# Exploring the Potential of LC3 Cement Incorporating Calcined Clay from Abitibi Region (Canada) for Cemented Paste Backfill Applications: a Preliminary Study

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#### **Abstract**

In the Abitibi-Témiscamingue mining region, underground mining operations extensively employ cemented paste backfill (CPB) as a crucial secondary support method in stopes following ore extraction. This method has become indispensable and widely adopted. However, its utilization of binders encompassing basically Portland cements and blast furnace slag contribute to elevated costs and an increased carbon footprint. These challenges are further exacerbated by the logistical complexities of transporting binders to remote mining sites. Consequently, there is a pressing need to explore alternative, cost-effective, and environmentally friendly binders that leverage locally available manufacturing materials.

This study explores the potential application of limestone calcined clay cement (LC3) in CPB using the calcined Abitibi clays. The Abitibi-Témiscamingue region in Canada is endowed with extensive clay reserves, presenting a valuable source to produce supplementary cementitious material (SCM). A recent preliminary investigation study has been conducted to assess the viability of utilizing a specific clay from Rouyn-Noranda city in Abitibi-Témiscamingue region as a precursor of SCM. The clay sample underwent calcination within the temperature range of 700–850°C for LC3 production. Numerous CPB specimens were manufactured using this new type of LC3, as well as the conventional LC3 based on metakaolin. Subsequently, these specimens underwent uniaxial compression tests to determine the unconfined compressive strength (UCS) after curing periods of 7 and 28 days. The results reveal comparable UCS values at 28 days between the new type of LC3 and the conventional LC3 incorporating metakaolin.

The obtained results show promising UCS at 28 days. However, additional samples are imperative to ascertain the quality of clays in Abitibi-Témiscamingue region and their suitability for incorporation into LC3 cements for CPB. The effectiveness of these binders is anticipated to yield significant economic and environmental benefits for the Abitibi-Témiscamingue mining region. Furthermore, this project promotes the local supply of cements with a minimized carbon footprint.

Key words: cemented paste backfills, eco-friendly binders, limestone calcined clay cement, Abitibi clay, binder cost, carbon footprint reduction

#### Introduction

CPB is an engineered paste used for secondary support in underground mines, ensuring safe operations and increased ore extraction. CPB is a mixture with a solid percentage of 70–85%, a slump range of 6–10 in, and UCS of approximately 1 MPa after 28 days of curing. CPB comprises filtered fine tailings, mixing water and a hydraulic binder (Belem and Benzaazoua, 2008). Acquiring binders represents most of the expenses in the backfilling operation (Benzaazoua et al., 2005). In the Abitibi-Témiscamingue region of Quebec, a common binder is a blend of general use Portland cement (GU) and ground granulated blast furnace slag. However, the escalating costs of these binders (Curry, 2020 a, b) significantly raise the overall expenses for backfilling. Moreover, the use of GU contributes to a high carbon footprint. Transporting these binders to remote mines adds extra costs and further amplifies the backfilling

operation's carbon footprint. In such a scenario, exploring locally accessible, cost-effective binders with lower carbon footprints stands as a promising alternative.

The LC3 is part of the Portland cement family. What sets it apart is its significantly lower clinker content, leading to a noteworthy reduction in carbon footprint. Within the LC3 category, different compositions exist and they are collectively referred to as LC3-X, where X represents the clinker percentage in the cement. For example, LC3-50 is comprised of 50% clinker, 30% calcined clay, 15% limestone, and 5% gypsum. While LC3-65 and LC3-30 are also part of this cement family, LC3-50 has received more attention and thorough examination in the literature (Sharma et al., 2021). LC3 represents a cement blend that merges the pozzolanic reaction of calcined clay with the filler effect of limestone, creating a synergistic relationship among clinker, calcined clay, and limestone. LC3 presents two distinct advantages compared to regular Portland cement: a diminished clinker content without compromising mechanical performance, enhanced durability, and resistance owing to refined porosity. This porosity refinement is attributed to the simultaneous formation of C-A-S-H gel, ettringite, and additional carbo-aluminates during the synergetic reaction (Antoni et al., 2012).

For LC3 production, clays with a minimum of 40% kaolinite are required (Avet and Scrivener, 2018). The most of LC3 research has predominantly focused on kaolin type clays, neglecting exploration into other varieties like montmorillonite, illite, and vermiculite, as indicated by the literature (Sharma et al., 2021). These alternative clays have been employed in replacing Portland cement but not in the LC3 context. Studies suggest that their reactivity is comparatively lower than that of kaolinite (Fernandez et al., 2011). Ambroise et al. (1985) demonstrated that illites and montmorillonites are suitable only for applications requiring low mechanical strength when used as cement replacements. Furthermore, Ambroise et al. (1985) noted that thermal activation at 750°C of well-crystallized micas and micaceous minerals, such as muscovite, lacks significance in the domain of supplementary cementitious materials. However, an intriguing avenue emerges through prolonged grinding, known as mechanochemical activation, for these clays (eg, muscovite, vermiculites, and montmorillonites). Mechanochemical activation induces dehydroxylation, amorphization, an increase in aluminum concentration on the mineral surface, and a reduction in bonding forces between silicon and aluminum elements. This process contributes to enhancing pozzolanic reactivity (Baki et al., 2022).

For mining paste backfills, the need for a high percentage of kaolinite in LC3 might be reconsidered, especially considering the lower strength requirements support in underground mines (Belem and Benzaazoua, 2008) for secondary support comparatively to civil engineering (Kosmatka et al., 2011). Diverse clays sourced from various countries, originating from rock weathering (Akindahunsi et al., 2020; Lin et al., 2021), marine deposits (Dixit et al., 2021), kaolin residues (Huang et al., 2020), along with limestone derived from marble dust (Krishnan et al., 2018) or stone waste (Lin et al., 2021), have been employed in LC3 production. This opens the possibility of utilizing mine tailings containing clays or clays found near mines, which can be calcined and employed in mine backfill applications. In mine tailings, there is a clay fraction ( $< 2 \mu m$ ) which ranges from 5–14% (Bussiere, 2007).

It is reported in the literature that a type of clay known as pozzolana clay can replace Portland cement in hydraulic fill up to 25% without loss of mechanical performance (Eshun et al., 2018). Also, research suggests that brick and marble residues can replace Portland cement in paste backfill up to 15% with a net improvement in mechanical strength at different ages (Ercikdi et al., 2015; Külekçi et al., 2016). Knowing that the essential raw material for making bricks is essentially clay, these two examples reported in the literature show that calcined clays as a partial replacement for Portland cement is a promising alternative in CPB. A recent study evaluated two varieties of LC3 cements in CPB (Dhers et al., 2023). These were LC335 (composed of 40% calcined clay and 20% limestone) and LC3-65 (composed of 15% calcined clay and 15% limestone). Both types of cement demonstrated significant UCS up to 56 days. However, a

decrease in strength was observed for both cements after this period (at 91 and 182 days). This phenomenon, due to sulphide tailings, was also observed by Külekçi et al. (2016) when replacing Portland cement with a calcined clay-based waste (brick waste) at rates higher than 15%. The same trend was also noted by Eshun et al. (2018) when the replacement of Portland cement by calcined clay (pozzolana clay) reached 40%. According to Külekçi et al. (2016) and Eshun et al. (2018), the resistance of cement containing calcined clays to sulfate attack is related to the proportion of calcined clay in the cement, the nature of the natural clay, the calcination temperature, and the proportion of the amorphous phase in the calcined clay. It is therefore essential to note that the proportions of calcined clay, clinker, and limestone, as well as gypsum, are of extreme importance, particularly in the case of sulphide tailings based CPB.

The mining region of Abitibi-Témiscamingue in Quebec, Canada, has substantial quantities of clays that can be utilized in the production of LC3. The average thickness of the clay layer in Abitibi-Témiscamingue is ~ 10 m for a regional volume exceeding 100 km³ (Cloutier et al., 2016). In reports by Cloutier et al. (2016), the characterization of clays was not determined by their mineralogical composition; instead, the focus was on their hydrogeological and geotechnical properties, such as permeability and particle size. Some studies on the mineralogy of clays in the Barlow-Ojibway Lake, which includes Abitibi-Témiscamingue (Vincent and Hardy, 1977), indicate that these clays contain trace amounts of kaolinite. The abundant minerals in these studies are primarily illites and chlorite, but they also contain varying amounts of smectites and vermiculites, along with quartz, feldspars, and carbonates (calcite and dolomite), as well as hornblende in the non-clay fraction. These results align with geological reasoning. Illites are characteristic of glacial and desert climates, while kaolinites are typical of lateritic, tropical, and subtropical climates (Al Ani and Sarapää, 2008; Carroll, 1970).

Based on the soil classification by the United States Department of Agriculture (Baillie, 1999), there are generally five soil types in Quebec: gelisols in the extreme north, inceptisols in the north, spodosols in the south, and histosols and alfisols in the southeast. Among these soil types, alfisols are soils where kaolinite can occur (Lal, 2006), and are predominantly located in Abitibi-Témiscamingue and the James Bay region. Considering that kaolin deposits have already been discovered in Quebec, there is a high probability of finding more, especially in Abitibi-Témiscamingue. This hypothesis is reinforced by the discovery of ceramic artifacts related to an ancient ceramic industry in the region, as the production of these artifacts requires specific clays, preferably rich in kaolin (Beaulieu, 2020; Côté and Inksetter, 2001). Furthermore, Laverdiere and De Kimpe (1984) conducted a characterization of six soil samples in Abitibi for agricultural use and showed that these clays contain illites, chlorite, and vermiculites in the clay fraction, as well as quartz, feldspars, hornblende, and carbonates in the non-clay fraction.

To the best knowledge of the authors, this research represents the inaugural application of LC3-50 cements in cemented paste backfill. It seeks to assess the suitability of Abitibi-Témiscamingue clay N°1 (ATC1) collected in the city of Rouyn-Noranda (Esker d'Évain) in the Abitibi-Témiscamingue region as a supplementary cementitious material in the formulation of LC3-50. Initially, the clay was characterized, followed by calcination tests. The calcined samples were then used to produce LC3-50, which in turn were utilized to create simulated paste backfill specimens. These specimens underwent subsequent mechanical compression tests. Additionally, two methods were assessed to enhance the reactivity of the calcined clay in LC3-50.

## Materials and methods

Since this study focuses solely on assessing the UCS of the LC3-50 formulation, Sil-Co-Sil 106® was chosen instead of mine tailings. The aim is to prevent any influence from the mineralogy of mine tailings on the UCS of the CPB. This formulated CPB is designated as simulated cemented paste backfill (SCPB). Sil-Co-Sil 106® is a high-purity fine sand comprises 99.8% silicon oxide and can be sourced from US SILICA (www.ussilica.com). With a density of 2.65 and a BET specific surface area measuring 880 m²/kg.

Peyronnard and Benzaazoua (2011) compared the particle size distribution of Sil-Co-Sil 106® with that of a tailings sample from LaRonde (in Abitibi, Quebec, Canada), collected in 2011. This analysis shows that the particle size distribution of Sil-Co-Sil 106® closely mirrors that of tailings from the LaRonde mine

The production of LC3-50 cement requires four components: clinker (CL; 50%), limestone (LS; 15%), gypsum (GYP; 5%) and metakaolin (MK) or calcined clay (30%). In this study, calcined ATC1 under various calcination and treatment (grinding, concentration) conditions was used, while MK served as the reference. The ATC1 is sourced from the city of Rouyn-Noranda (at Esker of Évain) in Abitibi-Témiscamingue. The MK was supplied by WhiteMud Inc in Saskatchewan (Canada), while CL, LS, and GYP were provided by Ciment McInnis in Port-Daniel-Gascon, Quebec, in granular form. These materials were ground using a laboratory rod mill with a capacity of 9 L. The grinding charge constitutes approximately 25% of the mill volume and consists of three types of rods: 1/2 in, 3/2 in, and 5/2 in.

Three distinct types of analysis on the cementitious materials in question were conducted: physical, chemical, and mineralogical. Physical characterization involved measuring density and specific surface area. Density measurements were performed using the Micrometrics AccuPyc 1330 helium pycnometer, while specific surface area was determined via the BET method using the Micrometrics GEMINI analyzer, following the method established by Brunauer, Emmett, and Teller. Chemical characterization involved X-ray fluorescence (XRF) analysis using a Bruker S8 TIGER spectrometer on fused beads prepared using an automatic fusion machine (Autofluxer®).

Table 1. Results of physical and chemical characterizations

Materials		CL	LS	MK	GYP
	SiO <sub>2</sub>	21.99	4.38	63.21	1.45
	$Al_2O_3$	5.08	1.17	28.90	0.5
	CaO	65.03	51.22	1.95	44.97
	$Fe_2O_3$	4.15	0.32	1.17	0.24
	MgO	1.29	0.70	0.45	0.10
	MnO	-	-	-	< 0.01
	$K_2O$	0.43	-	1.81	0.08
Oxides	$Na_2O$	-	0.27	-	< 0.01
	$P_2O_5$		-	-	< 0.01
(%)	$SO_3$	0.87	0.13	-	46.76
	TiO <sub>2</sub>	0.47	-	0.70	< 0.01
	$Cr_2O_3$	-	-	-	< 0.01
	BaO		-	_	0.49
	ZnO	-	-	_	-
	SrO	0.05	0.04	_	-
	$ZrO_2$	-	-	-	-
	$V_2O_5$	-	-	_	-
LOI* 950 °C (%)		0.49	41.66	1.69	5.29
d(g/cm <sup>3</sup> )		3.19	2.70	2.71	2.32
SSA BET (m²/kg)		518.00**	2,078.00	15,279.00	17,543.00
SSA Blaine (m²/kg)		180.00**	562.00	604.00	140.00

<sup>\*</sup>LOI = loss on ignition; ¶BET SSA = BET method specific surface area

For mineralogical analysis, X-ray diffraction (XRD) was carried out using a Bruker A.X.S. Advance D8 instrument with  $CuK\alpha 1$  radiation ( $\lambda = 1.54056$  Å). The operational parameters were set at 40 kV and 30

<sup>\*\*:</sup> Portland cement GU (General Use) type used in the thesis project associated with this study has a BET specific surface area (SSA) of 1,462.00 m<sup>2</sup>/kg and a Blaine fineness of 354.00 m<sup>2</sup>/kg.

mA, with an increment of  $0.54^{\circ}$ /min within  $5-70^{\circ}$  (20). Diffractograms were indexed using Diffract Eva 6.0 software along with the PDF 2023 database

Table 1 presents the results of physical and chemical characterizations on the materials used in this study. Clinker is mainly made up of CaO and SiO<sub>2</sub>, which represents approximately 87% of its composition. Its Blaine specific surface area of 182 m<sup>2</sup>/kg is lower than that commonly reported in the literature (Laurent, 1999): 280–500 m<sup>2</sup>/kg. Limestone is primarily composed of CaO and CO<sub>2</sub> (manifested by  $\sim$  42% loss on ignition at 950°C). As for MK, it is mainly made up of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, distinguished by its fineness with a BET surface area of 15279 m<sup>2</sup>/kg. Gypsum is mainly made up of CaO and SO<sub>3</sub>, displaying a very high BET specific surface area of 17,543 m<sup>2</sup>/kg.

Regarding ATC1 clay, the predominant oxides are silicon (SiO<sub>2</sub>, 60%), aluminum (Al<sub>2</sub>O<sub>3</sub>, 15%) and iron (Fe<sub>2</sub>O<sub>3</sub>, 6%). There is also calcium oxide (CaO, 3.24%) and magnesium oxide (MgO, 3.18%). These five oxides represent 88% of the chemical composition of this clay.

Figure shows the XRD patterns of LS, GYP, MK, CL and ATC1 from bottom to top, respectively. The main phases of the clinker include alite, belite, and brownmillerite. Within the metakaolin, the prominent phases are an amorphous phase (52.6%), quartz (20.7%), and muscovite (25.1%), accompanied by traces of kaolinite (0.9%) and rutile (0.6%). The gypsum is almost entirely pure, while the limestone is predominantly made up of calcite, with quartz present as an impurity.

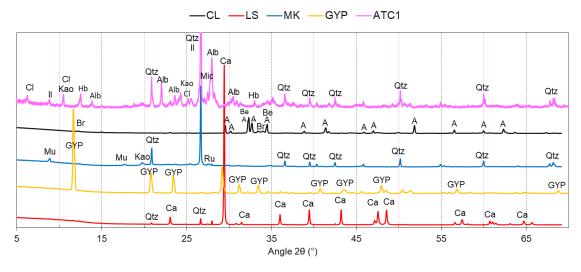


Figure 1. XRD plots of materials used in LC3-50 cement, where A:alite; Be:belite; Br:brownmerillite; Mu:muscovite; Qtz:quartx; Kao:kaolinite; Ru:rutile; GYP:gypsum; Ca:calcite; Cl:clinochlore-1MIIb, Fe<sup>2+</sup> bearing; Alb: albite, Ca-bearing; Hb:magnesio-ferri-hornblende; and II:illite.

Preliminary analysis of ATC1 revealed the presence of minerals such as quartz, feldspars (albite and microcline), Magnesio-ferri-hornblende, and dolomite in the non-clay fraction. As for the clay fraction, the potential minerals identified were clinochlore, illite, and kaolinite. To verify the clay minerals in ATC1, two treatments were carried out: one sample was placed in an ethylene glycol environment for 12 hrs and another sample was heated at 550°C for one hour. A third sample remained natural (untreated) and was used as a reference. These treatments were applied to three glass slide-oriented samples (natural, ethylene glycol-treated, and calcined at 550°C). To prepare slides, a gram of clay was introduced into a beaker containing 20 mL of deionized water. This mixture was stirred for an hour. Then, using a pipette, a few drops were taken from the top of the mixture and poured onto the glass slide. The aim of this process was to separate the clay fraction as much as possible. All three slides were air dried. Then, one was heated to 550°C for one hour, another was left in ethylene glycol medium for 12 hours, and the third did not

undergo any treatment. The results of these treatments are shown in Figure 2 where the three diffractograms can be seen. On the figure, the corresponding peaks to the (001) and (002) planes of the kaolinite at 7Å (12.61°) and 3.5Å (25.21°), respectively, have disappeared after calcination at 55°C. However, it should be noted that certain types of iron chlorite can be affected by heat treatment (Thiry et al., 2013), which seems to be the case here. The ATC1 clay does, in fact, contain a high concentration of Fe<sub>2</sub>O<sub>3</sub> ( $\approx$ 6%) although distinct iron minerals were not detected. The probable mineral is Clinochlore1MIIb, Fe<sup>2+</sup> bearing (Figure 2).

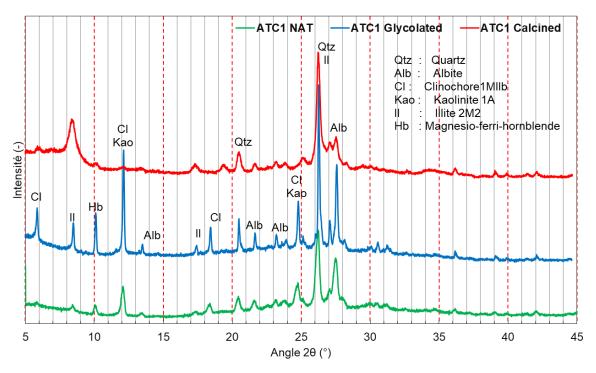


Figure 2. Diffractogram of natural ATC1 (NAT) and after treatment by calcination (C) and glycolates (G), the samples are oriented with glass slides.

The ATC1 calcination was carried out in a Nabertherm LT/9/13/P330 muffle furnace. Its impact was assessed on the unconfined compressive strengths of SCPB at 7 and 28 days. LC3-50 type cements based on calcined ATC1 under various conditions defined via a Taguchi array (Table 2). Parameters explored encompass temperature (T) from 700–850°C, temperature gradient (Gr) set at 5 and 10 °C/min, and calcination time (t in h) representing the duration at the specified temperature level (Figure 3). The selection of optimal calcination conditions was made according to the satisfaction index presented by Equation 1.

$$IS_{UCS} = \sqrt{\frac{UCS 7}{UCS 7_{REF}}} \times \frac{UCS 28}{UCS 28_{REF}}$$

$$(Equation 1)$$
Equation 1

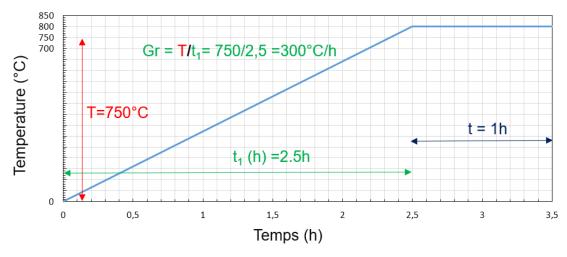


Figure 3. Schematic representation with example of the different calcination factors studied.

Table 2. Design of experiment using Taguchi array L<sub>16</sub> (4<sup>1</sup> 2<sup>2</sup>) for ATC1 calcination.

Table 2. Design of experiment using Taguem array $L_{16}$ (4-2-) for ATC1 calcination.						
N° Experiment	Factor					
	T° (°C)	Gr (°C/h)	Gr (°C/min)	t (h)		
1	700	300	5	0.5		
2	700	300	5	1		
3	700	600	10	0.5		
4	700	600	10	1		
5	750	300	5	0.5		
6	750	300	5	1		
7	750	600	10	0.5		
8	750	600	10	1		
9	800	300	5	0.5		
10	800	300	5	1		
11	800	600	10	0.5		
12	800	600	10	1		
13	850	300	5	0.5		
14	850	300	5	1		
15	850	600	10	0.5		
16	850	600	10	1		

UCS7 and UCS28 are respectively the 7 and 28 day unconfined compressive strength of the SCPB specimens with LC3-50 binder based on calcined ATC1. UCS7<sub>REF</sub> and UCS28<sub>REF</sub> are respectively the UCS at 7 day and 28 day of the reference SCPB made with LC3-50 based on MK. Protocol of unconfined compressive strength test was previously described in Ouffa et al. (2023).

# **Results and discussions**

Table 3 presents the results of the UCS tests carried out on the SCPBs using LC3-50 cements. The variation between these cements lies in the calcined ATC1, produced under various conditions. To evaluate the reactivity of these calcined clays (from ATC1), a blank sample (M5SB), was created. It has the same

composition as the other specimens, except that an equivalent amount of fine sand Sil-Co-Sil106® was integrated to replace the corresponding amount of calcined clay.

Table 3. Results of UCS tests of SCPB according to the calcination conditions of ATC1.

N°	Coloined alere	UCS7(kPa)		UCS28(kPa)	
specimen	Calcined clay type	Mean	Standard deviation	Mean	Standard deviation
REF	MK	792	16	1088	83
M5SB	Sil-Co-Sil	518	47	990	54
M5S1	CATC1-1	474	8	958	54
M5S2	CATC1-2	475	49	986	65
M5S3	CATC1-3	473	35	958	24
M5S4	CATC1-4	488	25	1028	42
M5S5	CATC1-5	422	3	1008	15
M5S6	CATC1-6	518	38	1040	70
M5S7	CATC1-7	484	29	1037	50
M5S8	CATC1-8	488	55	1083	8
M5S9	CATC1-9	528	27	1211	127
M5S10	CATC1-10	584	58	1163	64
M5S11	CATC1-11	440	25	1043	9
M5S12	CATC1-12	568	32	1156	46
M5S13	CATC1-13	526	62	1203	72
M5S14	CATC1-14	549	34	1152	55
M5S15	CATC1-15	542	74	1238	36
M5S16	CATC1-16	557	11	1207	20

From Table 3, the overall average UCS7 of SCPBs incorporating LC3-50 with calcined ATC1 across various conditions is UCS7<sub>mean</sub> = 507 kPa. Recorded standard deviations for UCS7 values range from 3–74 kPa, averaging 35 kPa. Consequently, UCS7 does not exhibit significant variations; the maximum UCS7 value is  $584 \pm 29$  (ie, +77 kPa compared to UCS7<sub>mean</sub>), while the minimum value is  $422 \pm 3$  kPa (ie, -85 kPa compared to UCS7<sub>mean</sub>). Upon comparing the UCS7 values of SCPB specimens incorporating calcined ATC1 with that of the blank specimen, we observe minimal differences, ranging from -96 kPa–66 kPa. The UCS7 values vary from 82% to 113% compared to the blank specimen. The reference specimen displays a UCS7<sub>REF</sub> = 792  $\pm$  16 kPa. The UCS7 of SCPB specimens incorporating calcined ATC1 varies between 53–74% of UCS7<sub>REF</sub>. The comparison of SCPB specimens containing calcined ATC1 with the blank and the reference specimens indicates low reactivity at seven days under different calcination conditions.

By analyzing Table 3, it can be noted that the UCS28 of the SCPBs produced with LC3-50 based on calcined ATC1 clays have a general average of UCS28<sub>mean</sub> = 1092 kPa, with a maximum value of 1238 kPa and a minimum value of 958 kPa. The extreme deviations (maximum and minimum) from the general average are +146 kPa and -134 kPa, respectively. Considering the standard deviations recorded, which reach a maximum of 127 kPa, it can be concluded that the variations in UCS28 of SCPBs based on LC3-50 with calcined ATC1 clays are not significant. The different UCS28 values of SCPBs based on LC3-50 with calcined ATC1, in comparison with the blank specimen, reveal slight variations ranging from 100-130% compared to the blank specimen. The reference specimen shows a UCS28<sub>REF</sub> =  $1088 \pm 83$  kPa. The UCS28 of SCPBs based on LC3-50 with calcined ATC1 vary between 90-110% of UCS28<sub>REF</sub>. Comparison with the blank and the reference specimens indicates a slight reactivity at 28 days of the calcined ATC1 under different calcination conditions. This reactivity is comparable to that of industrial metakaolin at 28 days in SCPB.

Given the significance of UCS28 within the mining context, attributable to the mining cycle enduring approximately one month, our analysis will focus on UCS28. Figure 4 illustrates the main effects plot for UCS28; the dotted line represents the average of the whole experiment (Table 3), ie, UCS28<sub>mean</sub>.

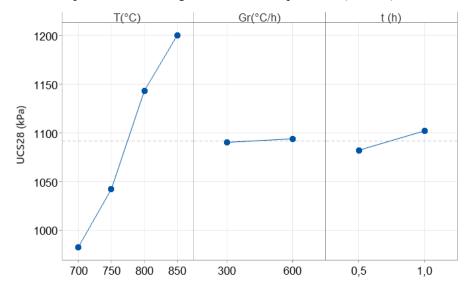


Figure 4. Diagram of the main effects on the UCS28 of SCPB based on LC3-50 with CATC1-x calcined clays (x = 1, ..., 16). The temperature gradient (Gr) has almost no effect (a line identical to the dotted line), and the calcination time (t) has a weak effect (a slight inclination compared to the dotted line).

Only temperature had a significant effect. On average, calcination tests at  $850^{\circ}$ C show high UCS28. Nevertheless, through the examination of potential interactions among distinct factors (T, Gr, and t) and guided by the satisfaction index formulated in Equation 1, condition 10 emerged as the optimal calcination condition (T =  $800^{\circ}$ C, Gr =  $5^{\circ}$ /min, t = 1h). Therefore, the selected sample is **CATC1-10**.

In conclusion, the results of UCS tests (Table 3) conducted in accordance with the adopted calcination experimental design reveal that the calcined ATC1 exhibited negligible reactivity with respect to UCS7. Indeed, the UCS7 values are comparable to those of the blank specimen (fine sand) and, notably, lower than those of the reference sample (MK). After 28 days, a slight increase in reactivity was observed as the corresponding UCS28 values slightly surpass those of the blank specimen and are comparable to those of the reference specimen (MK). It is important to highlight that the materials, excluding metakaolin (clinker, limestone, gypsum), are ground using a laboratory grinder. Their physical properties, particularly specific surface area and particle size distribution, may differ from industrially ground materials. Consequently, the observed UCS may be lower than those anticipated. Furthermore, the study aims to conduct a comparative analysis between calcined ATC1 and industrial metakaolin, justifying the selection of LC3-50 with MK (LC3-50 (MK)) as the reference for this investigation.

To enhance the reactivity of the **CATC1-10**, two approaches were considered: the first consists of grinding CATC1-10 at variable durations, while the second involves the concentration of the ATC1 (uncalcined) before its calcination. For the first method, equivalent masses of calcined clay (100 g) were pulverized for distinct durations (1–12 mins). The second method was applied by concentrating the clays using a 90  $\mu$ m diameter mechanical sieve for half an hour prior to calcination. Due to the difficulty of concentrating these clays by sieving, the ratio between pass and reject was not evaluated. The sieving time was determined so as to obtain a sufficient quantity of material for the tests. It should be noted that in the industrial context, hydrocyclones are used, while sieving is carried out in the laboratory.

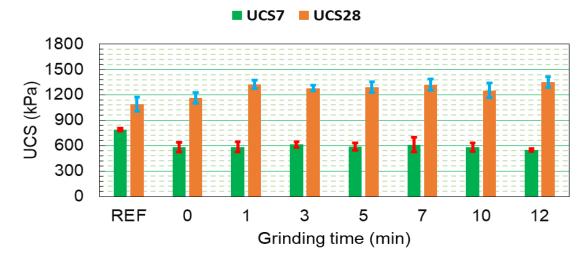


Figure 5. UCS7 and UCS28 results of the SCPB specimens based on LC3-50 with CATC1-10.

At 7 days, the grinding process had little impact on the reactivity of CATC1-10 (GCATC1-10-0). Indeed, the UCS7 of SCPB based on LC3-50 incorporating the different ground calcined clays shows practically no change. At 28 days, slight increases are observable, varying from +8% to +16% in the UCS28 of the unground specimen (GCATC1-10-0). These increases do not follow any notable trend. It should be noted that grinding of clays, often referred to as 'mechanochemical treatment' in the literature, is particularly effective in increasing the pozzolanic activity of 2:1 clay, such as muscovite, vermiculite, and montmorillonite, which are often difficult to activate effectively by calcination. Furthermore, this treatment leads to the amorphization of clay, the enrichment of the aluminum particle surface, and a decrease in the binding energy between Si and Al elements, thereby making a substantial contribution to enhancing pozzolanic reactivity (Baki et al., 2022; Balek et al., 2007). However, the result obtained here can be attributed to insufficient grinding time or to the specific types of clays present in the ATC1 sample, notably chlorites (clinochlore), which may exhibit different behavior due to the grinding process. Figure 6 presents the results of the UCS7 and UCS28 of the SCPB based on LC3-50 produced with calcined concentrated ATC1 (CCATC1-10), in comparison with calcined clay without concentration (CATC1-10) and MK.

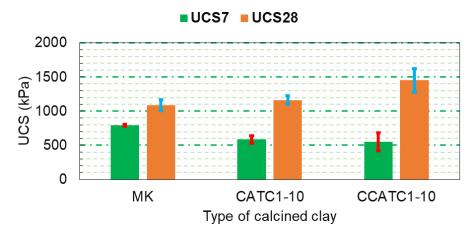


Figure 6. Effect of [ATC1] before calcination on reactivity in SCPB based LC3-50 with CCATC1-10.

# **Concluding remarks**

This study seeks to assess the applicability of MK and a calcined clay from Abitibi-Témiscamingue, collected in the city of Rouyn-Noranda, as a SCM in LC3-50 cements intended for CPBs. Specifically, the study aimed to conduct a preliminary characterization of this clay to determine its potential to contain clay minerals of interest, especially kaolinitic minerals. Unconfined compressive tests were also performed on simulated paste backfill specimens based on LC3-50 cements containing this calcined clay under various conditions. XRF chemical analyses revealed that this clay is rich in silicon (SiO<sub>2</sub>, 60%) aluminum (Al<sub>2</sub>O<sub>3</sub>, 15%), and iron (Fe<sub>2</sub>O<sub>3</sub>, 6%), and also contains lesser amounts of calcium oxide (CaO, 3.2%) and magnesium oxide (MgO, 3.2%). These five oxides constitute 88% of the chemical composition of this clay.

Although iron minerals were not detected by XRD, iron oxide represents 6%. It is believed that iron is concentrated in various clay minerals. The results of mineralogical characterization, along with data available in the literature, indicate that this clay is rich in chlorites and illite. It may contain kaolinite in minor concentration, which requires confirmation through specific treatments. In the non-clay fraction, this clay contains quartz, feldspars, carbonates (calcite, dolomite), and hornblende.

UCS tests on simulated backfill specimens showed that this calcined clay is minimally reactive, especially at 7 days. At 28 days, the UCS under different calcination conditions are similar to those of MK. Concentration treatment before calcination or grinding after calcination of this clay does yield significant improvements. Results obtained show promising UCS at 28 days. The effectiveness of these binders could bring significant economic and environmental benefits to the mining region of Abitibi. However, additional samples are imperative to verify the quality of clays in the Abitibi region and their suitability for incorporation into LC3 cements for CPB. Studies are ongoing by the same authors to achieve these objectives.

This study paves the way for the exploration of the calcination of certain mine tailings, as they contain a significant clay fraction and are finely ground. Eventually, the clay fraction can be concentrated by centrifugation of tailings before deposition.

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