Value-added Alumina Product from Low-grade Feedstock: From Concept to Test Work and Beyond

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https://doi.org/10.71659/icsoba2025-aa051

Abstract

Currently, the Bayer process is the predominant technology used to produce smelter grade alumina. Due to the dwindling supply of high-grade bauxite, coupled with the challenges of processing red mud generated by the energy intensive Bayer process, there is an interest in exploring options of alumina extraction from "low-grade" mineral resources. The Thor Polymetallic Black Shale deposit is a sizeable low-grade aluminium deposit, located in Tisdale, Saskatchewan, which is under development by the Canadian Energy Metals Corp. (CEM). Hatch has developed a preliminary flowsheet to process the Black Shale deposit to produce value-added alumina products. The flowsheet comprises acidic leaching, hydrometallurgical purification and recovery, and pyrometallurgical upgrading. This paper presents results from a related, iterative, proof-of-concept bench-scale test-work program, conducted at the Saskatchewan Research Council (SRC) in 2024. CEM commissioned a second, larger-scale, semi-continuous pilot program which is currently underway at SRC.

Keywords: Black Shale, Aluminium Chloride Hexahydrate (ACH), Smelter Grade Alumina (SGA), Chemical Grade Alumina (CGA), High Purity Alumina (HPA).

1. Background and Motivation for Study

Currently, the energy-intensive Bayer process is the predominant method for the production of Smelter Grade Alumina (SGA). The process involves leaching of bauxite in an alkaline solution at elevated temperatures and high pressure, followed by precipitation of gibbsite which is then calcined to produce alumina. The Bayer processing typically requires that the bauxite Al_2O_3 to SiO_2 mass ratio is greater than 8 (Jiang et al., 2012) – this necessitates the use of high-grade bauxite. The global reserves of high-quality bauxite are in decline; thus, the development of flowsheet concepts that are alternative to the classical Bayer process is of interest (Cheng et al., 2012, Kyriakogona et al., 2017), which has led to extensive research since the 1940's.

Alternative paths to produce alumina as described in the literature include the processing of clay-based feedstock with hydrochloric acid (Hoffman et al., 1946). Hoffman and co-workers showed that roasting of an aluminous (kaolinitic) clay at 700 °C, followed by leaching of the calcined product with hydrochloric acid led to the efficient separation of siliceous impurities from solution. The resulting solution was then crystallized in the presence of gaseous hydrogen chloride (HCl) to form an "intermediary" Aluminium Chloride Hexahydrate (ACH) product, which upon further calcination, formed alumina.

In the 1970s and 1980s, the US Bureau of Mines conducted independent bench-scale and mini-plant scale research to improve processing methods for a kaolin-based feedstock and obtained preliminary data for the subsequent design of a 25-tonne-per-day alumina pilot plant. The flowsheet comprised hydrochloric acid leaching, and gas sparging crystallization technology (Maysilles et al., 1982). The work demonstrated the technical feasibility of ACH production which was partially compliant with purity criteria for Smelter Grade Alumina, though the study did not examine the calcination of the ACH product.

Advanced Energy Minerals Inc. produces value added alumina products using the patented Chlorine Leach Crystallization Purification (CLCP) process. The Cap-Chat plant in Quebec is a full-scale plant that produces specialty alumina products including 3N and 5N high-purity alumina (HPA) from an aluminium tri-hydrate (ATH) feed.

Anorthosite (a rock type primarily comprised of feldspar) is another feedstock that has been researched as a source of alumina, primarily through processing by the lime-soda-sinter process and the melt-quench technologies routes. The former process involves sintering anorthosite with soda ash and limestone followed by leaching, desilication, and precipitation prior to alumina recovery as a tri-hydroxide (Brown et al., 1947). Calcination of the hydroxide product is expected to yield alumina The latter process involves arc melting of anorthosite at 1650 °C followed by rapid cooling to form an amorphous product that is amenable to leaching (Leitch et al., 1965). Acid leaching of anorthosite has been previously tested using a lixiviant composed of hydrochloric acid (HCl) and fluorides and has shown promise as a potentially viable process route (Bremner et al., 1982).

A common, and important feature of hydrochloric acid processes is the regeneration of the lixiviant for re-use; thereby, reducing processing costs (Demopoulos et al., 2008). This typically involves pyrohydrolysis of aluminium chloride hexahydrate (ACH), and in some cases iron chloride ($FeCl_3$), to regenerate the HCl, which is captured, condensed and recycled back to the process. A further advantage of processes using hydrochloric acid is the ability to achieve effective silica separation during leaching (Wu et al., 2014, Zhang et al., 1950).

2. Introduction

In the present study we establish an experimental basis for a hydrochloric acid-based technology, to valorise a low-grade "black shale" type of feedstock from Tisdale, Saskatchewan, for the production of Chemical Grade Alumina and High Purity Alumina.

According to a mineral resource estimate (NI 43-101 Technical report, Thor project-2024 Stantec Consulting, January 15, 2025), the "black shale" deposit, also known as the "Thor" deposit, spans 987 square miles. To date, exploration and evaluation of a portion of the deposit representing approximately 22% of the deposit surface area has identified an estimated resource of approximately 43 billion tonnes (measured and indicated, ore basis). Accordingly, the resource has been estimated to contain approximately 6 billion tonnes of contained alumina equivalent. A photo of "black shale" core samples located at the CEM warehouse in Saskatoon, Saskatchewan is shown in Figure 1. Composites generated from core samples were used in test-work described herein.

The mineral resource estimate established an average stripping ratio of 0.3 waste tonnes per tonne of resource and potentially all resource is surface mineable. Furthermore, the mineral resource estimate reports that the "black shale" deposit includes additional metallization of high value elements such as V, Sc and other REE (rare earth elements). Future study may evaluate whether this additional metallisation can be exploited to improve project viability. At the time of writing this manuscript, the Thor processing flowsheet design has been updated to accommodate the production of a mixed oxide product which would include scandium oxide, vanadium oxide, and rare earth oxides (discussion of same is outside the scope of this technical summary).

The Thor black shale resource is situated in the province of Saskatchewan, which is a favourable jurisdiction that has implemented a streamlined project development/approval process as well as policies to encourage critical metals development. Furthermore, Thor's Tisdale location is particularly advantageous due to its proximity to power, rail, highway infrastructure, and a highly skilled labour force.

Figure 1. Core sample of the black shale deposit at the CEM warehouse in Saskatoon.

Thor-related metallurgical test-work (methodology, conditions, and results) is elaborated upon in the following sections of this paper. The objective of the test work program is to establish a conceptual design for the production of high value alumina products from mineral-rich black shales.

Subsequent sections of this paper describe the methodology and key findings of a bench-scale test-work program that simulated key unit operations of a preliminary flowsheet design for extraction of alumina from a black shale feedstock wherein a predominant fraction of the aluminium is deported to clays. The flowsheet simulated comprises comminution, followed by atmospheric stirred-tank leaching, followed by solvent extraction, followed by crystallization to obtain a high-purity intermediary (ACH), followed by calcination of the intermediary to obtain CGA.

At the time of writing this manuscript, analysis and test-work is in progress to evaluate the potential for producing SGA.

3. Materials and Methods

3.1 Comminution

Three ore types were identified (namely, low, medium, and high-grade, which are of relatively similar mineralogy, and varying in metallization, with high-grade representing highest aluminium content). Accordingly, representative composites were prepared, and each was subjected to crushing, with a target P_{80} of 25 mm. Figure 2 (cumulative mass fraction vs. particle diameter) describes the Particle Size Distribution (PSD) of six representative samples of the Medium-Grade Composite. Additional test work is currently in process to evaluate the benefits of processing a finer grind ore, P_{80} 200 microns (and finer).

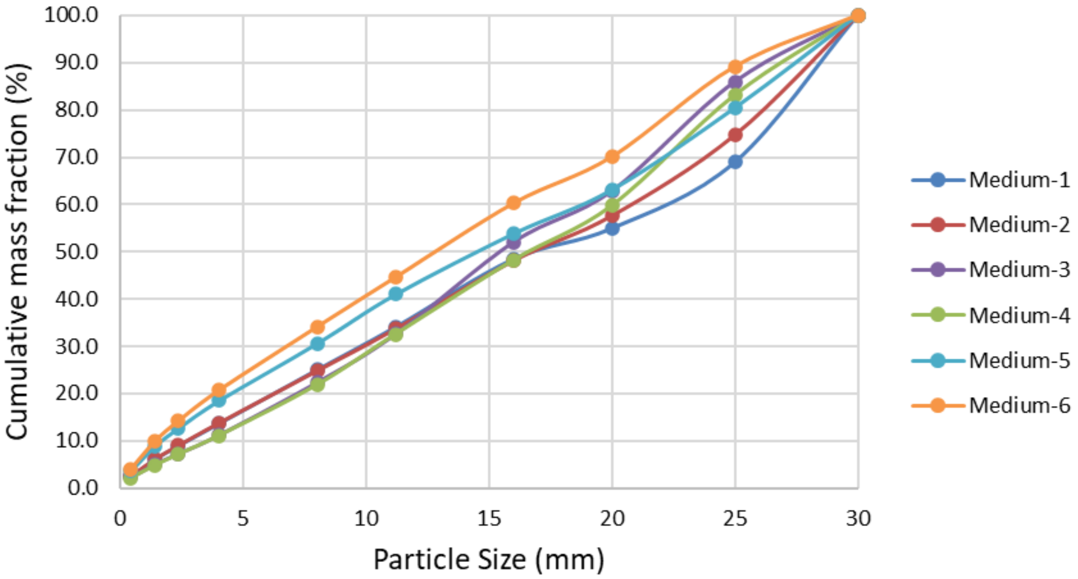


Figure 2. Particle Size Distribution (PSD) of the medium-grade composite.

Core samples of the high- and low-grade composites were analysed for their “abrasion index” and “crusher work index” estimate and were determined to be relatively soft (average crusher work indices of 11.1 and 9.9 kWh/t, respectively). The composites were categorized as “mildly abrasive” (measured bond abrasion indices of 0.003 and 0.005 for the high- and low-grade composites, respectively).

3.2 Feed Characterization

Feed characterization tests were completed to quantify elemental composition and modal as well as to determine specific gravity (SG), and bulk density of the black shale feed.

Representative samples of the composites underwent acid digestion and Inductively Coupled Plasma (ICP) scan to obtain the composites elemental composition. Elements present at wt. % level were assayed by Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) whereas elements present at ppm level were assayed by Inductively Coupled Plasma-Mass Spectrometer (ICP-MS).

Table 1 summarizes the chemical composition of the composites (certain elements are reported as oxide equivalent, which does not necessarily denote presence of the oxide). The high-grade composite was measured to have the highest aluminium content, whereas the low-grade composite, the lowest. A reverse trend is seen in vanadium and base metals (e.g. - nickel and cobalt). Of note, the black shale contains minor amounts of potentially radioactive elements (U, Th).

A detailed radionuclide assessment is planned as part of an on-going pilot test work program. Tailings and residue streams will be closely monitored to determine the extent of deportment of radionuclides in them. In addition, the present leach test work has confirmed that the leach recovery of U and Th is low and the organic extractant used in the iron solvent extraction does not load these radionuclides. In consideration of the above, the radionuclides, if any, are expected to report to a final mixed oxide calcined product. Consequently, accumulation of radionuclides in the system is not expected.

Table 1. Chemical composition of the black shale feed.

Element/Phase	Units	Low-grade composite	Medium-grade composite	High-grade composite
Aluminium Oxide	wt.%	12.9	13.1	14.0
Calcium Oxide	wt.%	9.61	8.33	0.47
Iron Oxide	wt.%	4.86	5.25	5.13
Titanium Dioxide	wt.%	0.51	0.54	0.60
Scandium	ppm	10.5	11.1	12.0
Vanadium	ppm	862	652	110
Magnesium Oxide	wt.%	1.22	1.25	1.52
Nickel	ppm	140	122	37
Cobalt	ppm	18.3	17.4	11.9
Manganese Oxide	wt.%	0.023	0.026	0.027
Copper	ppm	97.7	85.2	31.2
Lithium	ppm	62	53	40
Cerium	ppm	60	67	72
Dysprosium	ppm	5.56	6.47	5.71
Erbium	ppm	3.66	3.96	3.52
Europium	ppm	1.34	1.44	1.22
Neodymium	ppm	39.7	57.1	42.8
Praseodymium	ppm	12.3	17.9	15.9
Lanthanum	ppm	40.0	44.8	40.1
Lutetium	ppm	0.53	0.60	0.54
Niobium	ppm	13.8	15.0	16.8
Uranium	ppm	26.9	21.2	6.7
Thorium	ppm	8.77	9.79	12.84
Silica	wt.%	43.2	47.2	63.6

Quantitative XRD (Rietveld XRD) was used to obtain a preliminary quantitative estimate of mineralogical phases of the composites. Subsequently, QEMSCAN (Quantitative Evaluation of Materials by Scanning Electron Microscopy) was used to quantify the modal mineralogy.

The results of a QEMSCAN analysis indicate that most of the aluminium is present in the clay minerals and that kaolinite is the major aluminium bearing mineral phase of the high-grade composite. Other clay phases identified by QEMSCAN include: illite, biotite, and montmorillonite. Iron was found to report to pyrite (primarily) as well as to silicate phases.

3.3 Leaching and Solid Liquid Separation

The initial leaching tests were carried out in a 2 L Stirred Tank Reactor (STR) operated in batch mode. The STR was equipped with an agitator and a reflux condenser. The lixiviant was a 20 wt.% HCl solution, the leaching was operated at a solid loading of 20 wt.% and the leach temperature was maintained at 95 °C for 40 h. During the tests, kinetic samples were withdrawn, filtered, and analysed for the dissolved metal contents by ICP scan. At the termination of a leaching test, the reactor contents were vacuum filtered, and the resulting filtrate was weighed, and a sub-sample of the filtrate was analysed for the dissolved metal contents by ICP scan. The leach residue was washed, dried, and acid digested to quantify the presence of metals in the residue to compile an overall mass balance.

3.4 Iron Solvent Extraction (SX)

The filtrate (PLS) from leaching was first oxidized using hydrogen peroxide to convert ferrous chloride in solution to ferric chloride (as readily extractable species by the SX extractant). Hydrogen peroxide was added to maintain an ORP of 800–900 mV vs. Ag/AgCl and the residence time was maintained at 1 h.

The oxidized Pregnant Leach Solution (PLS) was fed to the solvent extraction step. Batch-type bench-scale iron solvent extraction (SX) tests were performed using an organic phase composition consisting of 10 vol.% TBP extractant, 85 vol.% Calumet 400–500 diluent, and 5 vol.% Exxal 10 modifier. The extraction temperature was maintained at 40 °C and the mixing time was 10 min. The O:A ratio was maintained at 1:1. The raffinate collected from the test was weighed and analysed by ICP scans to estimate the concentration of dissolved metals and to quantify iron and impurity loading on the SX extractant.

3.5 ACH Crystallization - Stage 1 and Stage 2

The raffinate from the solvent extraction stage was processed in a two-stage ACH crystallization process. In a first stage of crystallization, iron SX raffinate was acidified to selectively precipitate "Stage 1 ACH" crystals. A portion of the "Stage 1 ACH" was redissolved and subjected to a second stage of crystallization, through acidification, to yield a 'Stage 2 ACH". Acidification tests with both concentrated HCl (36.5 wt.% HCl) solution and gaseous HCl (99 wt.% HCl) were conducted. Crystallization tests involving the use of concentrated HCl solution were conducted at 4 °C, while crystallization tests involving the use of gaseous HCl were conducted at 60 °C.

In Stage 1 tests employing concentrated HCl solution, acid was added to the iron SX raffinate at a mass ratio of approximately 8:1.

In Stage 2 tests, "Stage 1 ACH" was dissolved in a 125 g/L HCl solution and re-crystallized under the same conditions as the Stage 1 crystallization process.

Of note, ACH crystallization tests were conducted on relatively small batches of the raffinate using gaseous HCl. Due to the limited availability (and the cost) of HCl gas, relatively larger batches of iron SX raffinate were processed with concentrated acid in order to expediently generate a sufficient quantity of ACH for calcination testing (as described below).

3.6 ACH Calcination

Bench-scale calcination tests were conducted to evaluate the potential for acid regeneration and the properties of the alumina thus obtained.

Figure 3 shows the apparatus used for executing the first calcination test. The apparatus consists of a tube furnace operated under an inert atmosphere of nitrogen. ACH crystals were placed on an HPA "boat" of 4N-5N purity, which was situated in the middle section of an alumina tube in the furnace. Sweep nitrogen gas was introduced at one side of the tube (at the left in Figure 3) and the off-gas flowed from the tube (at the right in the figure) to a water-cooled condenser which adjoined a round-bottomed flask used to collect condensed acid of the off-gas (i.e. HCl liquid collection flask shown at bottom right in the figure). A vent port of the flask was routed to an NaOH scrubber to neutralize any residual acid in off-gas discharging from the flask.

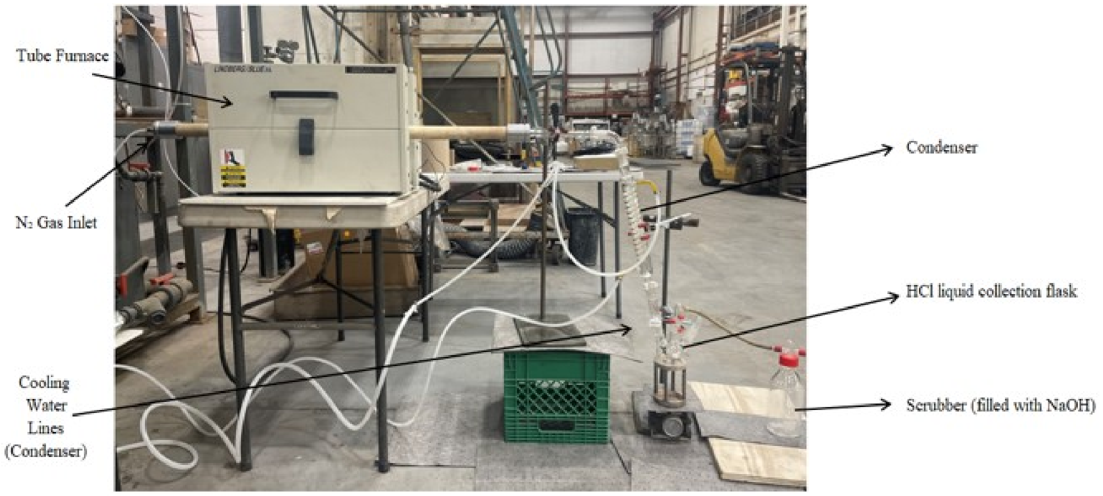


Figure 3. Batch calcination test apparatus.

The following procedure was used to prepare and calcine the ACH sample obtained from crystallization:

- The ACH crystals are washed with a high purity concentrated HCl solution at a S/L (Solid to Liquid) ratio of 1:4 prior to calcination.
- The ACH crystals are weighed, placed on an HPA "boat" and then inserted into the middle of the tube of the furnace.
- The flow of nitrogen is started, and the flow rate is carefully adjusted so that the flow is just sufficient to form gas bubbles in the "scrubber flask" filled with NaOH.
- The temperature in the middle section of the furnace is set to 150 °C and held at 150 °C for 30 minutes.
- The temperature is then ramped up at a rate of 50 °C/10 min and held at the new set point temperature for 10 minutes.
- This ramp up is continued until the set point temperature is 500 °C. This set point is maintained for 2 hours.
- The temperature is subsequently ramped up to 1000 °C and maintained at the set point for 1 hour and then further ramped up to 1200 °C and held there for 2 hours.
- At the end of the test, the furnace is shut down and the calcined alumina sample is allowed to cool down.

The ramp up profile and the temperature described were chosen to allow sufficient time for reaction, in order to allow a first assessment of obtainable product purity.

4. Results and Discussions

4.1 Leaching of Metals from Black Shale

8 kg of the high-grade composite underwent STR leaching and solid/liquid separation according to the method of section 3.3. Approximately, 77 % aluminium, 79 % scandium, 79 % vanadium, and 62% iron were solubilized during the procedure. Figure 4 shows the apparatus used for leaching, as well as kinetic aqueous samples collected, and the final residue generated after filtration and drying.

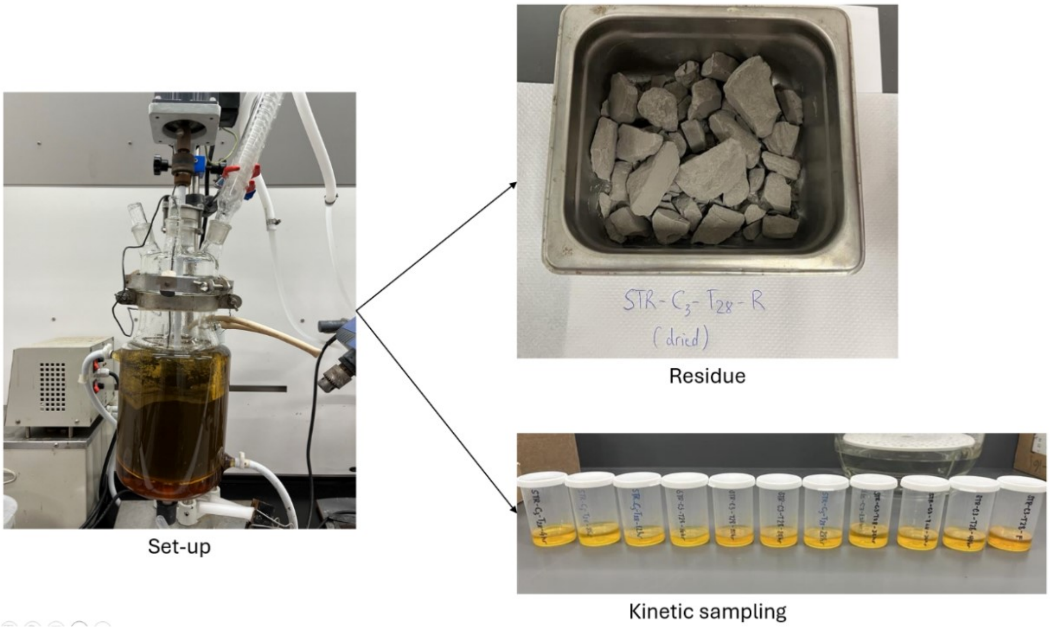


Figure 4. Stirred tank reactor used for the leaching test: the black shale feed, aqueous kinetic samples, and the high silica residue.

The residue was primarily composed of SiO_2 (73 wt.%) and undissolved metals (Table 2). Of note, the leach residue composition is similar to the composition of "pozzolans" – a typical additive used in the cement industry in concrete countertop mix designs.

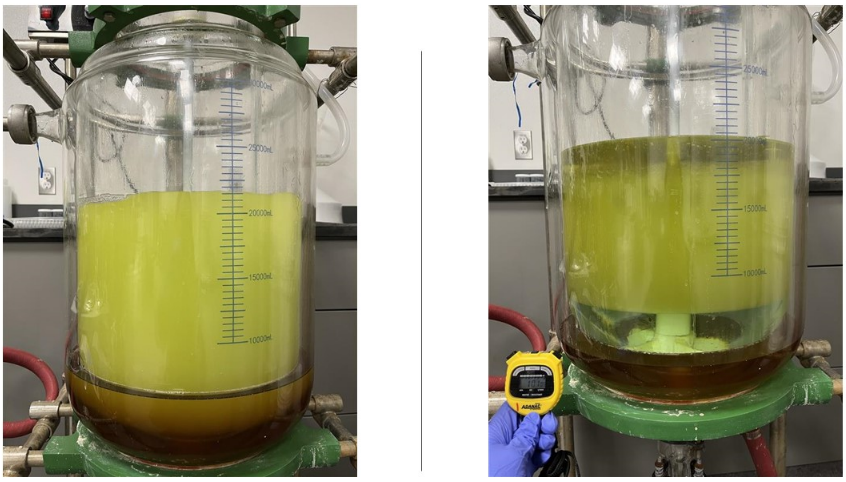
Table 2. Chemical Composition of leach residue.

Element/Phase	Units	Leach residue composition
Aluminium Oxide	wt.%	3.8
Calcium Oxide	wt.%	0.06
Iron Oxide	wt.%	2.33
Titanium Dioxide	wt.%	0.595
Scandium	ppm	3
Vanadium	ppm	28
Magnesium Oxide	wt.%	0.11
Nickel	ppm	7
Cobalt	ppm	2
Manganese Oxide	wt.%	0.01
Sodium Oxide	wt.%	0.26
Copper	ppm	18
Lithium	ppm	9
Cerium	ppm	14
Dysprosium	ppm	1.3
Erbium	ppm	0.9
Europium	ppm	0.3
Neodymium	ppm	5
Praseodymium	ppm	1
Uranium	ppm	< 2
Thorium	ppm	2
Sulphur	ppm	16 600
Silica	wt.%	73.05

Natural pozzolans include clay and diatomaceous earth and the major chemical components of natural pozzolans are silica, alumina, and iron oxide. The sum of the mass composition of these three oxide phases must be at least 70 wt.% for the material to be considered suitable as a "pozzolan". (Mohamed et.al., 2022). The combined mass composition of these phases in the leach residue is 79 wt.%. Contingent on further testing in accordance with TS 25 standards for pozzolan, the leach residue has the potential to be a value-add high volume pozzolan by-product that may be well suited to be used as a mineral admixture on further processing. The practice of using mineral admixtures as a partial replacement of clinker in Portland cement are increasingly being adopted by the cement industry to reduce the CO_2 footprint (Schneider et al., 2011; Damtoft et al., 2008; Scrivener et al., 2018).

4.2 Impurity (Iron) Removal by Solvent Extraction (SX)

Approximately 24.9 kg of the PLS generated by leaching was subsequently processed in an iron removal solvent extraction procedure, according to the method of section 3.4, using the apparatus shown in Figure 5.



Mixing

Settling

Figure 5. Apparatus used for the bench-scale iron solvent extraction test
Left: uniform emulsion of extraction phase; Right: loaded organic phase on top and iron-free raffinate at the bottom. jacketed reactor to maintain temperature at target value.

Solvent extraction tests achieved a raffinate iron concentration lower than 1 ppm. A comparison of the iron SX feed and raffinate shows that the loading process was relatively selective towards iron (Fe^{3+}) (Table 3). Minimal co-loading of impurities such as Na, K, Mg, and Ca was observed.

Table 3. Comparison of the feed and raffinate compositions.

Analyte	Concentration (ppm) in the feed to solvent extraction	Concentration (ppm) in the raffinate from solvent extraction
Aluminum Oxide	11700	10000
Iron	45	<1
Calcium Oxide	500	500
Magnesium Oxide	1600	1700
Sodium Oxide	1300	1200
Potassium Oxide	1500	1300
Scandium	0.985	0.049
Vanadium	9.24	8.63
Titanium	38.4	5.88

4.3 ACH crystallization

Iron SX raffinate was fed to the first stage of ACH crystallization. The crystallizer was operated according to the method of Section 3.5 to generate “Stage 1 ACH” and “Stage 2 ACH”.

“Stage 1 ACH” included the impurities Na, Mg, Cr, Mo, Pb, Ni, and S. “Stage 1 ACH” was washed with high-purity concentrated HCl solution to remove impurities while minimizing the dissolution of Al. Washed “Stage 1 ACH” crystals were estimated to have a purity of 99.97 wt.% ACH, which if calcined, is expected to yield a chemical grade alumina (CGA).

“Stage 2 ACH” was produced by dissolution of “Stage 1 ACH” and re-crystallization, which proved effective in reducing impurity concentrations: MgO composition was reduced from 0.04 to 0.01 wt.%, while the sodium oxide and the sulfur composition were reduced below the detection limit of the ICP-OES. A composition comparison of washed “Stage 1 ACH” and unwashed “Stage 2 ACH” is presented in Table 4.

Table 4. Comparison of STAGE 1 ACH and Stage 2 ACH.

Analyte	Units	Stage 1 crystal composition (unwashed)	Stage 2 crystal composition (washed)
Aluminium Oxide	wt.%	21.3	20.9
Calcium Oxide	wt.%	< 0.01	< 0.01
Iron oxide	wt.%	< 0.01	< 0.01
Potassium Oxide	wt.%	< 0.01	< 0.01
Magnesium Oxide	wt.%	0.04	0.01
Sodium Oxide	wt.%	0.02	< 0.01
Scandium	ppm	< 1	< 1
Titanium Oxide	wt.%	< 0.01	< 0.01
Phosphorous Pentoxide	wt.%	< 0.01	< 0.01
Chromium	ppm	10	7
Silver	ppm	< 0.2	< 0.2
Barium	ppm	< 1	<1
Molybdenum	ppm	1	1
Lead	ppm	2	2
Sulfur	ppm	11	< 10
Nickel	ppm	1	< 1
Cobalt	ppm	<1	< 1

Figure 6 shows the apparatus, and the ACH crystals produced in Stage 2.

Set-up ACH Product

Figure 6. Crystallization apparatus and ACH crystals.

4.4 ACH Calcination

"Stage 2 ACH" was prepared and calcined according to the method of Section 3.6.

"Stage 2 ACH" crystals were washed with concentrated HCl at a solid/liquid (S/L) ratio of 1:4 prior to calcination. Table 6 below compares the impurity composition of the unwashed and washed crystals. Elemental impurities are reported on an oxide equivalent basis. The purity of the ACH crystals before washing was estimated to be 99.97 wt.%. Washing resulted in an increase in purity to approximately 99.99 wt.%. The assays reported in Table 5 are an average of the assays from a combined fusion and a four-acid digestion protocol.

Table 5. Comparison of the Stage 2 ACH crystal composition before and after washing.

Analyte	Units	Composition before washing	Composition after washing
Aluminium Oxide	wt.%	21.1	15.85
Calcium Oxide	wt.%	0.0023	0.0022
Iron Oxide	wt.%	0.0028	0.0007
Potassium Oxide	wt.%	0.0043	ND
Magnesium Oxide	wt.%	0.0004	0.002
Sodium Oxide	wt.%	0.0083	ND
Scandium	ppm	ND	ND
Titanium Oxide	ppm	ND	ND
Potassium Oxide	wt.%	ND	ND
Chromium Oxide	wt.%	0.0008	ND
Barium Oxide	wt.%	ND	ND
Molybdenum	wt.%	ND	ND
Lead	wt.%	ND	ND
Sulfur	ppm	0.00025	0.0011
Nickel Oxide	ppm	0.0009	ND

ND = Not Detected - refers to any ICP measurements that are below the detection limits of the ICP-OES and the ICP-MS with reference to the element being analysed.

The alumina product obtained from calcination was further washed with De-Ionized (DI) water at a S/L ratio of 1:7.5 and assayed. Major impurities of the unwashed product included Na (354 ppm), Si (70 ppm), Ca (47 ppm), Mg (28 ppm), Fe (14 ppm), resulting in an estimated alumina purity of 99.924 wt.% Al_2O_3. In contrast, the washed alumina product showed a ten-fold reduction in sodium concentration and a 50% reduction in the Mg concentration resulting in a higher purity alumina product (99.975 wt.% Al_2O_3). Table 6 compares the composition of the unwashed and washed alumina product.

Table 6. Alumina product composition comparison – unwashed vs. washed.

Analyte	Units	Unwashed Alumina	Washed Alumina
Barium Oxide	wt.%	ND	ND
Calcium Oxide	wt.%	0.00658	0.00588
Cadmium Oxide	wt.%	ND	ND
Cobalt Oxide	wt.%	ND	ND
Chromium Oxide	wt.%	ND	ND
Iron Oxide	wt.%	0.002	0.0018
Gallium Oxide	wt.%	ND	ND
Potassium Oxide	wt.%	ND	ND
Lithium Oxide	wt.%	ND	ND
Magnesium Oxide	wt.%	0.0046	0.0021
Manganese Oxide	wt.%	ND	ND
Molybdenum Oxide	wt.%	ND	ND
Sodium Oxide	wt.%	0.047	0.0049
Nickel Oxide	wt.%	ND	ND
Phosphorus pentoxide	wt.%	ND	ND
Lead Oxide	wt.%	ND	ND
Antimony Oxide	wt.%	ND	ND
Scandium Oxide	wt.%	ND	ND
Silicon Oxide	wt.%	0.015	0.010
Titanium Oxide	wt.%	ND	ND
Alumina Purity	wt.%	99.924	99.975

ND = Not Detected - refers to any ICP measurements that are below the detection limits of the ICP-OES and the ICP-MS with reference to the element being analysed.

The Figure 7 shows the alumina product that was obtained from calcination of the “Stage 2 ACH”. Per the assay presented above, the purity of the washed calcined alumina was demonstrated to be between 3N and 4N (subsequent testing is investigating further improvements to purity levels by incorporating additional washing steps upstream of calcination to further increase the purity of the ACH precursor as well as incorporating additional washing steps on the calcined alumina product).

5. Conclusions and Future Work

The present study has established an experimental basis for preliminary flowsheet development to produce CGA and HPA from a low-grade, clay-based, black shale feedstock. Specifically, the test-work investigated the following unit operations of such a flowsheet critical to the production of a high purity alumina product:

- Leaching and solid liquid separation.

- Iron solvent extraction.
- Two-stage aluminum chloride hexahydrate crystallization.
- Calcination of aluminum chloride hexahydrate to produce High Purity Alumina.

Figure 7. High purity washed alumina product from calcination of “Stage 2 ACH”.

Leaching of the black shale in hydrochloric acid at 95 °C and atmospheric pressure yielded greater than 70 % aluminium recovery with a coarse grind ore sample.

Iron solvent extraction was demonstrated to be relatively selective (further optimization is in progress) and produced a raffinate containing less than 1 ppm of iron, as well as a loaded organic low in impurities.

ACH Crystals produced in Stage-1 and Stage-2 crystallization circuits have a purity of greater than 99.5 wt.%. Specifically, washed crystals produced from the Stage-2 crystallization circuit have a purity of 99.99 wt.% ACH.

Calcination tests of the “Stage 2 ACH” yielded an alumina product with (greater than) 3N purity. At the time of writing this paper, further optimization tests are underway to increase the purity of the alumina product.

The present test work program has informed the development of a preliminary flowsheet concept to valorise alumina from a low-grade black shale feedstock. The process has significant technical and economic advantages over traditional processing methods – the residue produced from leaching could potentially be a high volume pozzolan type by product that could be used in the cement industry; thereby, potentially reducing the tailings tonnage. Most significantly, the present process could be viewed as a sustainable alternative to the traditional methods of producing HPA: aluminium metal produced from bauxite is further processed to HPA. Such traditional methods involve energy intensive processing; in contrast, the present process is expected to have a lower CAPEX intensity and a lower carbon footprint.

In summary, whereas bauxite processing generates a red mud residue that requires disposal and long-term management, clay processing avoids such a material, and on the contrary may generate residues that are saleable.

The data from the test-work has provided the design criteria for a semi-continuous pilot operation initiated in 2025 at the SRC. Data from the pilot would be used to further process development and ultimately provide design criteria for a commercial demonstration plant in 2026.

6. Acknowledgements

The authors wish to acknowledge the Canadian Energy Metals management team (Christopher Hopkins, Director and CEO and President; Gary Billingsley, Director and Chief Operating Officer; and David Dukes, Resource Exploitation Manager) for their support as well as their very insightful comments and feedback on this paper and many topics, for reviewing this manuscript and for their insightful and critical feedback that helped improving the manuscript.

The authors wish to acknowledge the assistance of the Saskatchewan Research Council (SRC) team (in particular, Baodong Zhao, Manager and Supervisor of Mineral processing; Thi Thu Doan Nguyen, Technologist) for their diligent and timely completion of laboratory tests involving leaching, solvent extraction, and crystallization.

The authors wish to acknowledge the assistance of the Process Research Ortech team (in particular, Jonathan Chen, CEO; and Sahil Kumar, Chief Metallurgist) for their diligent and timely completion of laboratory tests involving calcination.

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