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An alternative to NaOH in the alkali-activation of ground granulated blast furnace slag in the formulation of cemented paste backfills

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Abstract

In the mining district of the Abitibi region (Canada), several underground mines use cemented paste backfill (CPB) for ground support. The binder typically used is a blend of 20% general use Portland cement (GU) and 80% ground granulated blast furnace slag (GGBFS), and qualified as the reference binder (RB). Using the RB always allows CPB to achieve the unconfined compressive strengths (UCSs) targets required. Due to its relatively high price, limited GGBFS availability and the high carbon footprint attributable to GU manufacturing, the search for alternative binders becomes imperative. Alkali-activated binders (AABs) made of GGBFS, and type F fly ash (FAF) activated using NaOH, were tested at the laboratory scale. This activation process achieved similar UCSs of CPB prepared with the RB. Unfortunately, the economic and environmental assessment of these AABs suffers from the high costs of NaOH, its high carbon footprint and GGBFS dosage, which is still significant. Consequently, the industrial application of these AABs in mine backfilling becomes challenging.

The proposed alternative to NaOH consists of using circulating dry scrubber dusts (CDSD) from the desulfurisation process of Rio Tinto Iron and Titanium operations at Sorel-Tracy (Quebec, Canada). This byproduct was successfully tested in a CPB formulation. Moreover, FAF and fine glass powder (FGP) are proposed as partial replacement to GGBFS. The results show that at 28 days the UCS values of CPB prepared with the GGBFS/CDSD mixtures are comparable to the ones of the RB. At 7 days, satisfactory UCS are obtained and can reach the same UCS of the CPB prepared with the RB by adding only 5% clinker to the GGBFS/CDSD mixture. In addition, several mixtures have shown comparable and even higher UCSs to the RB with lower costs and smaller carbon footprints (CO2eq). The results are promising and encouraging for a future industrial application.

Keywords: *cemented paste backfill, NaOH, alkali-activated binders, circulating dry scrubber dusts, binder cost, carbon footprint*

1 Introduction

Cemented paste backfill (CPB) technology has become very popular in underground hard rock mines worldwide. Indeed, CPB represents a very effective method for integrated management of mine tailings. As reported by Benzaazoua et al. (1999), CPB allows for the underground placement of up to 50% of the mine tailings through backfilling in stopes. Therefore, CPB facilitates the reduction of possible geotechnical and geochemical instabilities that the tailings could cause if they are stored on the surface (Benzaazoua et al. 2004a; Bowker & Chambers 2015; Simate & Ndlovu 2014). In fact, CPB helps stabilise local underground stresses, minimise the rock wall closure and optimise ore recovery (Grice 1998). CPB mechanical and hydro-geotechnical behaviours have been well-established through research over the last three decades. The main ingredients forming CPB are filtered mine tailings (up to 70–85 wt% solid), a hydraulic binder at dosages varying between 2 and 8 wt% (of the dry tailings) and potable or recycled process water (15–30 wt% of CPB). The required binder rate (B_w), the binder-to-dry tailings ratio, depends on the required unconfined compressive strength (UCS) for the ground support. This strength is generally around 1 MPa at 28 days. However, it can range from 0.5 to 5 MPa for specific applications (Belem & Benzaazoua 2003, 2008). The binders commonly used are general use Portland cement (GU) alone or GU mixed with a supplementary cementitious material (SCM). The latter can usually be ground granulated blast furnace slag (GGBFS), a byproduct of cast iron or types F and C fly ashes (FAF, FAC) from thermal power plants (Benzaazoua et al. 2010). The binder supply accounts for 75% on average of the backfill operation's cost (Gauthier 2004; Grice 1998). In the mines of the Abitibi region (Quebec, Canada), the commonly used binder is a blend of 20% GU and 80% GGBFS (20GU/80GGBFS). Due to its operational success in always achieving the mechanical strength targets required for ground control, this type of binder has been considered as the reference binder (RB) (Belem et al. 2010).

The acquisition price of GU and SCM, particularly GGBFS, is increasing. Moreover, because of its wide use in many applications, GGBFS reserves are limited (Scrivener et al. 2018). In addition, the carbon emission from the GU manufacturing process is very alarming (Andrew 2018). Indeed, 1 tonne of GU generates an average of 0.8 tonnes of CO_2 (Chen et al. 2010a). In the industrial mining context, it must be emphasised that mining operations are in increasingly remote regions removed from urban areas and conventional transport routes (e.g. northern Quebec). Therefore, significant additional costs are involved, and the carbon footprint is increasing while the mining industry is also evolving towards carbon neutrality. Finding alternative eco-friendly and cost-effective binders therefore becomes necessary. In addition, these alternative binders must be produced locally to avoid transportation issues. The aim is to make mine backfilling operations eco-friendly and economically viable. To address these issues, many alternative binders have been studied (Peyronnard & Benzaazoua, 2011, 2012). Among these alternatives is the alkaline activation of mine tailings with or without SCM. In the first case, unsatisfactory CPB compressive strengths have been obtained (Ouffa et al. 2020). High concentrations of alkaline solutions are necessary for achieving satisfactory UCS values (Falayi et al. 2018; Kiventerä et al. 2016). However, the handling of high concentrations of alkaline solutions is very costly, harmful and risky because of their corrosiveness. Special equipment (e.g. stainless steel pipeline) and highly qualified personnel are required. Alkaline activation of tailings with SCM and low concentrated alkaline solutions has been tested at the laboratory scale (Ouffa et al. 2022). The results were promising since the CPB UCS values are comparable or higher than those of the RB. The formulations of 75% GGBFS and 25% FAF alkali-activated (AA) by 0.5 to 1 Mol/L of concentrated NaOH were elaborated at the laboratory scale. Unfortunately, the economic and environmental evaluations of these alternative binders suffer from the high cost of NaOH, its high carbon footprint and the high dosage of GGBFS (up to 75%). Consequently, the application of these AABs in mine backfilling operations becomes challenging. Further investigations and optimisations are thus needed.

The objective of this paper is to propose an alternative to NaOH in GGBFS-based CPB alkali-activation, namely flue gas desulfurisation (FGD) dust. More specifically, the goal is to propose a circulating dry scrubber dust (CDSD) generated by Rio Tinto iron and titanium operations in Sorel-Tracy (Quebec, Canada) to replace NaOH in GGBFS alkali-activation. FGD dusts by-products are produced through three types of industrial processes: wet, semi-wet and dry processes. Thus, three by-products are obtained and each one is named according to its respective generation process (Galos et al. 2003). FGD gypsum, for example, is the first byproduct and is obtained by a wet process. As reported by Galos et al. (2003), this industrial gypsum has been recognised as a substitute for natural gypsum. It is utilised in gypsum binders and plasters, and as an additive in Portland cement production. FGD gypsum was also tested as an activator for fly ashes and steel slags in the CPB binder's formulation (Li et al. 2020). The second and third desulfurisation by-products are obtained with dry and semi-dry processes. They display varying mineral phases and chemical compositions, but do not have any industrial applications (Galos et al. 2003). Recently, in the concrete field, Adesanya et al. (2020) presented successful work related to the use of FGD dust from steel processing. The authors did not specify the process type that generates this byproduct, which was most probably generated from a semi-dry process. A circulating dry scrubber (CDS) process is a lime-based, semi-solidified FGD process that uses a circulating fluidised bed arrangement to contact a sorbent with the SO-laden flue gas under 'cooling' conditions (Neathery 1996). To the best of our knowledge, there is no available literature about the use of this byproduct in mine backfilling. While we refer to the work of Adesanya et al. (2020) the focus is on CPB, which is very different from concrete (Benzaazoua et al. 2004b).

2 Methodology

The methodology employed in this work is well-described previously in Ouffa et al. (2022). Indeed, to avoid the mineralogical variability of the mine tailings (Ouffa 2019), Sil-Co-Sil 106 fine pure silica was used (in dry form) as the skeleton of the simulated CPB (SCPB). The solids mass concentration (C_w) of the SCPB ($C_w = M_{solid}/M_{SCPB}$) was set at 75% (the water concentration is 25%) and the binder ratio B_w ($= M_{binder}/M_{Sil\text{-}Co\text{-}Sil106}$) at 7%. The corresponding binder concentration C_c ($= M_{binder}/(M_{Sil\text{-}Co\text{-}Sil106}+M_{binder})$) is of 6.54%. Since the goal was to find the formulation of a blended binder, the strategy of mixture design (Cornell 2002) was adopted. Firstly, NaOH was substituted by CDSD for the alkali-activation of GGBFS. Then, and to reduce GGBFS in the binary GGBFS-CDSD binder mixtures, FAF and fine glass powder (FGP) were added respectively as third components. As a follow-up to the results obtained, and to improve the 7-day UCS (UCS7), clinker (CL) was added to the binary GGBFS-CDSD binder mixtures. Statistical analysis was performed to interpret the results and to guide future work.

2.1 Material characterisations

The Sil-Co-Sil 106 fine pure silica was supplied by U.S. Silica. The FAF and FGP were used as SCMs for partial replacement of GGBFS in the binary GGBFS-CDSD binder mixtures. The FAF and GGBFS were supplied by Lafarge Canada Inc., while GU and CL (in granular form) were supplied by McInnis Cement at the Port Daniel–Gascons site (Quebec, Canada). FGP was obtained after collecting and grinding the glass fragments. Its pozzolanic properties are due to the high content of amorphous silica (SiO_2) (Chekireb 2015). This powder, designated as 6×25 (Zidol 2009), was provided to the group researching alternative binders at the Civil Engineering Department at the University of Sherbrooke (Quebec, Canada). The GGBFS is a byproduct of the cast iron process obtained by rapid quenching and grinding. It is an amorphous product which can contain few crystallised phases such as akermanite. Chemically, it is composed mainly of calcium, silicon, magnesium and aluminium oxides (Matthes et al. 2018). The FAF is a semi-amorphous product from coal-fired power plants. It contains quartz and mullite. It is mainly composed of silicon, aluminium and calcium oxides (Fernández-Jiménez & Palomo 2003). The CL is obtained in granular form and ground at the Université du Québec en Abitibi-Témiscamingue (UQAT) laboratory. Its Brunauer–Emmett–Teller (BET) model specific surface area (BET SSA) of 520 m^2/kg is less than that of GU (95% CL/5% gypsum) which is 1,460 m^2/kg. This can be explained by the efficiency of industrial grinding compared to grinding in a university laboratory.

Only physical characterisations have been carried out in this work. The chemical and mineralogical analyses presented are taken from the literature and/or the material suppliers. Specific gravity is determined using the UL TRAPYC 1200e helium pycnometer according to ASTM C128. The BET SSAs were determined using a GEMINI surface analyser from Micromeritics. Table 1 presents the physical properties obtained from the materials' characterisations.

Table 1 Specific gravity and specific surface area of the used materials

Material	Specific gravity (G_s)	BET SSA (m^2/kg)	Estimated Blaine SSA (m^2/kg)
Sil-Co-Sil 106	2.63	880	293
CDSD	2.38	13,440	4,480
GGBFS	2.88	2,230	743
FAF	2.07	1,880	627
FGP	2.49	940	313
CL	3.19	520	173
GU	3.08	1,460	487

The high-grade sand (Sil-Co-Sil 106) has a particle size distribution close to that of tailings generated by metallic hard rock mines (e.g. the LaRonde mine tailings) in Abitibi, Canada (Peyronnard & Benzaazoua 2011). It is mainly composed of silicon oxide (99.8%) (www.ussilica.com), its specific gravity is 2.63 and its BET SSA is 880 m^2/kg. The CDSD was supplied by Harsco Environmental, which provides environmental management services for Rio Tinto Iron and Titanium (the CDSD's producer). This byproduct contains mostly calcium sulphite ($CaSO3 \cdot x\ (H_2O)$), unreacted $Ca\ (OH)_2$ in the process, small amounts of $CaSO_4$ and $CaCO_3$, and other impurities. The material is very fine grained (BET SSA = 13,440 m^2/kg) and has a pH >12.

2.2 SCPB mixture designs

The definition of the mixture component bounds was made based on the CDSD scientific literature (Adesanya et al. 2020) and the blended binders containing GU, GGBFS, FAF (Sahi et al. 2015) and FGP (Zidol 2009). Table 2 presents the set of constraints (bounds) applied to the components of the ternary mixtures. For the GGBFS-CDSD binary mixture, the bounds of CDSD were set as 20% ≤ %CDSD ≤ 50%.

Table 2 Bounds of the different ternary mixtures' components

Binder (mixture) formulation	Byproduct (component)	Bounds (%)		Bounds (proportion)	
		Lower	Upper	Lower	Upper
GGBFS-FAF-CDSD	GGBFS	50	80	0.50	0.80
	FAF	0	45	0.00	0.45
	CDSD	5	30	0.05	0.30
GGBFS-FGP-CDSD	GGBFS	55	80	0.55	0.80
	FGP	0	15	0.00	0.15
	CDSD	5	30	0.05	0.30
GGBFS-CL-CDSD	GGBFS	55	80	0.55	0.80
	CL	5	15	0.05	0.15
	CDSD	5	30	0.05	0.30

Note: in this work, the formulation of the mixtures is presented as two formats, either in proportion or in percentage. For example, for the RB, the following ratios are equivalent: 0.8GGBFS/0.2GU and 80%GGBFS/20%GU or simply 80GGBFS/20GU. The transition from the proportional to the percentage format is done by multiplying the numbers by 100 (see Table 2).

The response analysed here is the 28-day unconfined compressive strength (UCS28). Indeed, UCS28 is an important indicator for a mining operation as the ore extraction cycle is about 28 days. The 7-day unconfined compressive strength (UCS7) is also presented to complete the discussion and to obtain an indication of early strength that is often needed for the design of the barricades.

2.3 Economic and environmental assessment

To evaluate the different binders' performances, a global satisfaction index (SI_G) was defined. For mixture B (binder B), this index includes UCS7, UCS28, the purchase price (P) and the carbon footprint ($CO2_{eq}$). This global satisfaction index is defined (proposed by the authors) in relation to the RB (20GU/80GBBFS) labelled REF in Equation 1.

$$SI_G(\mathrm{B}) = \left(\left(\frac{\mathrm{UCS7(B)}}{\mathrm{UCS7_{REF}}} \times \frac{\mathrm{UCS28(B)}}{\mathrm{UCS28_{REF}}}\right) \times \left(1 - \frac{\mathrm{CO2_{eq}(B)}}{\mathrm{CO2_{eq}(REF)}}\right) \times \left(1 - \frac{\mathrm{P(B)}}{\mathrm{P(REF)}}\right)\right)^{1/4} \tag{1}$$

This definition of the global satisfaction index is based on the following realistic assumptions:

- The carbon footprints of the new binders will be lower than that of the RB (REF).
- The prices (P) of the new binders will be lower than that of the RB (REF).

Indeed, these new binders are expected to be industrial by-products/wastes and could contain less GU (or CL) and less GGBFS. Therefore, the desirable SI_G value will tend towards a maximum value equal to 1. Parameter SI_G will be used for binders' selections for further experimental investigation. The calculation of parameter SI_G is based on the data presented in Tables 3 and 4.

Table 3 Carbon footprint of the materials used

Product/Byproduct	**Carbon footprint (kg CO_{2eq}/kg)**	**Reference**
GGBFS	0.0188	Chen et al. (2010b)
FAF	0.00877	Chen et al. (2010b)
CL, GU	0.8	Chen et al. (2010a) and Davidovits (1991)
FGP	0.063	Soliman & Tagnit-Hamou (2016)
CDSD	0	Considered as waste

Table 4 Prices of the cementitious additives used

Product	**CDSD**	**GGBFS**	**FAF**	**GU**	**FGP**
Price ($CAN/tonne)	49* (25%P(GU))	210	120	195	156* (80%P(GU))
Reference	Authors	(Belem 2017)			(Chekireb 2015)

*Estimated value

2.4 Statistical analysis

The purpose of the statistical analysis performed in this paper is to understand the contribution of each component (in the mixture) to UCS28 value and its trends. Thus the statistical analysis aims to also explain the interactions between the mixtures' components and their effect on UCS28. Different calculations are performed using R and Minitab software. The statistics are presented with a 95% confidence interval. The p-values were presented with a significance level $\alpha = 0.05$.

The response (UCS28) modelling was complex due to the small number of samples performed for each mixture design (≤ 16). However, modelling was performed. Considering the standard deviations (SD) obtained even for experimental values (see SD in Tables 5, 6, 8 and 9), the calculated values could be considered acceptable. The obtained models will be used for calculations but may be difficult to interpret physically.

Before modelling the response, linear correlation analysis was performed. The studied variables are the terms of the full quadratic model. Indeed, if a mixture design contains three components A, B and C, then the terms of the full quadratic model are: A, B, C, $A\times B$, $A\times C$, $B\times C$, $A\times B\times C$, $A^2\times B\times C$, $A\times B^2\times C$, $A\times B\times C^2$, $A\times B\times (A-B)$, $A\times C\times (A-C)$, $B\times C\times (B-C)$, $A\times B\times (A-B)^2$, $A\times C\times (A-C)^2$ and $B\times C\times (B-C)^2$.

2.5 Unconfined compression tests

For the SCPB mixtures' preparation, the components of the dry binders are mixed and homogenised first, then deionised water is added and mixed with a spatula until a homogeneous, single-coloured slurry is obtained. Fine pure silica, simulating the tailings, is then added, and manually mixed with the cementitious grout (binder slurry). Mixing is performed with an Eurodib mixer for a fixed duration of 5 minutes. The mixer is stopped to scrape the sides of the bowl to avoid settlement, then the mixing is continued for another 5 minutes. The total mixing time of 10 minutes is sufficient to obtain a homogeneous paste. The paste is transferred in a single layer into plastic moulds of 30mm diameter and 60mm height (aspect ratio = 2). The paste within the mould is pounded 25 times with a metal rod (15 times at the periphery of the mould and 10 times in the centre). The moulds are hand vibrated and tapped on the edges using a metal rod to remove air bubbles from the paste. The moulds are then placed in a humidity chamber for curing at typical underground mine conditions (20 ± 2°C, RH = 95%) for 7 and 28 days. The moulds were triplicated to ensure accurate unconfined compressive strength (UCS) mean value. The specimens were tested for UCS determination after they reached the target curing time. The UCS was determined in accordance with ASTM C39/C39M using an MTS 10/GL mechanical press with an axial loading capacity of 50 kN and run at a minimum displacement rate of 1 mm/min.

3 Results

In this section, the results of the mechanical characterisation, economic and environmental evaluation, statistical analysis and modelling of the UCS28 are presented. These results will be presented for each mixture design as follows: GGBFS-CDSD, GGBFS-FAF-CDSD, GGBFS-FGP-CDSD and GGBFS-CL-CDSD.

3.1 Binary GGBFS-CDSD binder mixture design

Table 5 shows the results of the compression tests on the different GGBFS-CDSD binary mixtures. As expected, the CDSD was able to activate the GGBFS. The UCS28 obtained are comparable to the one of the RB (REF) and even higher in the case of the 20CDSD/80GGBFS (M0S1) and 27CDSD/73GGBFS (M0S7). For these two mixtures, UCS28 is equal to 2,212 ± 134 kPa and 2,263 ± 41 kPa, respectively: the $UCS28_{REF}$ of the RB being equal to 2,016 ± 205 kPa.

The SCPB's UCS28 corresponding to the binary GGBFS-CDSD binders are higher than the one of RB when CDSD ≤ 35% and vice versa. The UCS7 of the binary GGBFS-CDSD mixtures are always lower than that of the RB. However, if CDSD ≤ 35%, the UCS7 values are acceptable for mine backfill applications because the typical required UCS28 value is only 1,000 kPa.

Correlation analysis reveals that UCS28 is positively correlated with GGBFS, GGBFS×CDSD× (GGBFS-CDSD) and GGBFS×CDSD× $(GGBFS\text{-}CDSD)^2$ with correlation coefficients (r) of 0.91, 0.94 and 0.86 respectively. Correlation analysis reveals also that UCS28 is negatively correlated with CDSD and GGBFS×CDSD, with correlation coefficients of -0.91 and -0.80 respectively.

In terms of SI_G, the optimal mixture to consider for future investigations is M0S3 (70GGBFS/30CDSD). This mixture has a maximum SI_G of 0.612.

Table 5 Results of compression tests on SCPB prepared with the binary binder mixtures GGBFS-CDSD solid percentage (C_w) = 75%, water content (w′) = 25%, binder ratio (B_w) = 7%, binder concentration (Cc) = 6.54%

No. sample	Proportions		UCS7 (kPa)		UCS28 (kPa)		P	kg	SI_6
	GGBFS	CDSD	Mean	Standard deviation	Mean	Standard deviation	($CAN/ tonne)	CO_{2eq}/kg	(–)
REF (RB)	0.2GU/0.8GGBFS		1,430	55	2,016	205	207	0.175	–
M0S1	0.80	0.20	856	26	2,212	134	178	0.015	0.539
M0S2	0.75	0.25	1,002	28	2,032	60	170	0.014	0.585
M0S3	0.70	0.30	983	59	2,033	41	162	0.013	0.612
M0S4	0.65	0.35	736	28	2,060	44	154	0.012	0.596
M0S5	0.60	0.40	594	51	1,837	94	146	0.011	0.569
M0S6	0.50	0.50	406	6	1,532	39	130	0.009	0.526
M0S7	0.73	0.27	806	31	2,263	41	167	0.014	0.581

3.2 Ternary GGBFS-FAF-CDSD binder mixture design

Table 6 presents the compression test results of the different ternary GGBFS-FAF-CDSD binder mixtures. Apart from M1S1 and M1S8 mixtures, the obtained UCS28 exceeds 1,000 kPa. These compressive strengths are appreciable, especially when considering the reduced proportion of GGBFS (<80%). However, the UCS28s values are still lower than those obtained when using the binary binder mixtures (i.e. M1S4, M1S5, M1S6) and the RB. However, very low UCS7 values are recorded for the mixtures prepared with a high proportion of FAF and a low proportion of CDSD (i.e. M1S1, M1S3, M1S8).

Table 6 Results of compression tests on SCPB prepared with the ternary binder mixtures GGBFS-FAF-CDSD solid percentage (C_w) = 75%, water content (w′) = 25%, binder ratio (B_w) = 7%, binder concentration (Cc) = 6.54%

No. sample	Proportions			UCS7 (kPa)		UCS28 (kPa)		P	kg	
	GGBFS	FAF	CDSD	Mean	Standard deviation	Mean	Standard deviation	($CAN/ tonne)	CO_{2eq} /kg	SI_G (–)
REF (RB)	0.8GGBFS/0.2GU			1,430	55	2,016	205	207	0.175	–
M1S1	0.50	0.450	0.050	121	–	261	10	161	0.013	0.217
M1S2	0.50	0.200	0.300	695	40	1,372	56	144	0.011	0.555
M1S3	0.80	0.150	0.050	95	32	1,065	64	188	0.016	0.231
M1S4	0.80	0.000	0.200	856	26	2,212	134	178	0.015	0.539
M1S5	0.70	0.000	0.300	983	59	2,033	41	162	0.013	0.612
M1S6	0.50	0.325	0.175	322	41	831	82	153	0.012	0.388
M1S7	0.75	0.000	0.250	1,002	28	2,032	60	170	0.014	0.585
M1S8	0.65	0.300	0.050	88	6	577	78	175	0.015	0.223
M1S9	0.80	0.075	0.125	471	22	1,774	24	183	0.016	0.418
M1S10	0.60	0.100	0.300	811	33	1,726	95	153	0.012	0.587
M1S11	0.66	0.160	0.180	548	11	1,358	41	167	0.014	0.464
M1S12	0.58	0.305	0.115	208	9	1,114	64	164	0.014	0.352
M1S13	0.58	0.180	0.240	652	34	1,363	111	155	0.012	0.517
M1S14	0.73	0.155	0.115	315	5	1,544	69	178	0.015	0.385
M1S15	0.73	0.080	0.190	670	29	1,783	82	172	0.014	0.503
M1S16	0.68	0.080	0.240	745	28	1,673	70	164	0.013	0.536

Correlation analysis between the UCS28 and the different variables is summarised in Table 7. As demonstrated, GGBFS, CDSD and their interactions are positively correlated with UCS28. All terms where FAF is involved are negatively correlated to the UCS28 and are all positive except FAF×CDSD×(FAF-CDSD). This term can take negative or positive values depending on the proportions of the components FAF and CDSD. Therefore, its contribution to the UCS28 will be positive when it is negative as the correlation is negative. Consequently, the effect of FAF on the UCS28 of GGBFS-FAF-CDSD mixtures may be positive depending on the other terms. However, it is not clear because all the measured UCS28 are lower than that of the binary GGBFS-CDSD mixtures. To ensure the positive (or negative) short-term effect of FAF on the GGBFS-CDSD binary mixture, it would be relevant to narrow the experimental domain to study only the convex polyhedron defined by 5% ≤ FAF ≤ 15% and 15% ≤ CDSD ≤ 35%.

Table 7 Correlation analysis of UCS28 from ternary mixtures GGBFS-FAF-CDSD

Positive correlations		Negative correlations	
Term	**Cor. coeff. r**	**Term**	**Cor. coeff. r**
GGBFS	0.61	FAF	-0.96
CDSD	0.66	GGBFS× FAF	-0.97
GGBFS×CDSD	0.85	FAF×CDSD×(FAF-CDSD)	-0.65
GGBFS×CDSD×(GGBFS-CDSD)	0.94	FAF×CDSD×(FAF-CDSD)^2	-0.73
GGBFS×CDSD×(GGBFS-CDSD)^2	0.82	GGBFS×FAF^2 ×CDSD	-0.68

The UCS28 of the ternary GGBFS-FAF-CDSD binder mixtures can be modelled by Equation 2 (R^2 = 0.96). The calculated maximum is 2,161 kPa reached at GGBFS = 0.8, FAF = 0 and CDSD = 0.2. The measured value is 2,212 ± 134 kPa. The Cox trace plot and ternary diagram using Equation 2 is presented in Figure 1. As one can observe, FAF addition decreases the UCS28 of the 66GGBFS/16FAF/18CDSD blend.

$$UCS28 = 1140\, X_1 - 1338\, X_2 + 8624\, X_3 - 13915\, X_1 X_3 + 18240\, X_1 X_3\, (X_1 - X_3) \quad (2)$$

where:

GGBFS = X_1

FAF = X_2

CDSD = X_3

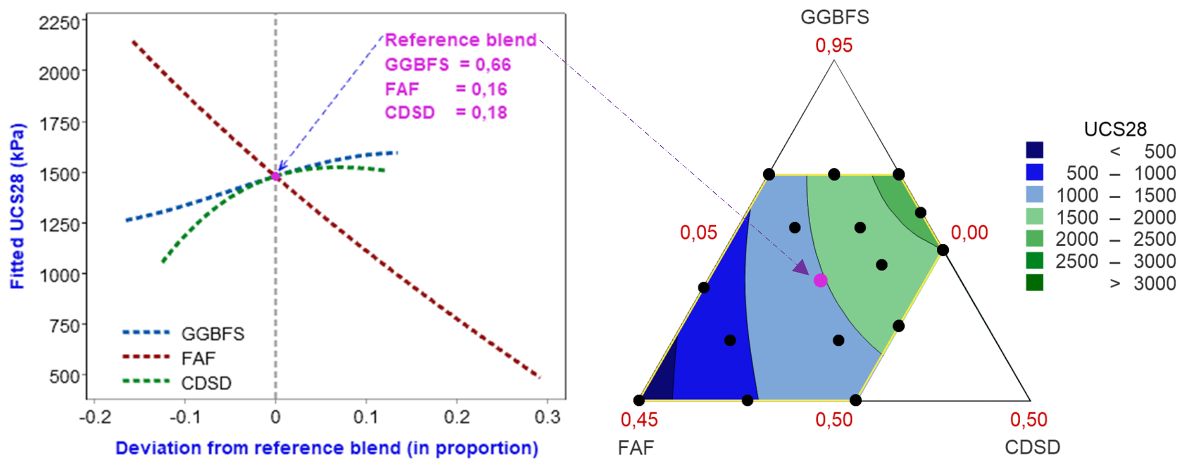

Figure 1 Cox trace plot and ternary diagram corresponding to UCS28 (kPa) of the ternary GGBFS-FAF-CDSD binder mixtures

Considering the SI_G, the optimal ternary binder mixture selected for further investigations is M1S10 (30CDSD/10FAF/60GGBFS). This mixture has a maximum SI_G of 0.587. M1S2 mixture (30CDSD/20FAF/50GGBFS) which present an SI_G of 0.555 and incorporate more FAF (20%) can be also considered.

3.3 Ternary GGBFS-FGP-CDSD binder mixture design

Table 8 presents the compression tests results of the different ternary GGBFS-FGP-CDSD SCPB binder mixtures. For this type of ternary binder, the recorded UCS28 values are comparable to those obtained with the binary binder mixtures (when FGP = 0%).

These mixtures are even equivalent when considering the M2S6 mixture (FGP = 7.5%). All of the UCS28 values recorded are again appreciable in the context of mine backfilling, especially if the proportion of GGBFS (replacement) is considered. The UCS7 values are also appreciable except in the case of the M2S4 mixture, which is probably due to the too-low proportion of CDSD (0.05).

Correlations analysis of UCS28 and the different variables reveals that UCS28 is positively correlated with GGBFS and FGP×CDSD×(FGP-CDSD) with a correlation coefficient r = 0.65 for both explanatory terms.

UCS28 is negatively correlated with FGP, GGBFS×FGP×$CDSD^2$, GGBFS×FGP^2×CDSD, $GGBFS^2$×FGP×CDSD, FGP×CDSD and GGBFS×FGP×CDSD with correlation coefficients of -0.60, -0.73, -0.63, -0.80, -0.75 and -0.78 respectively. The terms negatively correlated to UCS28 are all positive, while the term FGP×CDSD×(FGP-CDSD) positively correlated to the UCS28 can be positive and also negative.

Therefore, there may be an optimum at which the contribution of FGP can become either positive or negative. For example, for the mixture M2S6 (FGP= 7.5%) the UCS28 is equivalent to those of the binary binder mixtures M2S5 and M2S3 but GGBFS content is still significant (80%).

Table 8 Results of compression tests on SCPB prepared with the ternary binder mixtures GGBFS-FGP-CDSD, solid percentage (Cw) = 75%, water content (w′) = 25%, binder ratio (Bw) = 7%, binder concentration (Cc) = 6.54%

No. sample	Proportions			UCS7 (kPa)		UCS28 (kPa)		P ($CAN/ tonne)	kg CO_{2eq}/ kg	SI_G (–)
	GGBFS	FGP	CDSD	Mean	Standard deviation	Mean	Standard deviation			
REF (RB)	0.8 GGBFS/0.2 GU			1,430	55	2,016	205	207	0.175	–
M2S1	0.550	0.150	0.300	792	55	1,641	105	154	0.020	0.567
M2S2	0.800	0.000	0.200	856	26	2,212	134	178	0.015	0.539
M2S3	0.700	0.000	0.300	983	59	2,033	41	162	0.013	0.612
M2S4	0.800	0.150	0.050	46	12	1,896	117	194	0.024	0.202
M2S5	0.750	0.000	0.250	1,002	28	2,032	60	170	0.014	0.585
M2S6	0.800	0.075	0.125	417	35	2,044	126	186	0.020	0.405
M2S7	0.675	0.150	0.175	641	10	1,812	36	174	0.022	0.488
M2S8	0.625	0.075	0.300	891	50	1,797	87	158	0.016	0.589
M2S9	0.713	0.075	0.213	803	18	1,625	97	172	0.018	0.513
M2S10	0.631	0.113	0.256	699	91	1,364	26	163	0.019	0.501
M2S11	0.756	0.038	0.206	811	23	1,839	55	175	0.017	0.520
M2S12	0.706	0.038	0.256	874	28	1,818	65	167	0.016	0.559
M2S13	0.756	0.113	0.131	406	36	1,732	91	183	0.021	0.398

The UCS28 of ternary GGBFS-FGP-CDSD binder mixtures can be modelled as a function of GGBFS, FGP and CDSD based on Equation 3 ($R^2 = 0.97$). The calculated maximum is 2,078 kPa, achieved by using 80% GGBFS, 0% FGP and 20% CDSD. The measured value is 2,212 ± 134 kPa. The Cox trace plot and ternary diagram using Equation 3 is presented in Figure 2. As assumed in the study of the correlations, the addition of FGP can improve UCS28, provided that the appropriate concentration is chosen.

$$\begin{aligned} UCS28 &= 4096\,\boldsymbol{X_1} + 587863\,\boldsymbol{X_2} + 133289\,\boldsymbol{X_3} - 922616\,\boldsymbol{X_1}\boldsymbol{X_2} \\ &- 236289\,\boldsymbol{X_1}\boldsymbol{X_3} - 1298384\,\boldsymbol{X_2}\boldsymbol{X_3} + 1141646\,\boldsymbol{X_1}\boldsymbol{X_2}\,\boldsymbol{X_3} \\ &+ 306450\,\boldsymbol{X_1}\boldsymbol{X_2}\,(\boldsymbol{X_1} - \boldsymbol{X_2}) + 105239\,\boldsymbol{X_1}\boldsymbol{X_3}\,(\boldsymbol{X_1} - \boldsymbol{X_3}) \\ &- 302411\,\boldsymbol{X_2}\boldsymbol{X_3}\,(\boldsymbol{X_2} - \boldsymbol{X_3}) \end{aligned} \quad (3)$$

where:

X_1 = GGBFS

X_2 = FGP

X_3 = CDSD

For future works the M2S8 ternary mixture can be selected. This mixture (30CDSD/7.5FGP/62.5GGBFS) presents the maximum value of SI_G (0.590). The M2S1 mixture (30CDSD/15FGP/55GGBFS) which presents an SI_G equal to 0.567 can also be considered.

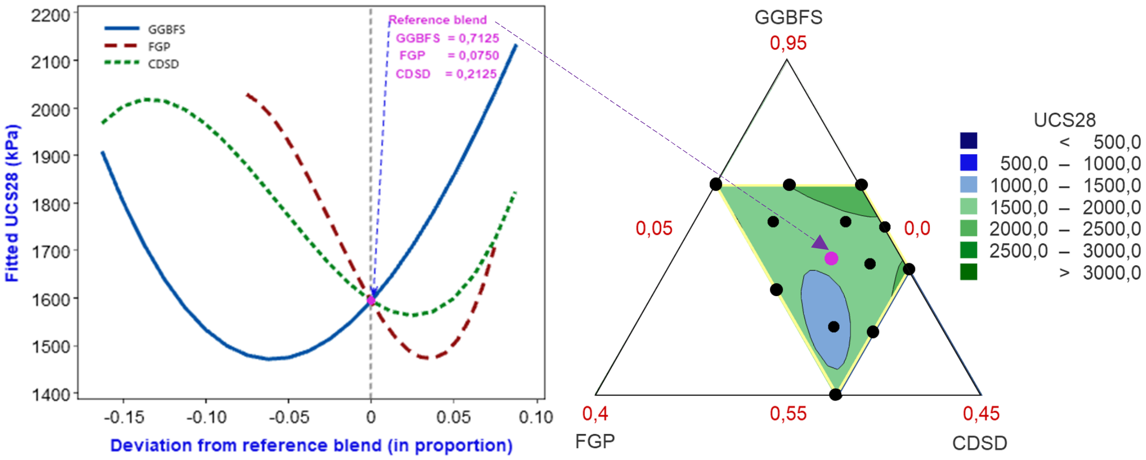

Figure 2 Cox trace plot and ternary diagram corresponding to UCS28 (kPa) of the ternary GGBFS-FGP-CDSD binder mixtures

3.4 Ternary GGBFS-CL-CDSD binder mixture design

As can be seen in Table 9, all of the UCS28 values of the ternary GGBFS-CL-CDSD binder mixtures are higher than those of the optimal binary 27CDSD/73GGBFS binder mixture and that of RB (20GU/80GGBFS). Moreover, several mixtures showed UCS7 values equivalent to that of the RB (M13S4, M13S7, M13S8, M13S9). Other mixtures showed UCS7 values close to that of the RB (M13S5, M13S6, M13S12, M13S13).

The maximum UCS28 is achieved with the M13S7 mixture where the UCS7 and UCS28 values are higher than those of the RB. M13S7 mixture (67.5GGBFS/15CL/17.5CDSD) is made with less than ≈5% CL and less than 12.5% GGBFS compared to the RB (20GU/80GGBFS). Other mixtures presented in Table 9Table also show UCS close to that of RB with only 5% CL (M13S5). For the M13S5 recipe, the UCS28 is higher than the one of the RB. In conclusion, the addition of a small amount of CL (≤10%) in the binder greatly enhance both UC7 and UCS28 of the binary GGBFS-CDSD mixtures.

Correlation analysis between UCS28 and the different explanatory variables reveal that UCS28 is positively correlated with GGBFS×CL^2×CDSD, GGBFS^2×CL×CDSD and GGBFS×CL×CDSD. The correlation coefficients (r) are 0.66, 0.59 and 0.55 respectively. No negative correlation is detected. These results show that the interaction between the three constituents (GGBFS, CL and CDSD) are positive and support the obtained results.

Table 9 Results of mechanical tests on ternary mixtures GGBFS-CL-CDSD, solid percentage (Cw) = 75%, water content (w′ = 25%, binder ratio (Bw) = 7%, binder concentration (Cc) = 6.54%

No. sample	Proportions			UCS7 (kPa)		UCS28 (kPa)		P ($CAN/ tonne)	kg CO_2eq/ kg	SI_G (–)
	GGBFS	CL	CDSD	Mean	Standard deviation	Mean	Standard deviation			
REF (RB)	0.2GU/0.8GGBFS			1,430	55	2,016	205	207	0.175	–
M13S1	0.550	0.150	0.300	1,077	63	2,646	29	159	0.130	0.491
M13S2	0.800	0.050	0.150	849	27	2,580	154	185	0.055	0.484
M13S3	0.650	0.050	0.300	1,134	57	2,286	98	161	0.052	0.612
M13S4	0.800	0.150	0.050	1,314	42	2,406	82	200	0.135	0.307
M13S5	0.725	0.050	0.225	1,270	32	2,474	48	173	0.054	0.594
M13S6	0.800	0.100	0.100	1,269	76	2,416	114	192	0.095	0.430
M13S7	0.675	0.150	0.175	1,583	144	2,735	53	180	0.133	0.468
M13S8	0.600	0.100	0.300	1,343	122	2,538	67	160	0.091	0.598
M13S9	0.700	0.100	0.200	1,410	67	2,479	56	176	0.093	0.538
M13S10	0.625	0.125	0.250	1,254	97	2,653	118	168	0.112	0.530
M13S11	0.750	0.075	0.175	1,120	75	2,515	159	181	0.074	0.517
M13S12	0.675	0.075	0.250	1,276	56	2,242	39	169	0.073	0.573
M13S13	0.750	0.125	0.125	1,299	27	2,486	22	188	0.114	0.435

The UCS28 of GGBFS-CL-CDSD ternary mixtures can be modelled as a function of GGBFS, CL and CDSD by the following Equation 4 (R^2= 0.77).

$$UCS28 = 4351\,X_1 + 67135\,X_2 + 10023\,X_3 - 91405\,X_1 X_2 - 17558\,X_1 X_3 - 163850\,X_2 X_3 + 203583\,X_1 X_2 X_3 \quad (4)$$

where:

X_1 = GGBFS

X_2 = CL

X_3 = CDSD

The calculated maximum value is 2,698 kPa achieved with 55% GGBFS, 15% CL and 30% CDSD. The measured value is (2,646 ± 29 kPa) (M13S1). The Cox trace plot and ternary diagram using Equation 4 are presented in Figure 3.

To better direct future investigations, the ternary mixtures M13S3 (30CDSD/5CL/65GGBFS), M13S5 (22.5CDSD/5CL/72GGBFS) and M13S8 (30CDSD/10CL/60GGBFS) could be selected. These mixtures present an SI_G value of 0.612, 0.594 and 0.598 respectively. The maximum value of 0.612 was achieved with M13S3.

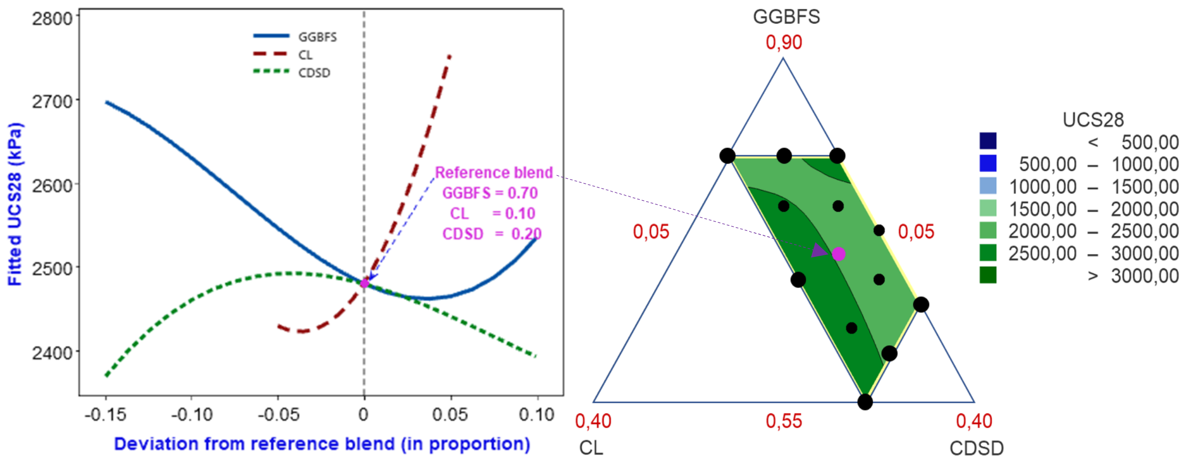

Figure 3 Cox trace plot and ternary diagram corresponding to UCS28 (kPa) of the ternary GGBFS-CL-CDSD binder mixtures

4 Discussion

The first aim of this paper was to propose an alternative reagent to NaOH for the alkaline activation of GGBF within underground mine paste backfill. The second aim was to reduce the content of GGBFS (expensive and less available) as much as possible in the formulation of alternative binders.

The results obtained from the uniaxial compression tests show that at UCS28, values of the binary GGBFS-CDSD binder mixtures are comparable to those of the RB. This is due to the alkaline potential of CDSD (pH > 12) which results in the formation of C-S-H gels and hydrotalcite (Adesanya et al. 2020). However, the UCS7 values were lower than those of the RB. In addition, the amount of GGBFS was still significant. For these two reasons, FAF and FGP (by-products) were tested as a partial replacement for GGBFS in the binary GGBFS-CDSD binder mixture.

It should be noted that FAF and FGP are pozzolanic materials that contribute to the long-term compressive strength because of their slow reactivity (Zidol 2009). Despite this, FGP can be added within the binder up to 7.5% and FAF up to 10% without greatly affecting the UCS28 of the binary GGBFS-CDSD binder mixtures. For FAF, a reduction of the investigation range is highly recommended to find the most suitable proportion according to the mixture design strategy. The latter consists in narrowing the domain of mixtures as knowledge is acquired.

The goal is to converge to the optimal value that verifies the maximum satisfaction index (SI_G = 1). The following constraints are suggested: 5% ≤ FAF ≤ 15% and 15% ≤ CDSD ≤ 35%. For both FAF and FGP, it is recommended to study the mixture in the long term (≥56 days) with the goal being able to quantify the contribution and hydration of FAF and FGP. In mine backfilling, the UCS28 is not always needed but is typically required at 90 days, depending on the mining sequences. Finally, in this study, CL was proposed to solve the problem of short-term strength (7 days).

According to the results obtained, 5% CL is enough to achieve a UCS7 value very close to that of the RB and a UCS28 higher than that of the RB. It should be noted that the CL used here is ground at the UQAT laboratory. Its fineness may be much lower than industrially ground CL. For example, the BET SSA of GU (95% CL/5% gypsum) is 14,600 m^2/kg, which is more than twice the BET SSA of the CL used here (520 m^2/kg).

5 Conclusion

The objective of this paper is to show the possibility of replacing NaOH with an industrial waste, CDSD, that is less expensive and has a smaller environmental footprint relative to the RB. In addition, this waste is available locally in Quebec (Sorel-Tracy). The paper also aims to propose FAF and FGP as partial replacements to GGBFS. From the research that has been carried out, it is possible to conclude that:

- UCS values comparable to that of the RB were obtained with less CL and a lower cost. Thus, the results of the compression tests presented in this article are encouraging.
- It is possible to replace NaOH with CDSD for alkaline activation of GGBFS in the context of mine backfill. It is also possible to partially replace GGBFS with FGP or FAF. However, the hydration and reactivity of FAF and FGP in the long term (≥56 days) must be ensured. If the required strength is higher or equivalent to the RB in the short term (≤7 days), the addition of 5% of CL could be a solution.
- In the continuation of this work, the new binders will be well characterised by an array of tests to understand the mechanisms responsible for the compressive strength development in order to better optimise the formulations and to adapt them according to the availability of by-products and the required compressive strength.

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