The Potential to Replace Cement with Nano-Calcium Carbonate and Natural Pozzolans in Cemented Mine Backfill

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The effectiveness of mine backfilling depends on the properties of its constituents. The high cost of cement, which is commonly used as a binder in mine backfill, has led researchers to seek alternatives to partially replace it with other binders. This study investigated the potential to use nano-calcium carbonate (NCC) and natural pozzolans (zeolite and pumice) along with Portland cement (PC) in mine backfill. Two types of experimental samples were prepared: (1) gold tailings and silica sand to investigate the effect of NCC and (2) nickel tailings to investigate the effect of natural pozzolans. The unconfined compressive strength (UCS) was measured for samples cured for up to 56 days. Moreover, selected samples were subject to mercury intrusion porosimetry to investigate microstructural properties. Results show that addition of NCC did not improve the UCS of backfill prepared with gold tailings and cured for 28 days, whereas a dosage of 1% NCC in backfill samples prepared with silica sand improved UCS by 20%, suggesting that the gold tailings negatively affected strength development. Natural pozzolans, in particular, 20% zeolite, had 24% higher UCS after 56 days of curing compared to samples prepared with PC and thus have the potential to partially replace cement in mine backfill.

1. Introduction

Mine backfilling is the practice of filling underground voids created during ore extraction in underground mining operations to (1) provide stability for the underground working environment [1], (2) reduce negative environmental impacts from tailings’ storage on the surface [2–4], and (3) maximize extracted ore volumes, such as in room and pillar mining [5, 6]. Three common mine backfill materials are rock fill (RF), hydraulic fill (HF), and paste fill (PF) [1, 7–9]. To make RF, waste rocks from the surface or underground are crushed (typically to 16 mm) and possibly cemented, depending on the required strength [10–12]. By decreasing waste rock piles on the surface, adverse environmental impacts and rock subsidence are avoided [11–14]. However, to ensure backfilling efficiency, binder consumption is high [15]. HF is produced during the classification of mill tailings using hydrocyclones: coarse, dense (>70 wt.% solids) underflow is obtained [16–19]. The high water content of HF facilitates transport through pipelines directly into stopes [19, 20]. According to Adiansyah et al. [21], drainage is the most critical factor in designing HF because ineffective drainage can pose a risk to workers if porous barriers are not used, and piping and liquefaction problems are common. PF consists of mill tailings with 15% of the material <20 μm in size and solid concentrations of 70–85 wt.% [15, 22, 23]. The high solids and low water content can cause issues for transport through pipelines and necessitate addition of a superplasticizer to enhance flowability [24, 25].

Mine tailings are waste products from ore concentration in mineral processing plants [26]. Their chemical and physical properties must be considered during backfill design because they can affect backfill mechanical properties [27]. For instance, tailings that contain sulfide minerals are prone to sulfate attack, which can decrease the unconfined compressive strength (UCS) over the long term due to ettringite formation [28]. Thus, binders that are compatible with the tailings are added to backfill at low concentrations to enhance UCS and other mechanical properties [29]. Cement is typically the main binder, but it is expensive. Therefore, alternative binders are required [30].
Recent advances in nanotechnology have presented the opportunity to use nanomaterials as supplementary cementitious materials [31]. The fine particles act as nuclei for cement to accelerate hydration and improve UCS at early stages [32]. For example, adding NCC to cement and silica fume accelerated the pozzolanic reaction at an early age and compensated for the low initial strength [36, 37].

Supplementary cementitious materials such as natural (e.g., volcanic pumice and zeolite) and artificial pozzolans (e.g., fly ash) could partially replace cement to prevent sulfate attack [38, 39]. Pozzolanic materials in mine backfill can reduce binder costs by more than 15% on an annual basis [30, 40–42]. At concentrations ranging from 10 to 30%, natural pozzolans improved concrete mechanical properties, permeability, durability, and transport properties [43–46]. Volcanic pumice properties vary by source and location due to different ash formation conditions, mineral components, and grain size characteristics [47–49]. Addition of zeolite enhanced the mechanical properties, durability, and performance of concrete [50–53]. Many industrial wastes also have pozzolanic properties [54]. For example, clay pozzolans, produced through calcination at temperatures ranging from 700 to 900°C [2, 55], are inexpensive, have low CO₂ emissions compared to cement, and improve backfill mechanical properties [56, 57]. Addition of 10% clay pozzolans to mine backfill produced maximum UCS and an annual savings of >7% [2, 58].

The application of NCC and natural pozzolans has primarily focused on concrete production: possible effects on backfill mechanical properties have not yet been widely investigated. Therefore, this paper investigates partial replacement of cement by NCC, pumice, or zeolite in cemented mine backfill. To investigate the effect of NCC, backfill samples were prepared with gold tailings and a superplasticizer to aid nanoparticle dispersion: various dosages of NCC were compared with reference samples containing only Portland cement (PC). To investigate the effect of natural pozzolans, backfill samples were prepared with nickel tailings and various dosages of pumice and zeolite and were compared against reference samples containing PC only or a combination of PC and fly ash. Samples were subject to UCS tests after up to 56 days of curing and selected samples were subjected to mercury intrusion porosimetry to investigate microstructural properties.

2. Methods

2.1. Materials. Tailings from gold and nickel mines and silica sand were used to make experimental backfill samples (Table 1). The particle size distribution (PSD) of these materials is shown in Figure 1.

The chemical composition of Type 10 PC with a specific gravity of 3.15, pumice (provided by Hess Pumice Company) with a specific gravity of 2.35, zeolite (provided by Bear River Zeolite), and fly ash are shown in Table 2. The chemical and physical properties of the NCC (provided by US Research Nanomaterials, Inc.) are shown in Table 3.

Table 1: Mineralogy and physical properties of silica sand and two types of tailings (% by weight).

| Characteristic | Gold tailings | Nickel tailings | Silica sand |
|---------------|---------------|----------------|-------------|
| Mineralogy (wt.%) |               |                |             |
| SiO₂          | 49.02         | 77.28          | —           |
| Al₂O₃         | 17.68         | 11.09          | —           |
| Fe₂O₃         | 9.15          | 2.13           | —           |
| SO₃           | 7.29          | —              | —           |
| CaO           | 6.52          | 2.22           | —           |
| MgO           | 3.79          | 1.14           | —           |
| K₂O           | 2.70          | 2.19           | —           |
| TiO₂          | 0.64          | 0.27           | —           |
| Physical properties |            |                |             |
| Specific gravity | 2.89          | 2.80           | 2.69        |
| D10 (μm)      | 35.22         | 3.54           | 20.14       |
| D30 (μm)      | 75.37         | 82.05          | 48.03       |
| D50 (μm)      | 97.72         | 9.92           | 75.60       |
| D60 (μm)      | 105.78        | 170.55         | 88.65       |
| D90 (μm)      | 200.09        | 189.66         | 185.95      |
| Cu (D60/D10)  | 3.003         | 48.177         | 4.407       |
| Cc (D30²/D10 × D60) | 1.52       | 11.15          | 1.29        |

Figure 1: Particle size distribution of gold and nickel tailings and silica sand.

NCC is characterized by high energy and surface area; thus, surface reactions occur, and particles are prone to adhere on direct contact via magnetic, electrostatic, and Van der Waals forces [37]. To aid NCC dispersion, a superplasticizer (sodium dodecyl sulfate, Thermo Scientific Company) was used, with the chemical and physical properties listed in Table 4.

Tap water (pH 7.5) was used to prepare experimental backfill samples. Compounds in mixing water can influence the mechanical properties of backfill and cement hydration [59]. For instance, dissolved calcium and magnesium enhance cement hydration, whereas salty brine decreases binder workability [60].
2.2. Experimental Design. Backfill samples containing NCC were prepared with 7 wt.% PC as a binder and 78% solids concentration using gold tailings (Table 5). Mixtures of PC and 1, 3, and 5% NCC (by dry weight of PC, equation (1)) were initially dry mixed to break up agglomerated nanoparticles that were visible to the naked eye. Dried tailings were then added, and water was gradually added to the mixture to make a paste. Finally, superplasticizer was added (by dry weight of NCC, equation (2)). Samples were poured into 5.08 × 10.16 cm cylindrical molds and cured for 7, 14, and 28 days at 25 ± 2°C and 90 ± 2% relative humidity:

\[ M_{\text{NCC}} = D_{\text{NCC}} \times M_{\text{PC}}, \]

\[ M_{\text{SP}} = D_{\text{SP}} \times M_{\text{NCC}}, \]

where \(M_{\text{NCC}}\) is the mass of NCC (g), \(D_{\text{NCC}}\) is the dosage of NCC (%), \(M_{\text{PC}}\) is the mass of PC (g), \(M_{\text{SP}}\) is the mass of superplasticizer (g), and \(D_{\text{SP}}\) is the dosage of the superplasticizer (%).

Backfill samples containing 10 and 20% pumice, zeolite, or fly ash were prepared using nickel tailings and PC with a solid concentration of 80% (Table 6; equations (3) and (4)). Samples were cured for 7, 14, 28, and 56 days at 25 ± 2°C and 90 ± 2% relative humidity:

\[ D_{\text{PC}} = \frac{M_{\text{PC}}}{M_{\text{PC}} + M_{\text{T}}}, \]

\[ M_{\text{POZ}} = R \times M_{\text{PC}}, \]

where \(D_{\text{PC}}\) is the dosage of PC (%), \(M_{\text{T}}\) is the mass of tailings (g), \(M_{\text{POZ}}\) is the mass of pozzolans (g), and \(R\) is the percentage of PC replaced by pumice, zeolite, or fly ash (%).

2.3. Unconfined Compressive Strength. On each curing date, the UCS of triplicate cured backfill samples was measured on a Wykeham Farrance 100 kN loading machine with a 50 kN load cell after ASTM D2166/D2166M-16 [61] (Figure 2). UCS values are reported as means.

2.4. Mercury Intrusion Porosimetry. Mercury intrusion porosimetry (MIP after ASTM D4404-18 [62]) was used to describe the pore structure of select backfill samples made with 20% pumice, zeolite, and fly ash after 56 days of curing (Table 6). The macrosize and volume distribution of pores inside materials are commonly measured using MIP, which is a well-known technique, although it has adverse environmental effects.

3. Results and Discussion

3.1. NCC Backfill Samples. The mean UCS of backfill samples increased with curing time at all NCC dosages and was consistently higher in reference samples than those made with 1% NCC and 10–100% superplasticizer (Figure 3), 3% NCC and 10–80% superplasticizer (Figure 4), or 5% NCC and 10–30% superplasticizer (Figure 5). Thus, the maximum UCS was observed for reference samples on day 28 (~0.80 MPa). The differences between treatment and reference sample means became more pronounced with NCC content, such that, on day 28, the UCS of the 30% superplasticizer sample was 5% (Figure 3), 25% (Figure 4), and 36% (Figure 5) lower for the 1, 3, and 5% NCC samples, respectively. At 1 and 5% NCC, addition of superplasticizer had a negative effect on UCS on each curing date (Figures 3 and 5), but at 3% NCC, it had a positive effect on UCS (Figure 4).

Figure 6 shows the samples with the maximum UCS on each curing day for 1, 3, and 5% NCC. Relative to the reference sample, increasing NCC from 1 to 5% decreased the UCS of backfill samples by 24–56% on day 7, 3–32% on day 14, and 5–30% on day 28.

Since adding NCC did not improve the UCS of backfill samples, the gold tailings were replaced with a more neutral material to see if the loss of strength was due to an interaction between the tailings and the NCC. Samples prepared with silica sand augmented with 7% PC or 7% PC+1% NCC were tested for UCS. The UCS increased with curing time as above (Figure 7). Addition of 1% NCC increased the UCS from 0.71 to 0.82 MPa by day 7, did not affect UCS on day 14, and by day 28, increased the UCS.

### Table 2: Chemical composition of Portland cement and supplementary cementitious materials (wt.%).

| Material        | CaO    | Al₂O₃ | SiO₂ | MgO  | Fe₂O₃ | TiO₂ | Na₂O | K₂O | SO₂ |
|-----------------|--------|-------|------|------|-------|------|------|-----|-----|
| Portland cement | 61.14  | 4.6   | 19.38| 3.35 | 2.02  | —    | 2.03 | 0.71| 2.28|
| Pumice          | 0.8    | 13.5  | 76.2 | 0.05 | 1.1   | 0.2  | 1.6  | 1.8 | —   |
| Zeolite         | 5.6    | 3.5   | 70.3 | —    | 3.05  | —    | 4.7  | 3.8 | 0.03|
| Fly ash         | 5.02   | 20    | 41.22| 1.4  | 23.84 | —    | 0.81 | 1.5 | 1.94|

### Table 3: Chemical and physical properties of nano-calcium carbonate.

| Appearance        | CaCO₃ (%) | HCl insoluble (%) | Particle size (nm) | Fe (%) | Mn (%) |
|-------------------|-----------|-------------------|--------------------|--------|--------|
| White powder      | ≥98       | ≤0.1              | 20–50              | ≤0.08  | ≤0.006 |

### Table 4: Chemical and physical properties of the sodium dodecyl sulfate superplasticizer.

| Appearance | Concentration (%) | Density (g/cm³) | pH | Boiling point (°C) |
|------------|------------------|----------------|----|-------------------|
| Liquid     | 73–79            | 1.08           | 5–7| 78                |
Silica sand had a higher PSD than gold tailings (Figure 1), which might contribute to the effectiveness of NCC. Previous studies have shown that NCC increased the UCS of concrete mixtures. Concrete has larger and relatively neutral in-earth components compared to backfill tailings. It is believed that physical (PSD) and chemical (components) differences contribute to NCC efficiency early in UCS development.

### 3.2. Natural Pozzolan Backfill Samples

As with the NCC experiments, the UCS increased with curing time from 7 to 56 days (Figure 8), whereas reference samples had the highest UCS on day 14, and backfill samples prepared with 20% zeolite or 10% fly ash after 56 days of curing had the highest UCS (0.66 MPa), 24% higher than the reference UCS on that day.

The porosity and pore size distribution of samples containing 20% pumice, zeolite, and fly ash are shown in Figures 9 and 10, respectively. Samples containing 20% fly ash had the highest UCS (0.66 MPa), 24% higher than the reference UCS on that day.

### Table 5: Mixing design for all backfill samples containing nano-calcium carbonate.

| Mixture ID   | Portland cement (%) | Nano-calcium carbonate (%) | Superplasticizer (%) |
|--------------|---------------------|----------------------------|----------------------|
| Reference    | 7                   | 0                          | 0                    |
| 1NCC         | 7                   | 1                          | 0                    |
| 1NCC10SP     | 7                   | 1                          | 10                   |
| 1NCC20SP     | 7                   | 1                          | 20                   |
| 1NCC30SP     | 7                   | 1                          | 30                   |
| 1NCC40SP     | 7                   | 1                          | 40                   |
| 1NCC50SP     | 7                   | 1                          | 50                   |
| 1NCC100SP    | 7                   | 1                          | 100                  |
| 3NCC         | 7                   | 3                          | 0                    |
| 3NCC30SP     | 7                   | 3                          | 30                   |
| 3NCC40SP     | 7                   | 3                          | 40                   |
| 3NCC50SP     | 7                   | 3                          | 50                   |
| 3NCC60SP     | 7                   | 3                          | 60                   |
| 3NCC80SP     | 7                   | 3                          | 80                   |
| 5NCC10SP     | 7                   | 5                          | 10                   |
| 5NCC20SP     | 7                   | 5                          | 20                   |
| 5NCC30SP     | 7                   | 5                          | 30                   |

### Table 6: Mixing design for all backfill samples containing natural pozzolans.

| Mixture ID | Tailings (kg) | Portland cement (g) | Tap water (g) | Pumice (g) | Zeolite (g) | Fly ash (g) |
|------------|---------------|---------------------|---------------|------------|-------------|-------------|
| Reference  | 2.5           | 131.6               | 657.9         | 0          | 0           | 0           |
| FA10       | 2.5           | 118.4               | 657.9         | 0          | 0           | 13.2        |
| FA20       | 2.5           | 105.3               | 657.9         | 0          | 0           | 26.3        |
| PU10       | 2.5           | 118.4               | 657.9         | 13.2       | 0           | 0           |
| PU20       | 2.5           | 105.3               | 657.9         | 26.3       | 0           | 0           |
| ZE10       | 2.5           | 118.4               | 657.9         | 0          | 13.2        | 0           |
| ZE20       | 2.5           | 105.3               | 657.9         | 0          | 26.3        | 0           |

**Figure 2:** Experimental setup for unconfined compressive strength tests.

**Figure 3:** Mean unconfined compressive strength (UCS) for triplicate backfill samples prepared with 1% nano-calcium carbonate (NCC) and 0–100% superplasticizer (SP). See Table 5, for mixture designs.
Figure 4: Mean unconfined compressive strength (UCS) for triplicate backfill samples prepared with 3% nano-calcium carbonate (NCC) and 0–100% superplasticizer (SP). See Table 5, for mixture designs.

Figure 5: Mean unconfined compressive strength (UCS) for triplicate backfill samples prepared with 5% nano-calcium carbonate (NCC) and 0–30% superplasticizer (SP). See Table 5, for mixture designs.

Figure 6: Maximum unconfined compressive strength (UCS) for backfill samples prepared with 0 (reference), 1, 3, and 5% nano-calcium carbonate (NCC) and 0, 10, and 60% superplasticizer (SP). See Table 5, for mixture designs.
ash had higher porosity (∼33%) compared to samples containing natural pozzolans (∼31%). This could explain the lower UCS value obtained for samples containing fly ash after 56 days of curing (Figure 8). Samples containing fly ash and natural pozzolans had similar range of pore size (∼0.006–10 μm), as shown in Figure 10. Adding 20% natural pozzolans decreased the number of pores, especially in the range of 0.01–0.02 μm (Figure 10).
4. Conclusions

This study investigated the potential replacement of cement by NCC and natural pozzolans in cemented mine backfill. Key conclusions are summarized as follows:

(i) Addition of NCC did not improve UCS relative to reference samples prepared with PC and gold tailings
(ii) 1% NCC without superplasticizer yielded the highest UCS among the experimental samples
(iii) Samples containing silica sand instead of gold tailings improved UCC, suggesting that the gold tailings had a negative effect on strength development
(iv) Addition of superplasticizer to NCC samples did not improve the UCS
(v) Natural pozzolans may have some potential to partially replace cement in mine backfill
(vi) Addition of 20% zeolite or 10% fly ash improved UCS the most on day 56 (24%) relative to the reference sample
(vii) Samples prepared with 20% natural pozzolans had a similar pore size distribution (~0.006–100 μm) and porosity
(viii) Additional studies are required to examine the effect of particle size and chemistry on the UCS of backfill samples prepared with NCC

Data Availability

The data used to support the findings of the study are available within the article.
Conflicts of Interest
The authors declare no conflicts of interest.

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