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# $CO_2$-assisted selective recovery of magnesium from brucite dolomites at Budureasa for the production of magnesium chloride as a raw material

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**Abstract:** This study evaluates $CO_2$-assisted magnesium recovery from Budureasa brucitic dolomites using historical laboratory records and three additional one-hour repeatability tests. The proposed route avoids direct hydrochloric-acid leaching of the calcium-rich feed: magnesium is extracted in a mechanical flotation cell, precipitated, and the recovered filter cake may subsequently be dissolved in HCl to produce $MgCl_2$ feedstock. Historical tests used thermal activation at 650 °C. The additional tests were conducted at 21 °C, with a feed grade of 22.76 wt.% MgO equivalent, a recorded dilution of 1:10, a $CO_2$ flow of 5 $dm^3$/min, a nominal cell capacity of 3 $dm^3$ and 800 $min^{-1}$ yielded extractions of 60.7%, 62.1% and 61.3% (mean 61.37%, sample standard deviation 0.70 percentage points, n = 3). These results support repeatability under a single condition. Calcination at 850 °C was used for analytical mass stabilization, not as a required downstream production step; the reported 99.06 wt.% MgO equivalent does not establish periclase phase purity. The historical 4.5 Mg/h product projection is distinguished from a recalculated theoretical maximum of 4.160 Mg/h at 72% Mg extraction and an assumed 99% MgO-equivalent product grade, before downstream losses. The proposed direct HCl conversion, environmental advantages, and overall experimental Mg balance remain unverified.

***Keywords:*** brucitic dolomite, $CO_2$-assisted leaching, flotation cell contactor, magnesium recovery, repeatability, magnesium chloride precursor, acid consumption

## 1. Introduction

Brucitic dolomites contain magnesium as both brucite and carbonate minerals. Differences in thermal response can activate the hydroxide-containing fraction while preserving much of the carbonate matrix. The required temperature depends on heating history, residence time, and atmosphere; therefore, carbonate preservation must be measured rather than inferred solely from furnace temperature.

$CO_2$-assisted processing and Mg/Ca separation are well-established research topics. Magnesium carbonate precipitation depends on temperature, gas pressure, and phase kinetics (Hänchen et al., 2008). Benzesik et al. (2026) investigated an integrated process comprising dolomite calcination at 1100 °C, hydration, Ca–Mg separation via carbonation and crystallization, and subsequent aluminothermic reduction of the recovered MgO to metallic magnesium under vacuum. This study provides a recent precedent for integrated Ca–Mg separation, although it differs from the present investigation in terms of heat treatment, reactor application, and the target end product. Thus, using $CO_2$ or producing MgO after precipitation cannot, in itself, constitute novelty.

$CO_2$-assisted leaching of calcined magnesium-containing materials and intensified contact have precedents in the literature. Zhu et al. (2008) developed a suspension-recirculation reactor for magnesium oxide leaching, which involves premixing the suspension with $CO_2$ and compares performance with operation without recirculation. Sevim and Atalay (2023) investigated ultrasound-assisted dissolution of magnesia in aqueous $CO_2$, providing a recent example of a different intensification approach. Freitas et al. (2021) introduced $CO_2$ via a bubble-generation system in flotation equipment to separate calcite from apatite at laboratory and pilot scale. This application pertains to

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flotation separation, whereas the present operation aims to transfer magnesium into solution. These precedents show that neither $CO_2$ leaching nor introducing $CO_2$ into flotation equipment, taken separately, is novel.

The contribution examined here is the documented application of a mechanical flotation cell as a gas-liquid-solid contactor to transfer magnesium from thermally activated Budureasa material into an aqueous carbonate system, supplemented by three one-hour tests of extraction repeatability under a single operating condition. The proposed processing strategy differs from direct acid leaching of the original Ca-rich rock: Mg is first separated via a $CO_2$-assisted aqueous route and subsequently recovered as a Mg-containing filter cake, which could be dissolved in HCl to produce $MgCl_2$ as a raw material. This separation aims to limit exposure of the original calcium-rich matrix to HCl and the formation of acid-leaching solutions rich in calcium chloride; the amount of calcium carried over into the intermediate product and its subsequent fate remain to be determined. An environmental benefit is a process hypothesis, not a proven result. Specific reactor improvements regarding $CO_2$ mass transfer, cavitation, or process intensification cannot be substantiated without adequate measurements and tests using reference reactors.

## 2. Materials and methods

Two technical reports from INCDMRR document the initial laboratory enrichment investigation and the subsequent $CO_2$ extraction investigation. The raw-material characterizations refer to distinct sampled materials and are not combined into a single raw-material analysis. According to the reports, the samples from the extraction study were preserved during a hiatus in the research and were subsequently included in the subsequent characterization. Tables 5–8 are derived from laboratory measurements conducted by the authors at INCDMRR, with a single determination for each historical operating condition (n = 1). In response to the review, three additional one-hour tests were conducted under fixed operating conditions; these are reported separately and are not retrospectively assigned to the historical experimental series. Results derived from reports, historical design quantities, new repeatability measurements, and hypothetical proposals regarding downstream processes are explicitly distinguished.

Table 1. Classification of samples in the datasets

| Data set | Sample status |
|---|---|
| Raw material mineralogy and MgO content | Enrichment report: chemical composition, qualitative XRD analysis, and thermal analysis; lot-specific material |
| 650 °C route and flotation cell tests | The extraction report documents previous tests (Tables 5–8: one determination per condition); three new one-hour tests performed are reported separately in Table 4 |

SC GEOASSET SRL supplied the enrichment sample, which was homogenized to collect laboratory subsamples. These subsamples represent the supplied batch, not the entire Budureasa–Valea Mare deposit. The report explicitly notes unusually low silicate and aluminum oxide contents and interprets the material as originating from a brucite-rich zone with more favorable beneficiation behavior than the deposit as a whole. The material for the extraction tests was sampled from a batch industrially crushed at Ștei; no documented sampling plan covers the entire deposit. Therefore, the results apply only to the materials tested.

### 2.1. Historical chemical characterization

Table 2 presents XRF analyses performed by SC PROSPECȚIUNI SA. Values are reproduced as reported, without normalization. Tests were performed using XRF. The loss on ignition was determined by heating to approximately 1000 °C—the listed oxides, together with the loss on ignition, total 99.52% by weight. The chemical composition equivalent to the oxides does not identify any mineral phases.

Table 2. Chemical composition of the main oxides and calcination loss of the supplied beneficiation batch (Source: INCDMRR beneficiation report)

| Component | Reported Weight Percentage | Detection limit (%) by weight |
|---|---|---|
| $SiO_2$ | 0.42 | 0.01 |
| $TiO_2$ | 0.01 | 0.01 |
| $Al_2O_3$ | 0.03 | 0.01 |
| MnO | 0.02 | 0.01 |
| $Fe_2O_3$ | 0.13 | 0.01 |
| MgO | 23.18 | 0.01 |
| CaO | 33.12 | 0.01 |
| $Na_2O$ | 0.09 | 0.01 |
| $K_2O$ | 0.06 | 0.01 |
| $P_2O_5$ | 0.18 | 0.05 |
| Loss on ignition | 42.28 | 0.10 |

### 2.2. Historical characterization by XRD and thermal analysis

Table 4 presents the reported distances. No synthetic diffractogram was constructed. The report includes a diffractogram of the initial sample (Fig. 9), but it provides neither the Rietveld quantitative refinement nor the raw diffraction file. At a heating rate of 10 °C/min up to 1000 °C, the thermal analysis reports effects around 420 °C for brucite dehydroxylation, 880 °C for calcite decomposition, and 800 °C associated with dolomite. The reported percentages of 23.2% brucite and 42.7% calcite are estimates from thermal analysis, not quantitative results from X-ray diffraction (XRD). However, to confirm carbonate preservation after the 650 °C treatment, the calcined product must be characterized.

Table 3. Qualitative identification of phases by powder X-ray diffraction for the enrichment batch (Source: INCDMRR Enrichment Report)

| Phase | Formula | Reported d-spacings (Å) |
|---|---|---|
| Brucite | $Mg(OH)_2$ | 4.77; 2.72; 2.36; 1.79; 1.57; 1.49 |
| Calcite | $CaCO_3$ | 3.03; 2.49; 2.28; 2.09; 1.91; 1.87; 1.60; 1.58; 1.52 |
| Dolomite | $CaMg(CO_3)_2$ | 2.89; 2.67; 2.54; 2.40; 2.19; 2.06; 2.01; 1.84; 1.80; 1.56; 1.54; 1.49 |

### 2.3. Methods and calculation basis

The extraction report documents exploratory tests on a gram scale, followed by trials involving hundreds of grams. Dry classification at 74 μm yielded approximately 5 kg of ultrafine paste, which does not correspond to a single documented reactor batch. Initial activation was performed in an electric furnace set to 650 °C; the residence time in the furnace and the composition of the calcination phase remain incompletely documented. In the historical $CO_2$ leaching process, a mechanical flotation machine was used at reported ratios of 1:9–1:10, with a $CO_2$ flow rate of approximately 5 $dm^3$/min and a contact time of 10–15 min. The diluent, reference conditions, and $CO_2$ purity in those historical runs are not specified. This was followed by decanting without flocculants and filtration. Without batch records, the distinct series lasting several hours in the historical time–temperature series should not be associated with the 10–15-min conditions. The additional one-hour tests and their available operational reference values are presented separately below.

#### 2.3.1. Design of the additional one-hour repeatability tests

To assess the reproducibility of the results, three additional one-hour carbonation tests were conducted to supplement the experimental evidence and evaluate repeatability under a single operating condition. Each test used a mechanical flotation cell with a nominal capacity of 3 $dm^3$, a dilution ratio recorded as 1:10, a $CO_2$ flow rate of 5 $dm^3$/min, a rotor speed of 800 $min^{-1}$, and a test temperature of 21 °C. The declared raw material

content was 22.76% by weight MgO equivalent (chemical analysis based on oxide equivalent, not elemental magnesium). The value of 3 $dm^3$ represents the cell's nominal capacity, not the independently measured volume of the working suspension. The reported 1:10 ratio is retained in its original notation; the mass/volume convention and actual loading quantities must be specified from the batch records before independently calculating the solid or liquid stock. The purity of $CO_2$, standard flow conditions, rotor geometry, and complete particle size distribution were not provided. These tests were new additions to the review, not retrospective replications of historical factorial or kinetic experiments.

### 2.3.2. Magnesium extraction and product recovery

Consistently use elemental magnesium. For an initial mass of dry raw material $m_0$ and a mass fraction of Mg $wMg,_0$, the raw material inventory is $M_0 = m_0 wMg,_0$. If the grade of the raw material is reported as MgO equivalent, $wMg_0 = wMgO_0 \times 24.305/40.304$. An oxide-equivalent grade expresses the elemental composition and does not establish the presence of the MgO mineral phase.

For a batch sampled repeatedly, the dissolved magnesium at time t is $Mdiss(t) = VtCMg,t + \Sigma(Vsample,j\ CMg,j)$, with volumes and concentrations expressed in compatible units and with dilution corrections applied. The net dissolved extraction is $XMg(t) = 100\ Mdiss(t)/M_0$. This differs from the gross mineral dissolution if the dissolved Mg subsequently precipitates. Vt accounts for replacement liquid, evaporation, and sampling.

The final product recovery is $Rglobal = 100\ mpwMg,p/M_0$. A process balance must include leaching residues, precipitates, mother liquors, wash waters, samples, and other external outputs. The balance equation is $B = 100\ \Sigma Mout/M_0$ for a complete batch; recycling streams must not be counted twice. Stage efficiencies may be multiplied only when they refer to consecutive transfers along the same defined product path.

For sequential treatment of the same residual solid, the cumulative extraction is $Xcum,n = 100[1 - \Pi(1 - xj)]$, where xj is the extraction fraction of magnesium entering stage j. Enriching the solution with fresh feedstock is a separate operation and requires its own input balance.

### 2.3.3. Chemical basis of $CO_2$-assisted extraction

The chemical interpretation is qualitative and is based on reported process observations and the established chemistry of Mg–Ca–carbonate. In this study, no thermodynamic equilibrium model was implemented, calibrated, or validated, and no numerical operating window for selective Mg/Ca separation is claimed to exist. Section 4.6 distinguishes between the historical process flow reported by the source and a recalculated 72% extraction scenario, using explicitly stated contents. Neither of these represents a thermodynamic model or an experimentally closed process balance.

$CO_2$-assisted extraction transfers Mg from the activated material into the aqueous phase. Heating and the release of $CO_2$ subsequently produce a precipitate containing Mg. These descriptions do not establish aqueous speciation, mineral saturation, or the precipitate's crystalline identity. Calcium partitioning and selectivity must be evaluated based on correlated analyses of the solution and the solid, rather than inferred solely from the ratio of MgO equivalent to CaO equivalent in the solution (Stumm and Morgan, 1996; Hänchen et al., 2008).

## 3. Results and discussion

Tables 5–8 present historical measurements from the INCDMRR laboratory, with one determination per condition (n = 1). The reported relative changes are arithmetic transformations; historical trends in the parameters remain descriptive and do not establish statistically validated optimal values. The three additional 60-min measurements presented below are tests reported separately at a single fixed reference point; they do not provide replicable evidence for Tables 5–8 and do not form a closed mass balance of product yield.

### 3.1. Additional one-hour tests: Repeatability at one condition

For illustrative purposes, three additional one-hour carbonation tests were conducted as supplementary experimental verifications under a common nominal condition. The equivalent MgO extraction results

were 60.7, 62.1, and 61.3%. Their arithmetic mean was 61.3667%; the sample standard deviation (denominator n − 1) was 0.7024%, and the coefficient of variation was 1.14%. The range was 1.4 percentage points. These descriptive statistics characterize repeatability only under this tested condition; the historical multifactorial data remain single determinations for each condition. The independence between batches regarding sample preparation and the complete closure of the mass balance should be confirmed based on individual laboratory records. The extensive historical screening was organized with a single test per condition because of the high experimental workload; this practical constraint does not replace replication.

Table 4. Additional one-hour $CO_2$-assisted extraction tests conducted after the second revision; feed with an equivalent MgO content of 22.76% by weight, with descriptive results regarding repeatability

| Parameter | Test 1 | Test 2 | Test 3 |
|---|---|---|---|
| Duration (min) | 60 | 60 | 60 |
| Temperature (°C) | 21 | 21 | 21 |
| Dilution ratio (according to records) | 1:10 | 1:10 | 1:10 |
| $CO_2$ flow rate ($dm^3$/min) | 5 | 5 | 5 |
| Nominal cell volume ($dm^3$) | 3 | 3 | 3 |
| Rotor speed ($min^{-1}$) | 800 | 800 | 800 |
| Feed rate (% by weight, MgO equivalent) | 22.76 | 22.76 | 22.76 |
| Mg extraction (%) | 60.7 | 62.1 | 61.3 |

For the reported extraction percentages, the mean = 61.37%, the sample standard deviation = 0.70 percentage points, the CV = 1.14%, and the range = 1.4% (n = 3). The mean is numerically comparable to values ranging from 61 to 63% in some historical dilution and agitation tests, but those studies differ in terms of documented or undocumented operating conditions and cannot be grouped into a single set of replicates.

Table 5. Reported ratio of MgO equivalent in solution to CaO equivalent. Authors' measurements, INCDMRR laboratory; a single determination per condition (n = 1). Derived columns represent arithmetic transformations; replicate variability is not available

| MgO Extraction (%) | MgO/CaO ratio in solution | Increase in the ratio compared to the first determination (%) |
|---|---|---|
| 10.0 | 2.70 | 0.0 |
| 24.0 | 3.45 | +27.8 |
| 35.0 | 4.80 | +77.8 |

The ratio increases from 2.70 to 4.80 based on the reported extraction values. This indicates a relative enrichment of the solution, but does not prove the precipitation of calcium. Appropriate Ca analyses and residue characterization are required. The mass ratio of oxide equivalents should not be confused with an elemental or molar Mg/Ca ratio.

The extraction rate increases from 45.0% to 63.5% as dilution increases. Values around the 10:1 ratio indicate smaller subsequent increases; without accounting for uncertainty and without a consistent basis for the ratio, neither an optimum nor statistical equivalence can be established.

The reported extraction increases between 300 and 600 $min^{-1}$, with smaller variations thereafter. Agitation can affect the suspension, liquid film transport, and gas dispersion. These results alone cannot identify the control mechanism, establish equilibrium limits, or validate a flotation contactor.

The reported endpoint covers the range of 38–40 h, not an exact sampling time. Exact endpoints are not available for kinetic fitting. With n = 1 per condition, the differences are descriptive, and their statistical significance cannot be assessed.

Table 6. Reported effect of the liquid-to-solid ratio. Authors' measurements, INCDMRR laboratory; one determination per condition (n = 1). Derived columns represent arithmetic transformations; replicate variability is not available

| Liquid-to-solid ratio | MgO recovery (%) | Increase compared to the previous stage (p.p.) |
|---|---|---|
| 2.5:1 | 45.0 | — |
| 5:1 | 52.5 | +7.5 |
| 7.5:1 | 58.5 | +6.0 |
| 10:1 | 61.5 | +3.0 |
| 12.5:1 | 62.8 | +1.3 |
| 15:1 | 63.5 | +0.7 |

Table 7. Reported effect of agitation. Authors' measurements, INCDMRR laboratory; a single determination for each condition (n = 1). Derived columns represent arithmetic transformations; replicate variability is not available

| Stirring speed ($min^{-1}$) | MgO recovery (%) | Relative increase compared to the previous stage (p.p.) |
|---|---|---|
| 300 | 38.5 | — |
| 400 | 45.8 | +7.3 |
| 500 | 54.8 | +9.0 |
| 600 | 61.8 | +7.0 |
| 700 | 62.2 | +0.4 |
| 800 | 62.5 | +0.3 |

Table 8. Final reported extraction at different temperatures. Authors' measurements, INCDMRR laboratory; a single determination per condition (n = 1). Derived columns represent arithmetic transformations; replicate variability is not available

| Temperature (°C) | Final MgO recovery (%) | Difference from 15 °C (p.p.) | Relative loss compared to 15 °C (%) |
|---|---|---|---|
| 15 | 63.0 | 0.0 | 0.0 |
| 20 | 59.0 | -4.0 | -6.3 |
| 25 | 55.0 | -8.0 | -12.7 |
| 30 | 42.5 | -20.5 | -32.5 |
| 40 | 36.0 | -27.0 | -42.9 |

### 3.2. Internal consistency checks

Using the ideal stoichiometry of brucite, 23.2% by weight of $Mg(OH)_2$ contributes approximately 16.0% by weight of MgO equivalent: 23.2 × 40.304/58.319. Relative to a total MgO content of 23.18% by weight, this represents approximately 69% of the reported magnesium stock. This is a calculated mineralogical check, not a measured recovery ceiling: the phase fractions and common base of the samples must first be confirmed. The extraction ratio indicates a 72% extraction rate and anticipates improvement to 80%; the source reports these without a complete mass balance, and they do not represent verified overall

recoveries. The reported MgO content in the residues (15–18%) cannot determine the extraction rate without the residue mass.

For illustrative purposes only, for 1 kg of raw material containing 23.18% MgO by weight and 10 $dm^3$ of final solution, a 55% extraction rate corresponds to approximately 0.316 mol/$dm^3$ of total Mg. A concentration of 0.50 mol/$dm^3$ on the same basis requires an extraction rate of approximately 87%. These values may coexist if raw materials with different contents, different final volumes, or different enrichment stages were used. Still, they cannot be combined as evidence for a single batch without its inventory.

The previously calculated cumulative extraction rate of 78.8% would also be below a total recovery of over 85% on the same feedstock basis, before losses in subsequent stages. Therefore, it cannot support the claim regarding the net recovery. These verifications show why the definitions of feedstock, concentrate, and stages must be harmonized.

### 3.3. Comparative activation and contactor concepts

Uncalcined material, material treated at 450 °C, and material treated at 900 °C are retained as the proposed comparison conditions. Their extraction curves previously presented in the tables, the predicted calcium concentrations, and the stage-wise recoveries have been removed, as a reproducible model, adjusted coefficients, and calculation files are not available. Numerical predictions should not be reinstated simply by reducing their displayed precision.

A mechanical flotation unit was used as a gas–liquid–solid contactor for $CO_2$-assisted leaching. Its application to this operation constitutes the process characteristic investigated in this paper. Although mechanical agitation and gas dispersion provide a plausible basis for efficient contact, their contribution to $CO_2$ mass transfer has not been quantified. No measurements of kLa, gas retention, or bubble size distribution were performed, nor was a controlled comparison made with a conventional stirred reactor. Consequently, the data do not support improved $CO_2$ transfer or superior reactor performance.

The literature comparison in the Introduction defines the scope of application for this equipment. Recirculation-based leaching, ultrasound-assisted dissolution, and $CO_2$-based flotation address aspects related to chemistry or gas dispersion but differ in terms of reactor configuration or process objective (Zhu et al., 2008; Sevim and Atalay, 2023; Freitas et al., 2021). The performance data reported by these authors cannot be directly applied to the Budureasa trials or used as direct recovery benchmarks, as the properties of the feedstock and the operating conditions differ. A controlled comparison would be necessary to attribute any improvement specifically to the flotation machine configuration.

### 3.4. Magnesium precipitation and chemical characterization

The authors note that the precipitation step, which involves heating the magnesium-containing solution to 105–110 °C, was carried out under pressure, rather than by boiling at atmospheric pressure in an open vessel. Under these conditions, heat treatment can accelerate the conversion of bicarbonate-containing species and the precipitation of magnesium-containing carbonate phases. The formation of hydromagnesite and improved crystallinity are plausible mechanistic interpretations, but neither the phase identities nor the crystallinity were verified by X-ray diffraction (XRD), and the actual pressure and the time-dependent chemistry of the solution were not documented. The recovered intermediate product was subsequently calcined at 850 °C solely for analytical characterization. In contrast, the proposed subsequent processing route treats the washed filter cake, which contains magnesium, with HCl to prepare $MgCl_2$, potentially avoiding intermediate calcination on an industrial scale; this route has not yet been experimentally validated.

A subsample of the magnesium-containing precipitate was heated in an electric furnace at 850 °C for 2 hours after the furnace reached the set temperature, in containers open to the air, followed by cooling of the furnace. Three parallel analytical portions of approximately 1 g each were processed and weighed on a balance with a resolution of 0.1 mg. The authors note that calcination was carried out to constant weight for analytical verification and to establish a fixed mass basis for chemical reporting in oxide equivalents. This was not intended as a mandatory industrial step in the proposed production route for $MgCl_2$. The exact masses per cycle and the operational tolerance used to confirm constant mass should

be provided from the original analytical records. The three portions are analytical preparations, not independent leaching replicates.

The authors note that repeated calcination/weighing steps were used to establish an analytical endpoint of constant mass; preliminary checks indicated mass differences of less than 1% in an additional cycle. These observations explain the purpose of the 850 °C treatment but do not quantify analytical uncertainty or the purity of the crystalline phase. Without successive individual weighing records, the manuscript cannot independently verify the precise criterion for constant mass or its attainment.

No XRD characterization was performed on the precursor or the calcined product. Since the intended marketable product stream is purified $MgCl_2$, rather than a crystallographically specified periclase powder, chemical analysis, dissolution behavior in HCl, insoluble residues, and impurity carryover are the more directly applicable quality indicators.

The chemical characterization of the final product is documented in Analytical Bulletin No. 01 of the Faculty of Mining at the University of Petroşani, sample 3.13, "FP1 MgO Reconstructed Product." The authors note that the research was interrupted, the samples from the initial study were preserved, and the research was subsequently resumed using those preserved samples. Therefore, the subsequent analysis continues the same research rather than an independent feed campaign.

Table 9. Selected chemical composition of product 3.13 from the preserved samples, as reported in analytical report No. 01. This selected set of parameters does not represent a complete transcript. LOD stands for limit of detection; NR stands for "not reported." Method identifiers are taken from the report

| Parameter | Reported weight percentage | Method specified in the bulletin | LOD % by weight |
|---|---|---|---|
| Mg | 59.7310 | SR ISO 5975:1999 | NR |
| MgO equivalent | 99.06 | Calculated based on Mg | Not applicable |
| Ca | 0.3170 | SR ISO 5975:1999 | NR |
| Fe | 0.0272 | SR ISO 6130:1998 | NR |
| Al | 0.0052 | SR ISO 8889:1999 | NR |
| Si | 0.0788 | SR ISO 5997:1999 | NR |
| $CO_2$ | 0.00 | STAS 1574/18-75 | NR |
| $H_2O$ | 0.00 | STAS 1574/19-75 | NR |

The cited international methods specify the scope of application for chromium ores and concentrates: Mg >3.0% and Ca >0.1% (ISO 5975), Fe 0.5–32% (ISO 6130), Al 1.5–20% (ISO 8889), and Si 0.05–0.5% by molecular absorption spectrometry or 0.5–15% gravimetrically (ISO 5997). These are application ranges, not detection or quantification limits. The reported values for Fe (0.0272%) and Al (0.0052%) fall below these ranges. The method can be extended, but no laboratory confirmations, adaptation details, or validation at these concentrations are available. The Si result (0.0788%) falls within the spectrometric range. The suitability for this Mg-rich matrix and the laboratory's LOD/LOQ have not been established. The Fe and Al values are retained as reported values in the certificate, with these limitations; no validated extension is claimed. No verified numerical LOD or LOQ was obtained for the cited ISO SR or STAS methods.

The value of 99.06% represents the MgO equivalent calculated from the measured Mg, not a directly measured periclase fraction or mineralogical purity. No XRD analysis was performed on either the magnesium-containing precipitate or the calcined product, as the investigation focused on the chemical extraction of Mg; their phases remain unidentified. The reported values of 0.00% for $CO_2$ and $H_2O$ cannot be interpreted as an absence or as being below the detection limit without documented analytical limits.

### 3.5. Integrated process flow and thermal treatment

Fig. 1 shows the calculated historical allocation of raw material alongside the revised upper limit, consistent with the extraction rate. The initially designed output of 4.5 Mg/h is retained as the design

value reported by the source. Separately, at a Mg solubility of 72%, a processed material flow rate of 26 Mg/h, and a feed grade equivalent to 22% by weight MgO, the maximum product flow rate is 4.160 Mg/h if all dissolved Mg were recovered in a product equivalent to 99% by weight MgO. The corrected upper limit does not represent a complete reallocation of all historical solid streams. The proposed direct conversion of the Mg-containing washed filter cake into aqueous $MgCl_2$ using HCl is presented as an unvalidated downstream option, distinct from laboratory-scale analytical calcination.

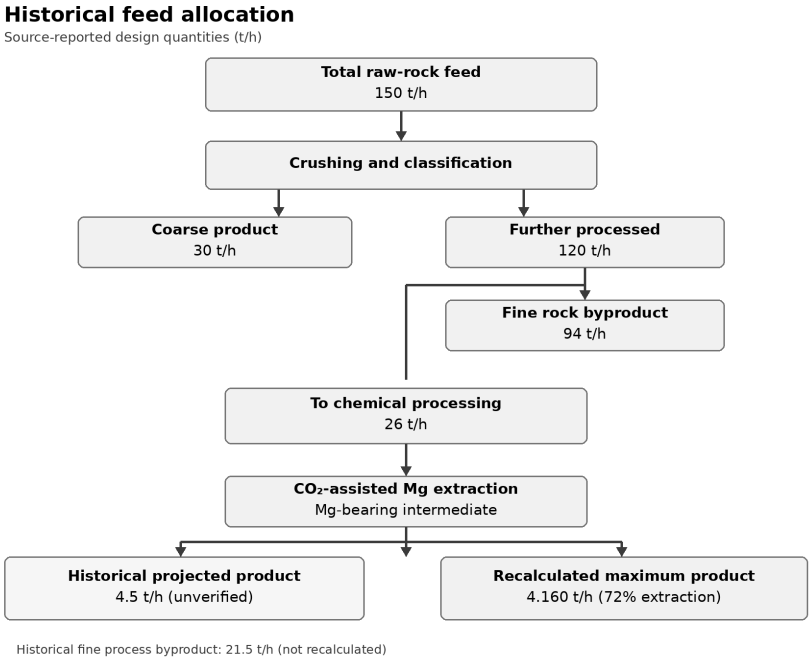



Fig. 1. Calculated historical material allocation (feed rate of 150 Mg/h, 26 Mg/h to chemical treatment, initial projected production of 4.5 Mg/h) and the separate maximum value of 4,160 Mg/h, calculated based on 72% Mg solubility and an assumed product equivalent to 99% MgO by weight. The recalculated value represents an upper limit, not a measured production rate or a closed mass balance. Direct conversion of the Mg-containing filter cake with HCl is a proposed but untested further processing route

After heating and degassing, the magnesium-containing precipitate is separated by hot filtration. The previously proposed route specifies filtration at temperatures above 80 °C; this is a condition proposed by the source, not a confirmed operating record for the characterized product. A controlled washing step is proposed to remove soluble salts. Mg in the mother liquor and in the wash effluent must be measured, as the removal of impurities can also cause Mg losses. Drying removes free water and must be distinguished from solid-phase conversion.

The isolated magnesium-containing precipitate was heated to 850 °C for 2 hours in three parallel analytical portions of 1 g each to facilitate the characterization of the oxide equivalent. The more direct chemical conversion proposed for future testing is: Mg-containing carbonate/hydroxide + HCl → $MgCl_2$ in aqueous solution, with the release of $CO_2$ from the carbonate-containing phases and the possible co-dissolution of Ca.

The authors specify that the Mg-containing precipitation was carried out at 105–110 °C under pressure, separately from the subsequent analytical calcination at 850 °C to constant weight. The proposed historical scheme also mentions a 20–30-min precipitation period and hot filtration above 80 °C. Chemical analyses support Mg recovery and the reported MgO equivalent, but do not establish the purity of the hydromagnesite or periclase phase.

Based on a processed feed rate of 26 Mg/h, with an MgO equivalent content of 22% by weight, the initial stock is 26 × 0.22 = 5.720 Mg/h of MgO equivalent (3.449 Mg/h of elemental Mg). Applying the solubilization rate of 72% Mg reported by the source, this yields 5.720 × 0.72 = 4.1184 Mg/h MgO equivalent dissolved (2.484 Mg/h elemental Mg). If, as an optimistic upper limit, all of this dissolved magnesium were recovered in a product with a concentration of 99% by weight MgO equivalent, the

corresponding maximum product flow rate would be 4.1184/0.99 = 4.160 Mg/h. Any Mg loss during precipitation, washing, filtration, or subsequent treatment would reduce actual production below this maximum threshold. This scenario is calculated based on extraction, not measured yield.

The equivalent MgO stock that has not been solubilized is 5.720 − 4.1184 = 1.6016 Mg/h (0.966 Mg/h of elemental Mg), allocated here solely as a calculated undissolved stock. The fine byproduct of the process, with a historical nominal value of 21.5 Mg/h, cannot be used to determine a verified residue composition without analysis of dry matter and Mg. Therefore, this scenario does not resolve the missing Mg balance issue for each stream.

Table 10. Conditional Mg inventory at 72% extraction (theoretical maximum yield, assuming no downstream losses)

| Conditional Mg stock | MgO equivalent (Mg/h) | Elemental Mg (Mg/h) |
|---|---|---|
| MgO equivalent entering the chemical process | 5.720 | 3.449 |
| Mg solubilized at 72% | 4.1184 | 2.484 |
| Calculated stock of undissolved Mg | 1.6016 | 0.966 |
| Maximum equivalent MgO stock in the final product, assuming 100% downstream recovery | 4.1184 | 2.484 |

If we further assume that the entire quantity of raw rock fed into the process (150 Mg/h) contains 22% MgO equivalent by weight, the corresponding hypothetical stock would be 33 Mg/h of MgO equivalent. The dissolved Mg stock of 4.1184 Mg/h would then be 12.48% of this assumed total Mg stock in the raw feed. This is a conditional comparison of the total feed, not a measured overall recovery in the product; downstream losses would further reduce the recovery in the final product. The composition of the historical by-product streams of 30 and 94 Mg/h is not specified.

The original historical project also reports 4.5 Mg/h of product with an assumed content of 99% by weight MgO equivalent, corresponding to 4.455 Mg/h MgO equivalent and an arithmetic ratio of 77.88% relative to the assumed feed rate of 5.720 Mg/h MgO equivalent. This historical calculation is retained solely for traceability purposes, not as a selected recovery scenario. It exceeds the 4.1184 Mg/h of MgO equivalent solubilized at a 72% extraction rate on the same basis and, therefore, cannot represent the same complete product stream. The revised maximum value of 4.160 Mg/h represents the upper limit compatible with extraction, while the historical value of 4.5 Mg/h may reflect a transcription error, an assumption, or a reporting discrepancy that has not been independently verified. None of these conditional calculations establishes a closed experimental material balance or an overall recovery rate exceeding 85%.

The laboratory reports identify tests involving 5 kg of prepared material and the delivery of a product to the recipient, but do not report a corresponding mass of the recovered product. The three parallel 1-g portions used for the final heat treatment are analytical portions, not measures of batch yield. A verified Mg balance would still require corresponding inventories of the raw material, leaching residue, filtrate, precipitate, mother liquor, and wash water. Therefore, both the historical design value of 4.5 Mg/h and the recalculated upper limit of 4.160 Mg/h must be distinguished from the experimentally measured recovery.

### 3.6. Resource use and potential environmental performance

The rationale for $CO_2$-assisted extraction is to avoid direct HCl attack on the original Ca-rich mineral feedstock. Direct leaching of calcite/dolomite-bearing rocks with hydrochloric acid would consume acid to dissolve calcium carbonate and generate a $CaCl_2$-containing liquid, which might require additional separation and washing. Preferential extraction of magnesium in an aqueous medium,

assisted by $CO_2$, and subsequent isolation of a Mg-rich filter cake could reduce these burdens in later stages, provided calcium distribution and leaching efficiency are favorable. The proposed direct conversion of the HCl-washed filter cake would also eliminate the need for industrial-scale analytical calcination at 850 °C. Neither the anticipated reduction in $CaCl_2$ nor the net savings in energy, water, effluents, or $CO_2$ have been quantified, and the necessary $MgCl_2$ concentration/dehydration step could offset some of the avoided calcination effort.

The presumed benefit lies in changing the location and quantity of acid used, not in an acid-free process: HCl remains necessary to convert the isolated Mg-containing intermediate product into $MgCl_2$. The calcium carried along with that intermediate product can still produce $CaCl_2$ upon dissolution. A practical assessment requires analyses of the Ca/Mg ratio in the raw material, leaching liquor, leaching residue, precipitate, wash solution, and final $MgCl_2$ stream, along with acid consumption and the water/$CO_2$/energy balance. $CO_2$ temporarily dissolved as bicarbonate may be released during precipitation or acidification; permanent sequestration is not supported. The available data do not demonstrate any environmental superiority.

## 4. Conclusions

Previous tests document thermal activation at 650 °C and $CO_2$-assisted leaching in a mechanical flotation cell for the sample material from Budureasa. Three additional one-hour tests, conducted after the second review at 21 °C, with a reported dilution ratio of 1:10, a $CO_2$ flow rate of 5 $dm^3$/min, and 800 $min^{-1}$, using a feed containing 22.76% by weight MgO equivalent, yielded Mg extraction results of 60.7, 62.1, and 61.3% (average 61.37%, sample standard deviation 0.70%; CV 1.14%). These additional results demonstrate limited repeatability under a single nominal operating condition, not a statistical validation of the historical test series. The nominal cell capacity was 3 $dm^3$; individual feed masses, working volumes, gas purity, and complete records still require documented confirmation.

The process concept uses $CO_2$ to preferentially dissolve magnesium from a heat-treated, calcium-rich raw material, thereby potentially avoiding direct leaching of the entire rock with HCl. It is proposed that the isolated filter cake, which contains magnesium, reacts directly with HCl to produce aqueous $MgCl_2$, for subsequent purification, dehydration, and electrolysis in molten salt. It appears that the precipitation was carried out at 105–110 °C under pressure, but the phase identity of the hydromagnesite and its crystallinity were not verified. Three analytical samples were separately calcined at 850 °C to constant weight to determine the oxide equivalent; this is not a mandatory unit operation in the proposed chloride-oriented route. The chemical report indicates 99.06% MgO equivalent based on Mg analysis; the lack of XRD data for the product prevents confirmation of periclase purity. Direct conversion with HCl and the environmental benefits remain unverified.

The historical project reports a total feed rate of 150 Mg/h, of which 26 Mg/h are directed toward chemical processing. For an assumed content of 22% by weight MgO equivalent, the 72% solubilization reported by the source corresponds to 4.1184 Mg/h of dissolved MgO equivalent and a maximum product flow rate of 4.160 Mg/h at 99% MgO equivalent by weight, assuming all dissolved Mg were recovered. The historical figure of 4.5 Mg/h for the product is retained as an unconfirmed projection reported by the source and is not used as a corrected result; the implied recovery rate of 77.88% is incompatible with the 72% solubilization rate on the same basis. No claim is made regarding the existence of a closed experimental Mg mass balance or a validated overall product recovery. Independent measurements of the feedstock, residues, filtrate, mother liquor, and product remain necessary.

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