Extraction of Pure Silicon from Tiger River Sediments in Iraq by using Pure Aluminum As Reducing Agent

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Abstract: Year after year, the sediments of the Tigris River in Iraq are increasing, causing the river clogs, which requires the removal of these sediments from time to time. Unfortunately, they do not benefit from these deposits, although they contain high levels of silica, which concentrations were between 59 - 66%. Pure aluminum powder mixed with sediments at different ratio (1:1, 1.5:1 and 2:1) sediments/aluminum. The reducing temperatures were 900, 950 and 1000 °C. leaching process was used to purified the silica. This process was carried out by using different concentrations (3, 4 and 5)M of sulfuric acid to obtain silicon element. The purity of produced silicon reached to 98.9% with of extraction efficiency more than (88%).

1. Introduction
The sediments are formed on the banks of the rivers and at riverbed as a result of drift of the soil with the movement of water, in addition to the effect of streams and small rivers flowing into the main river [1]. The sediments are taken from the banks of Tigris river which contain a various compounds such as (SiO2, CaO, MgO, Fe2O3, TiO2, Al2O3, P2O5 and K2O) at different concentrations. The source of sediments in Tigris River come from several areas, The most important one as Anatolia Plateau in Turkey: Also Tigris tributaries continue as the source of sediments in the Tigris river in Iraqi territory such Khabur, Great zab, Little zab, Diyala river and Adhaim River [1]. The Sediments vary in Tigris river depending on the quality of the water and affected by different pollutants and type of soil and effect of Dayala river on these sediments [2,3,4].
The synthesis of high purity silicon from natural substances has been performed by traditional technology, using coke, which acts as the reducing agent. In the inner hot zone of the furnace where liquid silicon is produced and where the temperature range is 1900 to 2100 °C using metals like Mg, Ca, Al as reducing agents [5,6]. The thermodynamically, silicon forms an oxide of intermediate stability as can be seen from the Ellingham diagram (Figure 1). A number of metallic oxides eg. (Ca, Li Mg, Al, Ba, Zn, Ti and all the rare earth elements) are more stable than silica (SiO2). The elements of the oxides whose curve lies below that of SiO2 should thermodynamically be feasible to reduce silica to silicon at lower reducing temperature comparing with using coke as reducing agent.
Sadique[7] discussed a new approach for the generation of high purity silicon from silica fume (SF), These include purification of SF, reduction bymagnesium. Reduction was carried out successfully in a sealed reaction chamber with varying Mg/SF ratios, temperature and time. Mg/SF ratio, temperature and time affected the production of silicon from SF with the intermediate compounds Mg2Si and Mg2SiO4 also forming. Suitable reduction conditions were found to be within the temperature range 750-850°C and at approximately 2:1 ratio of Mg/SF.
Jehad & Jasim[8] studied the extraction of silicon with high purity of 99% by using Iraqi starting materials (quartzite rock, plant coal) via electric arc furnace at 1500°C.
Figure 1 shows Ellingham diagram.

Mishra et al. [9] produced Polycrystalline silicon from amorphous silica obtained from rice-husk white ash by using calcium at the reduction temperature equaled to 720°C. The final purity of silicon was 99.9% after acid leaching with concentrated HNO3 and HF. Kingsley Kweku Larbi [10] investigated metallothermic reduction of purified Rice Husk Ash (RHA) with magnesium within the temperature range of 500-950°C. The effects of temperature, magnesium amount and leaching agents optimized in this work.

The present work aims to extract Pure Silicon from Tiger River Sediments in Iraq by using Pure Aluminum As Reducing Agent to clean the environment from them and to utilize produced pure silicon, in many engineering applications like the fabrication of most commercially available solar cells, in waterproofing treatments, molding compounds, mold-release agents, mechanical seals, high temperature greases and waxes. Silicon is a component of some super alloys like ferrosilicon or silicon-calcium alloys, electrical steel, aluminum-silicon alloys (slumming alloys), which use in the automotive industry. Silicon is important to human health for their nail, hair, bone, skin tissues and in breast implants.
2. Experimental work

2.1 Sampling preparations

The specimens were taken from three locations at Tigris river in Baghdad City. The first is at the entrance to Baghdad City (Al-Muthanna Bridge), the second in central Baghdad (Adhamiya) and the third at the end of Baghdad (Diyala Jisr). The specimens were taken from the banks of the Tigris river at 1.5 m from the edge of the water and a depth of 2 m at three different locations along the Tigris River within Baghdad city, which are:

A. Al Muthanna Bridge
B. Al-Adhamiyah
C. Jisr Diyala

The samples were taken by 10 kg from each mentioned location, in depth of 2 m. These samples were taken using steel-mold. The samples then dried at 100°C for 4 hrs. This process was achieved by using a bender oven.

2.2 Chemical analysis

Chemical analysis of the river-sediments was carried out by using X-Ray Fluorescence (XRF) at the University of Baghdad / Iraqi as shown in tables (1, 2 and 3). In exception for SiO₂, all other compounds present in the sediments may be considered as impurities and it was also known that the color of sediments was attributed to the presence of these impurities.

**Table 1** X-Ray fluorescence (XRF) at the Al-Muthanna Bridge

| Compounds | Concentration % |
|-----------|----------------|
| SiO₂      | 59.974         |
| CaO       | 20.421         |
| Al₂O₃     | 8.035          |
| Fe₂O₃     | 4.94           |
| K₂O       | 1.447          |
| P₂O₅      | 0.702          |
| MgO       | 3.255          |
| TiO₂      | 0.739          |
Table 2 X-Ray fluorescence (XRF) at Al-Adhamiya Location

| Compounds | Concentration % |
|-----------|-----------------|
| SiO₂      | 59.29           |
| CaO       | 18.17           |
| Al₂O₃     | 8.739           |
| Fe₂O₃     | 5.848           |
| K₂O       | 1.523           |
| P₂O₅      | 0.688           |
| MgO       | 3.835           |
| TiO₂      | 0.752           |
| Others    | 0.165           |

Table 3 X-Ray fluorescence (XRF) at Jisr Diyala Location

| Compounds | Concentration % |
|-----------|-----------------|
| SiO₂      | 66.007          |
| CaO       | 16.26           |
| Al₂O₃     | 7.70            |
| Fe₂O₃     | 4.24            |
| K₂O       | 1.400           |
| P₂O₅      | 0.702           |
| MgO       | 2.54            |
| TiO₂      | 0.662           |
| Other     | 0.078           |

2.3 Grinding and sieving processes

These processes were accomplished to obtain particles with size equal to 63 μm by using Ball mill machine for grinding time of 1 hr. The speed of the ball milling was 300 rpm. Auto sieve shaker device at a specified range 53-710 μm was used for grinding process.
2.4 Washing process (primary leaching)

The samples are washing by using hydrochloric acid at 10% concentration for removal some metal oxides such as \((\text{MgO}, \text{CaO})\) and increases the silica ratio \([11,12]\). This process was done by a magnetic stirrer device for 80 °C at 2 hrs with a rotation speed of 500 rpm and solid – liquid ratio (1-4). After each experience the sample was filtered and dried in oven at 100 °C for 2 hr. The following equations illustrate the process of dissolving some of the impurities \([13,14]\).

\[
\text{CaO} + 2\text{HCl} = \text{CaCl}_2 + \text{H}_2\text{O} \quad (1)
\]
\[
\text{MgO} + 2\text{HCl}= \text{MgCl}_2 +\text{H}_2\text{O} \quad (2)
\]

2.5 Mixing and Pressing Processes

Different ratio of Al/Sediments were used. These ratio are \((1:1, 1.5:1, 2:1)\) the specimens were compressed using compression a die with diameter of 3 cm, the applied load was 20 ton for 1 min.

2.5 Reduction Process

The reduction process was carried out in carbolite furnace in university of technology at temperatures 900, 950, 1000 °C for 2 hrs. The specimens were tightly covers in steel crucible for dimensions \((11 \text{ cm diameter}, \text{ and } 15 \text{ cm length})\) to full the crucible. The cover was supplied with value allow for following for argon gas flow. All specimens were tested by atomic absorption spectrometry, X-ray in university of Baghdad. Diffraction Analysis test was done onetime for each reduction temperature.

2.6 Leaching process

The leaching process was done to remove impurities in the reduction process by using sulfuric acid at 3, 4 and 5 M using 100 °C as leaching temperature for 4 hrs and solid to liquid ratio of 1:6 via using Heating mantle device. The insoluble residue were separated from solution by vacuum pump using filtration paper. Than the filtered cake was washed with distilled water for several times and then dried in an oven at 100 °C for 1 hr. The purity of extracted silicon powder was tested by using X-ray fluorescence-XRF in university of Baghdad.

3.0 Results and discussion

3.1 Washing process (Primary leaching)

Tables \((4, 5 \text{ and } 6)\) show the increase of silica percentage in samples and decrease the percentage of others compounds after Primary leaching process according X-ray fluorescence analysis compared with tables \((1,2 \text{ and } 3)\)
Table 4 X-Ray fluorescence (XRF) at the Al-Muthanna Bridge location

| Compounds | Concentration % |
|-----------|-----------------|
| SiO₂      | 70.132          |
| CaO       | 13.251          |
| Al₂O₃     | 9.522           |
| Fe₂O₃     | 2.651           |
| K₂O       | 0.923           |
| P₂O₅      | 0.632           |
| MgO       | 1.554           |
| TiO₂      | 0.519           |
| Other     | 0.816           |

Table 5 X-Ray fluorescence (XRF) at the Al-Adhamiyah Location

| Compounds | Concentration % |
|-----------|-----------------|
| SiO₂      | 68.241          |
| CaO       | 11.216          |
| Al₂O₃     | 11.631          |
| Fe₂O₃     | 3.642           |
| K₂O       | 0.989           |
| P₂O₅      | 0.423           |
| MgO       | 2.415           |
| TiO₂      | 0.514           |
| Others    | 0.929           |
Table 6  X-Ray fluorescence (XRF) at the Jisr Diyala Location

| Compounds | Concentration % |
|-----------|-----------------|
| SiO₂      | 76.500          |
| CaO       | 8.800           |
| Al₂O₃     | 10.415          |
| Fe₂O₃     | 2.145           |
| K₂O       | 0.478           |
| P₂O₅      | 0.315           |
| MgO       | 0.918           |
| TiO₂      | 0.211           |
| Other     | 0.218           |

3.2 Reduction Process

The reduction process by mixing aluminum powder with sediments was carried out to obtain the silicon element from sediments in Tigris river. The effect of Al powder is to decrease the reduction temperature of silica. The compounds present in the products at different temperature were Si and some traces like Al₂O₃, and Al non-react. In addition to small amount of non-reacted silica as shown in tables (7, 8 and 9). The mixing aluminum powder with silica in reduction process, Al reacts with silica to produce directly Si element and alumina (Al₂O₃), because the aluminum element is a more active metal than silicon, according to the following reaction (3)[12,13]. The corresponding X-Ray diffraction analysis (XRD) was shown in Figure(2).

\[
3\text{SiO}_2 + 4\text{Al} = 2\text{Al}_2\text{O}_3 + 3\text{Si}
\]  

(3)

Figure 2  XRD pattern of reduction products at 1000°C and ratio of Al/sediments 1:1.
1. Effect of Al/sediments ratio in reduction process at different temperature

Tables (7, 8 and 9) show the amount of Al powder added to sediments at referent mixing ratio 2:1, 1.5:1 and 1:1 and different temperature 900, 950, 1000 °C. It was shown in an introduction that the reactive metals Mg, Ca, Al and Ti are thermodynamically favourable to reduce SiO2 at comparatively lower temperature and forming a mixture of condensed phase products.

**Table 7** Effect of Al/sediments ratio in reduction process at different temperature at the Al Muthanna Bridge location

| Particle size µm | Mixing ratio | temperature°C | Si%  | SiO2% | Al%  | Al2O3% | CaO%  | Others% |
|------------------|--------------|---------------|------|-------|------|--------|-------|---------|
| 63               | 1:1          | 900           | 18.2 | 0.9   | 32.5 | 42.2   | 5.3   | 0.9     |
|                  |              | 950           | 20.9 | 0.7   | 31.5 | 40.2   | 6.1   | 0.6     |
|                  |              | 1000          | 24.8 | 0.4   | 32.9 | 38.1   | 3.1   | 0.7     |
| 63               | 1.5:1        | 900           | 13.5 | 2.7   | 43.5 | 32.05  | 7.3   | 0.9     |
|                  |              | 950           | 14.9 | 1.5   | 44.1 | 35.9   | 7.1   | 1.1     |
|                  |              | 1000          | 16.1 | 1.2   | 43.1 | 30.6   | 6.8   | 2.2     |
| 63               | 2:1          | 900           | 11.2 | 3.2   | 40.2 | 34.4   | 9.2   | 1.8     |
|                  |              | 950           | 13.4 | 2.7   | 41.8 | 33.1   | 7.8   | 1.2     |
|                  |              | 1000          | 13.7 | 1.7   | 40.5 | 38.1   | 5.4   | 0.6     |
| 63               | 2.5:1        | 650           | 7.8  | 5.1   | 51.3 | 25.8   | 7.9   | 2.1     |
|                  |              | 1000          | 17.5 | 2.7   | 39.2 | 31.6   | 6.7   | 2.3     |
| 63               | 3:1          | 650           | 7.2  | 6.5   | 49.4 | 27.3   | 7.1   | 2.5     |
|                  |              | 1000          | 14.4 | 4.7   | 38.1 | 35.2   | 5.7   | 1.9     |
Table 8 Effect of Al/sediments ratio in reduction process at different temperature at the Al-Adhamiyah Location

| Particle size µm | Mixing ratio | temperature°C | Si%  | SiO₂% | Al%  | Al₂O₃% | CaO%  | Others% |
|-----------------|--------------|----------------|------|-------|------|--------|-------|---------|
| 63              | 1:1          | 900            | 20.7 | 1.2   | 31.2 | 41.9   | 4.1   | 0.9     |
|                 |              | 950            | 22.1 | 0.8   | 32.6 | 41.5   | 2.4   | 0.6     |
|                 |              | 1000           | 25.3 | 1.9   | 29.1 | 38.9   | 3.8   | 1       |
| 1.5:1           |              | 900            | 16.4 | 1.6   | 41.4 | 35.6   | 3.2   | 0.9     |
|                 |              | 950            | 14.8 | 1.5   | 46.8 | 32.6   | 2.7   | 1.6     |
|                 |              | 1000           | 18.2 | 0.9   | 35.1 | 40.3   | 4.1   | 1.4     |
| 63              | 2:1          | 900            | 14.1 | 2.7   | 35.3 | 37.6   | 7.9   | 2.4     |
|                 |              | 950            | 15.3 | 2.9   | 38.1 | 40.1   | 3.1   | 0.5     |
|                 |              | 1000           | 15.6 | 2.4   | 37.4 | 38.2   | 4.9   | 1.5     |
| 63              | 2.5:1        | 650            | 8.1  | 4.3   | 49.3 | 26.6   | 8.1   | 3.6     |
|                 |              | 1000           | 16.1 | 2.8   | 41.1 | 30.5   | 6.9   | 2.6     |
| 63              | 3:1          | 650            | 7.8  | 4.4   | 46.8 | 29.2   | 7.9   | 3.9     |
|                 |              | 1000           | 13.7 | 2.5   | 39.9 | 33.6   | 6.1   | 4.2     |

Tables (7 , 8 and 9) show that the amount of Al addition have large effect on the reduction of silica Located in sediments. It had been shown that at 1000 °C using ratio 1:1 Al/Sediments, Si appears very high amount in three location (24.8, 25.3, and 26.1) %, whereas at 1000 °C ratio with a 2:1 appears small amount in three location (13.7, 15.6, and 13.5)%. As a result of atomic absorption spectrometry and XRD analysis the percentage of extracted Silicon increase when mixing ratio decrease into 1:1 at constant temperature 1000 °C [7,15].

Sadique[7]:explain when mixing aluminum or magnesium powder with silica in reduction process, Al or Mg can reacts with Silica to produce Si element and alumina compound (Al₂O₃), because these elements is a more active metal than silicon in elligham diagram (ΔG of aluminum or magnesium ≥ ΔG of silicon). Sadique extracted high purity silicon (99.1% purity ) from waste of silica fume (SF) by used magnesium element to reduction of silica (SiO₂).

Figure (3) shows Si content as a function of Al/sediments. The maximum amount of Si is achieved at mixing ratio1:1. While the Si yield decrease at mixing ratio 2:1 because non-reaction all silica (SiO₂) with Al powder.
Table 9  Effect of Al/sediments ratio in reduction process at different temperature at the Jisr Diyala Location

| Particle size µm | Mixing ratio | temperature°C | Si%  | SiO₂% | Al%  | Al₂O₃% | CaO% | Others% |
|------------------|--------------|----------------|------|-------|------|---------|------|---------|
|                  |              | 900            | 19.6 | 1.6   | 35.3 | 36.1    | 5.5  | 1.9     |
| 63               | 1:1          | 950            | 23.4 | 1.2   | 32.6 | 33.4    | 6.8  | 2.6     |
|                  |              | 1000           | 26.1 | 2.4   | 26.9 | 36.8    | 6.1  | 1.7     |
| 63               | 1.5:1        | 900            | 15.1 | 2.1   | 31.2 | 40.6    | 8.2  | 0.9     |
|                  |              | 950            | 16.8 | 1.5   | 35.7 | 37.8    | 6.5  | 1.7     |
|                  |              | 1000           | 17.2 | 1.1   | 30.3 | 43.7    | 6.4  | 1.3     |
| 63               | 2:1          | 900            | 12.5 | 1.8   | 43.5 | 31.1    | 8.3  | 2.8     |
|                  |              | 950            | 13.7 | 2.8   | 40.9 | 32.8    | 9.2  | 1.8     |
|                  |              | 1000           | 13.5 | 1.3   | 41.5 | 34.4    | 5.4  | 3.9     |
| 63               | 2.5:1        | 650            | 8.1  | 5.3   | 48.3 | 27.8    | 8.9  | 1.6     |
|                  |              | 1000           | 16.9 | 3.7   | 44.2 | 25.1    | 7.7  | 2.4     |
| 63               | 3:1          | 650            | 7.6  | 4.9   | 49.4 | 26.2    | 9.1  | 2.8     |
|                  |              | 1000           | 15.4 | 4.7   | 43.9 | 26.9    | 7.2  | 1.9     |

Figure 3 Shows the effect of Al/sediments ratio on the silicon yield at three different locations
2. Effect of temperature in reduction process

As shown from Tables (7,8,9) that the effect of the temperature on the reduction process of SiO₂ at constant ratio of Al/SiO₂ (1:1). According to atomic absorption spectrometry and XRD analysis at 900°C appears silicon amount in three location (19.6, 20.7, and 19.6). whereas at high temperature of 1000°C the silicon amount increases (24.8, 25.3, and 26.1). As a result of atomic absorption spectrometry the amount of Si increase when the temperature increase into 1000°C at constant mixing ratio (1:1)[7].

Figure (4) shows Si content as a function of temperature. The maximum amount of Si is achieved 1000 °C. While the Si yield decrease at 900 °C.

![Figure 4](image)

**Figure 4** shows the effect of temperature on silicon percentage at three different locations.

3.3 Evaluation of silicon amount production

The extraction of silicon process was evaluated, since the main purpose of the reduction experiments is to achieve the highest amount of Si with high purity. The best condition for this process to obtain the maximum amount of Si 26.1 % with high purity 98.9% is achieved at Al/sediments ratio of 1:1 at 1000°C.

3.4 Hydrometallurgical Process

Hydrometallurgy (leaching process) is a method for obtaining metals from their ores via using different solutions. the Pyrometallurgical process for extraction of silicon which was made at different conditions led to production of various compounds in the reduction process such Al₂O₃.

For the purification of the Produced silicon, the leaching processes were used. Table (10) shows the purification of silicon produced and effect of sulfuric acid on dissolving impurities in the samples and figure (5) shows the EDX spectra of the silicon powder. Angus L. Daniels [16] and Paween Numluk
[17] explained that the aluminum oxide (alumina) react with sulfuric acid at different concentration (3–5)M and forming aluminum sulfate according to following equation (5) at 100 °C for 4 hrs, The efficiency of silicon extracted from sediments of Tigris river is greater than (88%).

\[
\text{Al}_2\text{O}_3\cdot\text{H}_2\text{O} + 3\text{H}_2\text{SO}_4 = \text{Al}_2(\text{SO}_4)_3 + 6\text{H}_2\text{O}
\]  

(4)

| Table 10 | XRF analysis of purification of silicon |
|----------|---------------------------------------|
| Samples  | Concentration | Temperature | Time | Si% | Si_2O_3 | O_2 | % |
|          | M             | °C          | hr   |     |         |     |    |
| Al-Mudanna Bridge | 3   | 100        | 4    | 95.9| 0.3     | 3.72| 8 |
|            | 4   | 100        | 4    | 98.9| 0.3     | 0.8 | 4 |
|            | 5   | 100        | 4    | 93.2| 0.4     | 6.36| 4 |
| Adhamiyah  | 3   | 100        | 4    | 96.4| 1.4     | 2.17| 3 |
|            | 4   | 100        | 4    | 98.4| 1.5     | 0.1 | 3 |
|            | 5   | 100        | 4    | 92.7| 1.8     | 5.44| 6 |
| Jirr Diyala| 3   | 100        | 4    | 96.9| 2.0     | 1.08| 2 |
|            | 4   | 100        | 4    | 97.3| 1.9     | 0.8 | 2 |
|            | 5   | 100        | 4    | 93.6| 2.1     | 4.25| 5 |

Figure 5 EDX spectra of the as-produced silicon
The SEM micrograph of the silicon powder as shown in figure (6) and it can be seen that the shape of particles is random and different size for 4 hrs of leaching time, 100 °C of temperature and (4M) concentration of sulfuric acid. Some identical forms of pure silicon molecules were produced in this work are also obtained in works [6&10].

![SEM micrograph of as-produced silicon](image)

**Figure 6** The SEM micrograph of as-produced silicon

The particle size distributions of silicon powder can be seen in Figure (7) shows that approximately 90 % volume of the produced silicon is ≤ 66 μm, 50% of the produced silicon volume ≤ 22μm and 10% volume of the produced silicon is ≤ 5μm.
4. Conclusions

1. Sediments of Iraqi tiger river have been found to be rich in silica, which is more than 60%. Therefore, it could be inferred that tiger sediments have the potential to be used as natural raw substances for preparing silicon.

2. Silicon has been successfully prepared from tiger river sediments as raw materials using metallothermal reduction process. The reduction of silica was carried out with aluminum powder (63 µm particle size) aluminum as the reducing agent. Different ratio of sediments / Al (2:1, 1.5:1 and 1:1) the best results obtained at ratio 1:1. to get pure silicon with high deposited weight and high purity.

3. The best reduction temperature was found at (1:1) mixing ratio of Al/Sediments was 1000˚C. The efficiency of extracted silicon from sediments of Tigris river is greater than (88%). with silicon purity about (98%).

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