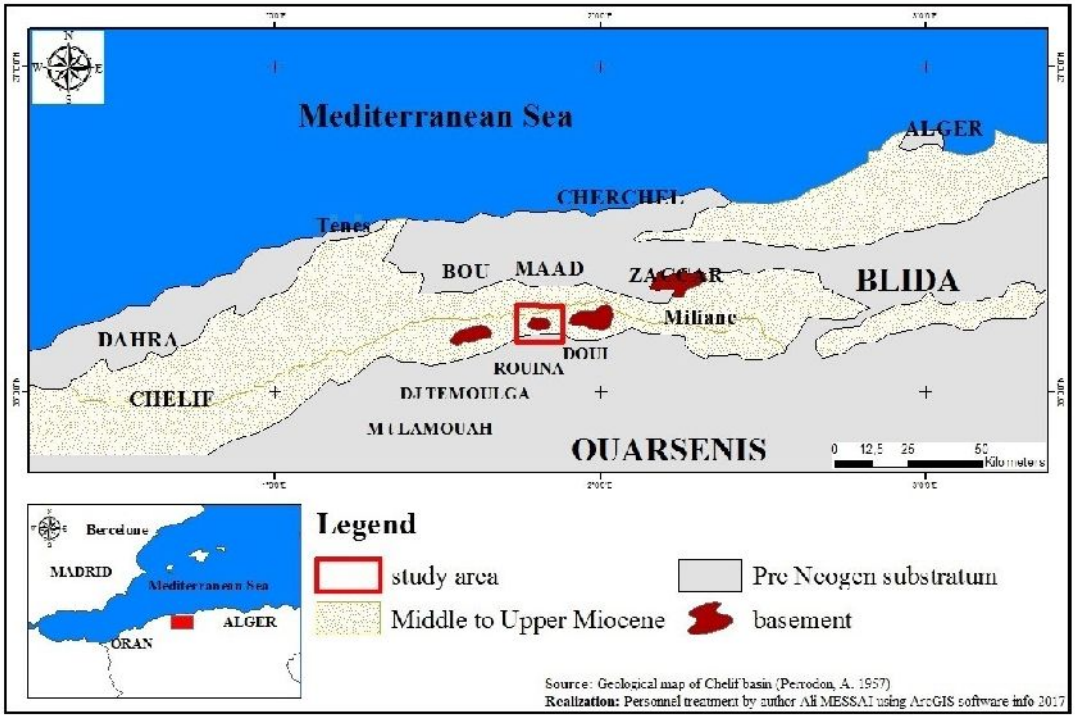


Fig.2. Simplified geological map of the Chelif basin (Personal treatment by author Ali MESSAI) (Perrodon, A. 1957)

The flanks of this anticline are composed of secondary carbonate soils with dips growing from the heart to the exterior including Paleozoic formations.

For the Rouina massif, it consists mainly of:

- The schist sandstone and conglomerates series of the Paleozoic;
- The carbonate benches (limestone and shale) of the Jurassic, which presents the majority of outcrops in Rouina;
- The marls outcrop of Lower Cretaceous;
- The basic conglomerate that marks the contact between the base and the cover.

We also note the absence of the Triassic and Tertiary and the Quaternary soils. Thus, the iron mineralization of Rouina appeared in the Triassic and Jurassic periods (Middle Lias) and it was formed before at least 245 million years ago. (RAACH Khadidja. 2010)

There are two major litho-stratigraphic formations; Jurassic and Cretaceous.

Jurassic. The Jurassic represents the majority of outcrops in Rouina. As everywhere in the western of Algeria, the Jurassic constitute of massive carbonate banks.

a- Lias : It is discordant on the Paleozoic basement in favour of a thin layer (few meters), including pale grey and purplish shale elements, it testifies the passage of Paleozoic schist to Jurassic limestone; its age is not precise. The basic conglomerate is followed by a rather thick layer of greyish limestone attributed to the Lower Lias.

Reddish limestone in the higher levels shows microscopically fine calcite ranges including digenetic quartz grains, coarser limestone, sometimes pigmented iron, crossed by fractures filled with oxides and hydroxides of iron. This formation is of Middle Lias age.

b- Dogger: A compact formation of bluish-grey massive limestone rich in flint nodules surmounts the Middle Lias; its strength is about 50 m. The microscopic study done by (Kireche, O. 1993) reveals the presence of jaw debris and microfilaments, found in the Dogger faces of the Tellian regions, which allowed it to be assigned a Dogger age.

c- Marl: A limestone and marl-limestone series in small banks, located above the Dogger series

d- *Cretaceous.* On the west of the Rouina valley, a narrow outcrop of green gray marl above the Jurassic limestone is recognized as Upper Cretaceous.

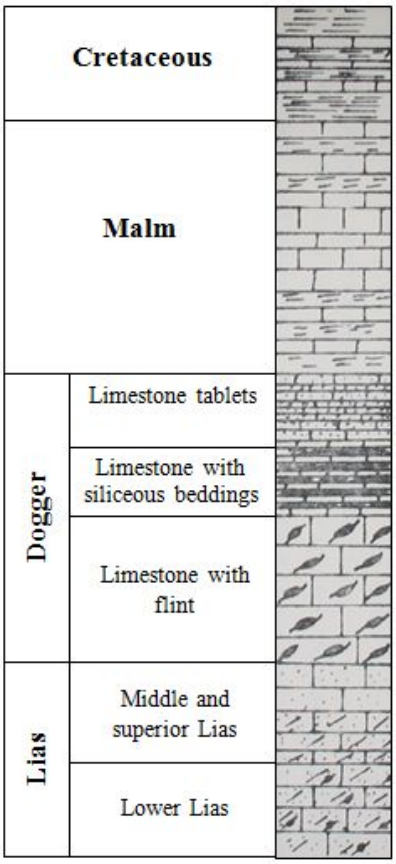


Fig.3. Stratigraphic column of Rouina (personal treatment by author Ali MESSAI) (Kireche, O. 1977)

Methods and Materials. The first task mineral characterisation

Sampling. The sample weighing 120 kg with the maximum diameter of lumps about 120 mm was selected from the open pit mining. The protocol of sampling was realized to prepare samples intended for definition of physico-chemical and mineralogical characteristics.

Figure 4 presents a geological map of the Rouina massif treated using the Geographical Information System (ArcGIS 10) 2017 software to illustrate the geology of the "BUTTE" deposit where the samples were collected.

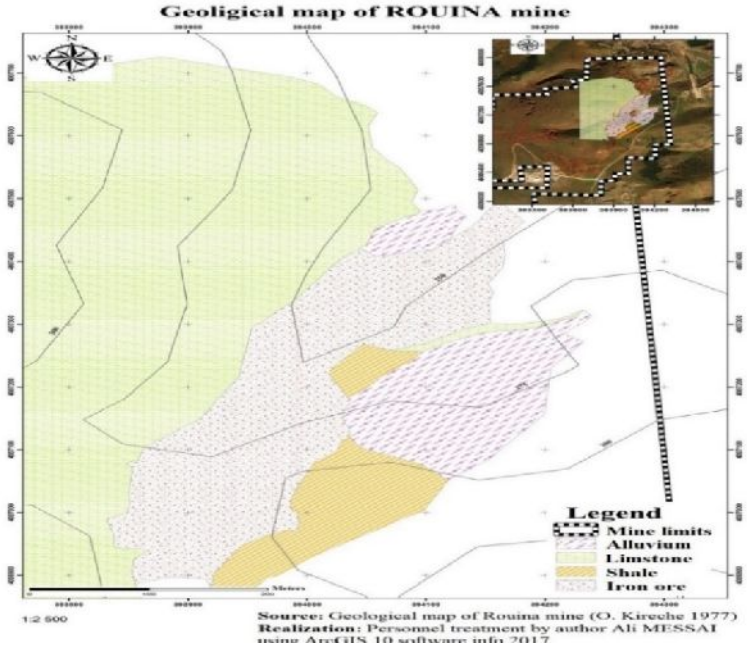


Fig.4: Geological map of Rouina massif (personal treatment by author Ali MESSAI) (Kirreche, O. 1977)

*Mineralogical characterization.*This task permits us to identify and quantify minerals contained in the material studied.

In this step, two different techniques are applied. X-Ray Diffractions (XRD) using PAN analytical Diffractometer : XPERT-PRO, equipped with Copper Anticathode Ceramic X-ray Tube, The current and voltage were 40 mA, 45 Kv respectively and on the other hand by observation of thin section using Scanning Electron Microscope (SEM) type SEM7001F.

Size analysis. This was conducted on quantity of 600 grams of dried raw material primarily crushed to 5 mm, a shaking machine, type RETSCH and sieves series assembly of: 2, 1, 0.5, 0.25, 0.125 and 0.063 mm were used. each sample is sieved for 30 minutes with magnitude of 60 mm/g. The refusal mass of each sieve is weighed using a scale with an accuracy of 0.01g.

Washing. The refusing masses prepared with size analysis washed using a sieve with an aperture of 0.074 mm (Figure 5), all of washed fractions were viewed under a petrographic microscope and the liberation sizes chosen are analysed by XRD (X-Ray Diffraction).

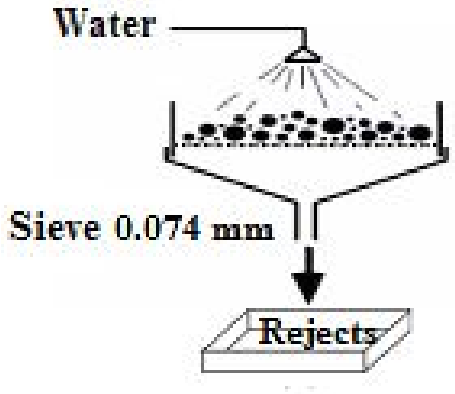


Fig.5. Washing of different fractions

Results and Discussion

X-Ray diffraction

Figure 6 represents the diffractogram obtained by the XRD, and demonstrates the presence of iron oxides (hematite Fe_2O_3 and goethite FeO(OH)) and quartz (SiO_2) as major mineral phases. It also proves the presence of illite (clay mineral) K ($Al_4Si_2O_9$ $(OH)_3$) besides calcite $CaCO_3$

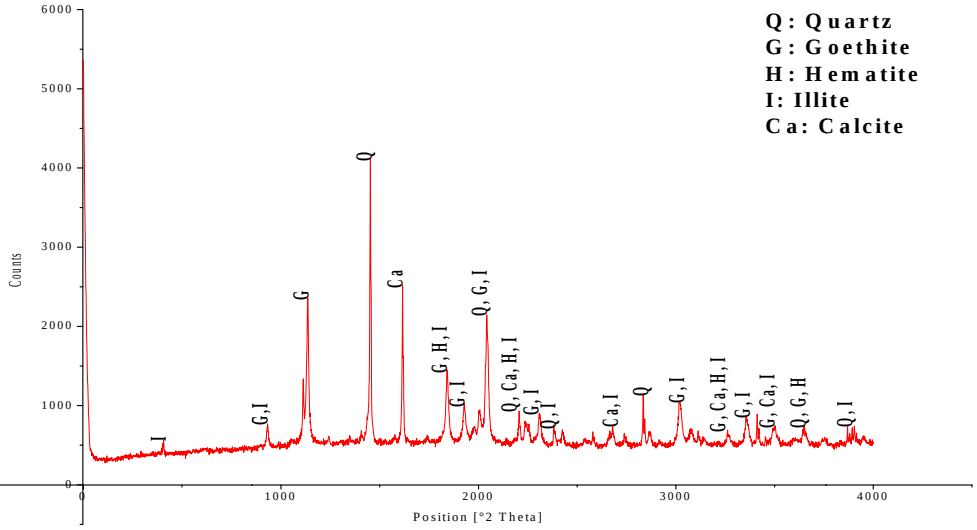


Fig.6. Diffractogram of raw material

Scanning Electron Microscope (SEM).

Observation of thin sections. Three series of observations were performed on the thin sections and the results are illustrated in Figures 7 (a, b and c). We noticed the presence of quartz (greyish black) like a dominant element related to iron oxides, that has a white colour (Figure 7.a). , we also noted the presence of quartz bathed in a mass of goethite pre-sents in hilly forms (Figure b) to erased structure of finely fibrous aggregates (Figure c).

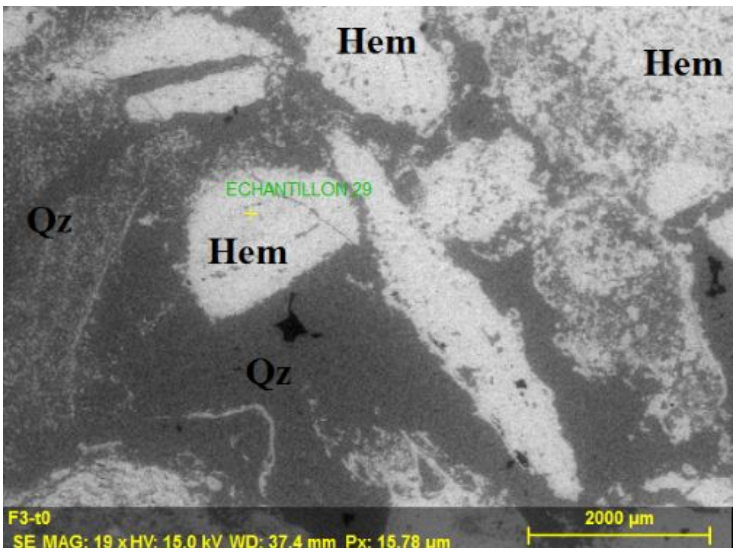


Fig.7a. Thin section 1 under SEM; Quartz associated with hematite (Hem: hematite, Qz: quartz) Whitney, D. L., & Evans, B. W. (2010).

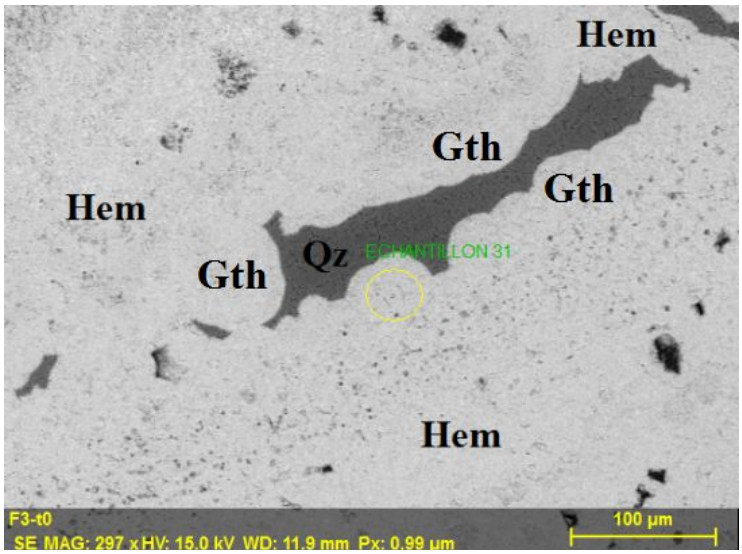


Fig.7b. Thin section 2 under SEM; quartz bathed in a hematite cluster and goethite, (Hem: hematite, Gth: Goethite and Qz: quartz) Whitney, D. L., & Evans, B. W. (2010).

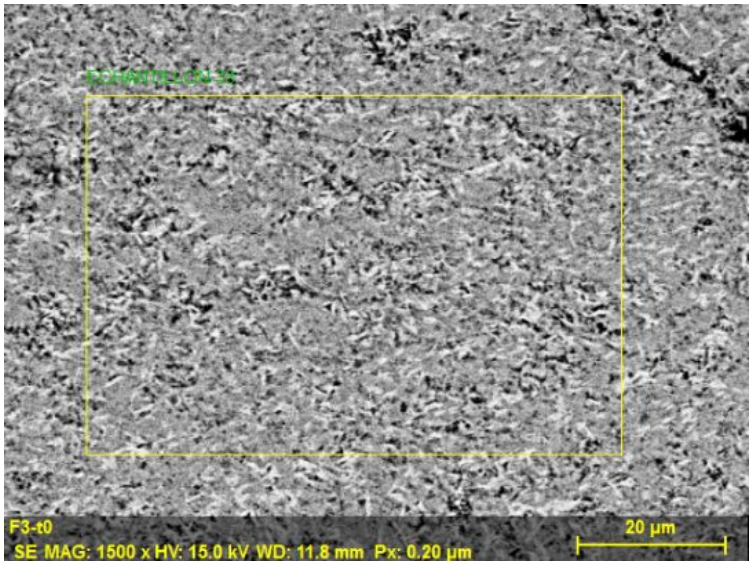


Fig.7c. Thin section 3 under SEM; Mass of iron oxides and hydroxides interacting with quartz

Particles Observation of particles. In order to confirm the results obtained previously of raw material samples and the thin sections, some particles were chosen in order to observe them with the SEM. The results are illustrated in Figure 8 (A_1, B_1, A_2, B_2, A_3, B_3, A_4, B_4, A_5, B_5), where hematite and goethite (white colour), quartz (black greyish colour), traces of clays and besides calcite (black colour) are observed, which is in good agreement with DRX results and results of analysis of this section.

A_1) B_1)

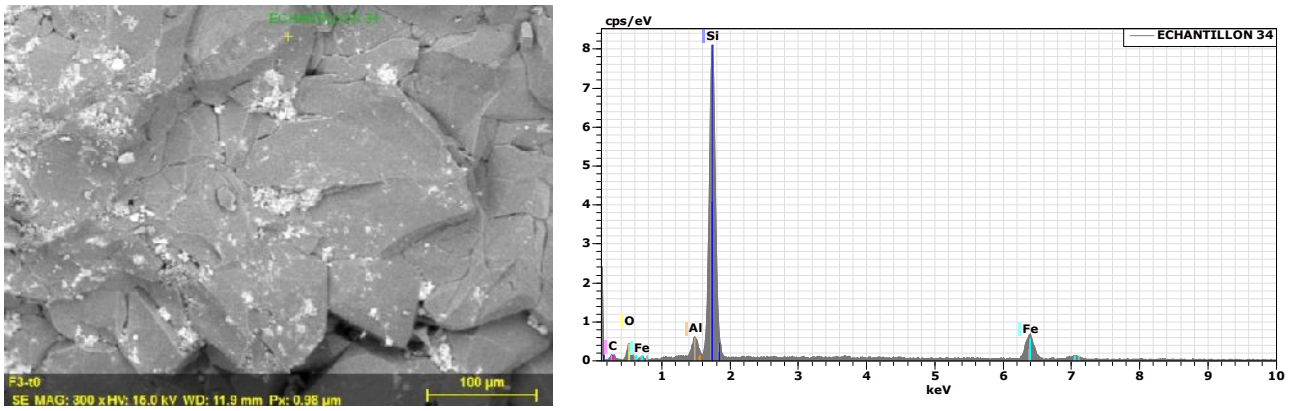


Fig.A_1. 1st particle under SEM; scanned point shows quartz as a dominant mineral

A_2) B_2)

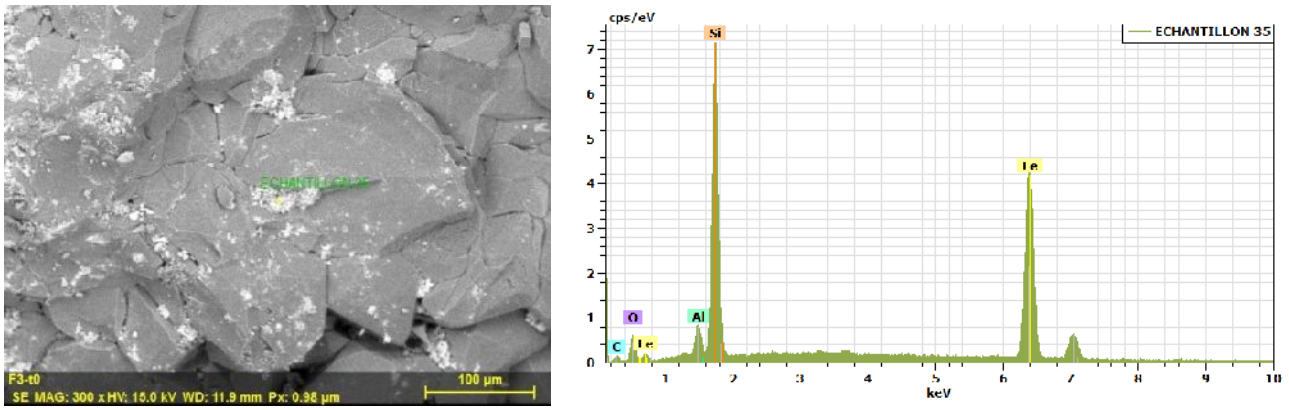


Fig.A_2. 1st particle under SEM; scanned point shows trace of hematite contained on quartz

A_3) B_3)

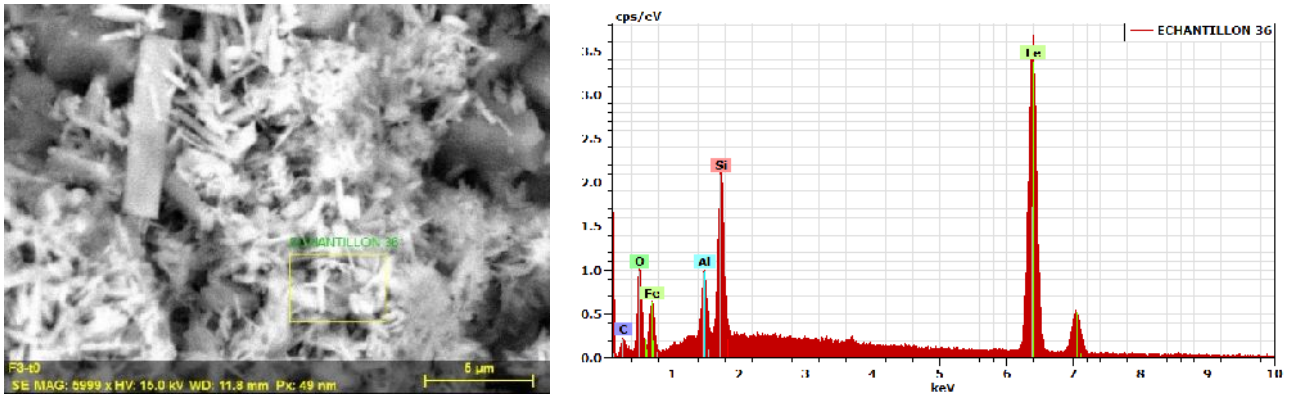
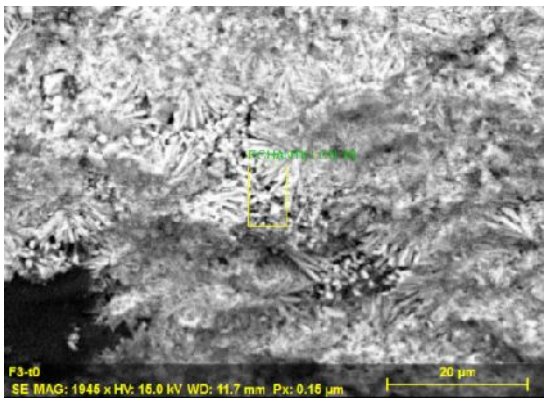


Figure.A_3. 2nd particle under SEM; scanned point shows fibrous goethite mass with quartz and clay material traces

A_4) B_4)



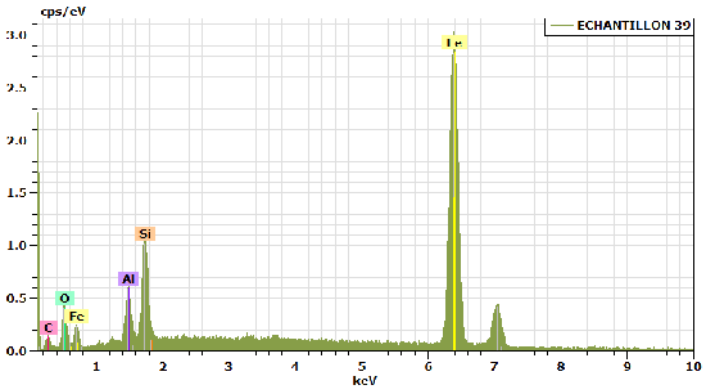
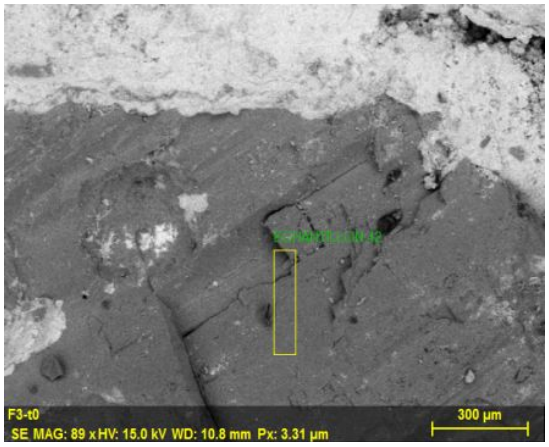


Fig.A_4. 3rd particle under SEM; scanned point shows quartz bathed in a hematite and goethite fibrous mass with presence of clay material traces

A_5) B_5)



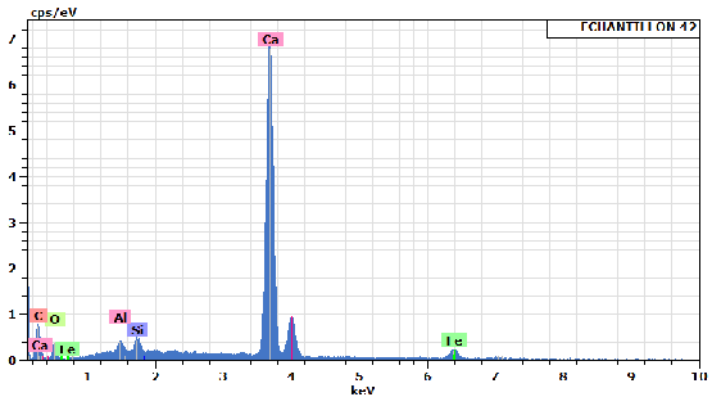


Fig.A_5. 4th particle under SEM; scanned point shows a calcite mass associated with hematite, quartz and clay material traces.

*Size analysis.*The results of the sieve analysis shown in Table 1 and Figure 9 show that the majority of the mass appears in the larger fractions [+2, +1, +0.5 and +0.25 mm] by 72.54% (486.47 grams), which confirms the iron ore hardness. The rest of the products appear in the finer fractions [+0.125, +0.063 and -0.063 mm].

Table.1. Results of particle size analysis of Rouina iron ore crushed to 5 mm

Size classes (mm)		Weight (g)	Yields (%)		
			Partial ∑γ	Passing cumulative ∑ γ↗	Refusing cumulative ∑ γ↙
-4	+2	281.77	46.96	100	0
-2	+1	89.42	14.91	53.04	46.96
-1	+0.5	64.03	10.67	38.13	61.87
-0.5	+0.25	51.25	8.54	27.46	72.54
-0.25	+0.125	50.72	8.45	18.92	81.08
-0.125	+0.063	41.44	6.91	10.47	89.53
-0.063	+0	21.37	3.56	3.56	96.44
TOTAL		600	100	/	/

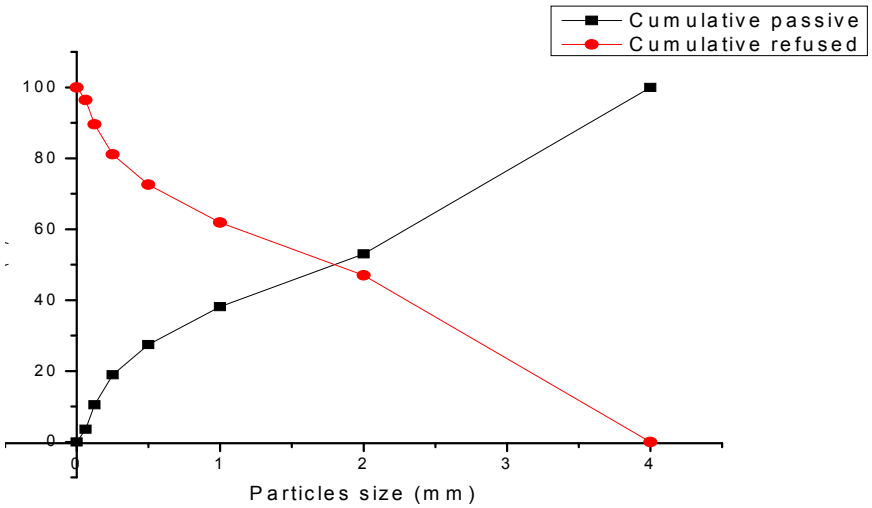


Fig.9. Graphical representation of particles size analysis results

Washed size classes.

XRD Analysis. The results shown in Figures 10 (a, b and c) prove that the washed size classes constitute essentially of iron oxides and calcite as a major component. However, few traces of quartz and illite are noted, confirming the effectiveness of washing in the reduction of the proportion of clay.

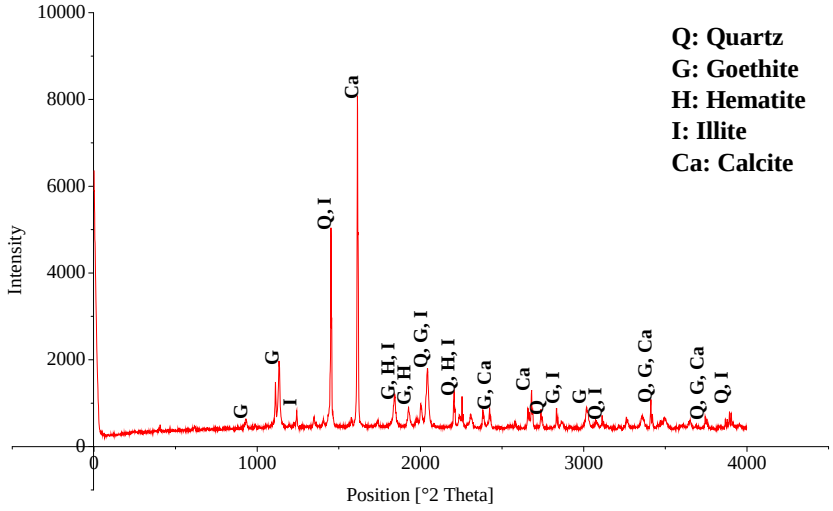


Fig.10.a: Diffractogram of size class [-1 +0.5 mm]

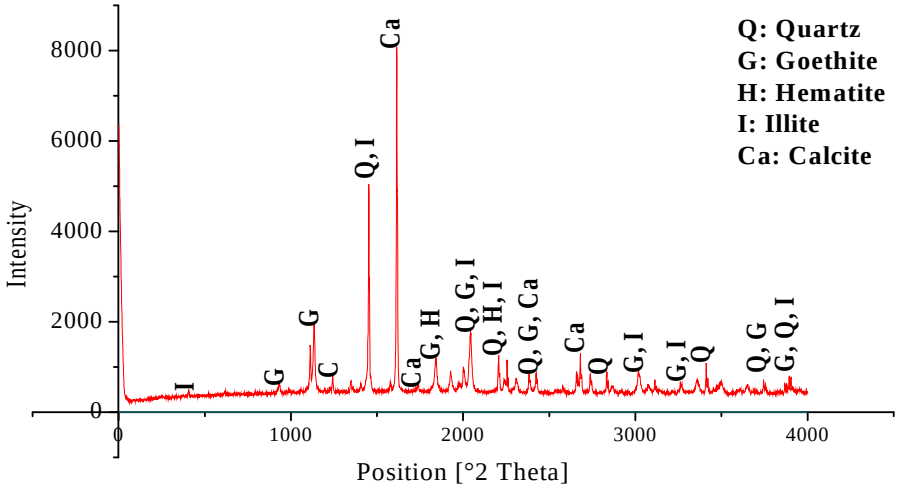


Fig.10.b. Diffractogram of size class [-0.5 +0.25 mm]

*XF Analysis.*The chemical analysis results of different size classes before and after washing are shown in Table 2. It is noted that the proportion of clay decreased after washing for the fraction -1 + 0.5 mm, it is also noted that the iron content was 51.03% against 44.18%, in the unwashed raw ore . Similarly, the alumina content decreased from 7.87% to 1.45%. The findings presented in Table 2 confirm the effectiveness of the washing process.

Table.2. FX analysis of the products before and after washing

Fraction (mm)	Process	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO
-1 +0.5	before washing	44.18	23.13	7.87	6.53	1.13
	After washing	51.03	24.20	1.45	7.57	0.40
-0.5 +0.25	before washing	43.78	22.26	6.96	6.22	1.06
	After washing	46.62	24.19	1.78	8.22	0.47
-0.25 +0.125	before washing	46.44	18.09	8.48	3.73	1.91
	After washing	41.99	30.81	1.96	7.69	0.59
-1 +0.125	before washing	45.44	22.06	7.53	5.08	1.45
	After washing	46.12	26.25	1.64	7.62	0.50

*Sludge XF Analysis.*It isnoted that rejects from the washing operation contain a high content of clays against a low content of iron oxide, whichmakes it possible to be used in other fields such as the cement and ceramic industry.

Table.3. FX analysis of the rejects from the washing test

Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO
28.97	15.37	22.02	0.33	3.45

Proposed enrichment diagram. The suggested preparation and pre-treatment diagram of Rouina iron ore are presented in Figure 11; this proposed scheme allows one to obtain a pre-concentrate, which will be subsequently enriched. It permits also the recovery of water for reuse in the washing step. The rejects obtained (+1 mm and dried sludge) will be used in cement production.

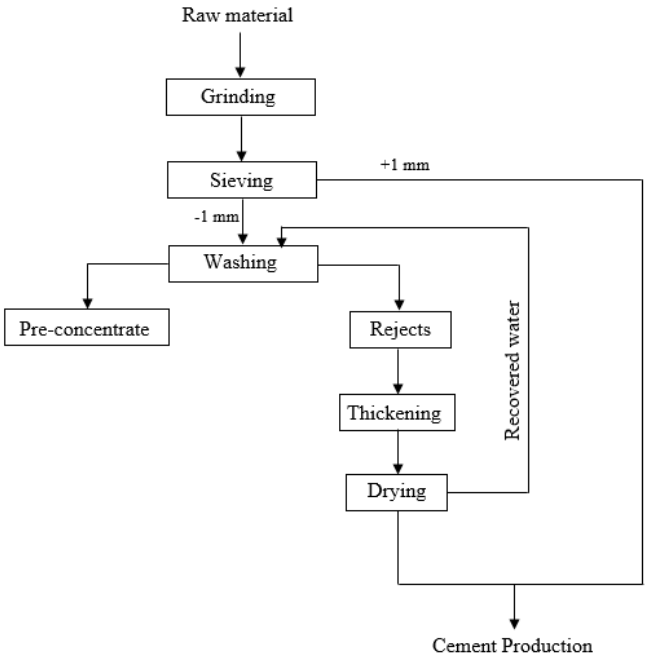


Fig.11. The proposed scheme of iron ore pre-treatment

Conclusions. The experimental results in the present study lead to the following conclusions:

1) The Rouina iron ore is classified as a low-grade clayey iron ore, which contains hema-

tite and goethite as useful minerals with quartz, calcite and clays as gangue minerals.

2) Application of washing as a preliminary enrichment method is effective for decreasing clay content and other associated gangue minerals (calcite and quartz) from the raw material, where the results obtained from the chemical analysis show a significant decrease in clay percentages after washing. It is also noted that the iron content is 51.03% against 44.18% in the raw material before washing. Similarly, the content of Al_2O_3 decreases from 7.87% to 1.45%, which confirms the significant results obtained by this preliminary enrichment (wet sieving).

3) On the one hand, the sludge residue from the washing process will be used as an adjuvant in the cement industry and on the other hand, the pre-concentrate will be enriched with the aim of recovering the maximum of useful minerals and obtaining a high-grade concentrate.

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