PRILIMINARY UPGRADING OF SILICA SAND FOR SILICON AND SILICONES INDUSTRIES FROM ARDHUMA, IRAQI WESTERN DESERT

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ABSTRACT

In this research a preliminary beneficiation study was conducted on a sample from Ardhuma silica-sand deposit in the Iraqi Western Desert, to provide a raw material for silicon industries. The beneficiation processes applied were vibration screening (dry and wet), attrition scrubbing and magnetic separation (dry and wet). These techniques were used individually or in combination to upgrade the sand to produce quartz-sand meeting the requirements of Metallurgical-Silicon production. This would be the base material for silicon materials and Solar-Grade Silicon.

Quartz-sand fulfills the requirements of the State Company for Mining Industries and Aquatic Insulation for the raw materials of MG-Silicon production, which was produced by applying one of the following beneficiation routs: The first consists of autogenesis grinding, dry screening on 150 µ, attrition scrubbing and wet screening on 150 µ. Whereas, the second includes autogenous grinding, dry screening on 150 µ and dry magnetic separation using magnetic field intensity of 16.5 Kilogauss.

دراسة أولية لتنقية رمال السليكا لأغراض صناعة السليكون والمركبات السليكونية من منطقة أرضمة في الصحراء الغربية العراقية

علاء محمد خليل مصطفى، ابتهال خليل فليح و تانيا فاروجان خاجيك

المستخلص

تم في هذا البحث إجراء دراسة أولية لتنقية نموذج من خام رمال السليكا لمنطقة أرضمة الواقعة في الصحراء الغربية العراقية لإنتاج مادة أولية تستخدم في صناعة السليكون الميتالورجي، حيث طبق في هذا البحث عدد من عمليات المعالجة شملت الغربلة الجافة والرطبة، الرج والحك الميكانيكي الشديدين والفصل المغناطيسي الجاف والرطب. هذه التقنيات استخدمت بشكل منفرد أو بصورة متتالية (متعاقبة) لإنتاج رمال زجاج مطابقة لمواصفات المواد الأولية (الكوارتز) الداخلة في إنتاج السليكون الميتالورجي الذي يعتبر الأساس في الصناعات السليكونية والألواح الشمسية المستخدمة في توليد الطاقة.

ولقد وجد أن بالإمكان إنتاج رمال كوارتز مطابقة لمواصفات الشركة العامة للصناعات التعدينية والعزل المائي الخاصة بالمواد الأولية الداخلة في صناعة السليكون الميتالورجي بإتباع أي من المسلكين التكنولوجيين التاليين: الأول يتضمن عملية طحن رملي ذاتي وغربلة جافة على غربال 150 مايكرون، ثم عملية الرج والحك الميكانيكي الشديدين وأخيراً غربلة رطبة على غربال 150 مايكرون. فيما يشمل المسلك الثاني عملية الطحن الرملي الذاتي والغربلة الجافة على غربال 150 مايكرون، ثم عملية الفصل المغناطيسي الجاف باستخدام شدة مجال مغناطيسي 16.5 كيلوگاوس.

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INTRODUCTION

Silicon (Si) is a gray metallic lustrous element with atomic number 14 and atomic mass 28.086. After Oxygen, silicon is the second most abundant element (27.5%) in the lithosphere. It does not occur in an elemental form, but only in forms of oxides and silicates. Since the turn of the 20th century, silicon has been produced, almost exclusively, by carbothermal reduction of silicon dioxide:

$$SiO_2 + 2C \longrightarrow Si + 2CO \qquad \Delta H_{298} = 695\ KJ$$

The carbothermal process yields silicon with a purity of 98% (metallurgical-grade silicon, M.G-Si), for which a typical analysis is 0.5% Fe; 0.4% Al; (100 – 400) ppm each of Ca, Cr, Mg, Mn, Ni, Ti and V; (20 – 40) ppm each of B, Cu, P and Zr. The usual starting materials are chunks of quartzite, coke coal, as well as charcoal and wood chips for good ventilation of charge. To produce 1 ton of silicon, (2.9 – 3.1) ton of quartz or quartzite, (1.2 – 1.4) ton coke (gas coke and petroleum coke) and (1.7 – 2.5) ton of charcoal and wood are required. The purity of the produced silicon depends primarily on the purity of these materials and secondarily on the purity of the graphite electrodes and the furnace lining. The purity of silicon can be increased by using purer starting materials (Zulehner, 2005).

Silicones have been used directly or indirectly in many industries; therefore, it is a life necessity. In 2002, statistics reported by Dow Corning Company showed that, world production is around one million ton of silicones, which is equivalent to 9 billion US Dollars covering 50% silicon oils, 40% silicon rubbers, 10% silicon resins.

Silicon element having high degree of purity has a special importance for the world economy. A rough estimation stated that Iraq, the Arab World and the Middle East import silicones of several hundred million dollars, for this reason and due to the presence of high reserves of pure silica sand (SiO_2 > 98%), as well as oil and petrochemical industries in Iraq, silicon and silicones technology become vital for the future national economy (Ibn Sina State Company, 2010).

The grade of quartz raw materials depends on the required grade of the products. According to Ullmann's encyclopedia of industrial chemistry, high-grade raw materials (quartz) are required to produce metallurgical grade silicon (Florke and Graetsch, 2007) as shown in Table (1)

Table 1: Chemical composition of the quartz raw materials required for the production of M.G-Silicon*

Component	**SiO_2**	**Fe_2O_3**	**Al_2O_3**	**TiO_2**	**CaO**	**MgO**
(%)	≥ 99	≤ 0.1	≤ 0.2	≤ 0.02	≤ 0.1	≤ 0.1

* According to the Ullmann's Encyclopedia of Industrial Chemistry (Florke and Graetsch, 2007)

The State Company for Mining Industries and Aquatic Insulation as a part of a project for the production of silicon for solar cell and silicones industries indicated their own specification for the silica sand raw materials required for the production of M.G-Silicon, (Table 2) (State Company of Mining Industries and Aquatic Insulation, 2011).

Table 2: Chemical composition required for M.G-Silicon production*

Component	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	P_2O_5	SO_3	Na_2O	K_2O	L.O.I
(%)	99 min	0.03 max	0.5 max	trace	0.1 max	0.006 max	0.4 max	0.5 max	0.25 max	0.05 max	0.2 max

* According to State Company for Mining Industries and Aquatic Insulation

PREVIOUS WORKS

According to our knowledge, there is no study concerning the processing of Ardhuma silica sand for production of silicon in Iraq yet. However, many works have been conducted for the up-grading of Iraqi silica sand particularly Ardhuma and Kilo 180 sites. Nevertheless, most of these works were carried out to produce silica sand that fulfill the requirements of different types of glass production (Al-Ajeel *et al*., 1988; Daykh *et al*., 2007; Mustafa, 2008 and Mustafa *et al*., 2011).

Furthermore, Sontakkey and his co-worker presented multiple process routs to up-grade Indian silica-sand from Ratnagiri region, and they obtained a high-grade concentrate of silica sand (Table 3), by following a process rout, which includes wet screening, attrition scrubbing of – 590 µ fraction, followed by magnetic separation of the de-slimed sand fraction (– 590 + 150 µ) (Sontakkey *et al*., 2010).

Table 3: The raw sample is a chemical composition as well as the up-graded sand

Constituents	Silica sand raw sample (%)	Up-graded sample (Concentrate) (%)
SiO_2	98.31	99.40
Al_2O_3	0.61	0.106
Fe_2O_3	0.28	0.042
CaO	0.023	0.013
MgO	0.01	0.003
K_2O	0.077	0.02
TiO_2	0.028	0.003
Cr_2O_3	79 (ppm)	5 (ppm)
L.O.I.	0.23	0.10

Al-Maghrabi, suggested attrition milling, screening followed by conduction flotation of the (– 500 + 100) µ to up-grade Saudi silica sand of Jeddah area. The concentrate containing 98.6% SiO_2 and 0.1% Fe_2O_3 from a feed, contains 81.4% SiO_2 and 0.6% Fe_2O_3 (Al-Maghrabi, 2004).

Venkatraman and his colleagues applied a floatex spiral circuit in processing Indian silica sand. The application comprised of floatex density separator and Humphreys glass sand spiral-model LC-3700. The compositions of the sand products as well as the silica sand feed are shown in Table (4) (Venkatraman *et al*., 2000).

Table 4: Floatex/ spiral circuit products weight distribution and chemical compositions

Compositions	weight	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO + MgO	$Na_2O + K_2O$
	(%)						
Silica sand feed	100	98.4	0.637	0.143	0.439	0.030	0.043
Spiral lights	85.4	99.6	0.222	0.025	0.030	0.015	0.016
Spiral heavies	9.6	97.9	1.430	0.110	0.465	0.017	0.018
Floatex	25	95.7	1.188	0.548	2.296	0.023	0.090

MATERIALS AND METHODS

▪ Materials

─ **Sand Samples:** Raw silica sand sample of 50 Kg from Ardhuma site was subjected to mixing, quartering and dividing using rotary sample divider to obtain representative samples of 1 Kg each. The chemical analysis of the raw silica sand is shown in Table (5).

Table 5: Chemical composition of Ardhuma silica sand

Chemical Composition	SiO_2	Fe_2O_3	Al_2O_3	CaO	TiO_2	SO_3	L.O.I.	MgO	Na_2O	K_2O
(%)	98.76	0.085	0.48	0.23	0.18	0.07	0.20	0.032	0.03	0.01

▪ Methods

─ **Sieve Analysis:** One Kg of a representative sand sample was subjected to sieve analysis using vibratory sieve device type RETSCH and B.S sieve opening (850, 600, 250, 150 and 75) µ. The results of sieve analysis are shown in Table (6). The chemical analysis of each sand fraction is shown in Table (7).

Table 6: Sieve analysis of the Ardhuma silica sand

Grain Size (µ)	Weight (gm)	Weight (%)	Accumulative Weight (%)	
			Retain	Pass
+ 850	0.35	0.082	0.028	99.94
– 850 + 600	0.21	0.0016	0.044	99.92
– 600 + 250	251.50	20.35	20.39	79.58
– 250 + 150	771.35	62.43	82.82	17.15
– 150 + 75	208.78	16.89	99.71	0.62
– 75	3.27	0.26	99.97	0.00

Table 7: Chemical analysis of each sand fraction

Sieve Opening (µ) \ Chemical Composition	SiO_2	Fe_2O_3	Al_2O_3	CaO	TiO_2	SO_3	L.O.I.	MgO	Na_2O	K_2O
	(%)									
+ 850*	–	–	–	–	–	–	–	–	–	–
– 850 + 600*	–	–	–	–	–	–	–	–	–	–
– 600 + 250	98.98	0.022	0.43	0.16	0.02	< 0.07	0.13	0.016	0.02	0.004
– 250 + 150	98.58	0.034	0.30	0.17	0.06	< 0.07	0.16	0.022	0.01	0.005
– 150 + 75	98.0	0.17	0.32	0.16	0.45	< 0.07	0.13	0.038	0.045	0.007
– 75*	83.28	0.87	–	–	–	–	–	–	–	–

***** These size fractions retain very small amount; not or partially enough for chemical analysis

─ **Screening:** Two types of screening were applied, these are:

Dry Screening: Several experiments were conducted using sieves with openings of (850, 600 and 150) µ and fractions in the ranges of (– 850 + 150) µ, (– 600 + 150) µ and + 150 µ were collected as product.

Wet Screening: Wet screening was carried out on 150 µ (ASTM) sieve opening, using a sieving device type RETSCH, where + 150 µ fraction was collected as a product.

─ **Dry Magnetic Separation:** Raw silica sand samples of 100 gm each were subjected to induced roll dry high intensity magnetic separator type OUTOTECH supplied with hopper and vibrating feeder. The magnetic intensity (Kilogauss) can be adjusted from the electric current that induced the magnetic field, where current is ranged from (0 – 3) Ampere, which is equivalent to a magnetic field intensity ranged from (0 – 16.5) Kilogauss. The sand passes over a controlled speed rotating induced disc, and then separated into magnetic and non-magnetic fractions.

▪ **Up-grading Techniques**

In addition to the screening, several techniques were used in combination so as to improve the upgrading efficiency. These techniques are as follow:

─ **Autogenous Grinding:** It is a process whereby the sand grains act as a grinding media. This process provides a suitable mean for fine impurities (e.g. clay) separation from hard sand particles due to self-abrasion between them. This process was applied to improve the up-grading process, through the liberation of clay fine particles and removing it from the sand particles by screening, and thus, minimizing the impurities incorporated with the sand.

─ **Attrition Scrubbing:** This process was done at a single cell attrition scrubber type DENVER. The device consists of two stainless steel opposed pitch turbine-type propellers on a stainless steel shaft, shaft collar, and a stainless steel tank of one liter capacity square with a cover for performing high density batch sand scrubbing tests. The action of the pitch propellers forces the particles to impinge on each other giving multiple grain-to-grain attrition scrubbing. Silica sand samples were mixed with water in a solid percentage of 75%, scrubbing time 60 minutes in a scrubbing speed of 1000 rpm. The products were either further processed by magnetic separation or simply passed through 150 µ sieve.

─ **Wet Magnetic Separation:** Wet high intensity magnetic separator type CARPCO was used to separate magnetic and non-magnetic particles from each other within the sand particles. The process was carried out by allowing sand slurry of 25% solid to pass through a chamber (can be loaded with steel balls of different diameters) enclosed between two magnetic coils capable of producing about 7 Kilogauss magnetic field intensity.

RESULTS AND DISCUSSION

In order to up-grade Ardhuma silica sand for silicon industry, several techniques were used, the first technique tested was screening (dry and wet), which is the simplest method in sand beneficiation. Other techniques; like scrubbing – desliming and magnetic separation were used, individually or in combination. Furthermore, autogenous grinding of the raw silica sand was found to be of a significant effect on the up-grading process.

▪ **Dry and Wet Screening**

Several dry and wet screening experiments were conducted to upgrade the raw silica sand of Ardhuma region using sieves of different openings (850, 600 and 150) µ. Table (8), shows the chemical composition of the sand products obtained.

As it is shown in Table (8), the results reveal that the screening process has the major contribution towards silica sand up-grading. However, the sand samples of + 150 µ, which were autogenously ground showed better results than the other tested samples, particularly in increasing the silica content. Thus, sand products of the dry and wet screening of the raw materials that were autogenously ground were found compatible to the requirements of State

Company of Mining Industries and Aquatic Insulation for the quartz raw material for M.G-silicon production in the term of silica content. MgO and TiO_2 as well as Fe_2O_3 contents remained critical. Therefore, further processing may require improving the up-grading efficiency.

Table 8: Chemical composition of products obtained from screening (wet and dry)

Treatment	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	L.O.I.
	(%)										
-Dry Screening (– 850 + 150) µ	98.45	0.083	0.39	0.15	0.25	0.038	–	0.02	0.02	–	0.21
-Dry Screening (– 600 + 150) µ	98.45	0.065	0.31	0.12	0.20	0.040	–	0.03	0.02	–	0.12
-Dry Screening (150 µ)	98.24	0.039	0.33	0.03	0.19	0.040	–	–	–	–	0.20
-Wet Screening (150 µ)	99.15	0.035	0.32	0.07	0.16	0.040	0.06	0.03	0.04	0.01	0.21
-Auto. Grind. -Dry Screening (150 µ)	99.21	0.024	0.28	0.03	0.16	0.020	0.07	0.03	0.02	0.01	0.10
-Auto. Grind. -Wet Screening (150 µ)	99.21	0.035	0.27	0.07	0.04	0.020	0.07	0.03	0.02	0.01	0.20

- **Attrition Scrubbing**

To improve the specifications of the screening sand products to match the requirements of M.G-Silicon raw materials, several experiments were performed using a combined technique of screening and attrition scrubbing. (Table 9), shows the results of the attrition scrubbing plus dry/ wet screening experiments.

Table 9: Chemical composition of the sand products (+ 150 µ) obtained from attrition scrubbing and wet screening combined experiment

Treatment	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	L.O.I.
	(%)										
-Attrition Scrubbing -Wet Screening (150 µ)	99.00	0.03	0.44	0.08	0.05	0.03	0.03	0.03	0.04	0.01	0.16
-Dry Screening (– 600 + 150) µ -Attrition Scrubbing -Wet Screening (150 µ)	99.10	0.027	0.46	0.06	0.05	0.03	0.03	0.03	0.04	0.01	0.16
-Dry Screening (150 µ) -Attrition Scrubbing -Wet Screening (150 µ)	99.11	0.027	0.35	0.06	0.04	0.02	0.03	0.02	0.03	0.01	0.16
-Autogenous Grinding -Dry Screening (150 µ) -Attrition Scrubbing -Wet Screening (150 µ)	99.48	0.011	0.23	0.03	0.03	< 0.01	0.04	0.02	0.02	0.01	0.09
-Wet Screening (150 µ) -Attrition Scrubbing -Wet Screening (150 µ)	99.49	0.026	0.21	0.06	0.04	0.01	0.03	0.02	0.04	0.01	0.08

As it can be seen from the results in Table (9), the chemical characteristics of the screened sand were highly improved after the incorporation of attrition scrubbing (1000 rpm for 1 hr). This led to produce silica sand that fulfills the requirements of quartz materials for M.G-Silicon production, according to the aforementioned company requirements, which are given in Table (2). It is worth mentioning that the removal of impurities were enhanced by autogenously grinding of silica sand prior to screening. While attrition scrubbing facilitates the removal of the remaining impurities, especially Fe_2O_3 that stains silica sand particles via high attrition activity.

- **Magnetic Separation**

─ Dry High Intensity Magnetic Separation: In this work, high intensity magnetic separation was used at dry and wet conditions with a magnetic field intensity of about (16 and 7) Kilogauss, respectively. A set of experiments consisting of screening (dry and wet) and/ or attrition scrubbing followed by dry high intensity magnetic separation were conducted. Table (10) shows the results of the chemical analyses of the produced sand.

Table 10: Chemical composition of the products obtained from dry high intensity magnetic separation, attrition scrubbing, dry and wet screening combined experiments

Treatment	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	L.O.I.
	(%)										
-Dry magnetic separation	99.02	0.04	0.5	0.05	0.12	0.01	0.09	0.01	0.08	0.01	0.25
-Dry magnetic separation -Attrition Scrubbing -Wet Screening (150 µ)	99.27	0.03	0.38	0.03	0.04	< 0.01	0.07	0.01	0.01	0.01	0.2
-Dry screening (– 600 + 150) µ -Dry magnetic separation	99.11	0.04	0.44	0.03	0.06	< 0.01	0.04	0.01	0.07	0.01	0.22
-Dry screening (150 µ) -Dry magnetic separation	99.20	0.03	0.41	0.03	0.04	< 0.01	0.04	0.01	0.06	0.01	0.2
-Autogenous Grinding -Dry screening (150 µ) -Dry magnetic separation	99.24	0.02	0.29	0.03	0.04	< 0.01	0.04	0.01	0.01	0.01	0.2
-Wet screening (150 µ) -Dry magnetic separation	99.21	0.03	0.39	0.03	0.04	< 0.01	0.04	0.01	0.01	0.01	0.2
-Autogenous grinding -Dry magnetic separation -Attrition Scrubbing -Wet screening (150 µ)	99.34	0.02	0.24	0.02	0.03	< 0.01	0.04	0.01	0.01	0.01	0.12

According to the results shown in Table (10), it can be seen that all the results of rows 4 – 7 are compatible to the chemical requirements of quartz materials used for M.G-Silicon production according to the State Company of Mining Industries and Aquatic Insulation, while those of rows 1 – 3 were partially failed. However, autogenous grinding of the raw silica sand and screening on 150 µ sieve opening followed by dry magnetic separation of the + 150 µ fraction seems to be technically and economically attractive route for silica sand up-grading. The processing sequence, allows to liberate clay materials from the sand grains, that were removed by the screening process. While, magnetic separation removes large portion of iron-contaminants.

– **Wet High-Intensity Magnetic Separation:** Another set of experiments were performed using wet high-intensity magnetic separation, with a magnetic field intensity of about 7 Kilogauss. Table (11), shows the results of the sand products obtained from these experiments.

Table 11: Chemical composition of the products obtained from wet high-intensity magnetic separation scrubbing and wet screening combined experiments

Treatment	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	L.O.I.
	(%)										
-Autogenous grinding -Wet Screening (150 µ) -Wet magnetic separation	99.06	0.04	0.41	0.04	0.12	0.01	0.07	0.01	0.09	0.02	0.20
-Autogenous grinding -Dry Screening (150 µ) -Wet magnetic separation	99.08	0.03	0.32	0.04	0.06	0.01	0.06	0.01	0.06	0.01	0.2
-Attrition Scrubbing -Wet Screening (150 µ) -Wet magnetic separation	99.13	0.03	0.3	0.03	0.03	< 0.01	0.04	0.01	0.01	0.01	0.15

From the results of Table (11), it is obvious that the wet magnetic separation did not show any improvement concerning Iron removal, as it is compared with the previous up-grading methods, of Ardhuma sand. This may be attributed to the low magnetic intensity, which was applied in this course of experiment comparing with the dry magnetic separation.

Figure (1), shows suggested technical routs, that lead to produce silica sand, which fulfills the requirements for M.G-Silicon production according to the requirements of State Company of Mining Industries and Aquatic Insulators.

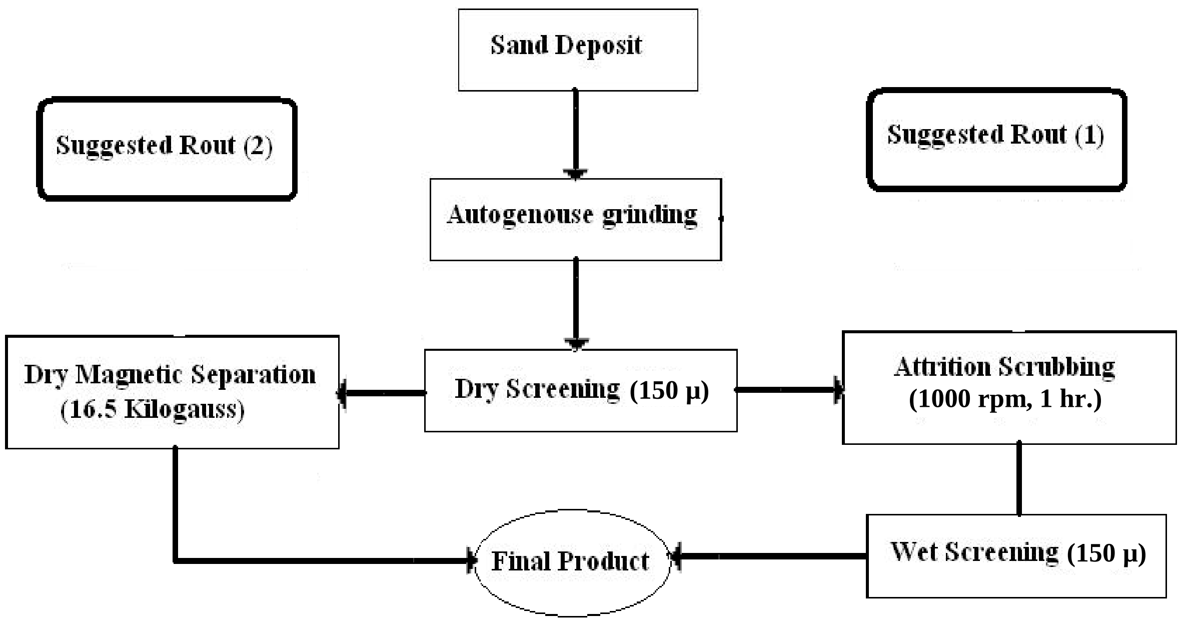

Fig.1: Suggested routs of silica sand beneficiation for M.G-Silicon production

CONCLUSIONS AND RECOMMENDATIONS

- The most efficient techniques in the sand up-grading are attrition scrubbing and magnetic separation; these techniques can be used individually or in combination with screening to produce sand compatible to the requirement of silicon raw materials.
- It was found that autogenous grinding has a high contribution to Ardhuma sand up-grading, by liberating clayey materials from the sand grains.
- From this research, two process routes can be recommend for the treatment of Ardhuma silica sand to fulfill the requirement of M.G-Silicon raw materials. The first consists of autogenous grinding; dry screening on 150 μ sieve, followed by attrition scrubbing at 1000 rpm for 1 hr., and finally screening on 150 μ sieve. The second is represented by autogenous grinding, dry screening on 150 μ sieve; followed by dry magnetic separation using magnetic field intensity about 16.5 Kilogauss.
- There were no significant improvements occurred by using wet magnetic separation at field intensity of 7 Kilogauss, compared with dry magnetic separation and attrition scrubbing techniques.

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