Nanostructured silicon production from quartzite ore by low-energy wet blending of the reagents, reduction in controlled atmosphere, and hydrometallurgy

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**Abstract**

High-purity porous nanostructured silicon particles were successfully produced from quartzite rock via the multi-step processing route including primary acid leaching of crashed quartzite feedstock, wet blending of quartzite and magnesium powder, reduction, and final multi-stage hydrometallurgical purification of the products. Laboratory-grade silica was also treated for comparison. The effect of silica purity, reactants’ molar ratios (Mg:SiO\(_2\)), and hydrometallurgical refining on reaction products were investigated and discussed through the results of X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM) equipped with energy dispersive spectroscopy (EDS) and inductively coupled plasma-mass spectrometry (ICP-MS). The results indicated that the combination of primary acid treatment, combustion synthesis in controlled atmosphere, and final special acid leaching process is an efficient route for the production of porous nanostructured elemental silicon particles with a uniform structure that can be used in several applications in the energy sector with or without further processing.

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**Keywords:**
- Porous nanostructured silicon particles
- Reduction by magnesium
- Hydrometallurgical treatment
- Quartzite rocks

1. **Introduction**

Energy and environmental issues will be the most important future problems of humankind. The major source of energy comes from fossil fuels which are not only the cheapest but also the most contaminant and diminishing of all sources. Alternative energy resources are rather diverse, but their widespread application is hindered by technological and economic issues. Elemental pure silicon is an important material in alternative energy technologies and can be used in three major fields of application: photovoltaic (PV) power panels which are manufactured using mono- and multicrystalline pure silicon as the base material [1]; anodes for...
lithium-ion batteries which are usually made of carbon, but recently porous pure silicon has been shown to be a promising replacement due to high theoretical capacity, electrochemical cycle stability, and low electrochemical voltage [2–4]; and as secondary energy carrier [5].

The classification of industrial silicon is based on purity level: metallurgical grade (MG, >98% purity), solar grade (SG, at least 5 N purity), and electronic grade (EG, at least 9 N purity). The generally accepted purity level of SG silicon is 5–7 N with P and B levels reduced to less than 1 part per million (ppm) [1,6–8]. MG silicon (also called raw silicon or silicon metal) is generally produced by the carbothermic reduction of quartzite rocks in a submerged arc furnace (SAF) and is the main source of silicon for different industries. Aluminum casting industries are the major consumer of MG silicon (about 55%). There are several methods for purification of MG silicon to EG and SG purity levels: (a) metallurgical approaches that include the metallothermic reduction of silicon halide (using zinc, aluminum, and alkali/alkaline earth metals as reductants) and halothermic reduction (reduction by aluminum subchloride) [9–13]; (b) chemical approaches (Siemens-based processes) [1,7,14–16]; (c) electrochemical reduction [17,18]; and (d) refinement of MG silicon by acid leaching, slag process, electron-beam melting, and solidification [19–22].

Although the prevalent technology – Siemens and allied processes – are quite mature, several drawbacks such as cost, complexity, environmentally hazardousness, and the purity level of the product (which is much more than the level required for PV cell manufacturing) have made them inappropriate for PV cell industry [1,7,15]. Metallothermic reduction processes have some issues too, such as difficulty in the removal of Zn/Al remaining as a solid solution, difficulty in silicon/salt separation, and formation of intermetallic compounds [13].

In the acid leaching process, MG silicon is crushed to nano-size and leached with acid. In the slag process, melted MG silicon is covered with a slag that dissolves the impurities better than melted silicon does. In the electron-beam method, the beam energy is high enough to evaporate the impurity elements and a blowing oxygen stream converts the evaporated atoms to oxide while transferring them from the silicon stock. In the solidification method, MG silicon is alloyed with melted Al or some other elements and the resulting alloy is then cooled to the mushy zone where solidified, floated silicon-rich crystals are gathered from the melt. The main problem of refinement methods is the difficulty of obtaining high-purity silicon [13,16,23,24]. The purity level of B and P is the main problem in these methods and they are normally coupled with Czochralski, Bridgman, or zone-refining methods to achieve the desired purity level [1,7,15]. Using other reductants rather than carbon to directly reduce silica has been the subject of some studies [3,13,25–34]. Ca, Al, and Mg can reduce SiO2 due to the higher stability of CaO, Al2O3, and MgO in comparison to SiO2. Among these reactions, the magnesiothermic reaction is highly exothermic and can propagate in a self-sustainable mode called combustion synthesis (CS). CS is a process which uses the exothermic reaction of reactants, properly ignited to spontaneously transform into products. CS is subdivided into two main modes: volume combustion synthesis (VCS) and self-propagating high-temperature synthesis (SHS). In the VCS mode, oxide and reductant powders are mixed and/or cold-pressed and heated to the combustion temperature. The whole powder pack transforms simultaneously as the CS temperature is reached. In the SHS mode, pressed powder is ignited from one side and the reaction propagates over the whole sample volume as a solid flame wave. The main benefits of the combustion synthesis processes are: low processing cost, low emission, low energy consumption, short processing time, and the higher purity level of the product [35,36]. There are some reports on the magnesiothermic production of silicon, mostly in the VCS mode [27,37–43]. Eliminating reaction by-products is the main challenge in producing silicon by alumino- and magnesiothermic combustion synthesis processes. Al compounds are highly stable and their elimination process is very difficult and expensive. Meanwhile, magnesium by-products can be easily eliminated by acid leaching [3,27,34,41,42,44,45].

In the present research, high-purity nanostructured silicon particles were produced by magnesiothermic reduction in the SHS mode. The blending of reactant powders was carried out in a wet medium because of the reactive nature of Mg. The aim of the research was to study the response of different silica sources to the magnesiothermic CS and subsequent acid leaching, as well as determining the suitability of the wet blending method for the CS process.

2. Materials and methods

Two sources of silica were explored in this research: laboratory-grade silica (LS) (d50 = 15 μm, 99% purity, Fluka) and mineral silica ore (MS) from Hamadan region quartzite mines, Iran. LS was used as-received and MS was mechanically ground to <1 mm in diameter and subsequently ball-milled and sieved. In order to remove some parts of impurities and non-silica oxides – that are naturally abundant in quartzite ore – and increase the yield of the subsequent CS process, the material was pre-treated by acid leaching which has been shown effective for obtaining higher-quality silica [30,34,46,47]. The optimum route leading to the highest purity of the product was selected as described as follows: First, 40 g of MS was leached with 400 mL of hydrochloric acid solution (2 M) for 4 h at 70 °C. The product was filtrated by #9 Whatman filter paper, washed several times with deionized water, and dried in an oven for 4 h at 105 °C. High-purity magnesium powder (<100 μm, 97% purity, Merck) was used as the reductant. Powders with various Mg-to-silica molar ratios of 1, 1.5, 2 (stoichiometric), and 2.5 were weighed and mixed together in benzene (99.99%, Merck) as the liquid agent on a heater/magnetic stirrer at 70 °C. The blend was dried in an oven at 120 °C for 6 h and cold pressed under 100 MPa pressure for 15 s to the final cylindrical samples (20 mm in diameter, 20 mm in height) ready for the combustion synthesis process. Combustion synthesis was conducted inside a sealed stainless steel chamber under a flowing argon atmosphere (15 L/min). The chamber was thoroughly purged with argon for 2 min and the argon flow maintained during combustion and the subsequent cooling down to room temperature. The samples were ignited at one end by electric arc using a TIG (Tungsten Inert Gas) welding torch. Acid leaching was utilized to eliminate the
by-products as well as the elements that are proven to be deleterious to the performance of solar cells. By comparing similar studies in the literature [13,25–27,30,32,39,48–52], three successive passes of acid leaching were carried out on the powder right after hand-milling in a mortar: (a) leaching in 2 M HCl for 1 h at 60 °C, (b) leaching in a solution of 1.25 M HCl + 4.38 M CH₃COOH mixture (4:1) for 1 h at 60 °C, and (c) leaching in a solution of 2.8 M HF + 4.38 M CH₃COOH mixture (1:9) for 2 h at 70 °C. Between stages, the leached slurry was passed through #9 Whatman filter paper and the residue was washed out and dried for the next step. A schematic diagram of the preparation process is shown in Fig. 1.

The final residue was subjected to scanning electron microscopy (SEM), inductively coupled plasma mass spectrometry (ICP-MS), and X-ray diffraction (XRD). Average crystallite sizes (D) were determined from XRD peaks using Scherrer’s formula (Eq. (1)) [53] where K is the shape coefficient (value between 0.9 and 1.0), λ is the wavelength, β is the full width at half-maximum (FWHM) of each phase, and θ is the diffraction angle.

\[
D_{hkI} = \frac{K \lambda}{\beta \cos \theta}
\]  

(1)

Moreover, the quantitative analysis was performed using Rietveld refinement with Maud software. FE-SEM micrographs were obtained using a VEGA MIRA3 TESCAN tool equipped with an energy dispersive spectroscopy (EDS) detector. XRD was carried out with a Bruker D8 XRD system using Cu-Kα radiation (λ = 1.5405 Å) with a nickel filter, Bragg-Brentano geometry, the 2-theta range of 10–90 degree, and step size of 0.05 degrees⁻¹. A Perking-Elmer Elan6000 DRC instrument was utilized for ICP-MS. Sample preparation for ICP-MS measurements was carried out as follows: First, 0.1 g of acid-leached product was digested in 15 mL of the 1:1:1 solution of deionized water, 17 M acetic acid, and 28 M hydrofluoric acid.
showed an irregular shape, characteristic of mechanically grinded powders. Image analysis on SEM microphotographs led to the estimation of the particle size (d50) as ≈ 7 µm. Fig. 3 presents the SEM micrograph of the magnesium powder, suggesting that the final manufacturing step was mechanical grinding. The d50 of the powder was measured as ≈30 µm.

The XRD patterns of LS and MS powder are illustrated in Fig. 4a and b, respectively. MS contained 14 wt% complex Mg, Al, Fe, and Si oxides, while LS was quite pure. ICP-MS tests were carried out on LS and MS to find the concentration of the most negatively affecting elements on solar PV performance: Fe, Al, Ba, Mg, Mn, Ni, Ca, K, B, and Zn. The results are presented in Table 1. To obtain higher-purity silicon by combustion synthesis itself, it would be beneficial to use silica with higher purity. Acid leaching is one of the most effective methods to purify minerals [46]. Some impurities such as Al, K, Fe, Ti, Ca, Mg, and Na that generally exist as oxides in mineral silica microstructure – depending on the origin of the ore deposit – can be eliminated through a series of acid leaching steps. The selection of appropriate acid leaching parameters (type of acids, time of leaching, temperature, and sequences) depends on the oxide type, the purity level needed, and economic issues. Details of the acid leaching procedure carried out on MS were described in the previous section.

The XRD pattern of acid-leached MS is shown in Fig. 4c. One can see that the concentration of impurities has been lowered below the detection limits of the test method. The SEM micrographs of MS particles after primary acid leaching, along with the EDS point analysis results of raw and leached silica, are depicted in Fig. 5. As the semi-quantitative analysis shows, treatment with the described one-step acid leaching led to a significant decrease in impurity level. ICP-MS results of the leached sample are listed in Table 1. These results also show the effectiveness of acid leaching parameters. As also stated by other researchers, leaching by high-acidic solutions is a simple and yet quite efficacious in Al, Fe, Ni, Zn, Ca, Ba, Mn, and Mg removal, and Ba can be eliminated in both basic and acidic solutions [46]. As shown in Table 1, K is moderately eliminated by the leaching process.

3. Result and discussion

SEM micrographs of silica from two different sources are depicted in Fig. 2. Particles in the LS sample were angular-shaped. As stated by the manufacturer, these particles were produced through acid leaching of silica sands. MS particles for 24 h. The solution was initially heated to 50 °C and, after digestion, diluted by 2 vol.% acetic acid to 50 mL. The blank solution was prepared by the same ratios of constituents and the same dilution method.

3.1. Combustion synthesis

Wet-blended and cold-compacted powders with different Mg silica ratios were ignited by a TIG torch in an argon-protected chamber. Laboratory silica (LS) was processed at first in order to determine the optimum Mg/SiO2 ratio leading to suitable reaction products for the following leaching steps. In contrast to the results of a previous report [27] which is about the combustion synthesis of ball-milled Mg/SiO2 powder, the combustion in experiments took place very rapidly and the velocity of the combustion wave was very high. The combustion velocity was calculated from the v = x/t equation (where t is the time of combustion process and x is the height of cylindrical sample) and shown in Table 2. Diluting the mixture with neutral compounds could help, but it was out of the scope of the present research.

The adiabatic temperature (T_a) of the mixtures as calculated by Thermo Software by [27] was in the range of 1800 to 2000 °C with a semi-linear relationship with Mg content. [27] Also, the same trend in the experimentally measured
combustion temperature ($T_c$) of different Mg/SiO$_2$ mixtures to be between 1200 to 1700 °C. The expected reaction products here were Si, MgO, Mg$_2$Si, Mg$_2$SiO$_4$ and residual SiO$_2$, also confirmed by software simulation [30,43]. According to the following reactions, Mg vaporized as the local high temperature created by the arc. The exothermic reaction between vapor Mg and SiO$_2$ created enough heat to overcome the activation energy of Mg sublimation ahead of the reaction front, and this reaction cycle was repeated until reaching the end of the sample [2,27,29,31,38,44,53]:

$$\text{Mg(s)} = \text{Mg(g)}, \Delta H = 147 \text{ kJ/mol} \quad (2)$$

$$\text{SiO}_2(\text{s}) + 2\text{Mg(g)} = \text{Si(s)} + 2\text{MgO(s)}, \Delta H = -586 \text{ kJ/mol} \quad (3)$$

$$2\text{Mg(g)} + \text{Si(s)} = \text{Mg}_2\text{Si(s)}, \Delta H = -373 \text{ kJ/mol} \quad (4)$$

Table 1 – ICP-MS results of different sources of silica.

<table>
<thead>
<tr>
<th>Element</th>
<th>Laboratory silica (ppm)</th>
<th>Mineral silica (ppm)</th>
<th>Acid leached MS (ppm)</th>
<th>Removal percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>&lt;1</td>
<td>7068</td>
<td>153</td>
<td>97.8</td>
</tr>
<tr>
<td>Ba</td>
<td>66</td>
<td>1731</td>
<td>159</td>
<td>90.8</td>
</tr>
<tr>
<td>Ca</td>
<td>242</td>
<td>4583</td>
<td>321</td>
<td>93.0</td>
</tr>
<tr>
<td>Fe</td>
<td>366</td>
<td>6924</td>
<td>530</td>
<td>92.3</td>
</tr>
<tr>
<td>K</td>
<td>558</td>
<td>536</td>
<td>480</td>
<td>10.4</td>
</tr>
<tr>
<td>Mg</td>
<td>50</td>
<td>3616</td>
<td>53</td>
<td>98.5</td>
</tr>
<tr>
<td>Mn</td>
<td>23</td>
<td>944</td>
<td>52</td>
<td>94.5</td>
</tr>
<tr>
<td>Ni</td>
<td>471</td>
<td>1227</td>
<td>252</td>
<td>79.5</td>
</tr>
<tr>
<td>Zn</td>
<td>585</td>
<td>141</td>
<td>40</td>
<td>71.6</td>
</tr>
<tr>
<td>B</td>
<td>&lt;2</td>
<td>&lt;3</td>
<td>&lt;1</td>
<td>66.7</td>
</tr>
</tbody>
</table>

Table 2 – The velocity of combustion wave in different Mg/SiO$_2$ molar ratios.

<table>
<thead>
<tr>
<th>Mg/SiO$_2$ molar ratio</th>
<th>Velocity (cm s$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.36</td>
</tr>
<tr>
<td>1.5</td>
<td>0.80</td>
</tr>
<tr>
<td>2</td>
<td>1.05</td>
</tr>
<tr>
<td>2.5</td>
<td>1.14</td>
</tr>
</tbody>
</table>

Combustion synthesis products consisted of loosely sintered powder and were hand-ground in a mortar before XRD test to obtain a fine powder. XRD results for the combustion-synthesized samples with different Mg:silica molar ratios are presented in Fig. 6 and the quantitative analysis of the results by Rietveld refinement with Maud software are summarized in Table 3. The results show that, at the substoichiometric Mg:SiO$_2$ of 1:1, there was some unreacted SiO$_2$ in the products. As Mg ratio increased to 1.5, SiO$_2$ was eliminated and Mg$_2$Si appeared. Mg$_2$Si was formed in all other mixtures, being minimum in the stoichiometric mixture (Mg/SiO$_2$ = 2). Rietveld refinement also showed that Mg$_2$Si by-products tend to decrease with Mg/SiO$_2$ equal to this stoichiometric value.

Fig. 5 – SEM micrograph of acid-leached MS along with the point analysis for a) milled MS and b) leached MS.
Fig. 6 – XRD pattern of the products of combustion-synthesized lab-purity silica (LS) and Mg mixture in different Mg:SiO₂ molar ratios of 1, 1.5, 2, and 2.5, respectively for (a), (b), (c), and (d).

Beside this, leaching of Mg₂Si was difficult and its formation reduced the efficiency of the process. Accordingly, the stoichiometric ratio was selected for the combustion synthesis of Mg and acid-leached mineral silica (MS).

The XRD pattern of combustion-synthesized Mg/MS samples with the Mg:SiO₂ of 2:1 is demonstrated in Fig. 7 and the resulting composition is listed in Table 4. There was no Mg₂Si in the products and some Mg₂SiO₄ was produced instead. The formation of the latter was based on the following reactions [30, 53]:

\[
3\text{SiO}_2(s) + 2\text{Mg}(g) = \text{Mg}_2\text{SiO}_4(s) + 2\text{SiO}(g), \quad \Delta H = -1757 \text{kJ/mol}(5)
\]

\[
4\text{SiO}(g) + 2\text{Mg}(g) = \text{Mg}_2\text{SiO}_4(s) + 3\text{Si}(s), \quad \Delta H = -2078 \text{kJ/mol}(6)
\]

Table 3 – Quantitative analysis of XRD patterns of Fig. 6 by Maud software.

<table>
<thead>
<tr>
<th>Mg:SiO₂ ratio</th>
<th>XRD quantification</th>
<th>Si</th>
<th>MgO</th>
<th>SiO₂</th>
<th>Mg₂Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>15.09</td>
<td>69.72</td>
<td>15.19</td>
<td>–</td>
</tr>
<tr>
<td>1.5</td>
<td></td>
<td>11.68</td>
<td>57.16</td>
<td>–</td>
<td>31.16</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>20.59</td>
<td>64.71</td>
<td>–</td>
<td>14.70</td>
</tr>
<tr>
<td>2.5</td>
<td></td>
<td>11.59</td>
<td>70.18</td>
<td>–</td>
<td>18.23</td>
</tr>
</tbody>
</table>

Fig. 7 – XRD pattern of the products of combustion-synthesized acid-leached MS and Mg mixture at the stoichiometric ratio.

Table 4 – Quantitative analysis of the XRD patterns of Fig. 7.

<table>
<thead>
<tr>
<th>XRD Quantification</th>
<th>Si</th>
<th>MgO</th>
<th>Mg₂Si</th>
<th>SiO₂</th>
<th>Mg₂SiO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>28.14</td>
<td>66.66</td>
<td>–</td>
<td>–</td>
<td>5.18</td>
<td></td>
</tr>
</tbody>
</table>
local elemental Mg content was not enough at the MgO/SiO₂ interface.

\[
2 \text{MgO(s)} + \text{SiO}_2(\text{s}) \rightarrow \text{Mg}_2\text{SiO}_4(\text{s})
\] (7)

3.2. Final purification

The final acid leaching process was carried out in order to achieve purer silicon toward the solar-grade silicon. From among all the advantages of combustion synthesis, its capability to eliminate some volatile impurities such as Mg, S and Na. The combustion temperature is high enough to vaporize these elements and guide them out of the powdery product. However, more cleanliness needs extra processing which, as other researchers suggest, is composed of several acid leaching stages [48–50,54].

The optimum acid leaching procedure is a three-stage process inspired by [32]. The leaching process was performed for 1–4 h at the temperature of 60–90 °C in a glass jar on a heater/magnetic stirrer using laboratory-grade hydrochloric acid (HCl), acetic acid (CH₃COOH), and hydrofluoric acid (HF) in order to omit MgO-, Mg₂Si-, and silica-containing compounds.

Fig. 8 shows the XRD pattern of the product after the first stage of acid leaching consisting of products stirring in 2 M HCl for 1 h at 60 °C. The reaction of Mg₂Si and HCl leads to silane or chlorosilane gas production [44]:

\[
\text{HCl(aq)} + \text{H}_2\text{O(aq)} = \text{H}_3\text{O}^+(\text{aq}) + \text{Cl}^- (\text{aq})
\] (8)

\[
\text{MgO(s)} + 2\text{H}_3\text{O}^+(\text{aq}) = \text{Mg}^{2+}(\text{aq}) + 3\text{H}_2\text{O}
\] (9)

\[
\text{Mg}_2\text{Si} = 2\text{Mg}^{2+}(\text{aq}) + \text{Si}^{3+}(\text{aq})
\] (10)

\[
\text{Si}^{3+}(\text{aq}) + 2\text{H}_3\text{O}^+(\text{aq}) = \text{SiH}_4 + \text{H}_2\text{O}
\] (11)

Mg₂⁺ ions, produced by the dissociation of Mg₂Si and MgO in diluted HCl, transformed to MgCl₂ which is soluble in the solution [54]:

\[
\text{Mg}^{2+}(\text{aq}) + 2\text{Cl}^- = \text{MgCl}_2(\text{aq})
\] (12)

As observed in Fig. 8, Mg₂Si and Mg₂SiO₄ were considerably decreased in both LS and MS.

In the second stage of acid leaching, the product from the first stage was treated using a mixture of 1.25 M HCl + 4.38 M acetic acid in a volumetric ratio of 4:1 for 1 h at 60 °C. Fig. 9 illustrates the XRD pattern of the product after the second acid leaching processing. The addition of acetic acid at this stage improved the solubility of the magnesium-containing compounds, particularly MgO, in acid due to the wetting effect provided by acetic acid, and the following reactions describe the process:

\[
\text{CH}_3\text{COOH} + \text{H}_2\text{O(aq)} = \text{H}_3\text{O}^+(\text{aq}) + \text{CH}_3\text{COO}^-
\] (13)

\[
\text{Mg}^{2+}(\text{aq}) + 2\text{CH}_3\text{COO}^- = (\text{CH}_3\text{COO})_2\text{Mg}(\text{aq})
\] (14)

In the third stage, to eliminate SiO₂ as the only remaining impurity and increase the final purity of the product, 4.38 M acetic acid and 2.8 M HCl mixture were used in the volumetric ratio of 9:1 for 90 min at 70 °C. The XRD pattern of the products after this stage of acid leaching is shown in Fig. 10. According to the results, it can be seen that all impure compounds such as MgO, Mg₂Si, SiO₂ and Mg₂SiO₄ from the combustion process for MS and LS samples were reduced to the detection limit of XRD analysis and the peaks of silicon were the only peaks which were identified. The crystallite size of the final
product was also calculated using Scherer’s equation and equaled 344 Å.

Scanning electron micrographs of silicon particles after three acid leaching stages are presented in Fig. 11a and b for different silica sources. By acid leaching, particles were dissolved in the acid and holes appeared on the surface of solid particles which resembled a honeycomb structure [55]. The size of these holes is directly related to Mg concentration and their depth was estimated at nearly half the diameter of particles. It can be noticed that the majority of holes were spherical. Since particles containing MgO, Mg2SiO4 and Mg2Si were formed at high temperatures, they were mainly large and rough. Therefore, they created big holes after acid leaching.

The quantitative elemental analysis was employed for a detailed survey of remaining impurities in the final product. The results of ICP-MS are shown in Table 5. By comparing the results of quantitative elemental analysis, it can be concluded that it is possible to eliminate impurities to a large extent (better than 99.8%) using the above-described hydrometallurgical treatment. This purity may not meet the levels needed for SG silicon, but it is several times better than the purity of MG silicon and fewer additional refining steps are needed when using it as feedstock in the production of SG silicon.

It is noteworthy that the nature of high-temperature combustion synthesis causes volatile elements such as Mg, Zn, S, and Na to be omitted to a large extent from the reacted sample. Other parameters such as low density of the sample and external pressure can also help the self-purification during the combustion synthesis process. This unique characteristic of the process can also lead to a gradual decline in the concentration of impurities before hydrometallurgical purification.

### Table 5 – Concentration of elements in the product after final hydrometallurgical treatment (ICP-MS).

<table>
<thead>
<tr>
<th>Element</th>
<th>Si produced from LS (ppm)</th>
<th>Si produced from MS (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>&lt;LOQ</td>
<td>&lt;LOQ</td>
</tr>
<tr>
<td>Ba</td>
<td>56</td>
<td>130</td>
</tr>
<tr>
<td>Ca</td>
<td>&lt;LOQ</td>
<td>&lt;LOQ</td>
</tr>
<tr>
<td>Fe</td>
<td>300</td>
<td>460</td>
</tr>
<tr>
<td>K</td>
<td>&lt;LOQ</td>
<td>&lt;LOQ</td>
</tr>
<tr>
<td>Mg</td>
<td>90</td>
<td>48</td>
</tr>
<tr>
<td>Mn</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>Ni</td>
<td>180</td>
<td>78</td>
</tr>
<tr>
<td>Zn</td>
<td>460</td>
<td>&lt;LOQ</td>
</tr>
<tr>
<td>B</td>
<td>&lt;LOQ</td>
<td>&lt;LOQ</td>
</tr>
</tbody>
</table>

LOQ, limit of quantification.

### 4. Conclusion

The combustion synthesis process of wet-blended silica and magnesium powder along with hydrometallurgical treatment were successfully utilized to produce high-purity silicon nanostructured particles from quartzite ore and laboratory-grade silica. Wet blending proved an effective method for mixing reactive magnesium and oxide. The proposed sequences can be used to produce pure silicon by a simple yet economical combustion synthesis process on a stoichiometric mixture of acid-leached quartzite and Mg powder and three subsequent stages of acid leaching on the powdery product. Rietveld refinement shows that the maximum reduction of SiO2 occurred in the ratio of 2 for Mg:SiO2. In addition, the crystallite size of the final product was equal to 344 Å. The mean size of the porosity of silicon particles achieved from laboratory- and metallurgy-grade silica was 180 and 90 nm, respectively. The results of ICP-MS showed that the obtained powder can be employed in several applications in the energy sector with or without further processing.

### Conflicts of interest

The authors declare no conflicts of interest.

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