International Journal of Engineering Trends and Technology
ISSN: 2231–5381 / https://doi.org/10.14445/22315381/IJETT-V72I10P129
Volume 72 Issue 10, 316-322, October 2024

Original Article

Magniothermal Methods of Silicon Extraction from Quartz Sands of "Jerdanak" Mine

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Received: 13 June 2024 Revised: 05 October 2024 Accepted: 14 October 2024 Published: 25 October 2024

Abstract *- In this article, the chemical and mineralogical composition of the sands of the local quartz mine "Jerdanak" in Sherabad district (Surkhondarya, Uzbekistan) is analyzed by X-ray phase analysis (XRD), Scanning Electron Microscopy (SEM), Emission Semi-qualitative Spectral (EYaS), Infrared Spectroscopy (IR) analysis methods were determined. Then, the sand was washed several times in distilled water for enrichment and dried. The obtained clean sand was crushed, sieved and reduced with magnesium and aluminum at different temperatures up to 1800℃ for up to 8 hours, and technical silicon (93%-94%) was obtained. Magnesium metal and silicon (IV) oxide in a 2:1 mole ratio and aluminum, magnesium metals and silicon (IV) oxide in a 1:0.1:1.33 ratio for obtaining technical silicon by the magnoalluminothermic method; 1:0.2:1.33; It was obtained in 0.2:2:1 mole ratio. The resulting reaction was washed three to four times in distilled water, hydrochloric acid, and alkali to remove silicon from the samples. The purity of the isolated silicon was reanalyzed using XRD, SEM, EyaS, and IR-analysis methods. The optimal reaction temperature was 800°C, the reaction duration was 6 hours, and the reaction yield was the highest (75%) when the mole ratio of silicon oxide and magnesium was 1:2.*

Keywords *- Quartz sand, Magnesium, Aluminum, Reaction mixture, Reducer, Reactor.*

1. Introduction

One of the urgent problems today is to meet the need for energy without having a negative impact on the environment[1]. The use of alternative energy is the solution to such problems, and, at the same time, the negative impact of greenhouse gas emissions on the environment reduces the secret. The evaluation showed that if the installed photovoltaic cells with a power of 6 kW require an investment of about 10,000 euros, the system will pay for itself in 6 or 7 years, reducing the annual energy bills of the population by 60%. The European Union's Renewable Energy (RTE) authorities have set a target of increasing the share of RTE in Europe by 20% by 2020. It has been studied that Norway's target for the share of renewable energy in 2020 is 67.5% from 60.1% in 2005 [2, 3]. Crystalline silicon is the main element for the production of solar batteries, solar panels, and photovoltaic elements [4]. More than 90% of solar cells manufactured in 2012-2013 were based on this crystalline silicon. Thus, the natural occurrence, price and quality of silicon raw materials are the most important factors for all photovoltaic elements [5, 6]. Silicon is the second most abundant element on Earth, making up 75% of the Earth's crust. However, silicon is not found in nature as an element but combines with oxygen to form various forms of silicates and sands [7, 8]. Due to its stable physicochemical properties, quartz sand (SiO_2) is widely used in fields such as silicate glass, quartz ceramics, silicon metal, solar cells, and quartz fiber. SiO2 used in these areas is required to be of high purity. Obtaining high-purity quartz is one of the important areas of quartz processing. There are several stages of metallurgical treatment of quartz sand, one of which is effective for boron removal, and the other is effective for phosphorus removal [9, 10]. Quartz sands are ground into powder through various grinding devices.

The produced silicon (IV) oxide powder was treated with magnesium granules in the temperature range of 500-900℃. Analysis of the results using X-ray diffraction revealed the formation of Si and other by-products, such as MgO, Mg_2Si and Mg_2SiO_4 (Forsterite) [11, 12]. A two-step washing method is used to remove additional products. When the process of magnesiothermic reduction of amorphous and crystalline silicon is carried out at different temperatures, the yield of silicon at 800°C is determined to be 22.5%. An acid solution was used to wash silicon [13-17]. The onset temperature of the reaction was found to be about 465°C, and the temperature of the system increased sharply from 535 °C to 1270 °C[18, 19]. When the temperature is lowered to 792°C, they found that Mg_2Si and MgO are formed in the outer layer, and Si and MgO are formed in the grains inside the SiO_2 aggregate. It has been studied that it takes about 8 hours for newly formed Mg_2Si to be converted back to Si with a yield of up to 92% [20, 21].

Metallurgical grade silicon containing ~99.3% Si was washed with hydrochloric acid to produce metallurgical grade silicon (MG-Si) powder from Al-Si alloys produced by the aluminothermic reduction of Egyptian sand under the following conditions: particle size 100% -0.1 mm, reaction temperature 50℃, reaction time 20.0 minutes, solid/liquid ratio 1:9 g/ml and acid concentration 6.24 M.

The amount of iron in aluminum-silicon alloy is almost soluble in solution (except aluminum), and iron content (~0.2%) was found in purified silicon metal [22, 23]. The main goal of this study is to extract silicon from the quartz sand of the "Jerdanak" quartz mine in Sherabad District, Uzbekistan, using a magnethermal method.

2. Materials and Methods

2.1. Materials

Quartz sand from the "Jerdanak" quartz mine, located in Sherabad District, Uzbekistan, was used as the main raw material in this research. Aluminum and Magnesium were purchased "technically pure" from "Merit Chemicals" company.

2.2. Methods

2.2.1. X-ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) is an analytical method that provides information on crystal structures, phases, crystal orientations (texture) and other structural parameters, such as average size, crystallinity, deformation and crystal defects. "SHIMADZU" XRD-6100 powder diffractometer manufactured in Japan was used.

2.2.2. IR Analysis

The composition of the sands was investigated using IR-spectra and elemental analysis with Shimadzu IR Tracer-100.

2.2.3. SEM-EDS Analysis

In this research work, the microstructure composition was analyzed using a Jeol JSM-IT200LA (Japan) SEM-EDS device.

3. Emission Semi-Qualitative Spectral Analysis (EYaS) Analysis

Emission Semi-Qualitative Spectral Analysis (EYaS) is a method of emission semi-qualitative spectral analysis with an accuracy range of 1-10-5 to 3%. It is used to determine the mass fractions of 54 elements in a sample, used for rocks, ores and minerals. Spectrograph PGS-2 (DFS-8) diffraction grating with 600 lines per mm was used in this research.

4. The Process of Preliminary Processing of Raw Materials

Minerals are separated from each other based on the difference in the properties of minerals. Preparation processes for enrichment, in turn, are divided into the following classes: grinding, chattering, crushing, and classification. The main (enrichment) processes, in turn, are divided into the following and constitute enrichment methods: gravity enrichment, flotation enrichment, magnetic enrichment, electric enrichment and special chemical enrichment [24, 25]. In the research, the methods of washing, sieving, grinding, and chemical enrichment of quartz sand were used.

4.1. Experimental Part

In this research work, IR, SEM, EYAS and XRD analyzes were carried out before the enrichment of quartz sand from the "Jerdanak" quartz mine of Sherabad district. The analyzed sand sample was sifted through a 0.63 mm sieve (D120/H38 steel mesh). The sieved sand was washed in distilled water and acids and dried in a drying oven. Then, it was ground to 200-300 nm in a nano mill (Retsch PM 400 ball mill), rotating 400 times per minute. Experiments were carried out by taking crushed quartz sand, magnesium and aluminum in different mole ratios: 1) 12 g of crushed quartz sand was taken and 9.6 g of magnesium (Mg: SiO_2 = 2:1 mole ratio), 2) 7.5 g of Al, 0.65 g of Mg and 12.5 g of SiO_2 (Al: Mg: SiO_2=1:0.1:1.33 mol ratio), 3) 7.5 g of Al, 1.3 g of Mg and 12.5 g of SiO_2 (Al: Mg:SiO_2=1:0.2:1.33 mol ratio), 4) 0.54 g of Al, 4.8 g of Mg and 6 g of SiO_2 (Al: Mg: SiO_2=0.2:2:1 mol ratio) were mixed and refluxed at 1200 ℃ for 6 hours. Each of the obtained samples was divided into two parts. One part was washed 4 times in distilled water and cleaned in hydrochloric acid and alkali. Then, the filtered products were dried in a drying oven at 100 ℃. The second part was not washed. The obtained products were reanalyzed. In the mixed experiment, the samples with a relatively high percentage were re-reacted by changing the temperature and time.

5. Results and Discussion

5.1. IR Analysis

The samples of the reaction product between Mg and enriched quartz sand (Mg: SiO_2=2:1) before and after cleaning were studied by the IR-spectroscopy method. When the reaction product is washed in acid, alkali, and water, it can be seen that due to the release of additional substances, the vibrations characteristic of aluminum and magnesium compounds are not observed (Figures 1 and 2). In the IR-spectrum analysis of the reaction product between Mg and enriched quartz sand presented in Figure 2, it is possible to observe a vibration characteristic of crustal silicon in the region of 1072.42 cm^{-1}. There are also vibrations attributed to Mg_2Si at 767.67 cm^{-1} and MgO at 694.37 cm^{-1}. At 800℃, the reaction yield was 75%. When the sample obtained as a result of the reaction was analyzed by the X-ray IR-spectroscopy method, it was found that the purity of technical silicon was ≈ 94% (Table 1). The experiment was conducted at different temperatures and times. At the temperature of 800 ℃, the production yield of technical silicon was 75%. When the sample obtained as a result of the reaction was analyzed by the X-ray fluorescence method, it was found that the purity of technical silicon was ≈ 94% (Table 2).

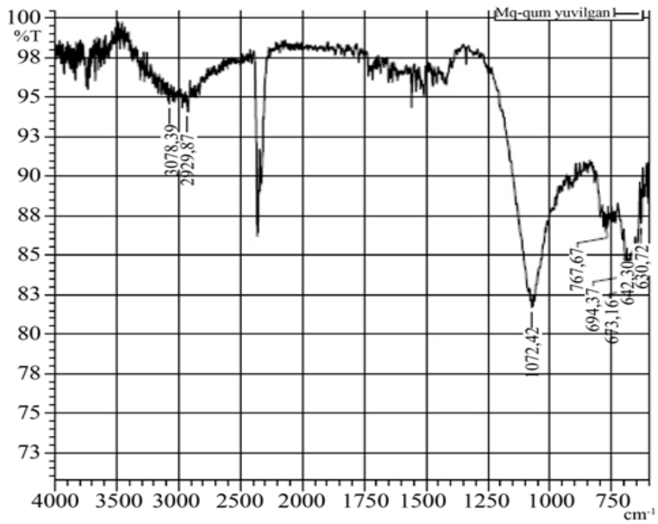


Fig. 1 IR spectrum images of the reaction product obtained at Mg: SiO_2=2:1 mol ratio before washing

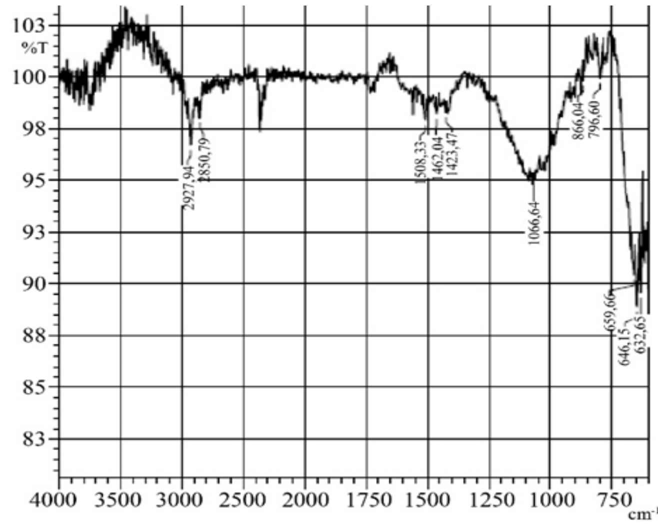


Fig. 2 IR spectrum images of the reaction product obtained in the ratio of Mg: SiO_2=2:1mol after washing

Table 1. Elemental analysis of the reaction product of Mg: SiO_2=2:1 mole ratio at 800°C

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
Si	93.896 %	[0.502]	Quan-FP	SiKa	21.5356
K	4.168 %	[0.068]	Quan-FP	K Ka	1.0906
Cl	0.663 %	[0.048]	Quan-FP	ClKa	0.0439
Al	0.662 %	[0.053]	Quan-FP	AlKa	0.0605
Fe	0.297 %	[0.007]	Quan-FP	FeKa	4.1029
S	0.173 %	[0.030]	Quan-FP	S Ka	0.0649

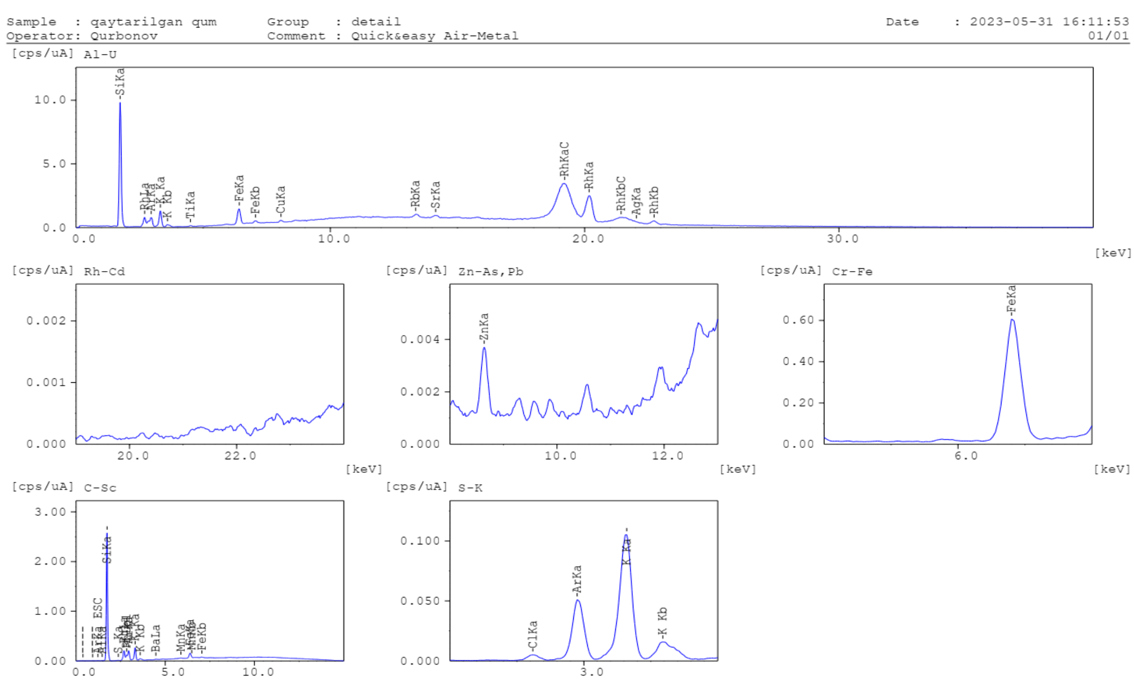


Fig. 3 X-ray image of the reaction product of Mg: SiO2=2:1 mole ratio at 800°C.

Table 2. EYaS analysis after purification of Mg:SiO_2=2:1 reaction products at 800°C

Element	Si	Al	Ca	Na	K	Fe	Mg	P	B	Mn	Cu	Ni
Amount, %	<94	0,3	0,03	1,0	<0,1	>0,3	>15	0,02	-	0,002	0,0008	0,0008

Technical silicon with a purity of 94% to 96% (GOST 2169-69) corresponds to the Kr3 brand. In recent years, companies producing solar panels have been trying to use low grade silicon Kr2 and Kr3 and fine fractions in order to reduce the cost of their products.

6. Methodology and Analysis of Silica Recovery in a Mixed Method

Crushed quartz sand, magnesium, aluminum were taken in different mole ratios and tested: 1) 7.5 g of Al, 0.65 g of Mg and 12.5 g of SiO_2 (Al:Mg:SiO_2=1:0.1:1.33 in mole ratio); 2) 7.5 g Al, 1.3 g Mg and 12.5 g SiO_2 (Al:Mg:SiO_2=1:0.2:1.33 mol ratio); 3) 0.54g Al, 4.8g Mg and 6g SiO_2 (Al:Mg:SiO_2=0.2:2:1 mole ratio) were mixed and refluxed at 1200 ℃ for 6 hours. Each of the obtained samples was divided into two parts. One part was washed 4 times in distilled water and cleaned in hydrochloric acid and alkali. Then, the filtered products were dried in a muffle furnace at 100 ℃. The second part was not washed. Re-analyses were performed on each sample [26, 27] (Table 3). Then, the reaction temperature and time were changed on the samples with a relatively high result, and the experiments were continued. Due to the low purity of silicon in the results of the above analysis, the temperature was changed, and a new experiment was conducted on the basis of a sample with a relatively high purity of Si. The reaction is 7.5 g of Al, 1.3 g of Mg and 12.5 g of SiO_2 Al:Mg:SiO_2=1:0.2:1.33 mole ratio and 0.54 g of Al, 4.8 g of Mg and 6 g of SiO_2 Al:Mg: SiO_2=0.2:2:1 mole ratio samples were made by raising the reactor temperature to 1800 ℃ for 8 hours [28].

6.1. SEM-EDS Analysis

After the reaction product was washed and purified, RXD and SEM analysis were again performed (Figure 4, Table 4).

Table 3. ESS analyzes of the products obtained as a result of the reaction (%)

The name of the sample	Si	Al	Ca	Na	K	Fe	Mg	P	B	Mn	Cu	Ni
Al:Mg:SiO_2 0,2:2:1 washed	25	0,2	0,03	0,03	<0,1	0,07	0,3	0,02	0,06	0,002	0,0006	0,1
Al:Mg:SiO_2 0,2:2:1 unwashed	25	0,4	0,006	0,15	<0,1	0,2	15	0,01	0,02	0,002	0,02	0,4
Al:Mg:SiO_2 1:0,1:1,33 washed	20	20	0,006	0,5	<0,1	3	0,2	0,02	0,006	0,02	0,001	8
Al:Mg:SiO_2 1:0,1:1,33 unwashed	18	23	0,01	1	<0,1	5	0,6	0,02	0,006	0,02	0,001	12
Al:Mg:SiO_2 1:0,2:1,33 washed	28	15	0,02	0,7	<0,1	3	0,3	0,01	0,005	0,01	0,01	6
Al:Mg:SiO_2 1:0,2:1,33 unwashed	25	15	0,008	0,1	<0,1	3	2	0,01	0,01	0,02	0,008	8

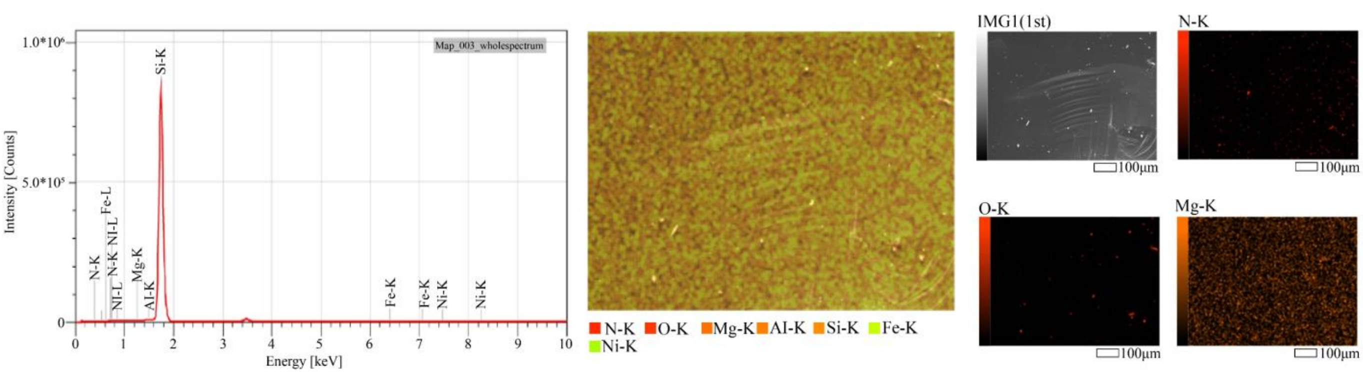


Fig. 4 SEM analysis of the sample formed by the mixture of Al:Mg:SiO_2=1:0.2:1.33 mol ratio

Table 4. Results of SEM analysis of reaction products carried out by mixed method at 1800°C

The ratios of mol	N	O	Mg	Al	Si	Fe	Ni
Al:Mg:SiO_2=**1:0,2:1,33**	-	1,55	-	0,13	99,24	0,05	0,03
Al:Mg:SiO_2=**0,2: 2:1**	-	18,42	0,30	0,47	80,48	0,30	0,02

From the RXD analysis, it was found that the intensity of the formed silicon was high when the X-rays fell at an angle of 20°-30° (Figure 5). So, the purity level of TK obtained as a result of the above reaction was equal to 99.24%. TK of this purity corresponds to the Kr00 brand. This brand of TK is used to improve the ductility, hardness and strength of aluminum. Addition of TK to aluminum alloys makes them strong and light. Therefore, they are used in the automotive industry to replace heavy cast iron parts. Automotive parts such as engine blocks and tire rims are most commonly made of aluminum-silicon casting. In addition, the technical silicon of this brand can be used as a basic material in the solar and electronics industries. For example, it will be possible to use it in the production of solar cells, semiconductors and silicon chips [29, 30]. It can be seen from Figure 6 that the purity of the silicon obtained in the reaction carried out in the ratio of Al:Mg:SiO_2=0.2:2:1 is 80.48% and should continue to give.

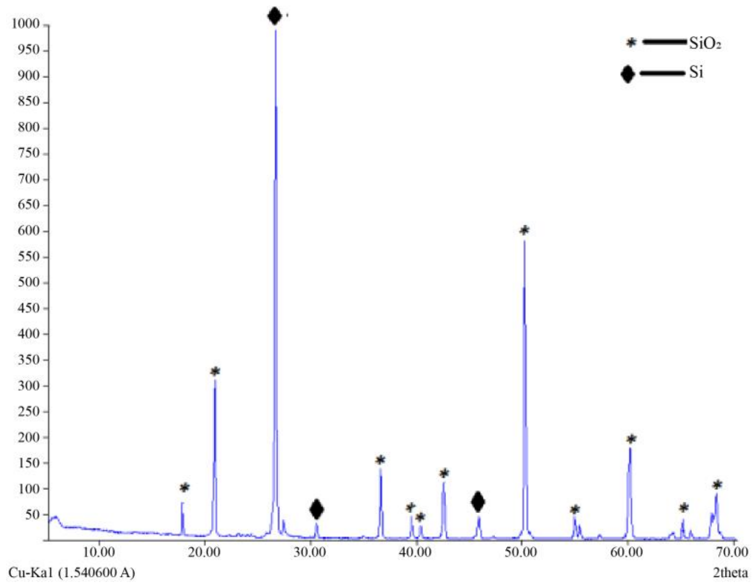
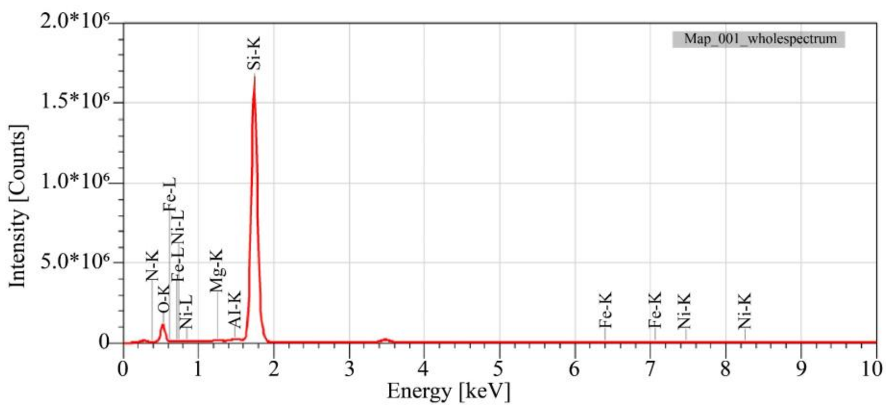


Fig. 5 RXD analysis of the sample formed as a result of the mixture of Al:Mg:SiO_2=1:0.2:1.33 mol ratio



Element	Line	Mass%	Atom%
N	K	nd	nd
O	K	18.42±0.02	28.41±0.04
Mg	K	0.30±0.00	0.31±0.00
Al	K	0.47±0.00	0.43±0.00
Si	K	80.48±0.03	70.70±0.03
Fe	K	0.30±0.00	0.13±0.00
Ni	K	0.02±0.00	0.01±0.00
Total		100.00	100.00

Fig. 6 SEM analysis of a sample with Al:Mg:SiO2=0.2:2:1 ratio

7. Conclusion

From the conducted research, it can be concluded that the "Jerdanak" mine, located in the Surkhandarya region can be used as an effective raw material for obtaining silicon due to the high content of silica in quartz sands. In this study, technical silicon was obtained as a result of the enrichment of quartz sands of the "Jerdanak" mine and the reaction with magnesium metal, a mixture of magnesium and aluminum metals. The highest purity was achieved at 800°C for 6 hours. The yield of the reaction and the purity of the extracted silicon increased up to 800°C. At a temperature above 800°C, the release of silicon decreased as the temperature increased due to the reaction of the formed silicon with excess magnesium. It was determined that the optimal temperature for obtaining technical silicon by magnoalluminothermic method is 1800°C.

Acknowledgment

Authors thank Termez State University for supporting this research work.

Funding

The research was supported by Termez State University, Termez, Uzbekistan.

Disclosure statement

We hereby confirm that all the figures and tables in the manuscript are ours.

Ethical Clearance: The project was approved by the local ethical committee at the Termez State University.

Authors' Contribution Statement

Jiyanova S.I: Conducted the drafting, Turaev Kh.Kh: Completed the conception, design, and drafting; Toshkulov A.Kh: responsible for the acquisition of data. Eshmurodov Kh.

E: data interpretation. Nabiev D.A: Participated in the conception, design, and drafting. Jiyanova S.I: Took part in revision and proofreading.

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