**ORIGINAL PAPER** 



# Strength and Rheology of Cemented Pastefill Using Waste Pitchstone Fines and Common Pozzolans Compared to Using Portland Cement

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Received: 24 May 2022 / Accepted: 18 August 2022 © The Author(s) 2022

## Abstract

Cemented pastefill is one of the most popular backfill types that use waste mill tailings to backfill and stabilise the mined-out voids in underground mines. Cement dosage in the range of 3–7% is added to the tailings to increase the strength of cemented pastefill to serve as wall support when extracting adjacent stopes. Cement is expensive and contributes significantly to the cost of backfilling even with small dosages. Moreover, the production of cement is energy intensive and contributes to the emission of carbon dioxide. Partial replacement of cement with supplementary cementitious materials can significantly reduce cost and make mine backfilling more environmentally friendly. This paper reports the findings from a laboratory test programme on the optimization of mix designs using fly ash, slag, pitchstone fines, and polycarboxylate plasticizer. Results indicated that apart from common pozzolans like fly ash and slag, the pitchstone fines attained comparable unconfined compressive strength when replacing cement by 10–20%. The findings are useful for the mining and civil industries trying to dispose the mine waste or reusing it as backfill or as construction material.

**Keywords** Cemented pastefill · Pozzolan · Supplementary cementitious material · Pitchstone fines · Polycarboxylate plasticizer · Tailings

# Introduction

## Background

Mine backfilling is mostly applied in underground mines that utilise either the supported methods like the cut and fill or the combination of methods like the primary-secondary transverse stoping. When the ore is extracted, large voids are created, which are then backfilled with mined waste rocks or tailings to stabilise the voids prior to extracting

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<sup>2</sup> Civil Engineering, James Cook University, Townsville, Australia the adjacent ore. The type and design of backfill material employed is dependent on the stope extraction method and mining sequence. Common backfill types are hydraulic fill, waste rock fill, cemented rock or aggregate fill, and cemented pastefill. Waste rockfill is used in cases where no significant strength of backfill is required. Competent backfill like cemented pastefill and cemented aggregate fill are used when the backfill is required to serve as a support pillar.

Cemented pastefill is one of the most popular backfill types that is environmentally friendly, attains desired strength within 7–28 days curing, and increases stope backfilling rate and production. Cemented pastefill constitutes waste mill tailings, cement binder, and water that are mixed at the surface paste plant and transported as slurry through pipe reticulations and disposed in underground stope voids. Cemented pastefill slurry is a non-Newtonian fluid and exhibits Bingham flow characteristics whereby the yield stress must be overcome to initiate plastic deformation for slurry flow [1, 2]. Typical yield stress of cemented pastefill is around 250 Pa, while the operating yield stress is site specific and can range from 50 to 500 Pa depending on the pumping, reticulation system, and cemented pastefill properties [3]. Cemented pastefill slurry flows through pipe reticulation over longer distances. The slurry has high solid content (70–80%) and with changing rheological properties and hydration process, the reticulation lines over longer distances can be potentially blocked [4]. Admixtures such as plasticizers are used to improve the flowability of the slurry and prevent pipe blockages.

Uniaxial compressive strength (UCS) limits for cemented pastefill have been adapted in mines for different strength requirements and are site-specific depending on the in-situ ground conditions and stress environment. A minimum UCS target of 100 kPa is maintained to prevent liquefaction from blasting or a seismic magnitude of 7.5 on the Richter scale and is adapted from the work of Clough et al. [5]. Freestanding cemented pastefill requires UCS up to 1 MPa, and ground support requires UCS value higher than 1 MPa [6]. An optimum cemented pastefill design should improve strength and rheological properties while reducing cement usage and the cost of backfilling.

# Significance and Objective

Although cemented pastefill is a popular backfill type that is competent for support, it is an expensive backfill type compared to other mine backfills. Thus, cemented pastefill requires optimisation through a well-planned mix design that can lead to cost savings for it to be continually used for wall support, establishing working level, and disposing waste mill tailings. The major cost driver in the application of cemented pastefill is the cost of cement binder. The common type of binder used in cemented pastefill is General Purpose Cement (GPC). Tailings used in cemented pastefill have substantial fines, and a small dosage of cement (3-7% by weight) is added to increase the strength. Even the small dosage of cement accounts for 75-80% of backfilling cost [4, 6]. In addition, the production of clinker cement from the calcination of limestone (CaCO<sub>3</sub>) is energy intensive and emits around 650-900 kg of greenhouse gas (CO<sub>2</sub>) per tonne of cement with 40% of the component coming from fossil fuel

[7]. Approximately 5% of  $CO_2$  emissions are from cement industries, and it is expected to increase in the future [8]. Therefore, this research intends to partially replace highcost clinker cement with pozzolanic waste products like fly ash, slag, or pitchstone fines to save operational cost and reduce  $CO_2$  emissions. This research focuses on optimizing the mix designs using pozzolans and polycarboxylate plasticizer to achieve a cemented pastefill with strength and flow characteristics comparable to using 100% of General Purpose Cement.

# Materials

## Water, Plasticizer, and Binder

Tap water and polycarboxylate plasticizer were added to the solid mix to produce the pastefill slurry. Polycarboxylate plasticizer is an admixture that disperses cement particles and reduces water requirement. Polycarboxylate plasticizers are comb-like polymers composed of polyethylene chains that get adsorbed onto the surface of the cement particles and prevent the cement particles from flocculating and thus slowing down the hydration process and improving workability [9, 10].

Binder types and dosages have a significant influence on the strength and cost of cemented pastefill [11]. The four (4) different binder materials that were used to design the blends in this study were General Purpose Cement (GPC), fly ash, slag, and pitchstone fines. The chemical compositions of different types of binders are summarized in Table 1. The use of binder in cemented pastefill is to create the physical bonding within the unconsolidated tailings particles to produce a more competent backfill material that can provide support.

GPC is produced from the calcination process of limestone with the addition of ingredients such as iron oxide, aluminium oxide, silica, and gypsum to improve the performance. Fly ash, slag, and pitchstone fines contain amorphous

Chemical composi- tion (%)	GPC	Fly ash (Class F)	Slag (Grade 100)	Pitchstone fines
CaO	63.90	3.50	41.30	0.90
SiO <sub>2</sub>	19.20	48.10	35.20	68.53
$Al_2O_3$	5.00	26.70	14.50	12.94
Fe <sub>2</sub> O <sub>3</sub>	3.20	15.20	0.30	1.04
SO <sub>3</sub>	2.40	0.10	1.09	-
MgO	1.10	1.40	5.00	0.02
Na <sub>2</sub> O	0.40	0.60	0.21	4.51
K <sub>2</sub> O	-	-	2.58	-
Others	-	-		7.90

 Table 1
 Chemical composition

 of binders
 Composition

(Sources: Niroshan et al. [31] and Vessala et al. [32]

silica mineral that can react with calcium hydroxide during the hydration process to form calcium silicates [12]. Fly ash is a waste product from coal burning, and the chemical composition can be either low or high in calcium. Low calcium (<10% of CaO) fly ash is from burning anthracite or bituminous coal, and high calcium fly ash is from burning lignite or sub-bituminous coal [13]. Fly ash used in this study is a low calcium (Class F) product. Slag is a waste product from metal smelting. Slag has several grades and can be graded as 80, 100, and 120 [14]. Slag type used in this research is classed as grade 100. Pitchstone fines are natural pozzolans which are obtained as a by-product while producing perlite [15]. During the production of perlite, around 30% of the crushed material produced has a particle size less than 500 µm and are called as pitchstone fines. Tuladhar et al. [16] have shown that pitchstone fines have pozzolanic properties and can be used as partial cement replacement. The pitchstone fines used in this research were waste products from perlite production at Nychum in North Queensland, Australia.

#### Tailings

Tailings properties are site specific and differ for each mine. Mill tailings used in this research were from George Fisher lead–zinc mine (GFM) in Mt Isa (Australia). The physical and chemical properties of the tailings affect the strength and rheology of the cemented pastefill and require testing and characterisation. Physical characteristics of mill tailings were determined using standard tests for particle size distribution, specific gravity, bulk density, and Atterberg limits [17–20]. Mineralogical compositions of the tailings were studied using x-ray diffraction (XRD).

Wet tailings received from GFM were oven dried at 105 C and over a 24 h period to remove the free water. The tailings were manually sieved using a 4.75 mm diameter sieve to separate the boulders and cobbles. Particle size distribution was done on the tailings that passed through the 4.75 mm sieve using mechanical sieving with openings from 75  $\mu$ m to 4.75 mm [21]. The fines less than 75  $\mu$ m collected from the pan were further analysed using Malvern Mastersizer 3000 (laser analyser). The particle size distribution for GFM tailings (Fig. 1) indicated that approximately 5% by mass of particles were less than 20 µm which indicated fewer fines. As a general rule of thumb, tailings should have a minimum of 15% finer than 20 µm for water retention and lubricating effect that aids flow, and prevents settlement and segregation of particles along pipelines [2, 22, 23]. On the other hand, excessive fines in tailings can consume higher cement to attain the desired strength [24].

The parameters of tailings like uniformity coefficient  $(C_u)$  and coefficient of curvature  $(C_c)$  measure the spread of the particle sizes and can influence the binder requirement. The



Fig. 1 Particle size distribution for GFM tailings

Table 2 Physical properties of GFM tailings

Physical property	Unit	Value
Bulk density of dry tailings	kg/m <sup>3</sup>	1300—1480
Specific gravity	-	2.77
D <sub>10</sub>	μm	33.50
D <sub>30</sub>	μm	78
D <sub>60</sub>	μm	200
Coefficient of uniformity, Cu	-	5.97
Coefficient of curvature, C <sub>c</sub>	-	0.91
Liquid limit, LL	%	22
Plastic limit, PL	%	15
Plasticity index, PI	%	7
Linear shrinkage, LS	%	2.5

 $C_u$  and  $C_c$  determined from the GFM tailings were 6.5 and 1.0, respectively. The GFM tailings contained 72% sand, and with a  $C_u$  value of 6.5, the tailings were within the range of 5–10 indicating a lesser spread of grain sizes. Higher  $C_u$ (10–20) represents a well-graded tailings and the requirement of binder is low to achieve the desired strength due to the minimum presence of voids [23, 24]. The physical properties of the tailings are summarized in Table 2. When combining the parameters ( $C_u$  and  $C_c$ ) with the Atterberg limits, the GFM tailings were classified as poorly graded sand with low plastic fines according to the Unified Soil Classification System (USCS).

Mineralogy of tailings is not necessarily inert and can influence the chemical reaction in the hydration process. X-ray diffraction (XRD) was done to study the chemical composition of the GFM tailings. Quantitative analysis of the XRD peaks was done and referenced to the known minerals to determine the chemical composition and source minerals as shown in Table 3. The dominant minerals were silica (40.74%), iron oxide (19.11%), sulphite (12.65%), dolomite (18.4%), and other lesser content of metal oxides. Galena

	1	e
Oxide	Percent (%)	Source rock or mineral
SiO <sub>2</sub>	40.74	Shale (pyritic)
Fe <sub>2</sub> O <sub>3</sub>	19.11	Pyrite
SO <sub>3</sub>	12.65	Pyrite
CaO	12.23	Calcareous-dolomitic siltstone
MgO	6.17	Calcareous-dolomitic siltstone
$Al_2O_3$	5.23	Shale (pyritic)
K <sub>2</sub> O	1.48	Shale (pyritic)
ZnO	0.69	Sphalerite (zinc mineral)
MnO	0.66	Shale (pyritic)
PbO	0.49	Galena (lead mineral)

Table 3 Chemical composition of GFM tailings

(PbO) and sphalerite (ZnO) are the source minerals of lead and zinc, respectively, and are hosted in pyritic shale and calcareous siltstone.

# **Experimental Procedure**

#### Mix Design

The mix design directly influences the rheological and strength properties of the cemented pastefill. Calculations of the mass of ingredients required for the mix were done in accordance with the equations used in a similar study done previously and as practised in the mines [25]. These mix equations outlined below have been used in this study.

Solid content (SC):

$$SC(\%) = \frac{(M_{ds} + M_b)}{(M_{ds} + M_b + M_w)} \times 100$$
(1)

Binder content (BC):

$$BC(\%) = \frac{M_b}{(M_{ds} + M_b)} \times 100$$
(2)

Water content (w):

$$w(\%) = \frac{M_w}{(M_{ds} + M_b)} \times 100$$
(3)

Admixture dosage (AD):

$$AD(\%) = \frac{M_a}{M_b} \times 100 \tag{4}$$

where  $M_{ds}$  is the mass of dry solids,  $M_b$  is the mass of binder,  $M_w$  is the mass of water, and  $M_a$  is the mass of admixture.

Increasing the solid content enabled more tailings to be placed, but made it difficult for the slurry to flow without pipe blockages. Through a series of laboratory trials, it



Fig. 2 Combination of different mix design

was found that the solid content of 74% produced an optimal mix with a slump value around 260 mm. Mix designs were carried out using these threshold values. The mix designs are summarized in Fig. 2. Solid contents of the majority of the mixes were set at 74%, while a few mixes at 75%, 76%, 77%, and 78% were done to study the influence of polycarboxylate plasticizer on the rheological properties. The binders were added at a dosage of 3%, 5%, and 7% for the different binder types. Polycarboxylate plasticizer was added to the slurry at various dosages of 2%, 4%, 6%, and 8% of the mass of the binder calculated for each of the mixes. A total of 68 different mixes were cast and tested throughout the study.

## **Sample Preparation**

A handheld electrical paint mixer was used to agitate the mix. Slump test was done immediately on the fresh slurry prior to casting the moulds. After the slump, the mix was scooped back into the bucket and agitated again to attain homogeneity. Spilt-cylindrical PVC moulds of 120 mm height and 50 mm diameter were used to cast the samples for UCS tests. Ten moulds were prepared to cast 2 samples per curing period for each mix for UCS test. Split-cylindrical PVC moulds of 40 mm height and 50 mm diameter were used to cast the samples for indirect tensile strength (ITS) tests. Four moulds were prepared to cast 1 sample per curing period for each mix for ITS test. The curing periods were 7, 14, 28, 56, and 112 days. The moulds were filled with the cemented pastefill slurry and positioned on the vibration table for a minute to expel any trapped air. The cast moulds were stored in containers. The bases of containers were filled with water to a height less than 1 cm to maintain a constant humidity. The containers were



Fig. 3 UCS test (a) sample loading (b) failed sample



Fig. 4 ITS test (a) sample loading (b) failed sample

tightly sealed off with sticky tape to prevent samples from exposing to air.

# **Strength Measurements**

UCS samples due for testing were trimmed to 100 mm height with 50 mm diameter to maintain a ratio of 1:2 [26]. The samples were trimmed using a straight blade or edge as normally done on soil samples. The sample was placed directly under the loading plate of the triaxial machine (Wykeham Farrance Tritech 50 kN) and loaded to failure as shown in Fig. 3. The loading rate for the UCS test was set at 2 mm per minute over the duration of 5 min. Load and deformation data from the triaxial data logger were analysed to plot the stress–strain curve. Tensile strength of materials like rock and soil are difficult to determine directly, so other indirect strength tests methods are used. The indirect tensile strength (ITS) or the Brazilian test was done to determine the tensile strength of the cemented pastefill sample [27].

The ITS sample was trimmed to get a diameter to thickness ratio ranging from 1.5 to 2.5 and was tested using a triaxial machine as shown in Fig. 4. The sample diameter was 50 mm and the thickness was trimmed to 25 mm to maintain a diameter to thickness ratio of 2. The sample was placed within the guide brackets prior to loading to prevent the sample from rolling over, and the bracket was removed prior to loading. The loading rate for the ITS test was set at 1 mm per minute over a duration of 5 min. Load versus deformation data were analysed to determine the tensile strength as:

$$\sigma_t = \frac{2P}{\pi Dh} \tag{5}$$

where  $\sigma_t$  is the tensile strength, *P* is the applied load, *D* is the diameter of the sample, and *h* is the thickness of the sample.

# **Microstructure Study**

Scanning electron micrograph (SEM) was done to study the growth and development of crystal structures from the hydration process over different curing periods. Small chips (100–200 g) were extracted from the failed UCS samples and were oven dried for 24 h. The dried samples were fully submerged into isopropanol solution for 24 h. Samples were removed from the isopropanol bath and oven dried at low temperature (40-60 °C) for 24 h and stored in sealed plastic bags. Samples were taken to an analytical laboratory for preparation for the SEM. The preparation of samples involved carbon coating on one side of the plate. Specimens were loaded into the machine Hitachi SU5000. Electron beams were directed to the specimen to produce images with high magnification and resolution. Images were captured at different magnifications (5 µm, 10 µm, 20 µm, 50 µm, and 100 µm).

#### **Rheology Measurements**

Rheological properties like yield stress and slump were measured to determine the flowability of the slurry. Slump measures the degree of wetness, consistency of batch, and workability. Rheological properties of cemented pastefill are difficult to determine in practice with experimental devices, thus slump cone is used as the quickest and conventional mode of relating the slump to the shear yield stress of the fresh pastefill slurry [28]. Cemented pastefill is a non-Newtonian fluid and exhibits Bingham flow characteristics. The relation of shear stress ( $\tau$ ), yield stress ( $\tau_0$ ), plastic viscosity ( $\eta$ ), and shear rate ( $\gamma$ ) is expressed as:

$$\tau = \tau_0 + \eta\gamma \tag{6}$$

Brookfield rheometer model DV3T-HA was used to measure the yield stress ranging from 20 to 200 Pa. Yield

stress was measured directly when the maximum torque was reached. Unlike the previous models of Brookfield viscometer, yield shear stress was recorded directly in dyne.cm<sup>-2</sup> and was converted to Pascals (Pa) for data analysis. A separate mix was done for yield stress and viscosity measurements. The slurry was scooped to fill a 250 mL glass beaker. The beaker was tapped at the base to expel any trapped air. The beaker with the slurry was positioned directly under the vane spindle attached to the DV3T rheometer for yield stress testing.

Two vane spindles were used, either the V73 or the V75 depending on the torque range. When the V73 spindle registered an out-of-range torque (> 100%), the smaller V75 spindle was used instead. Vice versa, when the V75 spindle registered a low torque (< 10%), the V73 spindle was used instead. The targeted torque range was between 10 to 100%. The V75 spindle of dimensions 0.803 cm width by 1.61 cm height was used for yield stress ranging from 80 to 800 Pa. The V73 spindle of dimensions 1.267 cm width by 2.535 cm height was cleaned after each test to remove the solids that coagulated around the spindle that can cause slippage and thus reduce torque. Brookfield's guidelines were used to set the parameters, as summarized in Table 4.

# **Results and Discussion**

# Effects of Polycarboxylate Dosage on UCS

Data from strength tests were analysed to ascertain whether the polycarboxylate plasticizer used has any influence on the strength development. Comparisons were made at a different dosage of polycarboxylate plasticizer (0%, 4%, and 6%) on different binder types used at constant 74% solid content and 7% binder dosage. The results from plotting the strength data separately for GPC (Fig. 5a) and pitchstone fines (Fig. 5b) indicated that there were no significant effects of the admixture polycarboxylate on the strength development of the cemented pastefill. Similar results were obtained

Table 4 Yield shear stress parameters

Parameter	Value
Spindle	V73 ot V75
Immersion	Primary
Pre-shear speed (rpm)	5
Pre-shear time (min)	1
Zero speed (rpm)	0.01-0.5
Wait time (sec)	30
Run speed (rpm)	0.05-0.5
Torque reduction (%)	100



Fig. 5 Effects of polycarboxylate on (a) GPC (b) Pitchstone fines

from the observations of strength data for fly ash and slag. Even though the UCS values and polycarboxylate dosage showed no apparent relationships, a general trend was observed when comparing the long-term strength (> 56 days curing) of cemented pastefill produced from GPC and pitchstone fines and that was the continuous strength development of samples containing pitchstone fines. The results (Fig. 5b) indicated that after 56 days, the silica in the pitchstone fines continued the pozzolanic reaction with the portlandite that increased the strength, which was not clear in the case of GPC.

# **Effects of Solid Content on UCS**

The UCS values of five (5) different mixes of varying solid contents (74, 75, 76, 77, and 78%) were compared to establish the relation of solid content and strength. At a constant binder dosage of 5%, the strength of the different mixes were analysed for curing periods from 7 to 112 days. The results (Fig. 6) clearly indicated that the strength increased with the increase in the solid content.



Fig. 6 Effect of solid content on UCS at 5% binder

#### Effects of Pozzolans and Dosage on UCS

UCS strength comparisons were done for the 5 binder types used. The 5 charts below (Fig. 7) compared the UCS of the 5 binder types used at a constant polycarboxylate dosage of 4% and at 74% solid content for curing periods from 7 to 112 days. The results (Fig. 7) indicated that UCS values for 5 binders at lower binder dosage (3%) were below 150 kPa, which were considered as incompetent and can liquefy when subjected to ground tremor. Samples with binder dosage at 5% and curing over the long term (>28 days) reached strength ranging from 400 to 800 kPa, which can serve as competent fill for adjacent ore blasting. Binder dosage at 7% and curing over 56 days attained strength above 1 MPa which can serve as crown, sill, or rib pillar support when mining adjacent stope. Generally, the increase in the curing period increased the strength of the cemented pastefill due to the continuation of the hydration process. Blends of pitchstone fines with replacement of GPC by 10 or 20% and at 7% binder dosage attained strength above 1 MPa over long-term curing periods (>56 days) and can be used in mine backfilling. Similar studies applying pitchstone fines in concrete studies confirmed its pozzolanic properties when partially replacing cement by 10 or 20% [15, 29].

#### **Strength Activity Index of Pozzolans**

Strength activity index (SAI) is a measure of reactivity of pozzolan with cement and is defined as the UCS of pozzolanic mix relative to the control mix over the same curing period and is expressed as percentage. The control mix was cast using ordinary Portland cement to compare the strength of the cemented pastefill to the other mixtures to determine the reactivity of the SCMs. Pozzolanic materials must attain SAI above 75% of the control mix at 7 to 112 days. Results (Fig. 8) indicated that the SAI values of the blended binders were below 75% for early curing periods (<28 days) in some cases but were above 75% for longer curing periods (>28 days). The pozzolans used have attained SAI values above the threshold of 75% for long-term curing periods and were in compliance with a requirement of materials for use in cemented pastefill [30].

#### **Relevance of Young's Modulus and UCS**

UCS and Young's modulus of materials are important parameters that are considered in the blast design of stopes that are adjacent to the cemented pastefill. UCS values were plotted against Young's modulus for several samples tested to establish a range where, with a known UCS value of cemented pastefill, the stiffness can be estimated. The results (Fig. 9) indicated the range of Young's modulus that can provide a first pass estimates in cases where there are absence of any better data. The possible range of values of Young's modulus (*E*) can be determined as:

$$E = (150 \text{ to } 250) * \text{ UCS}$$
 (7)

Most materials have a certain range of values for the ratio of Young's modulus (E) to UCS. According to Niroshan et al. [31], the ratio of E/UCS for cemented pastefill ranges from 150 to 350 which was within the range of values determined by this research.

#### **Relevance of Tensile Strength and UCS**

Indirect tensile strength (ITS) values were plotted against the UCS values for several samples tested to establish a range where ITS values can be estimated from UCS values. The results (Fig. 10) indicated the range of ITS values that can provide first pass estimates in cases where there are no reliable data on ITS. The possible range of values of ITS ( $\sigma_i$ ) can be determined as:

$$\sigma_t = (0.125 \text{ to } 0.25) * \text{ UCS}$$
 (8)

#### **Microstructure Development**

SEM images for each curing period can be related to the UCS values and can be useful in establishing the relationships between the microstructure and strength development. Microstructure developments for several mixes with higher strength were compared. Samples with lower strength crumbled during preparation and specimen could not be easily produced to make a good comparison. Ideally, the SEM of early curing days was preferred to view



Fig. 7 UCS for binder types and dosages (a) GPC (b) 10% pitchstone fines (c) 20% pitchstone fines (d) 25% fly ash (e) 60% slag

the growth of crystals precipitates and days beyond 28 days may have fully developed crystals and difficult to discern. Although SEM was performed on many of the mixes, only two mixes were used to compare in this paper, as the SEM observations were similar in trend (Fig. 11, Fig. 12, and Fig. 13). The control mix 16 was compared with pitchstone fines blend mix 29. Mix 16 has 7% binder

dosage of 100% GPC. Mix 29 has 7% binder in the ratio of 10:90 pitchstone fines to Portland cement, respectively. The SEM observations of the mixes compared indicated the development of ettringite minerals (needle like) during the early curing periods (7–14 days), and at later curing periods (> 28 days), precipitates of calcium silicates (C–S–H) were formed. The growth of calcium silicates occupied the voids, making the cemented pastefill



Fig. 8 Strength activity index comparison over curing periods



Fig. 9 Young's modulus (E) and UCS relations

denser, and that has increased the strength. Similar trend of growth of hydration products were observed for other mixes analysed.

#### Effects of Solid Content on Yield Stress

Cemented pastefill slurry flows when the driving head exceeds the pipeline wall resistance (or shear stress). In this study, the DV3T-HA Brookfield rheometer was used to measure the yield stress ranging from 80 to 200 Pa using the V73 spindle. With the thick fluids such as cemented pastefill, the yield stress measurements done on the majority of the mixes showed significant scatter due to the torque range. However, some data showed a general trend where



Fig. 10 ITS with UCS relations

500

450 400

350

300

200

150

100

50

0

0

ITS (kPa) 250

the increase in the solid content showed an increase in the yield stress. The results (Table 5) from this study indicated the range of values of yield stress measured for the various solid contents. The average values of the minimum and maximum yield stress values (Table 5) indicated a steep increase in the yield stress with increasing solid content. At lower solid content of 74%, the average yield stress was around 100 Pa and increased significantly to 400 Pa for a solid content of 78%. Further test work with the appropriate rheometer torque range is preferred to establish the relationship. Similar range of values (50 to 500 Pa) for yield stress was reported for cemented pastefill in mines [3, 25].

1000

UCS (kPa)

## Effects of Polycarboxylate Dosage on Rheology

Polycarboxylate plasticizer is an admixture that reduces the yield stress and increases the slump of slurry at high solid content. In this study, comparisons were made with several binder and polycarboxylate dosages on several solid contents. The mass of polycarboxylate plasticizer added to the mix was calculated based on the mass of the binder (Eq. 4) so that the ratio of polycarboxylate to the binder was maintained. Hence, the higher the binder content, the higher the amount of polycarboxylate. The results (Fig. 14) indicated that polycarboxylate plasticizer reduced the yield stress with increased dosage. When comparing 3%, 5% and 7% GPC dosages, it can be seen that the yield stress decreased significantly with the increase in polycarboxylate dosage. The results (Fig. 14) also indicated that the binder content affected the yield stress of the slurry. Without polycarboxylate, the yield stress reduced from 246 Pa for the 3% binder dosage to 174 Pa for the 5% binder dosage, and further reduced to 106 Pa for the 7% binder dosage.

GPC 100%

1500

PF 10% & G PC 90%

PF 20% & G PC 80% FA 25% & GPC 75%

SL 60% & GPC 40%

2000







Fig. 12 Comparison of SEM at 14 days curing (a) GPC mix 16 and (b) Pitchstone fines mix 29



Fig. 13 Comparison of SEM 28 days curing (a) GPC mix 16 and (b) Pitchstone fines mix 29

Solid content (%)	Yield stress range (Pa)	Average yield stress (Pa)
74	25-180	103
75	115-180	148
76	135–315	225
77	190–270	230
78	400	400



Fig. 14 Yield stress at varying binder and polycarboxylate dosages at 74% solid



Fig. 15 Polycarboxylate, slump and solid content

Slump height decreased with an increase in solid content. To improve the slump of the slurry at higher solid content, polycarboxylate plasticizer was added to the mixes of several solid contents (74, 75, 76, 77, and 78%) to study its influence on the slump. The results (Fig. 15) indicated that the polycarboxylate plasticizer had a significant effect on the slump and spread of the slurry. When the dosage of polycarboxylate was increased, the slump height and horizontal spread distance increased significantly. For instance, the slump value was low (55 mm) at a high solid content of 77% and 0% dosage of polycarboxylate. However, as the polycarboxylate dosage was increased to 6%, the slump increased to 250 mm, which was optimum for cemented pastefill. Slump height increased significantly for mixes



Fig. 16 Yield stress and 56 days UCS at 7% binder dosage

with high solid content (76–77%) when the polycarboxylate dosage increased (6–8%). A lower dosage of polycarboxylate (2–4%) was adequate to attain the desired slump (> 200 mm) for a mix with 74% solid content. At high solid content (77%) and with an increased dosage of polycarboxylate, the cemented pastefill transformed from cake to slurry. Polycarboxylate has proven to improve the workability of the slurry to maintain an optimal slump (> 200 mm) at higher solid content.

# **Relevance of UCS and Yield Stress**

The optimum mix of cemented pastefill should attain strength that can withstand blasting and flow through pipelines. Based on general practice, UCS of 1000 kPa and yield stress of 150 Pa were used as limits to identify optimum mixes for these specific tailings. Binder content of 7% was used for comparison of different binder types, as the majority of the mixes that used 3% and 5% binder dosage did not reach UCS values above the threshold of 1 MPa. Comparisons were made for 56 days of curing for 7% binder dosage (Fig. 16). Results indicated that the majority of the mixes reached 1 MPa and above, except 3 mixes of fly ash and slag blend with 4 to 6% polycarboxylate dosage. Interestingly, all the mixes with pitchstone fines were within the optimum mix zone.

# Conclusions

The overall aim of this research was to optimize the cemented pastefill using pozzolanic materials to attain the desired strength and rheological properties. The main outcome of this study is highlighted as follows:

Slag, fly ash, and pitchstone fines blends attained longterm strength activity index above the threshold of 75% compared to the control mix (100% GPC). These pozzolans have complied with the strength requirements as per ASTM C618 standard and can be utilized in backfilling underground mine voids.

Blends of pitchstone fines with replacement of GPC by 10 or 20%, and at 7% binder dosage attained UCS value above 1 MPa after 56 days curing and can be used in mine backfilling.

Unlike GPC mixes, all other pozzolans showed a significant strength increase between 28 and 56 days. Hence, pozzolans can be considered for stopes that are scheduled for production over a longer period (> 56 days). Pozzolans may not be ideal to be used in plug pour of a stope filling that require setting within a 24 h period prior to the main pour.

Polycarboxylate plasticizer significantly improved the flowability with the increase in dosages. However, higher dosages of polycarboxylate (>6% of cement content) increased the setting time and that can affect the early strength of pastefill. Polycarboxylate dosage ranging from 2 to 6% can be ideal for mix with low solid content (73–75%).

Scanning electron micrograph (SEM) images showed that at early curing days, ettringite (needle-like crystals) are formed, and calcium silicates are formed after 7 days. Growth of calcium silicates occupied the voids and increased the strength of the cemented pastefill over time.

The findings are useful for partial replacement of cement with pitchstone fines and other pozzolans in concrete and mine backfilling.

Acknowledgements The authors are grateful to several individuals who have assisted throughout the research. Shaun Robinson and Troy Poole assisted with laboratory strength tests, Shane Askew and Ruilan Liu assisted with SEM and rheology tests, Tayla Dinnison and Lucy Bombardieri assisted with strength tests of fly ash and slag. This research was made possible at James Cook University through the Australia Award Scholarship.

**Funding** Open Access funding enabled and organized by CAUL and its Member Institutions.

**Data Availability** The data analysed in this study are available from the corresponding author upon reasonable request.

#### Declaration

Conflict of interest The authors declare no conflict of interest.

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# References

- 1. Boger D, Scales P, Sofra F, Jewell RJ, Fourie A B (2006) Paste and thickened tailings, a guide. Australian Centre for Geomechanics, Nedlands, W.A.
- Sivakugan N, Rankine RM, Rankine KJ, Rankine KS (2006) Geotechnical considerations in mine backfilling in Australia. J Clean Prod 14(12):1168–1175
- Sivakugan N, Veenstra R, Naguleswaran N (2015) Underground mine backfilling in Australia using paste fills and hydraulic fills. Int J Geosynthetics Ground Eng 1(2):18
- Panchal S, Deb D, Sreenivas T (2018) Variability in rheology of cemented paste backfill with hydration age, binder and superplasticizer dosages. J Adv Powder Technol 29(9):2211–2220
- Clough GW, Sitar N (1981) Bachus RCJ (1989) Cemented sands under static loading. J Geotech Eng Div ASCE 107(6):799–817
- Belem T, Benzaazoua M (2008) Design and application of underground mine paste backfill technology. J Geotech Geol Eng 26(2):147–174
- Schneider M, Romer M, Tschudin M, Bolio H (2011) Sustainable cement production—present and future. J Cement Concrete Res 41(7):642–650
- Barcelo L, Kline J, Walenta G, Gartner E (2014) Cement and carbon emissions. J Materials Struct 47(6):1055–1065
- Nagrockiene D, Pundiene I, Kicaite A (2013) The effect of cement type and plasticizer addition on concrete properties. J Construction Build Materials 45:324–331
- Yu B, Zeng Z, Ren Q, Chen Y, Liang M, Zou H (2016) Study on the performance of polycarboxylate-based superplasticizers synthesized by reversible addition–fragmentation chain transfer (RAFT) polymerization. J Mol Struct 1120:171–179
- Nasir O, Fall M (2010) Coupling binder hydration, temperature and compressive strength development of underground cemented paste backfill at early ages. Tunnelling Underground Space Technol Incorporating Trenchless Technol Res 25(1):9–20
- Walker R, Pavía S (2011) Physical properties and reactivity of pozzolans, and their influence on the properties of lime–pozzolan pastes. J Materials Struct 44(6):11391–12150
- Nochaiya T, Wongkeo W, Chaipanich A (2010) Utilization of fly ash with silica fume and properties of Portland cement–fly ash– silica fume concrete. J Fuel 89(3):768–774
- 14. American Concrete Institute, (2001). Use of raw or processed natural pozzolans in concrete (ACI232.1R-00).ACI Committee, 232.
- 15. Tuladhar R, Smith M, Pandey GR, Joyce P (2011) Use of pitchstone fine as a partial replacement of Portland cement for sustainable concrete. Proceedings of 2011 international concrete sustainability conference, Boston, USA.

- 16. Tuladhar R, Sexton A, Joyce P (2013) Durability of concrete with pitchstone fines as a partial cement replacement. Concrete Institute of Australia's Biennial National Conference.
- 17. Australian Standards AS 1289.3.2.1 (2009) Methods of testing soils for engineering purposes-Soil classification tests- Determination of the plastic limit of a soil.
- Australian Standards AS 1289.3.4.1 (2008) Methods of testing soils for engineering purposes-Soil classification tests-Determination of the linear shrinkage of a soil.
- 19. Australian Standards AS 1289.3.5.1 (2006) Methods of testing soils for engineering purposes-Soil classification tests- Determination of the soil particle density of a soil.
- 20. Australian Standards AS 1289.3.9.1 Methods of testing soils for engineering purposes-Soil classification tests -Determination of the cone liquid limit of a soil.
- 21. Australian Standards AS 1289.3.6.1 (2009) Methods of testing soils for engineering purposes-Soil classification tests- Determination of the particle size distribution of a soil—Standard method of analysis by sieving.
- Deng X, Klein B, Tong L, de Wit B (2018) Experimental study on the rheological behaviour of ultra-fine cemented backfill. J Construction Build Materials 158:985–994
- Kuganathan K (2005) Rock Fill in Mine Fill, Handbook on Mine Fill, Australian Centre for Geomechanics, Nedlands, W.A: 101–115.
- Henderson A, Revell M (2005) Basic Mine Fill Materials, Handbook on Mine Fill, Australian centre for geomechanics, Nedlands, W.A: 13-20.

- Niroshan N, Sivakugan N, Veenstra RL (2018) Flow characteristics of cemented paste backfill. J Geotech Geol Eng Springer 36:2261–2272
- 26. Australian Standards AS 1289.6.4.1 (2016) Methods of testing soils for engineering purposes-Soil strength and consolidation tests -Determination of compressive strength of a soil.
- 27. Australian Standards AS 1012.10 (2000) Methods of testing concrete-Determination of indirect tensile strength of concrete cylinders (splitting test).
- 28. ASTM C143 Standard test method for slump of hydraulic-cement concrete, ASTM International.
- Ray A, Sriravindrarajah R, Guerbois JP, Thomas PS, Border S, Ray HN, Joyce P (2007) Evaluation of waste perlite fines in the production of construction materials. J Therm Anal Calorim 88(1):279–283
- 30. ASTM C618 Standard specification for coal fly ash and row or calcined natural pozzolan for use in concrete, ASTM International.
- Niroshan N, Sivakugan N, Veenstra RL (2017) Laboratory study on strength development in cemented paste backfills. J Mater Civ Eng ASCE 29(7):4017–4027
- 32. Vessalas K, Thomas PS, Ray AS, Guerbois JP, Joyce P, Haggman J (2009) Pozzolanic reactivity of the supplementary cementitious material pitchstone fines by thermogravimetric analysis. J Therm Anal Calorim 97(1):71

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