Analysis of Bauxite Fractions and Synthesis of Stable Sodium Silicate Solution

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Abstract

Caustic soda is a critical reagent in alumina refining, but its reaction with kaolinite in bauxite leads to the formation of sodium silicate, contributing to irreversible caustic losses due to sodalite formation in the Bayer process. To address this issue, this study analyses different bauxite fractions to identify high-silica fractions suitable for silica extraction and stable sodium silicate synthesis. Bauxite samples were fractionated and analysed using X-ray Fluorescence (XRF) to determine the silica-bearing fraction. Dissolution studies were conducted in process liquor to selectively leach kaolinite, while key process parameters—reaction time, temperature, and slurry concentration—were optimized to maximize silica extraction while minimizing gibbsite dissolution. The study establishes a basis for selective silica extraction and stable sodium silicate synthesis. Future research will focus on large-scale implementation and potential applications of the extracted silica in value-added products.

Keywords: Alumina refining, caustic soda consumption, bauxite fractions, silica extraction, sodium silicate synthesis.

1. Introduction

Caustic soda (NaOH) is a crucial component in the Bayer process, the main industrial method to produce alumina from bauxite. Caustic soda reacts with alumina bearing minerals like gibbsite, boehmite, diaspore to produce sodium aluminate. However, caustic also reacts with kaolinitic silica, known as reactive silica, to produce sodium silicate. Although sodium aluminate is the preferred intermediate in the production of alumina, simultaneous production of sodium silicate is undesirable as increases caustic losses and other downstream issues. One of the key problems with sodium silicate is that it can react with dissolved alumina and sodium ions to produce insoluble phases such as sodalite and cancrinite under specific process conditions. These sodium aluminosilicates tend to precipitate on the surface of heat exchangers, causing scaling, inefficiencies in energy, and periodic maintenance shutdowns. More importantly, these reactions cause irreversible caustic soda losses, which have a negative impact on the process economy and raises operating costs.

Earlier research has highlighted the negative effect of reactive silica in high-silica bauxite ores [1]. It is known that the extraction rate of silica and the formation of sodalite greatly depend on the bauxite feedstock mineralogy and particle size. Smaller fractions tend to have higher surface area and can accumulate reactive clay minerals, enhancing undesirable side reactions [2]. Pickering et al [3] have investigated different pre-treatment methods to reduce reactive silica prior to digestion, including selective leach and desilication methods, without much success in retaining caustic efficiency and product quality. In recent years, the alternative solution has been to valorise it, rather than simply remove reactive silica as waste: researchers have sought to extract and

convert it to value-added products such as stable sodium silicate solutions. Sodium silicate is used in a variety of industrial applications across detergents, adhesives, refractories, and as a feedstock in the manufacture of silica gel [4]. Yet its synthesis demands strict control of the reaction conditions like temperature, alkali concentration, and the composition of silica-bearing source, to maintain solubility and stability. Smith et al [1] suggested that size fractionation of bauxite particles can determine silica-rich components that are prone to controlled dissolution and conversion into sodium silicate under optimized hydrothermal conditions. Based on this idea, the current research explores the role of bauxite's particle size distribution in the silica behaviour during caustic leaching. It has a two-pronged objective: first to describe and separate high-silica fractions of bauxite liable for the loss of caustic soda during alumina refining, and secondly, to evaluate whether reactive silica can be extracted from such fractions for use in producing stable sodium silicate solutions. This two-way strategy is designed to alleviate the problems with reactive silica in bauxite while providing a promising route for resource valorisation in alumina refineries.

2. Methodology

2.1 Collection of Bauxite Samples

Fine bauxite samples with a particle size below 30 mm were obtained directly from the crusher feed stream of an existing alumina refinery. They are representative of the finely crushed fraction of run-of-mine (ROM) bauxite and are known to be a heterogeneous mixture of mineral phases, including gibbsite and reactive silica-bearing phases like kaolinite. After collection, the samples were air-dried under ambient laboratory temperature (~30 °C) for 48 hours to eliminate surface moisture. To dry completely and avoid agglomeration during further handling, the air-dried samples were additionally oven-dried for 12 hours at 105 °C in a hot-air oven. This procedure provided homogenous moisture content throughout all samples and reduced size classification variability. The dry bauxite was then mechanically sieved with a Ro-Tap sieve shaker (Model RX-29) following ASTM standard D6913. A set of regular stainless-steel sieves with mesh sizes of + 6.3 mm to - 75 µm were employed to separate the sample into well-defined size fractions.

Each sieving procedure involved continuous shaking and tapping for 15 minutes to induce uniform stratification and reduce clogging. Following sieving, the retained material on each mesh was collected with care, weighed, and kept in labelled airtight containers for examination. The obtained size fractions were measured for their mass distribution, and representative samples from each size class were taken for subsequent chemical and mineralogical analysis and for silica extraction experiments.

2.2 Elemental Analysis

All the bauxite size fractions that were sieved, were analysed for chemical composition to find the distribution of major oxides, specifically the content of alumina (Al_2O_3) and silica (SiO_2). For the solids analysis, X-ray Fluorescence (XRF) spectroscopy was performed on a PANalytical Axios Max sequential wavelength-dispersive XRF spectrometer. Before the analysis, representative sub-samples from each size fraction were ground finely to a particle size of < 75 µm using an agate pestle and mortar for homogeneity and improvement in analytical precision. Around 5 g of ground sample was subsequently mixed with an appropriate binder (e.g., boric acid) and pressed into a pellet form using a hydraulic press at a force of 20 t to create stable and smooth analytical discs. The calibration of the XRF analyser was accomplished with certified reference materials (CRMs) for bauxite and laterite ore to provide quantitative precision in the elemental determination. Every pellet was scanned in triplicates to validate repeatability and minimize instrumental error. The acquired spectra were reduced using the instrument software to determine the concentration of major oxides, such as Al_2O_3, SiO_2, Fe_2O_3, TiO_2, and other trace

impurities. This analysis gave insight into the variations of silica and alumina content in a range of particle sizes, which made it possible to identify the size fractions that are enriched with silica-bearing minerals. This helped in the choice of suitable fractions to be used for further silica extraction and synthesis of sodium silicate studies.

2.3 Selection of High-Silica Fraction

Based on X-ray Fluorescence (XRF) analysis results, the size fractions of bauxite with high silica (SiO_2) content and comparatively low alumina (Al_2O_3) content, were isolated and shortlisted for further investigation. These fractions were assumed to include a higher percentage of reactive silica minerals like kaolinite, halloysite, and amorphous silica, which are more susceptible to dissolution under alkaline conditions. Targeting these particular fractions, the aim was to extract as much silica as possible with minimal co-dissolution of alumina, thus enhancing the purity and stability of the produced sodium silicate solution. To enable the selective dissolution of silica, a diluted caustic liquor was used, instead of the normal concentrated liquor utilized in the Bayer process. Particularly, spent liquor – that was recovered from the precipitation process and has a low caustic soda (Na_2O) content – was chosen as the leaching agent.

This method provided two main advantages: (i) prevention of excessive dissolution of alumina due to the lower alkalinity, and (ii) enhancement of silica dissolution by ensuring a beneficial caustic and temperature condition without causing premature precipitation of aluminosilicate species. The chosen silica-enriched bauxite fractions were then leached in batch mode under controlled temperature, time, and solid-to-liquid ratio with the spent liquor. The experimental conditions were optimized cautiously to facilitate efficient extraction of reactive silica without compromising valuable alumina losses. The leachate was then analysed for content of dissolved silica and utilized for the synthesis of stable sodium silicate solutions.

2.4 Dissolution Studies

To analyse the leaching behaviour of reactive silica from chosen bauxite fractions, controlled dissolution experiments were conducted under laboratory-scale bottle tester equipment with a thermostatically controlled water bath. The system provided the precise control required for mixing conditions and solution temperature, which are essential to simulate the leaching environment and provide reproducible results.

For every test, an accurately weighed amount of the silica-rich bauxite fraction was added to a measured volume of spent liquor in 250 mL high-density polyethylene (HDPE) bottles. A predetermined value (e.g., 1:10 w/v) was used for the solid-to-liquid ratio to achieve proper slurry dispersion. The bottles were placed on the rotating rack of the bottle tester into the water bath, and the temperature of the water bath was kept constant during the experiment. The rotation speed of the bottle tester was fixed to provide uniform suspension of solids and to facilitate mass transfer between the solid and liquid phases. Samples were drawn at regular intervals (e.g., 15, 30, 60, 120, and 240 minutes).

The withdrawn aliquot was quickly cooled down to room temperature to stop further reaction and filtered through 0.45 µm membrane filters. The transparent filtrates were analysed to calculate the amounts of dissolved silica (as SiO_2), caustic soda (as Na_2O), and alumina (as Al_2O_3). Silica was measured by ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometry) whereas soda and alumina levels were obtained by standard potentiometric titration methods. The dissolution profiles in function of time gave information about the kinetics of silica extraction and extent of unwanted alumina leaching under the process conditions presented.

Figure 1. Water bath used for test work.

3. Results and Discussion

3.1 Bauxite Beneficiation and Fraction Analysis

Chemical analysis of different bauxite particle size fractions showed considerable differences in the distribution pattern of silica (SiO_2) and alumina (Al_2O_3) content over the particle size range. From all the fractions tested, the intermediate particle size range between -500 µm and +212 µm contained the highest level of silica and low percentage of alumina (Table 1). The preferential concentration of silica in this particular fraction can be caused by the mechanical disintegration properties of clay minerals under crushing and grinding processes, resulting in their preferential concentration in the finer size fractions.

Table 1. XRF analysis of bauxite fractions.

Bauxite Size Fraction	Al_2O_3, %	SiO_2, %
+2 mm	44.29	5.73
-2 to +1 mm	39.98	7.21
-1mm to +500 µm	38.21	8.37
-500 µm to +300 µm	37.83	9.67
-300 µm to +212 µm	36.45	9.60
-212 µm to +63 µm	36.80	8.86
-63 µm	38.11	7.79

Based on these observations, the -500 µm fraction (material smaller than 500 µm) was selected as the best candidate for further dissolution experiments (Table 2). This was done to achieve maximum silica recovery under controlled caustic leaching conditions while achieving a minimum alumina loss, thus meeting the dual requirements of reactive silica removal and synthesis of a stable sodium silicate solution.

Table 2. Variation in silica and alumina concentration.

Size Fraction	Al2O3, %	SiO2, %
-500 µm	37.31	8.89
+500 µm	42.56	6.55

3.2 Kinetics Study

A 100 g/L slurry of the chosen fraction of bauxite was made by mixing with spent caustic liquor and kept at a fixed temperature of 70 °C in a thermostatically controlled water bath. Dissolution over time was followed, and it was found that the maximum extraction of silica was at about 5 hours of reaction time (Figure 2).

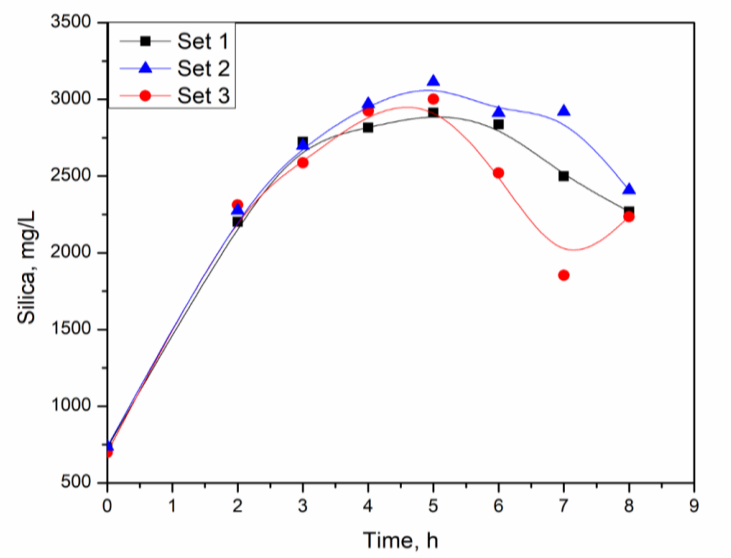

Figure 2. Variation in silica concentration with time at fixed temperature, 70°C.

To verify the reproducibility and reliability of the results, experiments were carried out in triplicates under the same conditions. The reproducibility of silica dissolution in multiple trials confirmed the experimental setup stability and ensured the best leaching time. The comparative uniformity of experimental sets suggests reproducibility of reported silica dissolution-precipitation kinetics, although Set 3 held slightly lower concentrations throughout the decreasing portion, possibly because of experimental variabilities, influencing nucleation or surface interaction processes.

3.3 Temperature Variation Study

The temperature variation study of silica concentration exhibits distinct patterns across the experimental sets at the temperatures of 60, 70 and 80 °C, respectively. Initial silica concentrations of approximately 700 ppm increased substantially during the first 5 hours of reaction time, reaching maximum values of about 3000 ppm for 70 °C. This significant increase by a factor of approximately 4.2 suggests a pronounced dissolution mechanism that dominates the desilication reaction stages, with a very slow rate of solids formation. Next, a steady drop in concentration was noticed with concentration reaching 2600 mg/L at the 6-hour mark (Figure 3), suggesting increased silica removal from solution, likely due to increased precipitation of solids. Other experimental results at temperatures of 60 and 80 °C, indicated a very slow dissolution of silica with under 1500 mg/L of silica in liquor up to the 8 hours (Figure 4, left) or a faster rate of

aluminosilicates precipitating after 2 to 3 hours, reducing the SiO_2 concentration in the liquor (Figure 4, right).

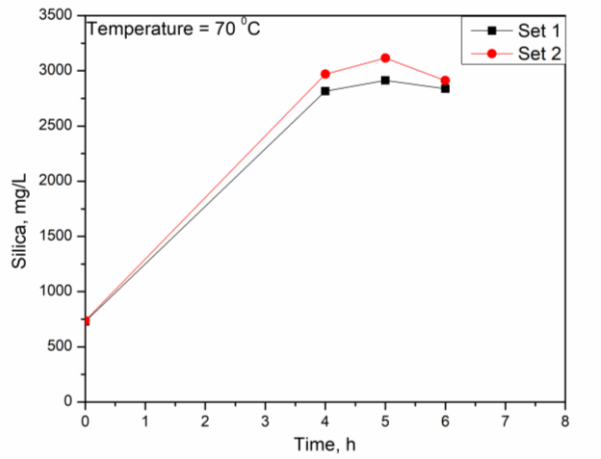

Figure 3. Change in Silica concentration with time at 70℃.

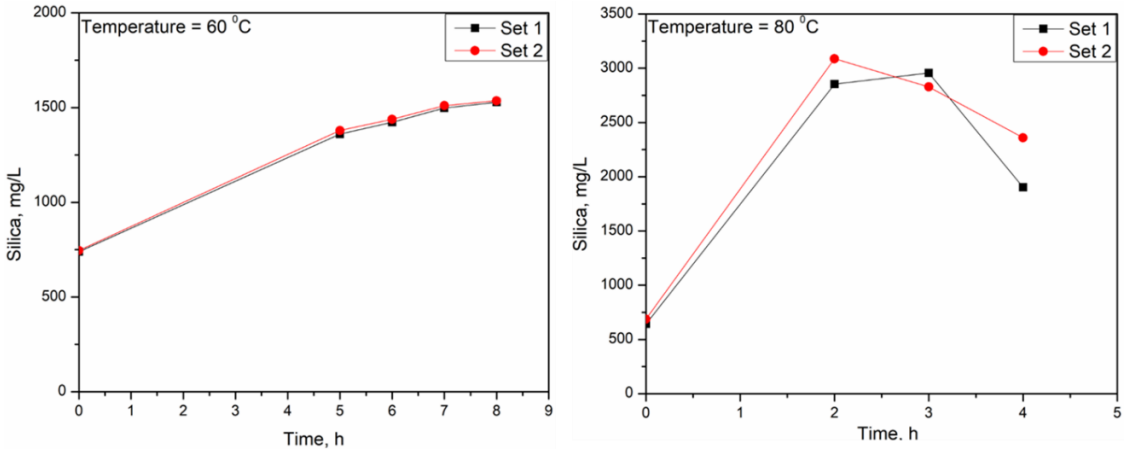

Figure 4. Change in Silica concentration with time at 60 ℃ (left) and at 80 °C (right).

3.4 Slurry Concentration Study

Table 3 indicates the impact of different solids feed concentrations (50, 100, and 200 g/L) on silica dissolution. The 100 g/L slurry yielded the highest silica concentration at 5 hours, confirming it as the optimal slurry concentration.

The 200 g/L slurry exhibited a rapid initial increase in silica concentration but experienced a sharp drop afterward, likely due to enhanced precipitation or silica removal at high solid loading. Due to this instability in silica concentration beyond the 3-hour mark, the 200 g/L condition was excluded from further studies. Collectively, these results support 70 °C, 100 g/L slurry concentration, and 5 hours of reaction time as the optimal leaching conditions for maximum extraction of silica from the chosen bauxite fraction.

Table 3. Change in Silica concentration with feed solids variation.

Time, h	50 g/L	100 g/L	200 g/L
0	621.4	698.6	621.4
3	1532.1	2586.4	3456.4
4	1725.0	2925.0	2582.1
5	1881.4	3002.1	1480.7

4. Conclusions

This research identifies the -500 µm bauxite fraction as the ideal for silica recovery, with better dissolution properties. The optimum dissolution conditions were determined at 70 °C, 100 g/L slurry, and 5 hours residence time, defining an accurate processing window for maximum silica recovery. Under this optimum temperature of 70 °C, thermal energy input adequately overcomes the activation energy barriers for silica dissolution without inducing competing reactions or accelerated precipitation processes seen at elevated temperatures. The 100 g/L slurry concentration is an optimal balance between low solids loading (50 g/L) that provides too little silica mobilization and high concentration (200 g/L) that speeds dissolution at first but then fosters fast precipitation, most probably as a result of greater nucleation sites and localized supersaturation conditions. The 5 hours residence time corresponds to maximum silica concentration values (averaging around 3010 mg/L within experimental sets), above which decreasing returns are realized as precipitation mechanisms take control of system dynamics.

These rigorously optimized parameters allow effective silica recovery for stable sodium silicate production, aiding in less caustic soda loss in follow-up Bayer process operations and providing potential paths for value-added silica products for applications as adsorbents, catalysts, and advanced ceramic materials. Systematic determination of these conditions offers both basic understanding of silica dissolution kinetics from aluminosilicate matrices and operating instructions for industrial utilization of selective recovery of silica from bauxite resources.

5. References

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