Particle growth of nano-silica below the isoelectric point

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Current production of nano-silica

Nano-silica is one of the compounds that is boosting the field of nano-materials with an annual rise of 5.6 % reaching 2.8 million metric tons in 2016 and with a total value of $6.4 billion [1]. The current production methods involve steps with high temperatures. To reach these temperatures huge amounts of fuel are consumed making these processes: a) non-sustainable because of the scarcity of fuels; b) not environmentally friendly because of the huge amount of CO₂ emissions released; and c) expensive because of the fuel price.

Production of olivine nano-silica

Initial research has demonstrated that nano-silica can be produced by dissolving olivine in acid at low temperatures. The acid is neutralized by olivine mineral, according to:

\[(\text{Mg,Fe})_2\text{SiO}_4 + 4\text{H}^+ \rightarrow \text{Si(OH)}_4 + 2(\text{Mg,Fe})^{2+}\]

The neutralization yields a slurry consisting of magnesium/iron salts, silica and unreacted silicates (more details in [2, 3, 4]).

Experimental Methods

The particle growth of silica during the olivine dissolution (negative pH) was investigated using olivine. These experiments were carried out at 20 °C in a vigorously stirred reactor of two liters with sulfuric acid. The experimental variables were the concentration of sulfuric acid and surface area of olivine (see Table 1). The silica particle size (or cluster size) was measured with a Malvern ZS after passing the samples through a filter of 1 µm in order to avoid the presence of olivine particles.

Table 1. Conditions of the nano-silica polymerization experiments.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>m₀(g/L)</th>
<th>[Si]ₐₘₐₜ</th>
<th>dₜₐₜ</th>
<th>SSAG</th>
<th>SA</th>
<th>[H⁺] (mol/L)</th>
<th>T (°C)</th>
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<tr>
<td>NS-PO-2</td>
<td>50</td>
<td>307</td>
<td>116</td>
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<td>9.0</td>
<td>4.0</td>
<td>20</td>
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<td>116</td>
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<td>2.0</td>
<td>20</td>
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<td>613</td>
<td>116</td>
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<td>429</td>
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</tr>
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<td>116</td>
<td>0.09</td>
<td>12.6</td>
<td>4.1</td>
<td>20</td>
</tr>
</tbody>
</table>

[Si]ₐₘₐₜ refers to the equivalent concentration of silica from the olivine amount, SSAG geometric specific surface area, and SAₜ total geometric surface area of the olivine in the reactor.

Results

Figure 1 the concentration of silica produced during the dissolution of olivine in the polymerization experiments. Figure 2 presents the average particle size of silica over time.

The cluster size of silica increased exponentially with time. The growth rate of silica accelerated with the increase of the hydrogen ion concentration and amount of nano-silica released.

Conclusions

The cluster size of olivine silica grows up to 420 nm after 4300 minutes of reaction in 2M H₂SO₄ solution. The particle growth should be much faster under the production conditions of olivine nano-silica (90 °C and 3M H₂SO₄), which cause olivine to dissolve within 4-6 hours. Olivine nano-silica does not gel. This behavior of olivine silica can be due to three reasons:

1) silica polymerizes fast at 90 °C in 3M H₂SO₄ solution, forming particles above 100 nm in a short time;
2) the concentration of small silica colloids is low;
3) the reactor is vigorously stirred.

References