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Investigation of inhomogeneous properties of backfill samples to explore a new quantitative criterion for cemented paste backfill identification

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Abstract

Cemented paste backfill (CPB) has been utilised globally in mines based on its benefits of non-segregation, non-bleeding and homogeneity. Due to a lack of research around the mechanism(s) driving anti-segregation properties, nowadays only some engineering empirical parameters including the slump or the fine particle content of backfill slurries can be used as the descriptive criterion for CPB. To better understand the anti-segregation mechanism of CPB, so a quantitative criterion for its identification can be determined, the segregation-induced inhomogeneous properties of cemented tailings backfill have been experimentally investigated. Samples (diameter 75 mm and height 150 mm) with different solid contents were poured, cured and cut into sections of equal height. Thereafter, titration measures of EDTA-2Na and helium porosimeter have been used respectively to test the cement content and porosity of each section. Results show that the cement contents decreased from top to bottom along the curing height of samples, while the porosities increased along the settling direction. The inhomogeneity of cemented samples is affected obviously by the solids content of the paste, and it is notable that there is a turning point for the slurry concentration value over which the homogeneity will be improved dramatically. The turning point could be used as a new criterion for CPB definition from the perspective of inhomogeneity inhibition.

Keywords: *cemented paste backfill, segregation, inhomogeneous properties, cement content, tailings*

1 Introduction

Mining is usually of great importance for a country's economy, industry and so forth. However, it can also cause many geohazards such as surface subsidence and/or groundwater contamination (Loupasakis et al. 2014; Zeng et al. 2018). Mining with backfill is common practice worldwide, because it can manage mine waste and refill underground mined-out voids and/or stopes, so that support and stable underground working environment can be provided and the related geohazards can be reduced or solved (Grice 1998; Belem & Benzaazoua 2008; Sheshpari 2015).

There are three main types of mine backfilling methods, including hydraulic fill, paste fill and rockfill respectively (Potvin 2005; Sheshpari 2015). Among them, cemented paste backfill (CPB), a relatively new backfill method, has been developed rapidly and widely utilised in recent years based on the benefits of non-segregation, non-bleeding and homogeneity (Barrett 2000; Ercikdi et al. 2009; Stone 2014; Sivakugan et al. 2015). Indeed, CPB is an inhomogeneous mix material commonly consisting of three ingredients; namely tailings, binder and water. To achieve the aforementioned benefits, the ingredients should be properly mixed in a backfill plant firstly producing the paste-like backfill slurry. After mixing, the paste slurry will then be transported into underground stopes by pumps or gravity through pipelines (Benzaazoua et al. 2004; Belem & Benzaazoua 2008).

In recent decades, CPB has been studied as a kind of cementitious material by many researchers, and lots of research findings concerning the properties and engineering performance of CPB have been worked out. For example, to ensure the transportability or pumpability of CPB, its rheological properties and the relative influence factors have been well explored (Verkerk & Marcus 1988; Bentz et al. 2012; Jiang & Fall 2017). To acquire explicit understandings of the working performance of CPB, its consolidation and hydration process, strength properties, etc. have been systematically studied as well (Benzaazoua et al. 2004; Nasir & Fall 2010; Yilmaz & Ercikdi 2016).

In the majority of cases, researchers usually ignore the inhomogeneity of CPB to simplify study contents. Thus, when it comes to the restricted definition of CPB, only some engineering empirical parameters including the slump, the fine particle content and the solids content of backfill slurries can be used as the descriptive criterion. In the *Handbook on Mine Fill* (Potvin et al. 2005), it accepts CPB has a range of definitions, including:

- Contains at least 15% of fine particles passing 20 microns.
- Paste-like slurry, no bleeding and settling or segregation in a pipeline.
- Non-Newtonian slurry.
- Has a slump of less than 230 mm (Henderson et al. 2005).

To ensure the CPB consistency, Belem & Benzaazoua (2004) described that the ideal slump should be in the range of 150–250 mm. Based on engineering experience, for mine stope fill, Sofrà (2017) suggests the yield stress of CPB slurry should more than 250 Pa. There are still some other views about the requirements of working performance of CPB (Cui & Fall 2016; Wu et al. 2016). Once a backfill slurry can fulfill the requirements, it will be treated as CPB which acquires the anti-segregation properties. Indeed, the empirical criteria are convenient for engineering practice, but for the study of the anti-segregation mechanism and structure properties of CPB, such descriptive definitions are ambiguous and hard to be used for further quantitative analysis.

Thus, in this paper, by using unclassified tailings and PO42.5 Portland cement as ingredients, backfill slurries with different solid contents will be prepared and cured. To experimentally study the segregation-induced inhomogeneous properties, the cured backfill samples will be cut evenly into five segments along the settling direction. Thereby, the cement content and porosity of each section will be measured, and based on the tested results of different sections, the segregation-induced inhomogeneous properties can be somehow compared and analysed. Moreover, through this experimental method and the measured results, a quantitative criterion for its identification can be explored from the perspective of inhomogeneity inhibition.

2 Experimental program

2.1 Materials used

This study focuses on the segregation-induced changes of cemented tailings backfill slurries. Thereby, materials used are tailings, binders and mixing water, and no aggregates or other additives are included.

The tailings utilised were unclassified tailings obtained from a copper mine in China. The main physical and chemical properties of the tailings are presented in Table 1. From the table, it can be found that the main component of the tailings is Si and the content of S is 4.9%, indicating the sulphate effect on slurries can be neglected. The tailings was homogonised prior to analysis to guarantee a representative of samples. The particle size distribution (PSD) of the tailings is monitored by a laser particle size analyser (Mastersizer 2000 series). From the results (Figure 1), it can be seen that more than 40% of the particles are passing 20 microns.

PO42.5 Portland cement is used as the binder. This cement is the ordinary binder used in many Chinese mines, and its properties have been well studied by researchers. As the chemical effect of mixing water is not the topic of this study, tap water is used as the water to mix the binder and tailings.

Table 1 Physical and chemical properties of tailings

Tailings type	Ca (wt%)	Si (wt%)	Al (wt%)	Mg (wt%)	Fe (wt%)	Mn (wt%)	S (wt%)	Specific gravity
Unclassified	3.0	20.3	5.5	7.0	16.5	0.1	4.9	3.06

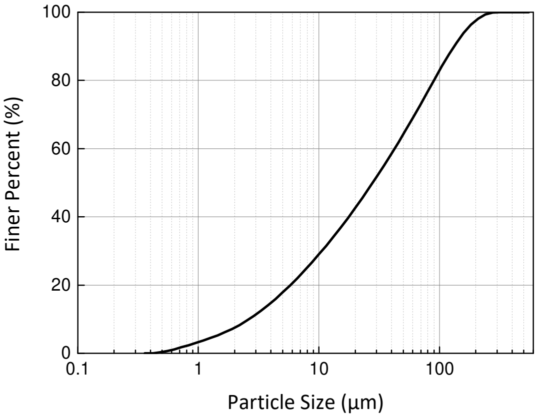

Figure 1 Particle size distribution of the tailings used in the study

2.2 Mix recipes and samples preparation

2.2.1 Mix recipes

Once the ingredients have been selected, the solids content of the backfill slurry will hugely affect the settling process of solid particles (Siddique et al. 2008; Cabrera et al. 2009), and then influence slurry segregation. Thus, the solids content was selected as the sole variable for mix recipes. As described above, the methology of this study is to directly measure and analyse the different properties of various sample segments. This is a new and nonstandard experimental method. Therefore, the solids contents were selected at a relatively large range here (not only limited at the range of paste) to ensure the differences of each segments can be well tested and compared, which are 50, 60, 65, 70, and 75 wt% respectively. To ensure comparability, the total mass of solid materials of each sample is fixed as 700 g, and by changing the amount of mixing water, the different solid contents can then be obtained.

Moreover, as cement content is one of the testing items, to strengthen the segregation-induced differences of specimen as well, the initial mixing cement content needs to be high, which is fixed at 20 wt% (proportion in total solid materials) for all slurries.

2.2.2 Samples preparation and curing condition

The tailings and cements were firstly weighed by a scale and mixed by an electric mixer. Then, the tap water was poured into the electric mixer based on the designed solid content. The ingredients of each sample should be mixed for more than 5 mins to ensure the mixing quality. After that, the backfill slurries were poured into cylinder moulds (diameter 75 mm and height 150 mm), and all the moulds were placed into a curing room, which maintains the curing temperature at 25±5°C and relative humidity at 95±5%, for three days.

2.3 Testing and monitoring

2.3.1 Cutting and numbering method of samples

After curing for a designed period, as described, each sample was cut into five equal parts in height, and these sample parts were numbered as 1 to 5 along with the particles settling direction for further testing. The cutting and numbering method is shown in Figure 2.

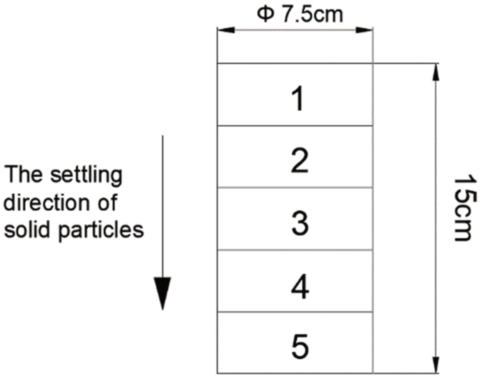

Figure 2 Sketch map of the cutting and numbering method of cured backfill samples

The cement content and porosity of each sample part were measured. For accurate discussion, the test results will be described in the form of initial letter of testing content–solid content of backfill slurry-sample part number. For example, 'c-60%-1' stands for the cement content of the no. 1 part of the sample which consists of 60% of solid materials.

2.3.2 Pre-treatments of samples

As cement will continue to react with water after curing, the pore structure of cemented tailings samples is changing over time. Therefore, to prevent the hydration process of cement, some pre-treatments have been done. Namely, once cut, each sample part has been merged into a hydrous ethanol for more than 12 hours and then been dried at the temperature of 105°C for 24 hours. After that, the sample part was sealed by plastic film for further experiments.

2.3.3 Cement content testing

The cement content of each sample part was tested by using the titration measures of EDTA-2Na (the details of this method can be derived from Chen et al. 2018, 2019). This is a chemical testing method, and its mechanism is to use 10 wt% NH_4Cl solution to react with the $Ca(OH)_2$, the main hydration product of cement in samples, to generate $CaCl_2$. Then, by using EDTA-2Na standard solution, the amount of Ca^{2+} produced by the dissolution of $CaCl_2$ can be titrated and measured to reflect the content of cement in the samples.

2.3.4 Porosity testing

The porosity of each sample part was measured utilising a helium gas expansion porometer (Vinci HEP-P). This kind of porometer has been used in many similar studies, such as the analysis of pore structures and permeability of coal samples (Li et al. 2012; Pan et al. 2019). All the measurements were operated under the routine core analysis methods following the Chinese Oil and Gas Industry Standard (SY/T)5336-1996 (National Energy Administration 2017). The pressure sensor accuracy of the Vinci HEP-P porometer can reach up to 0.1%, which can totally fulfill the accuracy requests of the testing.

3 Results and discussion

3.1 The inhomogeneous cement contents of the cured cemented backfill samples

To clearly demonstrate the inhomogeneous properties of cemented backfills, the cement contents of different parts of cured samples with different solid contents have been plotted against the sample part numbers (Figure 3).

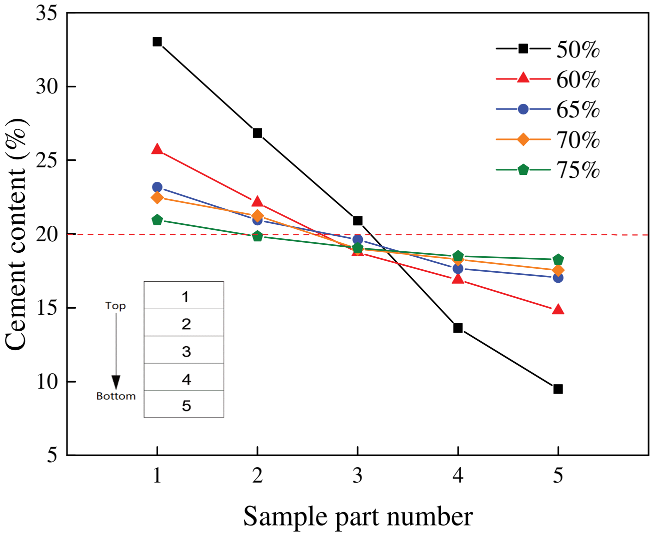

Figure 3 The cement contents of different parts of the cured samples with different solid contents

From the figure, it can be found that there are huge differences in cement contents among different sample parts. For all cured samples, the cement contents decreased along the settling direction from top to bottom. For the top two parts of the five samples, their cement contents are all more than 20% (shown as the red line in Figure 3), which is the designed content of mixing, while the values of other parts are commonly less than that. This indicates extra cement has been 'transported' to the top parts. Based on the studies of the settling properties of tailings (e.g. Peng et al. 2019, 2020), the reason can be explained as the sizes and relative density of cement particles are usually smaller than that of tailings. Thus, when backfill slurries are placed into the sample moulds, tailings particles have greater hindered settling rates than cement; therefore, more tailings will gather at the bottom and then extra cement will appear at the top part by contrast.

In terms of the effect of slurry solid contents on cement distribution, Figure 3 shows that the variations of cement contents decreased against the increase of solid contents; namely the sample which has a larger solid content shows a more homogeneous cement distribution. This can be explained as the results of particles settling as well. Through the theories of tailings hindered settling, the interactions of tailings will exponentially increase along with the increase of particles concentration (solid contents), which will enlarge the hinderance among particles and results in decreasing the segregation content. Thereby, the distribution of cement can be more uniform.

More specifically, from Figure 3, for sample part c-50%-1, its cement content is around 33 wt%, which is 13% larger than the initial designed value. While, by contrast, the value of c-50%-5 is only around 10 wt%, which is half of the designed content and is less than one third of the content of c-50%-1. Furthermore, when it comes to the sample with the solid content of 75%, the range value of different parts is only around 3 wt%, nearly a homogeneous distribution. The range values are meaningful as it quantitively describes the segregation-induced inhomogeneous cement content of cemented backfills. It is well accepted that the uniaxial compressive strength (UCS) of cemented backfill decreases with the decrease of solid content. Whilst, as mentioned in Section 2.2.1, the total solid mass of each sample is fixed; therefore, the range value comparison may also indicate that the inhomogeneous content is likely to be the determining factor affecting the UCS of cemented backfills. This is of great research value in further study.

3.2 The inhomogeneous porosities of the cured cemented backfill samples

To investigate the changes of porosities along with the backfill materials settling direction, the sample parts' porosities of the cured backfills with different solid contents have been plotted against the part numbers (Figure 4).

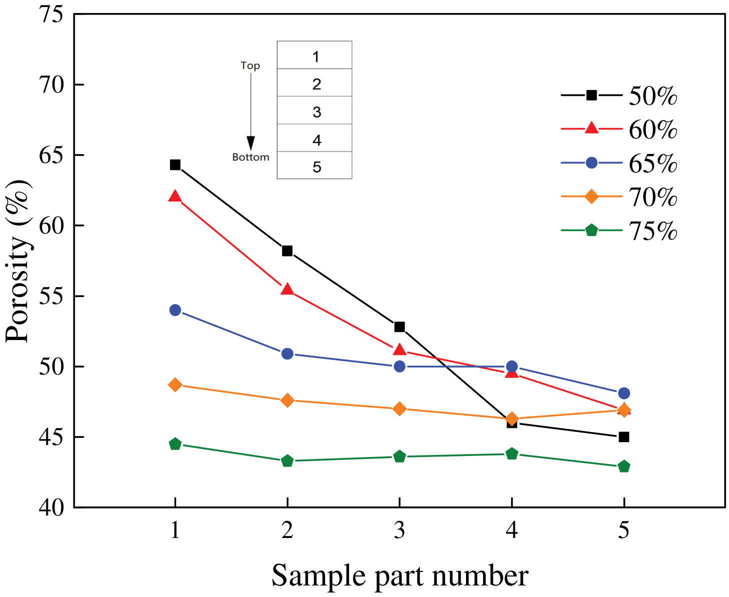

Figure 4 The porosities of different parts of the cured samples with different solid contents

Figure 4 shows that, except the sample with 75 wt% solid contents which has a fluctuation of porosities, the values of other sample parts all decrease with the increase of part numbers. Namely, alongside the cured samples, the porosities of top parts are usually smaller than that of bottom parts. This inhomogeneous feature can also be explained by the solid particles' settling properties. As discussed previously, in the hindered settling process, particles with larger sizes and/or relative densities will have higher settling rates and then precipitate more quickly at the bottom parts. As a result of this, more large particles will appear at the bottom parts, which will enlarge the uniformity coefficient of the bottom particles. Based on soil mechanisms, this increase of uniformity coefficient can then lead to the decrease of porosity.

Figure 4 also shows that the solid content of the cemented sample can dramatically affect the differences of the sample parts' porosities—the range value of porosities will decrease sharply with the increase of solid contents. This is similar to the effect of sample solid content on cement distribution, and then can be explained as the result of the effect of solid content on the particles' hindered settling as well. Once the solid content increased, the segregation induced by the particles' hindered settling can be restricted, resulting in more homogeneous particle distribution and much closer uniformity coefficients of different parts. Therefore, the differences in porosities can be decreased.

Moreover, for the sample with 50 wt% solid content, its top part, p-50%-1, has a porosity of 65% units, while the value of bottom part is 45%. Thus, the range value of porosities is around 20%. By contrast, the range value of the sample with 75 wt% solid content, as shown in Figure 4, is around 2%, namely only one tenth of the data of 50 wt% sample. Indeed, this quantitative inhomogeneous result is really essential as there is fewer study data that can be derived from former research. As the total mass of solid materials is fixed, the tremendous range value of porosities indicates that this inhomogeneous feature may also be a strength determining factor of cemented backfills.

3.3 Comprehensive discussion about the inhomogeneous porosities

Through the aforementioned discussions, it is obvious that the inhomogeneous properties of cement content and porosities have many similarities. It indicates the solid content of backfill slurry has the same impact on these two properties. Therefore, there is still a need to comprehensively compare and analyse the cement content and porosity testing results.

In statistics, the variance of experimental results is regarded as the value representing the differences of samples. Thus, the collected testing data was used to calculate the variances of cement contents and porosities, provided in Table 2.

Table 2 The variances of cement content and porosities of cured samples with different solid contents

Solid content (wt%)	Variance of cement content (%)	Variance of porosities (%)
50	8.55	6.03
60	3.85	5.14
65	2.23	2.23
70	1.86	1.94
75	0.98	0.81

Based on the above data, the variances are plotted against solid contents to comprehensively investigate the inhomogeneous properties (Figure 5).

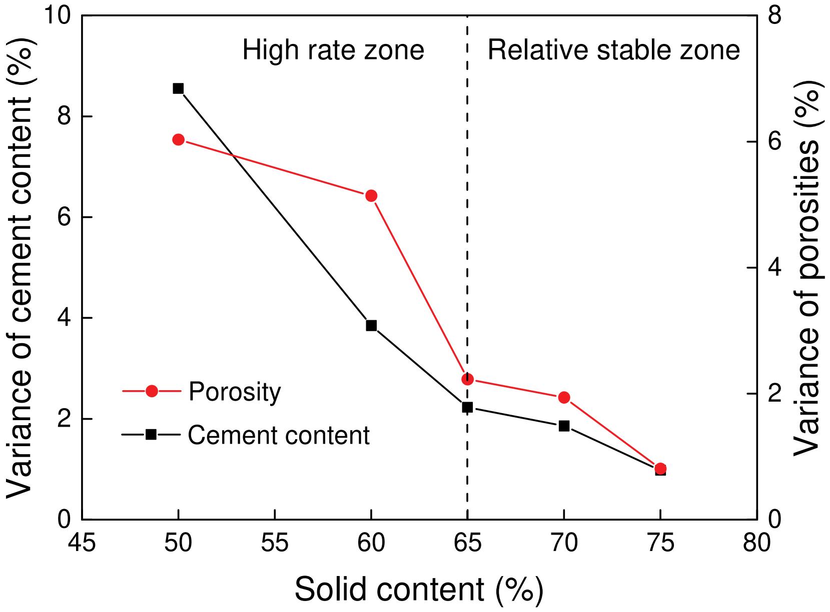

Figure 5 The variances of cement content and porosities of cured cemented backfills against the solid contents of samples

From the figure, it is obvious that the variances of both properties decrease along with the increase of solid contents; namely the inhomogeneity of cemented samples would decrease if there are more solid materials. This can still be explained by the hindered settling effect mentioned previously.

It can also be seen from Figure 5 that there is a turning point of solid content (65% in this study), lower than which the reduction rate of variances maintains at a high value. By contrast, when the concentrations of slurries are higher than the point, the reduction rate decreases dramatically, and the variances become relatively stable. Therefore, based on the phenomenon, this solid content turning point can be treated as an evaluation value of the inhomogeneities of cemented samples, and then be chosen as a quantitative criterion for the practical CPB identification from the perspective of inhomogeneity inhibition.

4 Conclusion

The cement content decreases from top to bottom along the settling direction of backfill materials. The solid content of backfill slurry can hugely affect the inhomogeneities of cement content. For a cured sample with low concentration, the cement content at the top part can only be one third of the value at the bottom part.

The porosities will decrease along the settling direction of solid materials as well. Similarly, the solid content has a dramatic effect on the inhomogeneity of porosities. For a cured sample with the concentration of 50 wt%, its porosities range value is 10 times larger than that of the sample with a solid content of 75 wt%.

Based on the variance analysis results, there is a turning point of solid content, lower than which the inhomogeneity of cured samples can decrease sharply with the increase of concentration. While, by contrast, when the solid content is higher than the turning point, the reduction rate decreases dramatically. Therefore, this turning point can be chosen as a new quantitative criterion for practical CPB identification from the perspective of inhomogeneity inhibition.

The experimental results reflected some properties of backfill slurry segregation, while there are still many improvements that can be made. For example, as explained, to ensure the validation of the testing method, the solids contents were selected at a huge range, which did not focus on CPB, and the rheological properties of sample segments were not measured. Therefore, future work will focus on densities and binder contents more in line with typical CPB including more thorough characterisation and rheological testing. More studies focusing on the details of 'turning point' will be designed and performed in the future.

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