CHEMICAL ENGINEERING TRANSACTIONS
VOL. 32, 2013

A publication of
AIDIC
The Italian Association
of Chemical Engineering
Online at: www.aidic.it/cet

Chief Editors: Sauro Pierucci, Jiří J. Klemeš
Copyright © 2013, AIDIC Servizi S.r.l.,
ISBN 978-88-95608-23-5; **ISSN** 1974-9791

DOI: 10.3303/CET1332149

Characterization and Quartz Enrichment of the Hoggar Deposit Intended for the Electrometallurgy

Abdelkrim Kheloufi, Mohamed Fathi, Hamid Rahab, Aissa Kefaifi, Aissa Keffous, Sid Ali Medjahed

[a]Centre de Recherche en Technologie des Semi-conducteurs pour l'Energétique (CRTSE). 2, Bd Frantz Fanon BP140, Algiers-7 merveilles, 16027, Algeria
dr_kheloufi@yahoo.fr

The actual advances in mineral processing technologies aim mainly to increase the supply of newly mined metals and reducing the cost of enrichment process. In this context, the solar grade silicon (SoG-Si) as feedstock for photovoltaic cells production requires a high purity. Its cycle of production consists on: a silica enrichment, carbothermic reduction for obtaining metallurgical grade silicon (MG-Si) and purification of MG-Si to get a high solar grade silicon.

However, in order to achieve this goal preliminary silica enrichment, it is necessary to reduce a maximum of impurities before the carbothermic and purification processes. This process will allow a high quality of silica required (99 % of SiO_2) as raw material for solar silicon grade production, which makes the processes downstream cited more efficient with a high yield.

In the present work, we have studied the silica enrichment process using a magnetic separation technology in laboratory scale, which consists firstly in silica characterization by X-Ray Fluorescence of Hoggar quartz samples in order to locate the ferromagnetic impurities incrusted in the crystal lattice or on its surface, secondly to use magnetic separation process to increase the SiO_2 content and to optimize its technological parameters before using leaching technology.

1. Introduction

The current environment is very conducive to the development of renewable energy: fossil fuel prices high, increased awareness of environmental issues, etc. Support measures expected by the state for the development of this important activity will certainly overcome the handicap of competitiveness in the renewable energy field [Wineganer et al., 2007; Muller ., 2010].

The continuous development of technology segments for the manufacture of silicon is a direct consequence to the decrease in silicon prices [Wineganer et al., 2010].

Whereas the continuity of the energy sector is based on values of clean energy, Algeria and through the group Sonelgaz has just taken its first step towards the development of renewable energy, by the launch of a manufacturing solar panels project with a capacity of 120 MW. Algeria wants to develop the renewable and creating incubators through the combined efforts from network of laboratories, companies and agencies. The introduction of renewable in the energy mix of countries with a special place for solar, is among the major challenges the country is thus committed to the development of photovoltaic industry in Algeria. Until now, silicon remains the essential material in the manufacture of solar cells [Dielt., 1987; Désindes., 2004]. Its use as a raw material reached 90 % compared to other materials (where it represents 40-50 % of the cost of a solar cell) [Wineganer et al., 2010].

The silicon cycle production consists of: enriching the silica, carbothermic reduction to obtain metallurgical grade silicon and finally to purify the MG-Si for obtaining high solar grade silicon (SoG-Si) [Banza et al., 2006

Please cite this article as: Kheloufi A., Fathi M., Rahab H., Kefaifi A., Keffous A., Medjahed S.A., 2013, Characterization and quartz enrichment of the hoggar deposit intended for the electrometallurgy, Chemical Engineering Transactions, 32, 889-894 DOI: 10.3303/CET1332149

Baoqi S et al., 1995; [Braga et al., 2008]. In the Algerian context, and to secure its supply of raw material for this industry, the production of solar grade silicon remains unavoidable, given the issues underlying it. Therefore, it is imperative from now to predict the development of treatment processes and enrichment by metallurgical silica (an abundant natural resource) to prepare the basis for future national solar industry while securing its supplies of raw material. In this context, a particular interest has been given to the magnetic separation process as a method of optimal extraction ferromagnetic impurities from used silica as raw material for photovoltaic field before using a chemical leaching process [Kheloufi et al., 2009; Kheloufi et al., 2010].

The aim of this work is to characterize silica (quartz from Hoggar Algeria) by X-Ray Fluorescence in order firstly to locate the ferromagnetic impurities, secondly using magnetic separation process to increase the SiO2 content and to optimize the technological parameters before using leaching technology.

2. Materials and Methods

In our work, quartz sand samples from the Algerian quartz silica from Hoggar deposit are used. After characterization of the raw material by XRF fluorescence X, it was washed, cleaned, and filtered than dried in order to undergo a magnetic separation at the laboratory scale. The samples with SiO_2<98 % were characterized and treated. Mineralogical research showed that this sand is composed of: limonite, tourmaline, hematite and biotite and martite are the most commonly found impurities in the quartz of Hoggar. All these impurities with low and high magnetism can be sorted by magnetic separation.

Then we use the magnetic machine wet type SHP-500 to purify the quartz with feed Volume hopper: 25 L vibrating trough/ - Oscillation amplitude: 0.2 mm ... 1.5 mm; Oscillation frequency: 50 Hz or 100 Hz drum - Diameter: 208 mm - Magnetic field: 190° - Speeds: 10...150 Rpm; Max. particles: 40 mm. Moreover, and in order to define the internal structure and morphology and the probable of existence of inclusions as well as the existence of impurities in the crystalline lattice, the imagery sound Backscattered was used (Figure 1).

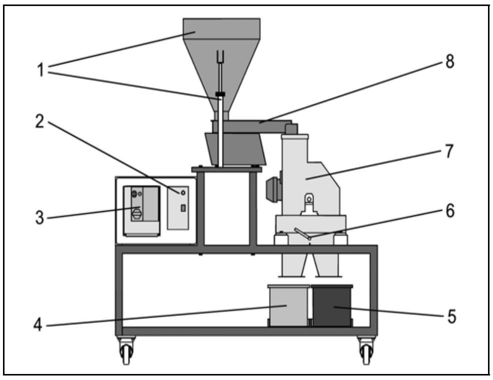

*Figure 1: Schematic of Laboratory magnetic separator type SHP-500***.** *(1) feed funnel height adjustable, (2) control elements of the trough, (3) vibrating three control elements of the magnetic separator, (4) tank (5) tank amagnetic material, (6) level plate separator, (7) magnetic separator) and (8) vibrating trough.*

2.1. Characterization by X-Ray fluorescence, XRF

Table 1 Quantification by XRF samples of Hoggar quartz silica deposit before enrichment.

Sample	Analysis of major elements (%)									
	SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**N_2O**	**K_2O**	**TiO_2**	**P_2O_5**	**ZrO_2**
H1	97.72	0.05	1.46	0.01	0.01	0.1	0.05	0.55	0.03	0.02
H2	97.78	0.05	1.58	0.04	0.05	0.04	0.05	0.30	0.01	0.1

The samples have a highest level of silica. Both samples contain a high level of iron and Titanium, which are 1.46% and 1.58%, respectively. The quartz samples, needs a cleaning step of enrichment by magnetic separation technology for its suitable future use.

3. Results and discussion

3.1. Quartz sand enrichment by magnetic separation process

The magnetic separation process removes almost of ferromagnetic impurities; the quartz raw ore containing 97-98 % of SiO_2 and suffered crushing and grinding in laboratory scale in ball mill to achieve required particles size (literature) in order to undergo the magnetic separation (Figure 2).

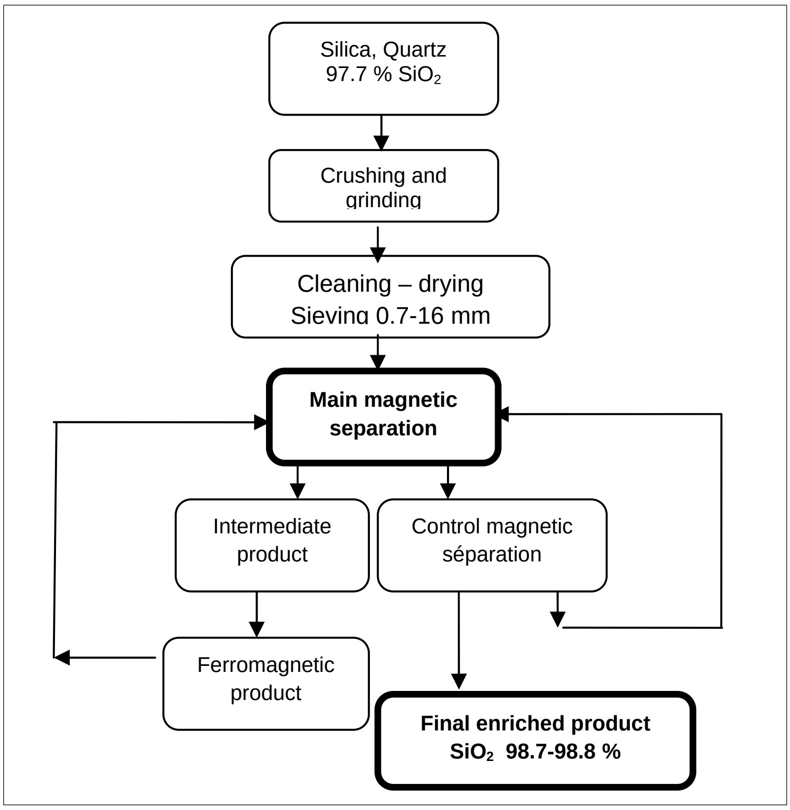

Figure 2: Flow-sheet of quartz silica enrichment of Hoggar deposit by magnetic separation process

In the table 2, we illustrate the measured concentration (%) of oxides impurities present into sample Hoggar quartz. Its indicates that SiO_2 presents into quartz after magnetic separation sample is 98.7% by report to the values before enrichment, which are in agreement with the literature [Technical and Technological Report., 2010].

Table 2: Quantification by XRF analysis samples of Hoggar H1 after enrichment

Analyte	Elements	Concentration (%)
Al	Al_2O_3	0.035
Si	SiO_2	98.7
N	N_2O	0.003
Ti	TiO_2	0.829
K	K_2O	0.023
Ca	CaO	0.05
Fe	Fe_2O_3	0.3
Zr	ZrO_2	0.01
P	P_2O_5	0.03
Mg	MgO	0.02

Table 3: Quantification by XRF analysis samples of Hoggar H2 after enrichment

Analyte	Elements	Concentration (%)
Al	Al_2O_3	0.045
Si	SiO_2	98.975
N	N_2O	0.04
Ti	TiO_2	0.195
K	K_2O	0.05
Ca	CaO	0.04
Fe	Fe_2O_3	0.525
Zr	ZrO_2	0.1
P	P_2O_5	0.01
Mg	MgO	0.05

For a same type of magnetic separator in industrial scale, the ZENITH Manning and Construction Company obtained after magnetic separation of quartz containing 0.36% Fe_2O_3 and 0.25% Ti_2O_3 enriched quartz with concentration 0.1% and 0.08%, respectively. We can therefore conclude that the obtained results are in agreement with those on an industrial (ZENITH Company) scale with the same working conditions.

3.2. The effect of particle size on the performance of the magnetic separation

The aim of this work is to make in evidence the role played by the size distribution in the process of magnetic separation and its impact on the level of concentration of impurities. In our case, we have chosen the Hoggar samples H2 and measured the rate of iron and titanium concentrations (Table 4) particle size in order to determine what class of size class the major removal of ferromagnetic impurities is optimal.

Table 4: Particle size distribution of the iron and titanium concentration of H2 samples

Quartz particle size, H2, mm	Iron Separation rate, %	Titanium Separation rate, %
16	1.58	0.3
10	1.50	0.25
6	1.12	0.225
3	1.0	0.217
1	0.525	0.195
0.8	0.525	0.195
0.7	0.525	0.195

The figure 3 depicts the separation rate of iron and titanium present in Hoggar quartz sheet versus particle size, where an optimum value of size 1 mm corresponding to a maximum reduction of iron and titanium impurities.

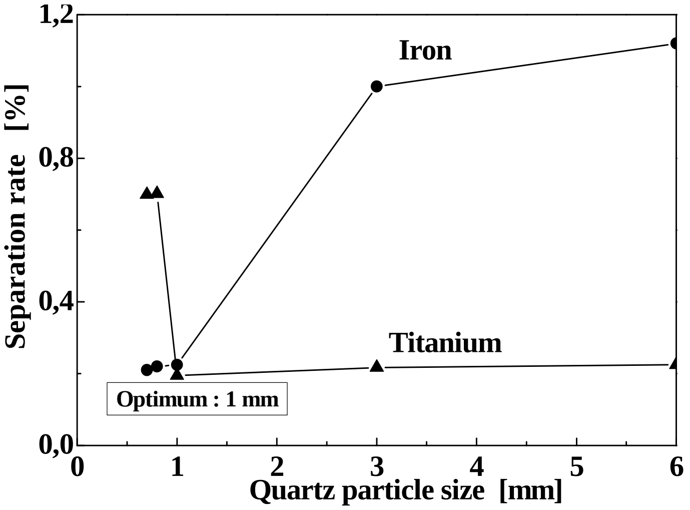

Figure 3: Iron and titanium separation rate versus Hoggar quartz particle size

Figure 3 depicts the separation rate function of particle size of Hoggar quartz for iron and titanium impurities, where the magnetic separation reaches an optimum value 0.525% and 0.195% , respectively, corresponding to the minimal value 1 mm particle size.

3.3. The Backscattered Electron image

The Backscattered Electron image (BEI) is used to locate the area of the silica. Bright areas correspond to high atomic Z number and dark areas correspond to low atomic Z numbers. This analysis are carried out before and after the enrichment of raw quartz in order to show the improvement of impurities removal from silica sand by using a magnetic separation process (Figure 4).

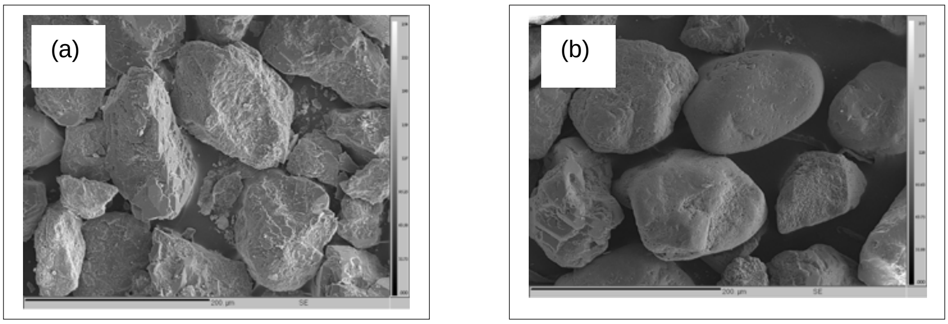

Figure 4: Images SEM quartz samples of Hoggar deposit (Algeria). (a) Before enrichment; (b) After enrichment

The results of BEI analysis (Figure 4) come to consolidate the XRF results. We can notice in these images, that the silica sand grains after enrichment by magnetic separation are clearer than those before enrichment. Bright areas correspond to high atomic Z number are eliminated. That is mean, that the heavy metals, especially iron and titanium are significantly removed, which confirmed by achieving magnetic separation.

Conclusion

The main objective of this work was to study the pure silica as a raw material for national electrometallurgy to ensure a better quality for its future application in photovoltaic (PV) area.

Within this framework and to achieve these objectives, a set of national silica samples (quartz from Hoggar deposit) have been investigated using X-ray fluorescence (XRF) and scanning electron microscopy (SEM). The samples H1 and H2 have raised the subject of magnetic separation enrichment by 'magnetic separator SHP-500 type, where the high gradient of separator and the grain size of the raw material played a very important role. During prospecting the previous works, we have noted that the use of a high gradient magnetic separator can separate different minerals and grains have almost the same magnetic susceptibility as magnetite or hematite-quartz-martite. The quartz raw material with Fe_2O_3 et TiO_2 concentration 1.58% et 0.3%, respectively, a value less than 0.6% of Fe_2O_3 and less than 0.2% of TiO_2. Have been reached, results in agreement with those given in the literature and confirm the effectiveness of the process.

The obtained results have shown that after granulometric separation (sieving) of the quartz samples reveal that the grain diameter of 1 mm coincides with the optimal concentration of Fe_2O_3 and TiO_2.

A considerable elimination of the aluminum was well noted using the XRF analyses, where the other impurities were no significant decreased. In addition, XRF and Backscattered Electron image analysis allows us to confirm that the used process was effectiveness for improving removal impurities from Hoggar deposit quartz silica intended for the electrometallurgy.

References

Wineganer R., 2007.17th workshop on crystalline silicon and solar cells and modules: materials and processes trends and future states of global polysicon production ", Vail, colororado august.
US Solar Market Trends, 1997-2006" (preliminary), Larry Sherwood, Interstate Renewable Energy Council, www.irecusa.com.
Muller. Chambre d'agriculture du Bas-Rhin Schiltigheim. Septembre 2010. Electricité photovoltaique : énergie propre de demain.
Wineganer R., august 2010. 17th workshop on crystalline silicon and solar cells and modules: materials and processes trends and future states of global polysicon production, Vail, colororado.
Yasuhico, Sakaguchi et al., ISIJ International Production of high purity silicon using Ac-arc furnace with by carbothermic reduction of heath shaft. 32, 1992, N° 5, 643-649.
Dielt J., 1987. Metallurgical ways of silicon meltstock processing. Elsevier Science Publisher B.V.
Department of Geology and minerals resources Engineering, IGBt, NTNU. (Norway). Strategic University Programme at Department of Geology and Minerals Resources Engineering. The value chain from mineral deposit to beneficiated product with emphasis on quartz. 2002.
Désindes L., 2004, Silica ultrapure for electrometallurgy: gitology, physical and chemical characteristics of the ore quartz, Henri Poincaré University, Nancy 1, 433, France.
Banza A.N., Quindt J. and Gock E., 2006, Improvement of the quartz sand processing at Hohenbocka, Min. Proc. 76– 82.
Baoqi S. and Zhengbing C.,1995, Chemical purification of industrial minerals. Proceedings of the XIX International Mineral Processing Congress, 2, 39, 207– 211.
Braga A.F., Moreira S.P., Zampieri P.R., Bacchin J.M.G. and Mei P.R. 2008, New processes for the production of solar-grade polycrystalline silicon: Solar Energy Materials & Solar Cells 92, 418–424.
Kheloufi A. 2009. Acid leaching technology for obtaining a high-purity silica for photovoltaic area. D. Sauro Pierucci. DOI 10.3303/CET0917119. Pp. 197-202.
Kheloufi A, Berbar Y, Kefaifi A, Drouiche N., (2010). Silica sand etching behaviour during a primary step of its leaching process, AIP journal, 1315, 271-276.
Technical and Technological Report Zenith Manning and Construction Company., 2010, N°4, 126.