


Article

Preliminary Beneficiation Studies of Quartz Samples from the Northwest Territories, Canada

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Abstract: Three quartz-rich geologic materials—vein quartz from the Great Bear Magmatic Zone, massive quartz from the Nechalacho rare earth deposit, and quartz sands from the Chedabucto silica sand deposit along the shores of the Northern Arm of the Great Slave Lake, Northwest Territories of Canada—were evaluated for their amenability to physical beneficiation into high-purity quartz (HPQ). The samples were subjected to various treatment processes, including crushing, grinding, calcining and quenching, acid leaching, wet high-intensity magnetic separation (WHIMS), and reverse flotation. After treatment, both the core and sand quartz samples met the requirements for HPQ, making them suitable for use in the production of semiconductor filters, liquid crystal displays (LCDs), and optical glass. However, the Al-bearing impurity content in the vein quartz products remained relatively high, and most of these impurities were dispersed in the quartz lattice, requiring further processing to meet the purity standards for HPQ required by these industries.

Keywords: vein quartz; core quartz; sand quartz; beneficiation; high-purity quartz; Northwest Territories



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1. Introduction

Silicon (Si) is one of the most abundant elements in the Earth's crust, comprising about 27.7% of its mass, second only to oxygen which makes up 46.6% [1]. Quartz is the most common crystal form of silicon dioxide (SiO₂), accounting for 12.6% of the Earth's crust by volume [2]. Due to its robust physical and chemical properties, quartz has extensive applications in both traditional and high-tech industries [3]. It is widely used in refractory materials, mechanical casting, and chemical industry, among others [4]. Furthermore, quartz is widely used in high-tech industries because of its exceptional properties. For example, quartz is extensively used in fiber optical transmission and photovoltaic devices due to its high refractive index [5]. Its exceptional transparency allows it to be used in the production of quartz crystal glass, known as the “crown of glass materials” [4]. However, the presence of impurities, such as Fe and Al, reduces its light transmittance, affecting the performance of quartz glass and quartz optical fibers [6]. Purity is therefore an essential factor in determining the applications of quartz. Production of high-quality optical devices, semi-conductors, synthetic quartz wafers, and crucibles for solar-grade Si production, among others, requires high-purity quartz (HPQ) [4]. In the production of quartz crucibles used to hold high-temperature Si melts, impurities can make the crucibles opaque and cause brown spots on the surface of the produced Si crystals [7]. Generally, quartz with a SiO₂ content of 99.9% (3N) [8] is referred to as HPQ, while ultra-high-purity quartz (ultra-HPQ) has a SiO₂ content exceeding 99.998% (4N8) [9].

The demand for HPQ is increasing steadily because of the rapid development of the semiconductor and photovoltaic industries. Currently, there are two main methods to produce HPQ: artificial synthesis [10,11] or purification of natural quartz mineral. Artificial synthesis relies on high-purity starter crystals and uses vapor deposition with CCl₄ or

sol–gel methods [11]. Due to the high costs of synthesis and the depletion of high-purity starter crystal resources, there is a growing focus on producing HPQ and ultra-HPQ by purifying the more abundant natural quartz minerals [3,12]. Natural quartz minerals often contain various inclusions and lattice impurities, which are incorporated into the quartz during crystallization by processes like alteration, irradiation, diagenesis, or metamorphism under varying temperatures, pressure, and humidity. These may include point defects, dislocations, planar defects, and melt/fluid or mineral micro-inclusions [2]. To obtain HPQ, these impurities must be removed through appropriate treatment methods. Key quartz purification techniques include physical, chemical, and biological methods [13,14]. The physical and chemical methods can be further subdivided into processes such as grinding, screening, microwave heating, calcining and quenching, magnetic separation, flotation, acid leaching, ultrasonic treatment, and chlorination roasting [15–24].

The beneficiation of quartz requires grinding to liberate quartz from mineral impurities. Fine grinding is an energy-intensive process; thus, a proper grind size needs to be established to balance mineral impurity liberation and energy consumption [15]. Fluid inclusions are often present in quartz. Calcining followed by quenching is a common method for breaking fluid inclusions and removing associated impurities. Notably, the smaller the grain size of inclusions, the more challenging it is to remove them [25]. Microwave heating offers a novel approach for treating fluid inclusions. Unlike conventional calcination, microwave heating provides internal selective heating and is especially efficient for polar substances like water, which are susceptible to microwave treatment [16]. Magnetic separation and froth flotation are also effective methods for quartz beneficiation. Magnetic separation can remove even weakly magnetic mineral impurities from quartz using a strong magnetic field, while froth flotation achieves separation by exploiting differences in hydrophobicity between quartz and mineral impurities, through the use of flotation reagents to modify surface hydrophobicity [4,17]. Selective flocculation [26] and two-stage flotation [27] have been investigated in recent studies to enhance quartz flotation efficacy. The above physical ore dressing methods can enrich quartz to a high purity. For further beneficiation, chemical methods, such as acid leaching and chlorination roasting, may be applied. Acid leaching is a chemical reaction at the solid–liquid interface, where impurities dissolve into the slurry and are subsequently removed through solid–liquid separation. Common acids used in leaching to purify quartz include HCl, H₂SO₄, and so on. HF may also be used in some cases to achieve more pronounced upgrading effects, but it reacts with SiO₂, leading to a decrease in quartz yield [4,18]. Chlorination roasting is a method for ultra-HPQ production, whereby roasting alters the quartz crystal structure and disrupts impurity structures, and chlorine gas can volatilize impurity elements from the quartz surface, facilitating their removal [28].

The Northwest Territories of Canada have many quartz-rich deposits, some with natural high quality. These include the giant quartz veins in the Great Bear Magmatic Zone (GBMZ), the massive quartz of the Quartz Core Zone (QCZ) in the Nechalacho rare earth mine, and the Chedabucto quartz sands along the shores of the Northern Arm of the Great Slave Lake. Byron [29] characterized the giant quartz veins at different zones in the GBMZ using electron microprobe analysis, scanning electron microscopy, X-ray diffraction, cathodoluminescence, and other methods. She found that although some of the zones hosted base metal and U mineralization, most were barren and only contained giant quartz veins. The quartz contained abundant fluid inclusions and some complex growth zones where Si was substituted by Al and Li. Quartz from the QCZ in Nechalacho contains very low concentrations of impurities, most of which were at the ppm level, including some problematic elements such as P, U, and Th [30]. Only Na had elevated concentrations at more than 100 ppm, which was predominantly observed in fluid inclusions. Hu [31] conducted a QEMSCAN mineralogical investigation of the –850 + 105 µm size fraction of quartz sand samples from Chedabucto sand. The primary mineral identified was quartz, with purity levels ranging from 95.88% to 99.54% SiO₂. Key impurities included orthoclase, Na-Ca feldspar, pyrite/marcasite, calcite/aragonite, kaolinite, and illite. The average

particle size of the quartz grains ranged from 150 to 236 μm , and impure mineral grains were generally smaller than 100 μm . QEMSCAN images showed that most quartz particles were pure and liberated, though some had intergrown impurities, mainly at the rim or in cracks, which may be removed by fine grinding and/or acid leaching. A very small number of particles had encapsulated impurities. The objective of this study was to upgrade samples from the above deposits to HPQ using simple and practical beneficiation methods.

2. Materials and Methods

2.1. Materials

Ten quartz samples from the Northwest Territories, Canada, were used in this study. These included three samples from a giant quartz vein in the GBMZ (labelled as S1, S2, and S3), four from the QCZ in the Nechalacho rare earth mine (S4, S5, S6, and S7), and three from the Chedabucto silica sand deposit along shores of the Northern Arm of the Great Slave Lake (S8, S9, and S10). Figure 1 shows photographs of the representative samples. As can be seen, the vein quartz samples showed irregular, blocky structures with noticeable surface textures. The samples were dense, primarily composed of translucent white to milky-white quartz interspersed with brown and grey coloration. The color variations were likely caused by lichens and mineral impurities. The surfaces exhibited some rough, uneven patches and occasional small cracks. The samples displayed a generally coarse grain size with vitreous luster in the milky-white area, suggesting their formation from HPQ materials. The samples from QCZ were HQ cores with a diameter of approximately 63.5 mm. The quartz appeared massive. The samples were translucent white to pale grey, with some fracture surfaces coated with reddish-brown iron oxide stains. Samples of the quartz sand were unconsolidated and appeared grey/brownish with particles generally measuring less than 500 μm . All quartz samples appeared to have high purity.

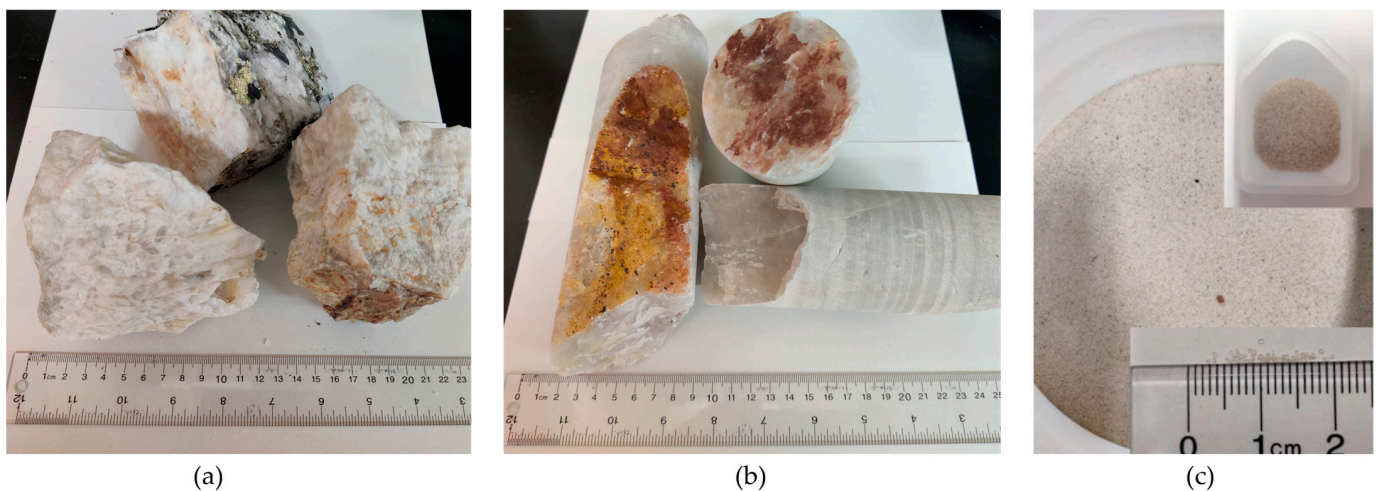


Figure 1. Photographs of quartz samples. (a) vein, (b) core, and (c) sand.

The particle size distribution of the three quartz sand samples was determined by sieve analysis. Figure 2 shows the cumulative particle size distribution and the weight retained in different size fractions. As can be seen, sample S8 was considerably finer than samples S9 and S10. The approximate median sizes of the three samples were 320, 390, and 450 μm , respectively.

The mineralogical composition of the ten samples was determined by powder X-ray diffraction (XRD) using a Bruker D8 XRD platform at the nanoFAB—Fabrication and Characterization Facility, located at the University of Alberta, after crushing and grinding. The 2θ range was from 10° to 80° with a scan speed of 5 degrees per minute. The patterns for all the samples are shown in Figure 3. The XRD patterns of all the samples exhibit

distinct characteristic quartz peaks with no impurity peaks, indicating the naturally high purity of the samples.

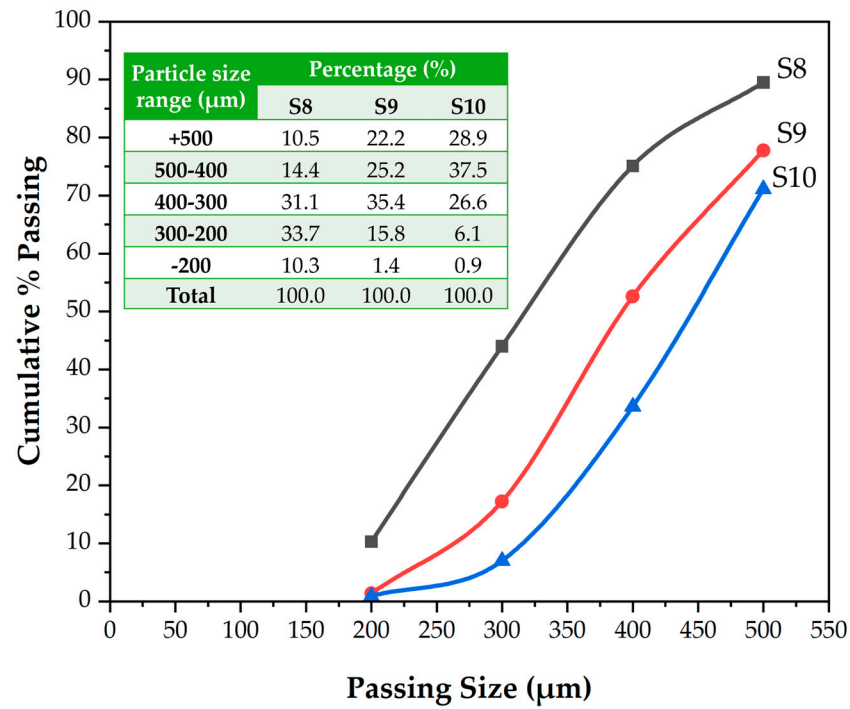


Figure 2. Cumulative particle size distribution of the three received quartz sand samples and particle size distribution table.

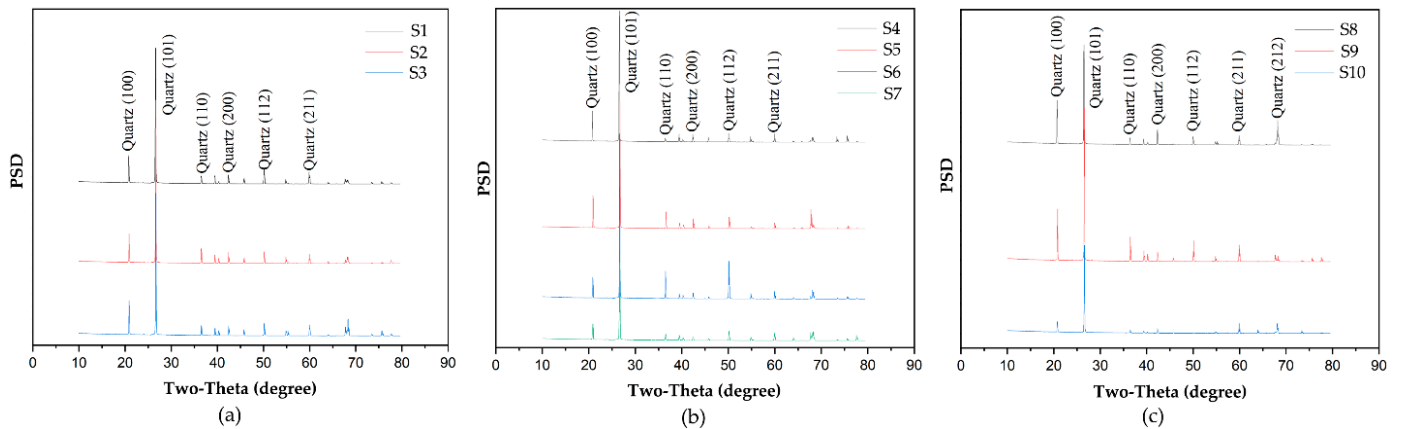


Figure 3. XRD patterns of quartz samples. (a) vein, (b) core, and (c) sands.

The chemical compositions of the samples were analyzed at the Geochem Laboratories of SGS Canada Inc. in Lakefield, Ontario. The SiO₂ content was directly measured by the gravimetric method according to ASTM C146, while the impurities were determined by whole-rock wavelength dispersion X-ray fluorescence (WD-XRF). The results are shown in Table 1. All ten samples had high SiO₂ content, ranging from 97.34% SiO₂ (Sample S1) to 99.19% (Sample S5). Vein and sand quartz samples had slightly lower purity, with Al and Fe being the main impurities. Therefore, removing Al and Fe should upgrade the SiO₂ purity.

Table 1. Composition of the ten quartz samples.

Sample	Composition (wt.%)							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	LOI
S1	97.34	0.73	0.14	0.05	0.06	0.09	0.12	0.55
S2	98.55	0.32	0.34	0.05	0.04	0.04	0.05	0.43
S3	98.35	0.33	0.35	0.06	0.03	0.02	0.06	0.49
S4	98.35	0.03	0.16	<0.01	0.02	0.03	<0.01	0.36
S5	99.19	<0.01	0.17	<0.01	0.02	0.03	<0.01	0.35
S6	98.63	0.02	0.3	0.04	0.02	0.04	<0.01	0.23
S7	98.06	0.02	0.42	0.06	0.21	0.03	<0.01	0.32
S8	97.79	0.18	0.03	<0.01	0.01	0.03	0.09	0.29
S9	98.03	0.27	0.06	<0.01	0.03	0.03	0.13	0.30
S10	98.02	0.25	0.05	<0.01	0.02	0.04	0.09	0.35

2.2. Beneficiation Processes

2.2.1. Reagents

Hydrochloric acid (HCl), sulfuric acid (H₂SO₄), oxalic acid (H₂C₂O₄), and sodium hydroxide (NaOH), all American Chemical Society (ACS) grade, were purchased from Fisher Scientific (Ottawa, Canada) and used for acid leaching and pH adjustment. Sodium oleate (active NaOl content ≥82%) was purchased from Millipore Sigma (Oakville, Canada) and used as a flotation collector. Dowfroth 250 (DF250) was acquired from Dow Chemical (Fort Saskatchewan, Canada) and used as a frother. Water used in all experiments was deionized (DI) water produced by Millipore Direct-Q® 5UV water purification system.

2.2.2. Experimental Procedures

Preliminary upgrading of quartz samples to HPQ is typically accomplished by conventional ore dressing techniques, including grinding, screening, magnetic separation, flotation, and acid leaching. More exotic treatment options could also be used for specific purposes, such as microwave heating or calcination followed by quenching to decrepitate fluid inclusions and expose their content, ultrasonic treatment to remove superficial contaminants, and chlorination roasting to volatilize trace metals that are difficult to remove by physical ore dressing processes. The objective of this work was to enrich the SiO₂ content of the samples to 99.9% or higher using simple ore dressing techniques. Figure 4 illustrates the complete beneficiation process for the quartz samples used in this study, including size reduction, calcination and quenching, the first acid leaching using HCl, wet high-intensity magnetic separation (WHIMS), reverse flotation, and the second acid leaching using mixed H₂SO₄ and H₂C₂O₄.

Size reduction and pre-treatment: The vein quartz samples (S1, S2, and S3) and core samples (S4, S5, S6, and S7) were crushed with a Retsch BB 200 Jaw Crusher, followed by pulverization with a Retsch DM 200 Disc Pulverizer to minus 500 µm. The pulverized quartz samples likely had surface contamination from the crusher and pulverizer. Therefore, prior to subsequent beneficiation treatment, the pulverized <500 µm quartz samples were leached with 5% HCl at 60 °C at a liquid-to-solid ratio of 5:1. The leach slurry was agitated in a Corning LSE benchtop orbital shaker at 200 rpm for 8 h. The acid-leached samples were further ground using a Fritsch Pulverisette 2 mechanized agate mortar/pestle grinder to the required size (either 80% or 100% passing 50 µm, depending on the progression of the study). Since the sand quartz samples were <500 µm, no crushing was necessary. They were directly ground using an agate mortar/pestle grinder to either 80% passing 106 µm or 80% to 100% passing 50 µm, depending on the requirements of subsequent experiments.

Calcination and quenching: 10 g of each ground sample was calcined in a muffle furnace in which the temperature was ramped at 3.5 °C/min up to 800 °C and maintained for 2 h, and then immediately quenched in 1 L DI water. After quenching, the solids were separated from the liquid and 10 mL of the collected liquid was analyzed to determine

its ionic composition. The solids (quartz samples) were dried and used for analysis and subsequent processing.

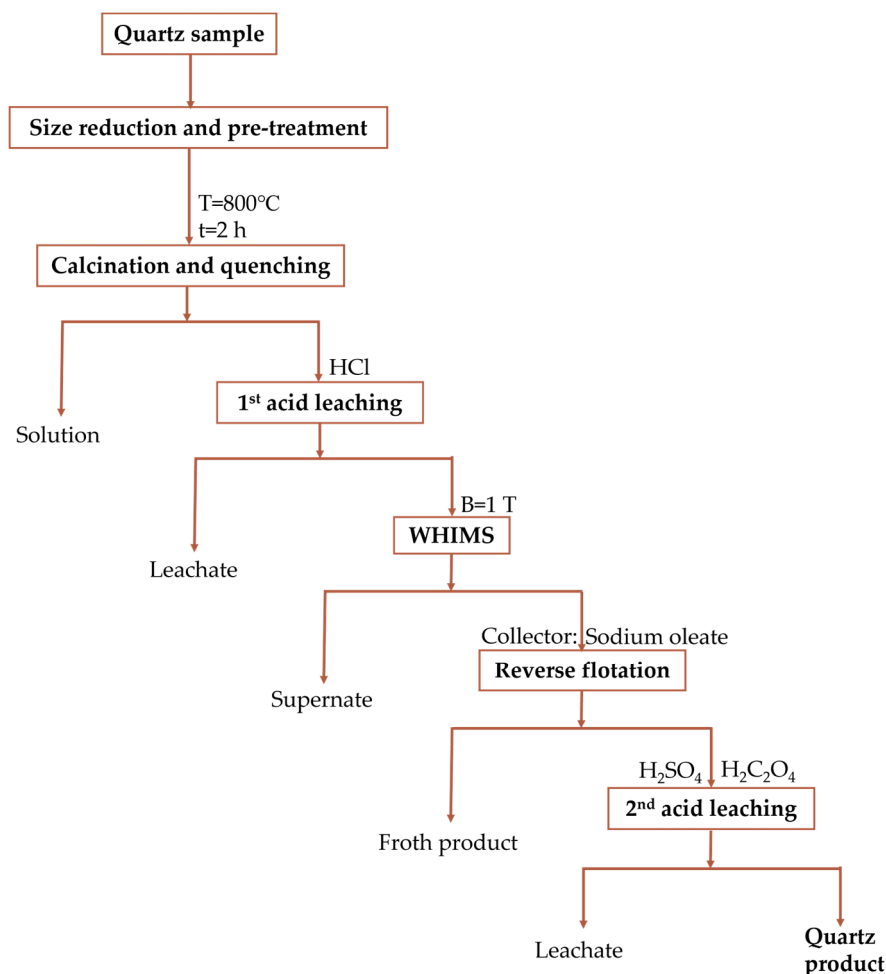


Figure 4. Flow chart of purification experiment.

Acid leaching: As shown in Figure 4, two steps of acid leaching were carried out. In the first step, a 10 g quartz sample was mixed with 10% HCl to create a slurry with a liquid-to-solid ratio of 10:1. The mixture was agitated using a Corning LSE benchtop orbital shaker at 200 rpm for 12 h at 60 °C. In the second acid leaching step, a 10 g quartz sample was leached with a mixture of 5% H₂SO₄ and 1% H₂C₂O₄, maintaining the same liquid-to-solid ratio of 10:1, at 70 °C and 200 rpm for 12 h. The experimental conditions mentioned above were determined through preliminary trials to achieve optimal efficiency, and the results of these trials are provided in Appendix A, Tables A1–A4. After each leaching step, 10 mL of the leachate was collected for chemical analysis, and the solids were filtered, washed, and dried for further analysis and processing.

WHIMS: Each sample was mixed with DI water to form a 20 wt.% solids slurry that was passed through the separation chamber of a WHIMS. The steel balls used in the chamber had a diameter of 10 mm, and the current was 5 A, i.e., enough to generate a strong magnetic field of approximately one Tesla. The samples that passed through the WHIMS while the current was turned on were the non-magnetic products. The current of the WHIMS was then turned off, and the retained solids in the separation chamber were flushed and rinsed to collect the magnetic product. Both streams of samples were collected and allowed to settle overnight and then decanted. Due to the high purity of the samples, the yield of the magnetic products was generally very low, in the 0.5–1% range.

Reverse flotation: Reverse flotation tests were performed in a 550 mm-tall and 50 mm-diameter flotation column, using 20 g of ground sample at 3 wt.% solids and at three different pH of 4, 5.6 (natural pH), and 10. Sodium oleate (NaOl) was used as a collector at dosages of 10, 100, 500, 1000, and 2000 g/t, with DF250 as the frother. The pulp was conditioned with NaOl for 5 min and floated for 1 min. The froth and tailing products were collected, filtered, and dried for analysis and further processing. The assay results of reverse flotation products under various conditions are provided in Appendix A, Tables A5–A8.

2.2.3. Analytical Methods

The compositions of liquid and solid samples were determined at the Geochem Laboratories of SGS Canada Inc., Lakefield, Ontario. The liquid samples were directly introduced into the inductively coupled plasma–optical emissions spectroscopy (ICP-OES) system to determine the concentrations of 30 elements, including Al, Fe, Na, and Ca. The SiO₂ content in the solid samples was measured directly by the gravimetric method according to ASTM C146. To measure the composition of impurities in the solid samples, the samples were fused with a lithium tetraborate/lithium metaborate mixture to form homogenous glass disks. The prepared disks were analyzed by WD-XRF, and the loss on ignition (LOI) was determined gravimetrically at 1000 °C using the G_PHY01V method (SGS Canada Inc.). We also used a benchtop Bruker CTX 801 X-ray fluorescence spectrometer to determine the compositions of interim quartz samples to guide the choice of upgrading methods and test conditions, and the results are reported in Appendix A. A Vega3 Tescan scanning electron microscope (SEM), equipped with energy-dispersive X-ray spectroscopy (EDS), and a Cameca SX 100 electron probe microanalyzer (EPMA) were used to analyze the form of Al impurities in the treated vein quartz samples, to determine whether they were present as discrete Al-bearing minerals or within the quartz lattice. The SEM-EDS was operated at an accelerating voltage of 20 kV, while the EPMA used an accelerating voltage of 15 kV, with a peak scan time of 60 s. The detection limit for Al was 12 ppm using the EPMA.

3. Result and Discussion

In this work, we did not apply all the processing steps described above to all ten quartz samples. Instead, the quartz sand sample S8 was used to scope the process conditions, and then one representative sample from each of the three sample groups was used to evaluate their responses to the conditions, and adjustments were made accordingly. Finally, all ten samples were processed using the established flowsheet.

3.1. Quartz Sand Sample S8

Sample S8 was selected for the scoping study because of its low SiO₂ content (97.8%). The sample was ground to 80% passing 106 µm, and then subjected sequentially to acid leach cleaning to remove superficial contaminants, calcining and quenching to remove fluid inclusions, HCl acid leaching, WHIMS, reverse flotation, and leaching by mixed H₂SO₄ and H₂C₂O₄. The change in SiO₂ content after each processing step is shown in Table 2.

As can be seen, acid leach cleaning increased the SiO₂ content of the sample from the original 97.79% to 99.13%. Subsequent processing steps, i.e., calcining and quenching, HCl leaching, and magnetic separation, increased the SiO₂ content to 99.67%, 99.68%, and 99.88%, respectively. However, reverse flotation did not lead to further enrichment, but rather resulted in a slight decrease in the SiO₂ content to 99.46%, indicating that further upgrading by reverse flotation was not possible. The second acid leaching using mixed H₂SO₄ and H₂C₂O₄ increased the SiO₂ content slightly to 99.64%.

The results of the various processing steps on Sample S8 showed that among the tested methods, acid leaching showed the best enrichment effect, followed by WHIMS. In contrast, the other methods showed limited effectiveness in enriching SiO₂ in the quartz sample. Regarding impurities, the most abundant contaminants were Al, Fe, K, and Na. While Fe

was removed almost completely after WHIMS, Al, K, and Na content remained unchanged. It is also worth noting that after reverse flotation, the quartz sample saw a slight increase in Na content, from 0.04% to 0.12%. This was likely caused by the addition of NaOl collector in the reverse flotation process.

Table 2. Assays of quartz sand sample S8 after sequential upgrading steps. SiO₂ content was determined directly by the gravimetric method according to ASTM C146, and impurity content was determined by whole-rock WD-XRF*.

Enrichment Process	Composition (wt.%)						
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	K ₂ O	P ₂ O ₅	LOI
Original sample	97.79	0.18	0.03	0.03	0.09	0.02	0.29
Pre-acid leaching	99.13	0.17	0.04	0.04	0.09	0.01	0.29
Calcination & quench product	99.67	0.18	0.02	0.04	0.09	0.02	0.05
1st acid leaching product	99.68	0.15	0.03	0.05	0.08	<0.01	0.17
Non-magnetic product of WHIMS	99.88	0.12	<0.01	0.04	0.08	<0.01	0.08
Tailings from reverse flotation	99.46	0.16	<0.01	0.12	0.07	<0.01	0.23
2nd acid leaching product	99.64	0.17	<0.01	0.08	0.08	<0.01	0.26

* In addition to the components in this table, all the samples contain about 0.01% CaO and very little (<0.01%) of MnO, Cr₂O₃, and V₂O₅.

The solution obtained from calcining and quenching, and the leachates from the two acid leaching steps, were analyzed by ICP-OES, and the results are shown in Table 3. The concentration of ionic species in acid leach solutions is expected to be 10 times higher than that in the solution obtained after calcining and quenching because of the difference in the volumes of solution used. Therefore, the results of the quench solution should be multiplied by 10 for comparison purposes.

Table 3. Ionic composition of solution samples collected during the step-by-step processing of quartz sand sample S8.

Elements	Concentration (mg/L)			Elements	Concentration (mg/L)		
	Quench Solution	1st Acid Leaching Solution	2nd Leaching Solution		Quench Solution	1st Acid Leaching Solution	2nd Leaching Solution
Al	<0.2	17.5	2.8	Be	<0.003	<0.003	<0.003
Ba	<0.007	0.63	0.042	Bi	<1	<1	<1
Ca	3.3	5.1	5.6	Cd	<0.09	<0.09	<0.09
Cu	0.2	0.2	0.3	Co	<0.3	<0.3	<0.3
Fe	<0.2	8.1	2.3	Cr	<0.1	<0.1	<0.1
K	<1	5	1	Li	<2	<2	<2
Mg	0.45	1.86	2.32	Mo	<0.6	<0.6	<0.6
Mn	<0.04	0.09	0.36	Ni	<0.6	<0.6	<0.6
Na	8	7	15	P	<5	<5	<5
Pb	<2	3	<2	Sb	<1	<1	<1
Sr	0.054	2.86	0.039	Se	<3	<3	<3
Ti	<0.02	0.32	0.06	Sn	<2	<2	<2
Y	<0.02	0.07	<0.02	Tl	<3	<3	<3
Ag	<0.08	<0.08	<0.08	V	<0.2	<0.2	<0.2
As	<3	<3	<3	Zn	<0.7	<0.7	<0.7

As can be seen from Table 3, the quench solution showed relatively high concentrations of Ca, Mg, and Na, which were likely released from fluid inclusions in the quartz sand. The leachate from the first acid leaching (10% HCl) contained a relatively high concentration of Al (17.5 mg/L), along with significant amounts of Fe (8.1 mg/L), Na (7 mg/L), Ca (5.1 mg/L), Pb (3 mg/L), and Sr (2.86 mg/L). This indicates that HCl removed a considerable amount of impurities from the sample, especially Al. Subsequent acid leaching using a mixture of H₂SO₄ and H₂C₂O₄ removed a significant amount of Al (2.8 mg/L) and Fe (2.3 mg/L) but noticeably less than in the HCl leaching stage. However, the leach solution from the second-stage mixed acid leaching contained more Ca (5.6 mg/L), Cu (0.3 mg/L), Mg (2.32 mg/L), and Mn (0.36 mg/L) compared to the first acid leaching by HCl, suggesting that the combination of H₂SO₄ and H₂C₂O₄ had advantages in removing these multivalent metals compared to HCl.

3.2. Vein (S1), Core (S4), and Sand (S8) Samples

The sequential multi-step tests on sand quartz sample S8 showed that acid leaching had the most pronounced effect on enriching the SiO₂ content of the sample. Further leaching with mixed H₂SO₄ and H₂C₂O₄ removed additional impurities, especially multivalent metals, including Al and Fe. The other effective processing step was WHIMS, which was effective in removing Fe-bearing impurities. To simplify the process, one sample from each sample group—S1 from vein quartz, S4 from core quartz (S4), and S8 from sand quartz—was sequentially leached with mixed H₂SO₄ and H₂C₂O₄, and subjected to WHIMS. To enhance liberation and achieve better results, the samples were ground to 80% passing 50 µm.

Figure 5 shows SiO₂ content in the three samples, before and after acid leaching and magnetic separation. As can be seen, the SiO₂ content in the original samples was lowest in the vein sample (S1) at approximately 97.3% and highest in core sample (S4) at around 98.4%. The SiO₂ content in the sand sample (S8) was about 97.7%. After WHIMS, the SiO₂ content increased significantly, reaching approximately 99.1% in S1 and approximately 99.9% in S4 and S8. This demonstrated the effectiveness of grinding the samples to 80% passing 50 µm, followed by WHIMS. Subsequent leaching with a mixture of H₂SO₄ and H₂C₂O₄ further increased SiO₂ content. However, for the vein samples, grinding to 80% passing 50 µm was not effective in removing impurities further.

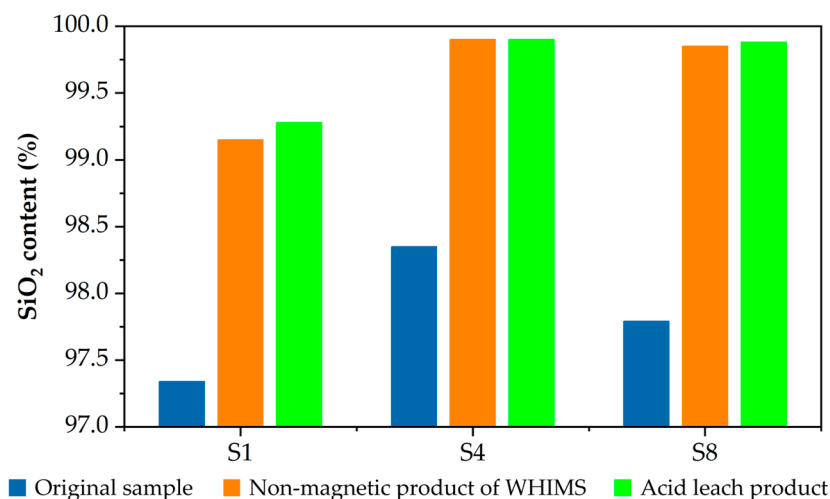


Figure 5. SiO₂ content, measured by the gravimetric method (ASTM C146) for S1, S4, and S8 after WHIMS and acid leaching with H₂SO₄ and H₂C₂O₄ mixtures.

3.3. Grinding, Magnetic Separation, and Acid Leaching of All Quartz Samples

Vein quartz samples were crushed and ground to 100% passing 50 μm , while the core quartz and sand samples were kept at 80% passing 50 μm . The beneficiation flowsheet relied on WHIMS followed by leaching with a mixture of H_2SO_4 and $\text{H}_2\text{C}_2\text{O}_4$.

As can be seen in Table 4, both WHIMS and acid leaching upgraded the SiO_2 content in all the quartz samples, with core quartz samples S4 and S5 showing the most significant improvement in SiO_2 content. The upgrade was lowest for the vein quartz samples, with SiO_2 content remaining below 99.9% even after reducing the particle size to 100% passing 50 μm . This indicated that magnetic separation and acid leaching are inefficient beneficiation methods for those vein quartz samples. The higher SiO_2 content of sample S1 after grinding to 100% passing 50 μm (99.49% SiO_2 , Table 4) compared to the same sample ground to 80% passing 50 μm (99.28% SiO_2 , Figure 5) indicates the effectiveness of particle size reduction in removing impurities and upgrading the SiO_2 content of vein quartz. The crushing/grinding step used in this study is easily achievable at an industrial scale, providing an efficient balance between impurity liberation and cost-effectiveness. Both WHIMS and acid leaching are standard processing techniques, widely recognized for their reliability and adaptability in large-scale production. Therefore, the approach developed here would be a feasible pathway for HPQ production from most of the tested quartz in an industrial setting.

Table 4. SiO_2 content of as-received quartz samples, as well as after magnetic separation and acid leaching. SiO_2 content was measured directly by the gravimetric method according to ASTM C146.

Sample	SiO_2 Content (wt.%)		
	Original Sample	Non-Magnetic Product	Acid Leach Product
S1	97.34	98.43	99.49
S2	98.55	98.76	99.05
S3	98.35	98.92	99.01
S4	98.35	>99.9	>99.9
S5	99.19	>99.9	>99.9
S6	98.63	99.65	99.76
S7	98.06	99.27	99.40
S8	97.79	99.85	99.88
S9	98.03	99.66	99.84
S10	98.02	99.79	99.86

Table 5 summarizes the SiO_2 content and major impurity content of each quartz sample after the beneficiation treatments (i.e., both magnetic separation and acid leaching. The data for the products of only magnetic separation is shown in Table A9). The vein quartz samples (S1, S2, and S3) were ground to 100% passing 50 μm , while the other seven quartz samples were ground to 80% passing 50 μm . As can be seen, after the complete beneficiation process, the vein quartz samples still contained a notable amount of Al_2O_3 impurities, ranging from 0.35% to 0.65%, while the concentrations of other impurities were negligible. In contrast, almost no impurities were detected in the core quartz samples, which showed the best beneficiation results. In addition, the sand quartz samples contained approximately 0.15% to 0.18% Al_2O_3 , while other impurities were successfully eliminated.

Depending on the intended use, HPQ has different tolerance limits for Al and Fe impurities. For example, the Al_2O_3 and Fe_2O_3 content of HPQ used in the production of semiconductor filters, (LCDs), and optical glass (used in cameras, optical instruments, and optical fibers for telecommunications), should be below 2000 ppm (0.2%) and 100 ppm (0.01%), respectively [32]. In extremely high-end applications, such as large-size silicon wafers, the Al content is required to be less than 8 ppm [4]; in this case, the SiO_2 content should be at least 99.99%. Achieving such high purity goes beyond simple physical separation and is outside the scope of this work. It can be seen from Table 5 that samples of core and sand quartz met the basic quality requirements of HPQ after the grinding–

magnetic separation–acid leaching treatment. However, for the vein quartz samples, the Al content remained too high for HPQ standards, even after effectively controlling Fe-bearing impurities through magnetic separation. To meet HPQ requirements, additional processing will be needed to further lower the Al content of the vein quartz samples.

Table 5. Assay results for SiO₂ (gravimetric method according to ASTM C146) and the main impurities (whole-rock WD-XRF) of the ten quartz samples after grinding–magnetic separation–acid leaching treatments.

Sample	Composition (%)							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅
S1	99.49	0.65	0.01	0.04	<0.01	0.02	0.12	<0.01
S2	99.05	0.35	<0.01	0.03	<0.01	0.02	0.07	<0.01
S3	99.01	0.36	0.02	0.04	<0.01	0.02	0.08	<0.01
S4	>99.9	<0.01	<0.01	<0.01	<0.01	0.03	<0.01	<0.01
S5	>99.9	<0.01	0.01	<0.01	<0.01	0.01	<0.01	<0.01
S6	99.76	<0.01	0.01	<0.01	<0.01	0.01	<0.01	<0.01
S7	99.40	<0.01	0.05	<0.01	<0.01	0.02	<0.01	<0.01
S8	99.88	0.15	0.01	<0.01	<0.01	0.03	0.08	0.01
S9	99.84	0.17	0.01	<0.01	<0.01	0.03	0.08	0.02
S10	99.86	0.18	0.02	<0.01	<0.01	0.05	0.07	0.01

3.4. Aluminum-Bearing Impurities in the Vein Quartz Sample

Vein quartz sample S1 was selected for SEM-EDS and EPMA analyses. The particle size of the treated S1 sample was below 50 μm, allowing direct compaction for SEM-EDS analysis. Figure 6 shows the SEM scan results for an area of 1.38 mm × 1.38 mm at 200 times magnification. The sample is dominated by silicon and oxygen regardless of particle size, with only two Al-rich particles shown in the area: one in the upper left and one in the lower right (circled in red in Figure 6b). The map sum spectrum shows only a very small Al peak, with an integrated result indicating an Al content of 0.14 wt.%.

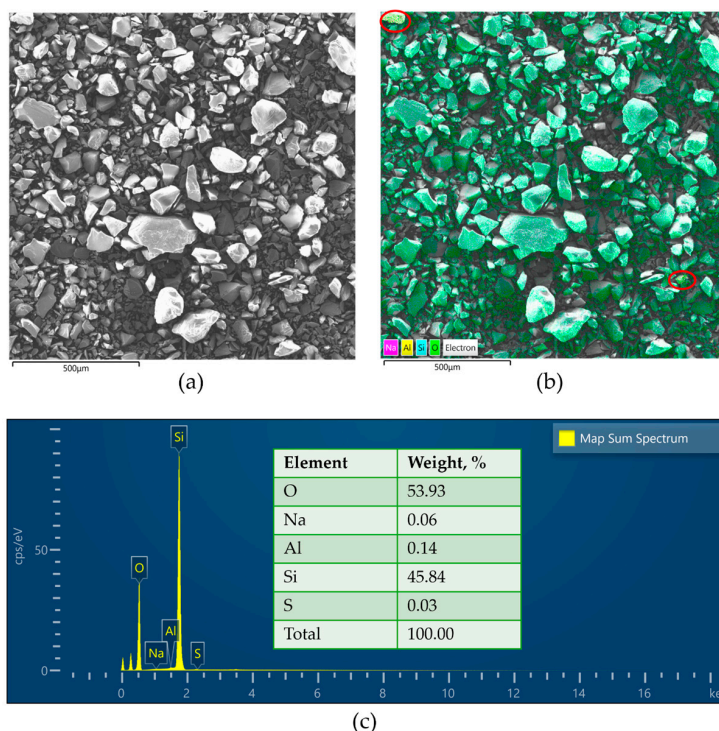


Figure 6. SEM-EDS surface-scanning images of an area of the treated sample S1 (magnified 200 times). (a) Electron image, (b) EDS elemental distribution map (the particles indicated by red circles show enriched Al), and (c) map sum spectrum and the integral results of elemental content.

The composition of one of the Al-rich particles (Figure 7) was analyzed. The EDS spectrum indicates that the particle contains Si (28.42 wt.%), Al (11.77 wt.%), O (50.02 wt.%), Na (6.02 wt.%), and Ca (3.77 wt.%). Based on the above compositional data, the particle is likely a plagioclase feldspar. This indicates the presence of discrete Al-bearing mineral phases. The Al distribution map (Figure 7b) shows many scattered yellow dots, resembling background noise. This probably indicates Al dispersed within the quartz lattice. EMPA was then used to scan quartz-dominated regions in sample S1 to determine the amount of Al in the quartz lattice.

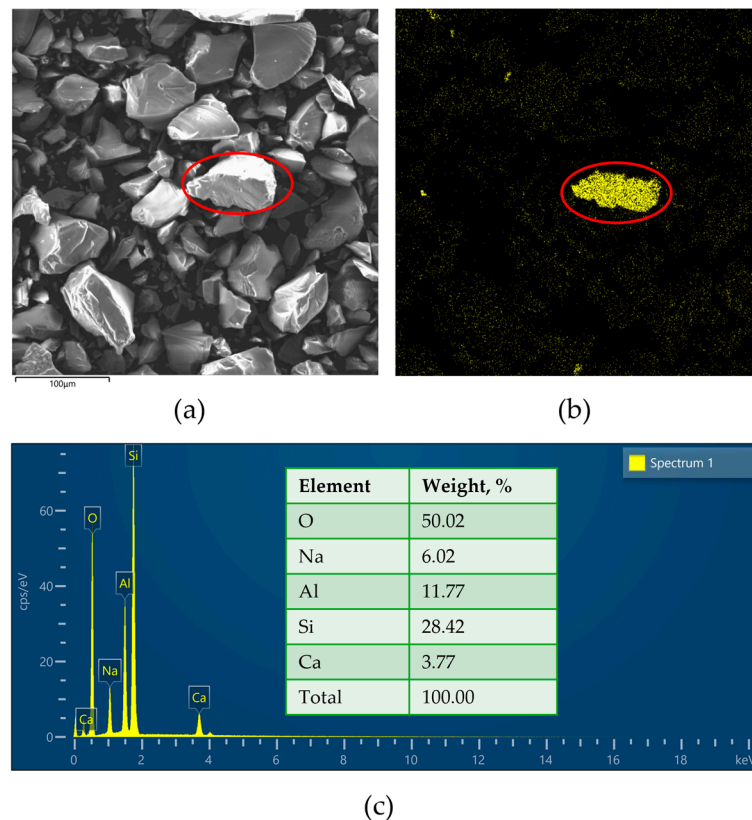


Figure 7. SEM-EDS surface-scanning images of a selected small area of the treated sample S1 (magnified 700 times). (a) Electron image, (b) EDS Al distribution map, and (c) EDS compositional spectrum of the highlighted particle (indicated in the red circle).

To prepare samples for EPMA analysis, the quartz vein sample S1 should be embedded in epoxy and polished to expose a smooth surface suitable for scanning. Due to the small particle size of the treated samples (less than 50 μm), this polishing process could not be effectively applied. A piece of untreated S1 sample without visible impurity was polished to produce a surface on which about 100 randomly selected points with no visible impurities were analyzed for trace amounts of Al. The analyses detected Al in all 100 points, indicating that part of the Al in the quartz vein sample is incorporated within the SiO_2 lattice rather than solely as discrete mineral phases. Figure 8 shows the cumulative distribution function of Al content measured in S1 (the Al content for each point is shown in Table A10). The detected Al concentrations ranged from 229 ppm (0.0229 wt.%) to 4323 ppm (0.4323 wt.%). The cumulative distribution function curve shows a relatively gradual slope at the lower and higher ends, with a steeper slope near the median values, suggesting that most points cluster around the median (2566 ppm, or 0.2566 wt.%). The mean Al content across the 100 points was 2466 ppm (0.2466 wt.%), almost coinciding with the median. Combining this with the SEM-EDS results, it can be seen that discrete Al-bearing minerals amounted to about 0.14 wt.% Al (about 36% of total Al), and Al in quartz lattice amounted to around 0.25 wt.% Al (about 64% of total Al) of the compositions in the processed vein quartz

sample. While EPMA was performed on untreated samples, Al within the quartz lattice was likely not removed by WHIMS and acid leaching using H_2SO_4 and $\text{H}_2\text{C}_2\text{O}_4$. Therefore, the values can be used as proxies for the Al content in the processed sample. The Al content measured by SEM-EDS and EPMA adds up to about 0.39 wt.% of Al, corresponding to an Al_2O_3 content of 0.73 wt.%, close to the 0.65 wt.% Al_2O_3 determined by whole-rock WD-XRF analysis. About two-thirds of the Al impurities in the vein quartz sample are dispersed within the quartz lattice, and this type of impurity cannot be easily removed through traditional physical beneficiation methods. Remaining discrete Al impurities, in theory, could be removed by conventional ore dressing techniques. However, the reverse flotation experiments did not seem to remove the discrete Al-bearing minerals, likely due to insufficient liberation. Finer grinding may enhance the liberation and removal of the discrete Al mineral impurities. Excessive fine particles may, however, adversely impact flotation performance. Combined SEM-EDS and EPMA analyses suggest that even when the discrete Al-bearing minerals were removed, the SiO_2 content of the sample would still not reach 99.9%, because lattice Al contributed to about 0.25 wt.% Al, or 0.48 wt.% Al_2O_3 . Indeed, our test work showed that all three vein quartz samples were upgraded to only 99–99.5 wt.% SiO_2 , which could be considered the upper limit of physical ore dressing techniques for these vein quartz samples.

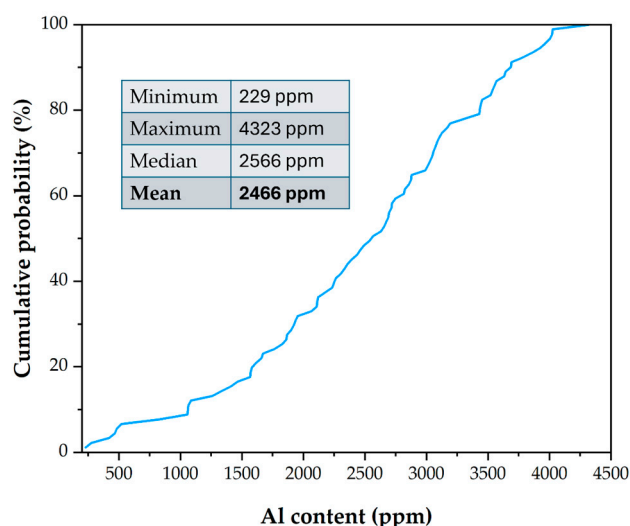


Figure 8. Cumulative distribution function of aluminum content measured at 100 points in quartz vein sample S1 using EPMA.

4. Summary and Conclusions

This work aimed to use simple and practical separation techniques for the preliminary beneficiation of ten quartz samples from the Northwest Territories, Canada. The samples included three samples from a giant quartz vein in the GBMZ, four from the QCZ in the Nechalacho rare earth mine, and three from the Chedabucto silica sand deposit along the shores of the Northern Arm of the Great Slave Lake. The separation techniques explored included grinding, calcining and quenching, acid leaching, WHIMS, and reverse flotation. The SiO_2 content in the original and treated samples was directly measured by the gravimetric method according to ASTM C146, and the contained impurities were determined by WD-XRF analysis. The impurities leached from the samples after calcining and quenching, and treatment with acids were analyzed by ICP-OES.

The initial SiO_2 content of the samples was already quite high at >97% SiO_2 . WHIMS and acid leaching proved highly effective in increasing the SiO_2 content. In contrast, calcining and quenching, as well as reverse flotation, were less effective. A mixture of H_2SO_4 and $\text{H}_2\text{C}_2\text{O}_4$ acids was effective in removing impurities, especially for multivalent metals relative to hydrochloric acid (HCl) only. Combined grinding, WHIMS, and leaching with mixed acids H_2SO_4 and $\text{H}_2\text{C}_2\text{O}_4$ were effective in upgrading the SiO_2 content of the

samples. The Al₂O₃ content in all four core quartz samples after beneficiation was reduced to less than 100 ppm (0.01%), and the value for the three sand quartz samples was from 1500 to 1800 ppm (0.15%–0.18%). This meets the <2000 ppm (0.2%) requirement for the production of semiconductors, (LCDs), optical glasses, optical instruments, and optic fibers for telecommunications.

Using the developed process, the SiO₂ content in the vein quartz samples increased to 99.5%, slightly less than the 99.9% SiO₂ standard. While Fe₂O₃ content was low at 100–200 ppm (0.01%–0.02%) after the upgrading treatment, likely due to the high-intensity magnetic separation, the Al₂O₃ content remained high, between 0.35% and 0.65% (3500 ppm to 6500 ppm). SEM-EDS and EPMA analyses showed that, in the vein quartz sample S1, discrete Al-bearing minerals (plagioclase group minerals) contributed 0.14 wt.% Al (0.26 wt.% Al₂O₃), and lattice Al substitution of Si contributed about 0.25 wt.% Al (0.47 wt.% Al₂O₃), accounting for 0.73 wt.% Al₂O₃ in sample S1. The 0.47 wt.% Al₂O₃ in the quartz crystal lattice in the vein quartz sample S1 represents the limit of physical ore dressing techniques. Therefore, this vein quartz sample could not be physically upgraded to more than 99.53% SiO₂.

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Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

Table A1. SiO₂ content (%) of S1 (vein quartz), S4 (core quartz), and S8 (sand quartz) leached with HCl at 60 °C for 8 h under various HCl concentrations (5% or 10%) and liquid-to-solid ratios (5:1 or 10:1), measured by a Bruker CTX XRF spectrometer.

Sample	5% HCl Liquid/Solid = 5	10% HCl Liquid/Solid = 5	5% HCl Liquid/Solid = 10
S1	98.37	98.66	98.87
S4	98.94	99.01	98.97
S8	98.53	98.58	98.67

Table A2. Major composition of the acid leaching products for samples S1, S4, and S8, treated with HCl at varying concentrations (5% or 10%) and liquid-to-solid ratios (5:1 or 10:1) at 60 °C for 8 h, measured by a Bruker CTX XRF spectrometer.

Conditions	Composition (wt.%)							
	SiO ₂	Al ₂ O ₃	Fe	MgO	Ca	Mn	K ₂ O	Ti
S1, 5% HCl, Liquid/solid = 5	98.37	0.46	0.05	<0.01	<LOD	<0.01	0.07	<LOD
S1, 10% HCl, Liquid/solid = 5	98.66	0.64	0.05	<0.01	<LOD	<0.01	0.06	<LOD
S1, 5% HCl, Liquid/solid = 10	98.87	0.60	0.05	<0.01	0.01	<0.01	0.06	<LOD

Table A2. *Cont.*

Conditions	Composition (wt.%)							
	SiO ₂	Al ₂ O ₃	Fe	MgO	Ca	Mn	K ₂ O	Ti
S4, 5% HCl, Liquid/solid = 5	98.94	<LOD	<0.01	<0.01	<LOD	<0.01	0.02	<LOD
S4, 10% HCl, Liquid/solid = 5	99.01	<LOD	0.02	<0.01	0.01	0.01	0.02	<LOD
S4, 5% HCl, Liquid/solid = 10	98.97	<LOD	0.01	<0.01	<LOD	0.01	0.02	<LOD
S8, 5% HCl, Liquid/solid = 5	98.53	0.13	0.07	0.01	0.01	<LOD	0.07	<0.01
S8, 10% HCl, Liquid/solid = 5	98.58	0.11	0.07	<0.01	0.01	<LOD	0.05	<0.01
S8, 5% HCl, Liquid/solid = 10	98.67	0.09	0.07	<0.01	<LOD	<LOD	0.08	0.02

Table A3. SiO₂ content (%) in sample S8 after acid leaching under different conditions, measured by a Bruker CTX XRF spectrometer. The tests were conducted using H₂SO₄, H₂C₂O₄, HCl + H₂C₂O₄, and H₂SO₄ + H₂C₂O₄, at acid concentrations of 10% HCl, 5% H₂SO₄, and 1% H₂C₂O₄. The liquid-to-solid ratio was maintained at 10:1, with a stirring speed of 200 rpm.

Conditions	H ₂ SO ₄	H ₂ C ₂ O ₄	HCl + OA	H ₂ SO ₄ + H ₂ C ₂ O ₄
60 °C, 8 h	98.12	97.79	98.06	97.95
70 °C, 8 h	97.89	98.19	97.76	98.48
60 °C, 12 h	98.25	98.09	98.32	98.31

Table A4. Major composition measured by a Bruker CTX XRF spectrometer of the acid leaching products for sample S8 after acid leaching with various acids under different conditions. The tests were conducted using H₂SO₄, H₂C₂O₄, HCl + H₂C₂O₄, and H₂SO₄ + H₂C₂O₄, at acid concentrations of 10% HCl, 5% H₂SO₄, and 1% H₂C₂O₄. The liquid-to-solid ratio was maintained at 10:1, with a stirring speed of 200 rpm.

Conditions	Composition (wt.%)							
	SiO ₂	Al ₂ O ₃	Fe	MgO	Ca	Mn	K ₂ O	Ti
H ₂ SO ₄ , 60 °C, 8 h	98.12	0.09	0.07	<0.01	0.01	<LOD	0.08	<0.01
H ₂ SO ₄ , 70 °C, 8 h	97.89	0.10	0.07	<0.01	<LOD	<LOD	0.09	0.02
H ₂ SO ₄ , 60 °C, 12 h	98.25	0.10	0.07	<0.01	0.01	<LOD	0.08	0.01
H ₂ C ₂ O ₄ , 60 °C, 8 h	97.79	0.11	0.07	<0.01	0.01	<LOD	0.08	0.01
H ₂ C ₂ O ₄ , 70 °C, 8 h	98.19	0.11	0.07	<0.01	<LOD	<LOD	0.14	0.02
H ₂ C ₂ O ₄ , 60 °C, 12 h	98.09	0.08	0.07	<0.01	<LOD	<0.01	0.10	0.01
HCl + H ₂ C ₂ O ₄ , 60 °C, 8 h	98.06	0.07	0.07	<0.01	<LOD	<LOD	0.09	<0.01
HCl + H ₂ C ₂ O ₄ , 70 °C, 8 h	97.76	0.07	0.06	<0.01	<LOD	<LOD	0.11	0.01
HCl + H ₂ C ₂ O ₄ , 60 °C, 12 h	98.32	0.07	0.06	<0.01	0.02	<LOD	0.12	0.02
H ₂ SO ₄ + H ₂ C ₂ O ₄ , 60 °C, 8 h	97.95	0.08	0.07	<0.01	0.01	<0.01	0.10	0.02
H ₂ SO ₄ + H ₂ C ₂ O ₄ , 70 °C, 8 h	98.48	0.10	0.07	<0.01	<LOD	<LOD	0.10	0.01
H ₂ SO ₄ + H ₂ C ₂ O ₄ , 60 °C, 12 h	98.31	0.07	0.07	<0.01	<LOD	<0.01	0.09	0.01

Table A5. SiO₂ content (%) in froth products and slurry products of the flotation tests of S8, measured by a Bruker CTX XRF spectrometer, using 1000 g/t sodium oleate as a collector under different pH values.

pH Value	SiO ₂ Content in Froth Product (%)	SiO ₂ Content in Slurry Product (%)
pH = 4	98.40	98.12
pH = 6.5	98.24	98.73
pH = 10	98.19	98.21

Table A6. Major compositions of froth and slurry products from the reverse flotation test of sample S8, measured by a Bruker CTX XRF spectrometer, using 1000 g/t NaOl as the collector at different pH levels.

Conditions		Composition (wt.%)							
		SiO ₂	Al ₂ O ₃	Fe	MgO	Ca	Mn	K ₂ O	Ti
Froth Product	pH = 4	98.40	0.13	0.07	0.01	<LOD	<LOD	0.07	0.03
	pH = 6.5	98.24	0.11	0.07	<0.01	<LOD	<LOD	0.05	0.01
	pH = 10	98.19	0.09	0.07	0.01	<LOD	<LOD	0.08	0.02
Slurry Product	pH = 4	98.12	0.07	0.06	<0.01	0.01	<LOD	0.11	<0.01
	pH = 6.5	99.73	0.10	0.07	<0.01	0.02	<LOD	0.12	0.01
	pH = 10	98.21	0.08	0.07	<0.01	0.01	<LOD	0.10	0.02

Table A7. SiO₂ content (%) in froth products and slurry products of the flotation tests of S8, measured by a Bruker CTX XRF spectrometer, using different dosages of NaOl.

NaOl Amount (g/t)	SiO ₂ Content in Froth Product (%)	SiO ₂ Content in Slurry Product (%)
10	98.41	97.65
100	98.05	97.61
500	97.60	98.04
1000	98.24	98.73
2000	98.29	98.18

Table A8. Major compositions of froth and slurry products from the reverse flotation test of sample S8, measured by a Bruker CTX XRF spectrometer, at varying NaOl collector dosages.

NaOl Amount (g/t)		Composition (wt.%)							
		SiO ₂	Al ₂ O ₃	Fe	MgO	Ca	Mn	K ₂ O	Ti
Froth Product	10	98.41	0.09	0.05	<0.01	0.02	<LOD	0.14	0.01
	100	98.05	0.13	0.05	<0.01	0.01	<0.01	0.14	0.01
	500	97.60	0.11	0.07	<0.01	<LOD	<LOD	0.10	0.02
	1000	98.24	0.10	0.07	<0.01	0.01	<LOD	0.12	0.03
	2000	98.29	0.11	0.06	<0.01	0.01	<LOD	0.09	0.01
Slurry Product	10	97.65	0.11	0.07	<0.01	0.01	<0.01	0.10	0.01
	100	97.61	0.10	0.06	<0.01	0.01	<LOD	0.11	0.02
	500	98.04	0.09	0.07	<0.01	<LOD	<LOD	0.12	0.01
	1000	98.73	0.08	0.06	<0.01	<LOD	<LOD	0.06	0.01
	2000	98.18	0.11	0.06	<0.01	<LOD	<LOD	0.06	0.01

Table A9. Major compositions of non-magnetic products of all the quartz samples, among which SiO₂ content, which was measured directly by the gravimetric method according to ASTM C146, while impurity content was determined by whole-rock WD-XRF.

Sample	Composition (%)							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅
S1	98.43	0.71	0.04	0.07	0.02	0.02	0.12	0.01
S2	98.76	0.39	0.08	0.06	0.02	0.01	0.08	<0.01
S3	98.92	0.39	0.14	0.07	<0.01	<0.01	0.08	<0.01
S4	>99.9	0.01	<0.01	<0.01	<0.01	0.02	<0.01	<0.01
S5	>99.9	0.05	0.02	<0.01	0.01	0.02	<0.01	<0.01
S6	99.65	<0.01	0.04	0.02	0.04	0.01	<0.01	<0.01
S7	99.27	<0.01	0.11	0.02	0.20	0.02	<0.01	<0.01
S8	99.88	0.15	0.01	<0.01	<0.01	0.03	0.09	<0.01
S9	99.66	0.24	0.03	0.01	0.02	0.05	0.10	0.02
S10	99.79	0.20	0.02	<0.01	0.02	0.05	0.07	0.02

Table A10. Data on the Al content at each point of the vein quartz sample S1 measured by EPMA.

Number	Al (wt.%)	Al (ppm)
1	0.3168	3168
2	0.2108	2108
3	0.2537	2537
4	0.2817	2817
5	0.2334	2334
6	0.3453	3453
7	0.3312	3312
8	0.3554	3554
9	0.4023	4023
10	0.4025	4025
11	0.1614	1614
12	0.2673	2673
13	0.3569	3569
14	0.2390	2390
15	0.2629	2629
16	0.2720	2720
17	0.2691	2691
18	0.2492	2492
19	0.3780	3780
20	0.1058	1058
21	0.1087	1087
22	0.2991	2991
23	0.1923	1923
24	0.2233	2233
25	0.2717	2717
26	0.1568	1568
27	0.2436	2436
28	0.0483	483
29	0.3644	3644
30	0.3430	3430
31	0.3073	3073
32	0.2879	2879
33	0.4323	4323
34	0.2248	2248
35	0.2462	2462
36	0.1828	1828
37	0.3087	3087
38	0.1954	1954
39	0.0229	229
40	0.2263	2263
41	0.3631	3631
42	0.3922	3922
43	0.1410	1410
44	0.2304	2304
45	0.4004	4004
46	0.3967	3967
47	0.3691	3691
48	0.3523	3523
49	0.2825	2825
50	0.1862	1862
51	0.3013	3013
52	0.3442	3442
53	0.1766	1766
54	0.2697	2697
55	0.1262	1262
56	0.1868	1868
57	0.1334	1334
58	0.2749	2749
59	0.3859	3859

Table A10. Cont.

Number	Al (wt.%)	Al (ppm)
60	0.3686	3686
61	0.3535	3535
62	0.2112	2112
63	0.3058	3058
64	0.2566	2566
65	0.1659	1659
66	0.2066	2066
67	0.1901	1901
68	0.3127	3127
69	0.3105	3105
70	0.2655	2655
71	0.2858	2858
72	0.3433	3433
73	0.2875	2875
74	0.3048	3048
75	0.1581	1581
76	0.1571	1571
77	0.2357	2357
78	0.3030	3030
79	0.2120	2120
80	0.3193	3193
81	0.0523	523
82	0.2175	2175
83	0.1466	1466
84	0.0277	277
85	0.1672	1672
86	0.0829	829
87	0.1065	1065
88	0.0467	467
89	0.1936	1936
90	0.1062	1062
91	0.0421	421

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