International Journal of Environmental Science and Technology (2022) 19:131–140
https://doi.org/10.1007/s13762-021-03659-7

ORIGINAL PAPER

Effect of different particle size distribution of zeolite on the strength of cemented paste backfill

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Received: 12 September 2020 / Revised: 18 February 2021 / Accepted: 8 September 2021 / Published online: 20 September 2021

Abstract

Cemented paste backfill system used for the storage of tailings underground also serves as support against subsidence due to roof loads. Therefore, paste backfill materials should be optimized according to strength, environment, and cost requirements. The main purpose of this study is to determine the ratio and optimal particle size distribution of zeolite which is one of the natural pozzolanic materials and can substitute cement in paste backfill. As a result of the experiments without zeolite, it was determined that paste backfill materials with the cement ratio of 9% and 11% can be used for ground support instead of the cemented paste backfill reference samples with 80% solid content. Then, zeolite-substituted samples were prepared in 2 different particle sizes of − 90 µm and − 180 µm at cement ratios of 5%, 10%, 15%, and 20%. Then, the effects of the paste backfill materials on the strength of curing periods of 28, 56, and 90 days were examined. As a result, it was found that 9% cemented paste backfills with 15% zeolite substitutes (− 90 µm) and 11% cemented paste backfills with 10% zeolite substitutes (− 180 µm) could be used in paste backfill. Also, 11% cemented paste backfills with 10% zeolite substitutes (− 180 µm) provide better strength depending on the curing times.

Keywords Cemented paste backfill · Waste and tailings management · Environmental · Strength

Introduction

The majority of the tailings dams failures can be attributed to factors such as type and material of construction, height, length, and total storage volume of the dam, as well as the inadequacy of drainage. The failure may ultimately have been triggered by a particular mechanism of one of these factors. Moreover, experiences showed that the failure of the tailing dams poses a serious environmental threat (Bowker and Chambers 2015, 2016; WISE 2020). As a solution to this problem, the filling of the tailings with the cement paste backfill (CPB) not only supports the storage of tailings in the underground but also provides roof support in underground mining. Therefore, the maximum strength value of the paste material has a significant role in the stability of the roof support. The strength of the CPB mixture should be ≥ 4 MPa for roof support and ≥ 1 MPa for free-standing (Hassani and Archibald 1998; Li et al. 2002; Fall et al. 2005; Li and Fall 2016). However, Belem and Benzaazoua (2008) stated that the required uniaxial compressive strength (UCS) of the fill should be at least 5 MPa to provide adequate ground support.

CPB material, which is filled into stopes, usually consists of a mixture of tailings with cement and its solid content (SC) that ranges between 70 and 85% (Yilmaz et al. 2011; Wu et al. 2015; Li and Fall 2016; Jiang et al. 2017). Variability in solids ratio is important for the pumpability of the paste, and therefore an adequate solids ratio is selected in the slump range of 15–25 cm (between 6″ and 10″) (Belem and Benzaazoua 2008; Cihangir et al. 2015; Ouattara et al. 2017). Moreover, it is seen that different cement binder additive ratios to paste backfill based on weight have been used in previous research (≤ 4% (Wang et al. 2017), ≤ 4.5% (Benzaazoua et al. 2002; Li and Fall 2016), ≤ 6.5% (Ouattara et al. 2018), ≤ 8% (Wu et al. 2015; Koohestani et al. 2017), ≤ 10% (Huang et al. 2011)).

However, CPB can be applied in two steps as plug-fill up to the first few meters and residual fill applied after about 1 week. While cement content in the plug-fill

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varies between 5 and 7%, the residual fill cement content can change between 2 and 5% (Yilmaz et al. 2015). The required strength values for CPB and the physical and chemical properties of the components in the paste mixture are the main factors accounting for the differences in cement ratios. It is expected that the strength of the paste backfill will increase with the amount of cement. However, the effect of calcium-rich cement on the strength may differ according to the sulfate attacks due to the sulfur content in the tailings (Dong et al. 2019).

The cost-effectiveness of cement used in the mixture of paste backfill is another important point. In general, CPB constitutes 10–20% of the total costs of the mining operation, and cement forms 75% of the total CPB costs (Belem and Benzaazoua 2008). Pozzolanic materials such as fly ash, blast furnace slag, and silica fume are added to the mixture to reduce the binder costs and increase the stability against sulfate attacks (Cihangir et al. 2015). Therefore, pozzolanic materials are used as additives in different ratios of dry tailings of fly ash, blast furnace slag, and silica fume at the rate of 1.5–5%, 2.4–8%, and 1.8–6% by weight, respectively (Jung and Biswas 2002; Benzaazoua et al. 2004; Yilmaz et al. 2009).

As an alternative, zeolite is a promising pozzolanic material. Natural zeolites (clinoptilolite and analcime) have the potential to be used in sustainable blended cement productions due to their favorable qualities such as silica-alumina content, high pozzolanic activity, low density, high specific surface area, and suitable mineralogical structure (Akgun 2017). As a result of these favorable properties, zeolite increases the strength of concrete through pozzolanic reaction with $Ca(OH)_2$, prevents unwanted expansion due to alkaline aggregate reaction, decreases the porosity of blended cement paste, and improves the interfacial microstructure properties of blended cement paste (Canpolat et al. 2004). In addition to these benefits in the mechanical properties of the cement paste, it has been indicated in several environmental studies that the negative ion content of zeolite is also suitable for wastewater treatment and the absorption of toxic gases ((Doula and Ioannou 2003; Trgo and Peric 2003; Mozgawa and Bajda 2005; Morali 2006; Bilgin and Kantarci 2018).

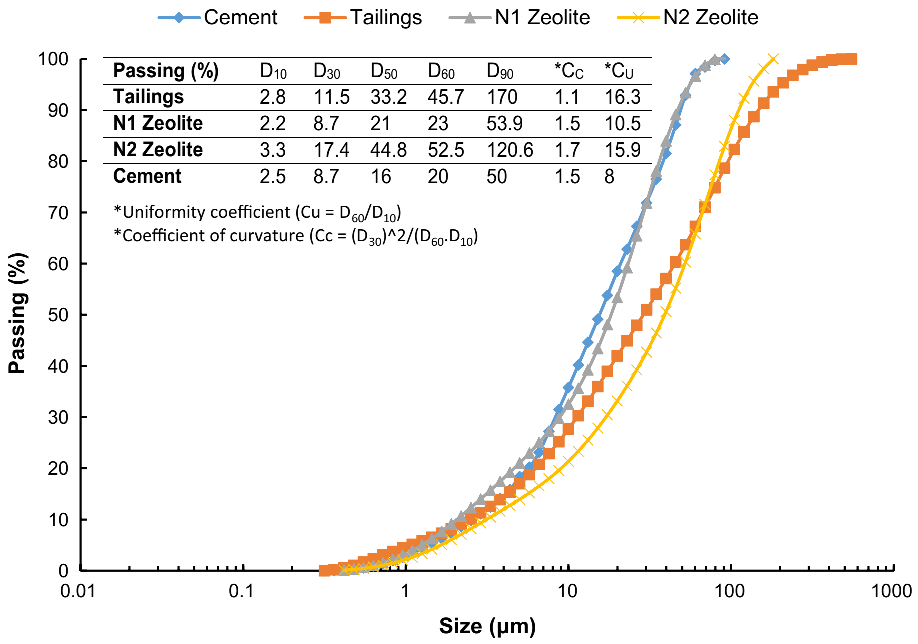
This study aimed to reduce the amount of cement that forms a high cost for CPB by using zeolite. At the same time, the optimal particle size of the zeolite, which will be used instead of cement, has been determined to obtain the necessary strength according to the purpose of use in CPB. The CPB material, which has a cement content between 3 and 11% and has a total solid content of 80%, was formed from the tailings of a Pb–Zn underground mine. Optimum cement ratios in paste backfill for use in the roof support were determined by the investigation of the curing periods of 28, 56, and 90 days. Based on these cement ratios, the effect of zeolite on the strength of paste backfill was investigated. Additionally, the effect of natural zeolite with different particle sizes on the strength of CPB was studied for the first time by this research. Furthermore, the possibilities of using zeolite in CPB were investigated in detail.

The studies of this research were conducted in Balıkesir and Istanbul between 2018 and 2020.

Materials and methods

Pb–Zn Pb–Zn mine process tailings were collected from the tailings discharge unit of a Pb–Zn mine in the Balıkesir/Balya region of Turkey. Meanwhile, zeolite samples were collected from the waste-rock dumpsite of a colemanite open-pit mine located in Balıkesir/Bigadic, Turkey. The collected zeolite samples were ground using a ball mill, and two size fractions of − 90 µm (named as N1) and − 180 µm (named as N2) were obtained with dry sieving to investigate the effect of its size on the strength of the CPB. As a cement, CEM I 42,5 R Portland cement was used. The experiments were carried out using tap water. The specific gravity of the samples was determined with a helium pycnometer. Meanwhile, a laser particle size analyzer (Malvern Mastersizer 2000 E) was used for the determination of particle size distribution. A multipoint Brunauer, Emmett, and Teller (BET) analyzer (Quantachrome Corporation, Autosorb-6) was used for the determination of the specific surface area, and an inductively coupled plasma mass spectrometer (ICP-MS) was employed in the elemental analysis. The specific gravities of the tailings, zeolite, and cement used in the mixture were 3.15, 2.32, and 3.13, respectively. The specific surface area of the tailings was 3.6 m^2/g, while it was 0.92 (m^2/g) for N1 zeolite and 0.62 for N2 zeolite. In the case of using cement, the specific surface area was determined as 0.3 m^2/g. The particle size distributions and chemical properties of the materials used in the paste mixture are given in Fig. 1 and Table 1, respectively.

One of the important parameters in the pumping of paste is its tailings material content sized below 20 µm (Brackebusch 1994). Adiguzel and Bascetin (2019) determined that the amount of the tailing material content < 38 µm in the paste should be at least 20% in order to ensure the efficient pumpability of the paste. As seen in Fig. 1, in this study, ≤ 20 µm fraction ratios for tailings,



Passing (%)	D_{10}	D_{30}	D_{50}	D_{60}	D_{90}	*Cc	*Cu
Tailings	2.8	11.5	33.2	45.7	170	1.1	16.3
N1 Zeolite	2.2	8.7	21	23	53.9	1.5	10.5
N2 Zeolite	3.3	17.4	44.8	52.5	120.6	1.7	15.9
Cement	2.5	8.7	16	20	50	1.5	8

*Uniformity coefficient (Cu = D_{60}/D_{10})
*Coefficient of curvature (Cc = (D_{30})^2/$(D_{60}.D_{10})$)

Fig. 1 The particle size distribution of tailings, cement, and zeolite (N1 and N2)

Table 1 Chemical composition of the tailings, zeolite, and cement

Compound	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	S*/SO_3**
Tailings (%)	13.6	36.2	8.1	23.3	2.5	2.6	0.2	7.5*
Zeolite (%)	1.4	70.5	11.1	2.8	1.2	4.2	0.3	0.2**
Cement (%)	3.4	19.9	4.8	61.6	1.3	0.9	0.3	3.7**

*indicates S content, **indicates SO_3 content

N1 zeolite, N2 zeolite, and cement are 42%, 53.5%, 33%, and 58.5%, respectively. Therefore, it can be said that both N1 zeolite and cement mainly consist of fine particles. On the other hand, it is seen that N2 zeolite consists of coarser particles. Thus, the tailings and N2 zeolites can be classified as "multi-graded," while the cement and N1 zeolites are "medium-graded" according to ISO 14688-2 standard. Meanwhile, according to EN196-6 standard, the cement should have less than 14% wt. of cement particles coarser than 90 µm. In addition, when the coefficient of uniformity (C_U) value increases in particle size distribution, it is expected that the early period strengths of mixtures should be improved. Particularly, the fineness ratio is a very effective parameter for early strengths. Therefore, higher strength is obtained after the hydration reaction depending on the activity of cement and pozzolan particles. To achieve this activity, the particles must be finely ground. Long-term strengths are more prominently affected by coarser particles compared to fine ones. In general, it is thought that proper particle size distribution does not only strengthen the structure of the CPB to optimum levels, but also increases freezing strength, corrosion resistance, flow property, and water requirements (Wu et al. 2018).

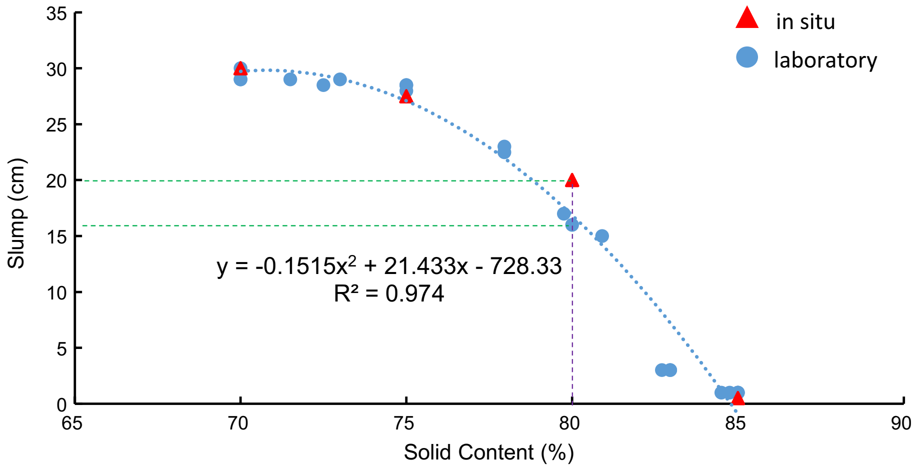
It is seen from the chemical analysis results of the zeolite material in Table 1 that the total SiO_2, Al_2O_3, and Fe_2O_3 content of zeolite material is 83%, which is higher than the minimum required content of 70% according to ASTM C618. Furthermore, the CaO (< 3%), MgO (≤ 5%), and LOI (< 10%) values of the zeolite are also fulfilling the related standard. The mineralogical content of the natural zeolite in the Balıkesir/ Bigadic region generally consists of clinoptilolite as the main component (~ 80%) with lesser amounts of quartz (~ 13%) and biotite (~ 5–6%) (Morali 2006; Bilgin and Kantarci 2018; Eker 2019). Eker (2019) showed that the zeolite obtained from the same region has a pozzolanic activity index of 84.07% on day 28 and 95.43% on day 90. According to ASTM C311 and ASTM C618 standards, the pozzolanic activity index values of the samples on day 28 should be at least 75%. Since the SO_3 content of the zeolite is under the maximum limit in ASTM C618 (4% SO_3) as seen in Table 1, it is clear that the zeolite provides pozzolanic

Fig. 2 Main stages of the study (a-slump test, b-mixing, c-molding, d-curing, e-UCS test)

activity conditions. Therefore, it was decided to use it as a substitute for cement within this study.

In this context, the optimum solid content of the paste mixture was determined with slump tests according to ASTM C143 at the solid content range of 70–85% (Fig. 2a). To determine the results of the effect of the amount of cement on the strength of CPB formed by the Pb–Zn tailings we used in the study, the minimum and maximum binding ratios of 3–11% were selected as appropriate to the literature. Subsequently, cement was included at various ratios (3%, 5%, 7%, 9%, and 11%) by weight of the solid material and thus the reference samples were obtained. According to the literature, either process water or tap water can be used in paste mixture (Fall et al. 2009; Hefni 2014). In the context of this study, tap water was used. A concrete mixer was employed for the homogenization of the mixture (tailings, cement, and water) as seen in Fig. 2b. The paste was mixed for 7 min according to the procedure detailed by Ghirian and Fall (2015). The prepared paste backfill mixture was transferred into cylinder sample molds with a diameter of 5 cm and a height of 10 cm (Fig. 2c). These reference samples were subjected to the uniaxial compressive strength (UCS) tests according to ASTM C39 (Fig. 2d, e) at the end of 28, 56, 90, and 200 days of curing at 80% moisture and 22 °C. Minimum 5 samples from each mixture were tested, and the arithmetic mean was obtained. The cement ratios of the reference samples were determined considering the minimum strength value for providing the roof support (4 MPa). Since strength is an important factor for the roof support, zeolite was added as a substitution of cement at the rate of 5%, 10%, 15%, and 20% by weight considering the strength activity index test according to ASTM C311 standard. The same zeolite sample was used in two different particle sizes of – 90 µm (N1) and – 180 µm (N2). UCS test was performed for 28, 56, and 90 days under the same curing conditions similar to the reference samples. In the reference samples with 9% and 11% cemented paste, the UCS results of 90 and 200 days were very close to each other. For this reason, in the experiments with zeolite, a maximum curing time of 90 days was applied.

Fig. 3 Slump test with respect to solid content



Results and discussion

The results of the paper were discussed under three sections which are the determination of solid content, strength of reference samples, and the effect of zeolite ratio.

Determination of solid content

It is a fact that fluidity is very important in the pumping and the placement of the paste backfill material into the stope. In the CPB method, generally, slump tests are carried out to have an opinion about the fluidity of the paste, and 18 cm slump value is frequently accepted for efficient pumping and placement (Tariq and Nehdi 2007; Fall and Pokharel 2010; Ghirian and Fall 2016). In addition, a paste filling material with an 18 cm slump value can be conveyed to stope with natural flow or its own weight at a flow rate of 100 tons/hour with a pipe system with a diameter of 15 cm, as stated by Belem and Benzaazoua (2008). The results of the in situ and laboratory-scale slump experiments are given in Fig. 3.

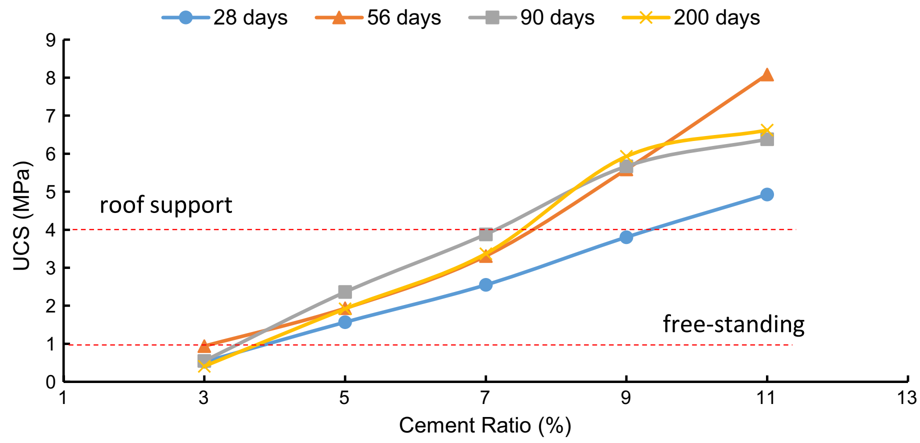
As seen in Fig. 3, the closest slump value to the 18 cm was obtained at 80% solid content. On the other hand, slight differences in slump values were observed at the same solid content in the in situ (20 cm) and laboratory-scale (16 cm) experiments. The main reason for the slump reduction in CPB at the same solid content can be explained by the increase in the water requirement apart from the increase in the surface area of the fine tailings particles (Fall et al. 2008). Clark et al. (1995) observed an increase of 5 cm in a slump which caused a decrease of 1% in the solid mass concentration. Moreover, it also reduced the pressure in the pipe during transportation. A similar situation can be seen in the slump results presented in Fig. 3. To accurately define the rheology of paste backfill, both shear yield stress and viscosity should be measured.

Strength of reference samples

UCS values of 28-day cured samples increased with the high rate of binder in the paste backfill. As seen in Fig. 4, the obtained results are in agreement with the previous studies (Ghirian and Fall 2016; Eker 2019). Higher binder ratios lead to an increase in the hydration products, which resulted in increased cohesion and reduced porosity. Thus, the UCS values were also increased (Ghirian and Fall 2016; Eker 2019). In addition, Fall et al. (2008) reported that the ratio of macropores (1–10 μm) in CPB increased significantly in the presence of a 25% fine particle. In this case, the fine particle content in the material increased the water-holding capacity of the mixture, which may affect the strength of the paste adversely. UCS test results of 28, 56, 90, and 200 days of the reference samples with 3%, 5%, 7%, 9%, and 11% cement binder ratio at 80% solids content are given in Fig. 4.

It is seen in Fig. 4 that paste materials with 9% and 11% cement can be used for roof support with a curing time of 28 days. It is also clear in Fig. 4 that at least 5% cement should be added to use the tailings as freestanding paste backfill for all curing days. The UCS values of 56 and 200 days were the same at 5% and 7% cement ratio. However, the UCS values of 90 and 200 days are significantly lower than that of 56 days curing at an 11% cement ratio. This decrease in the UCS has been attributed to the negative effects of sulfate attacks in the literature (Fall et al. 2004; Kesimal et al. 2005). Fall et al. (2004) stated that samples with more than 35% sulfur content were found to have lower strength values

Fig. 4 UCS results with respect to cement ratio



compared to the samples that have shorter curing times. Furthermore, Li and Fall (2016) reported that minerals with swelling properties such as ettringite cause the pore structure of the CPB to expand, which leads to a structural deformation as calcium silicate hydrate (C-S–H) absorbs sulfates, thus resulting in lower strength values in CPB.

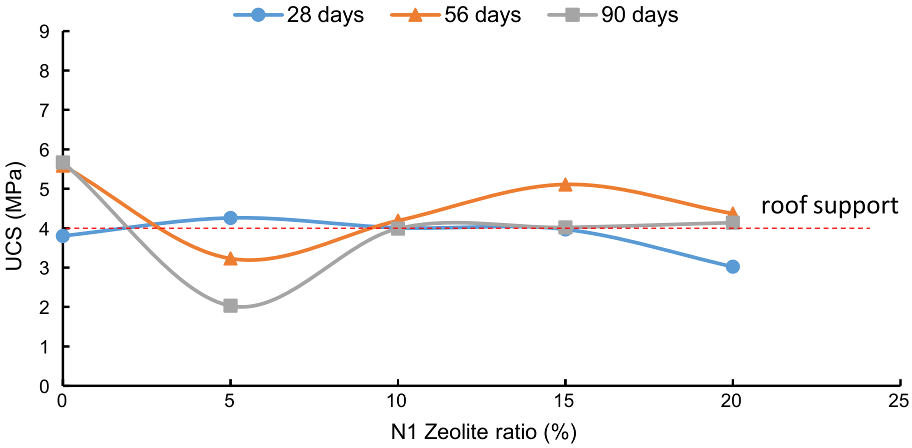


Fig. 5 UCS results as a function of N1 zeolite ratio of 9% cement

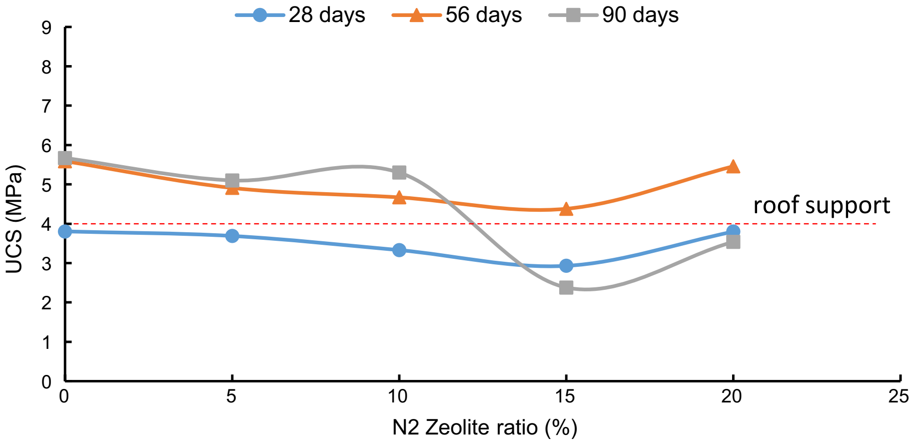


Fig. 6 UCS results as a function of N2 zeolite ratio of 9% cement

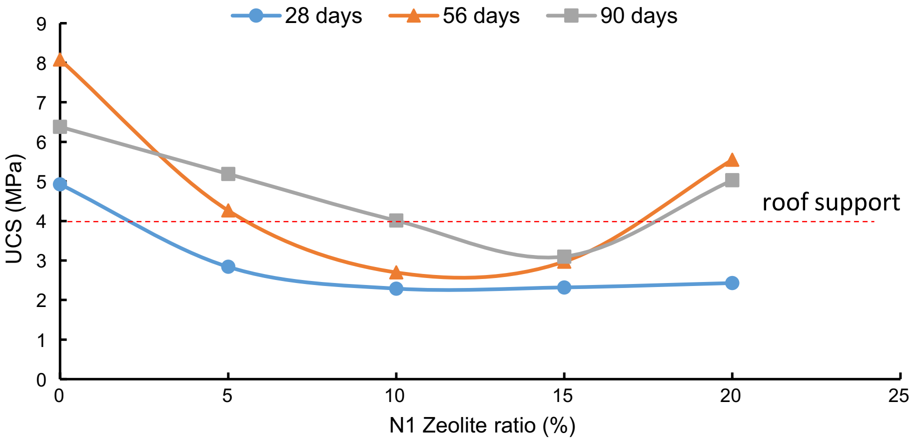
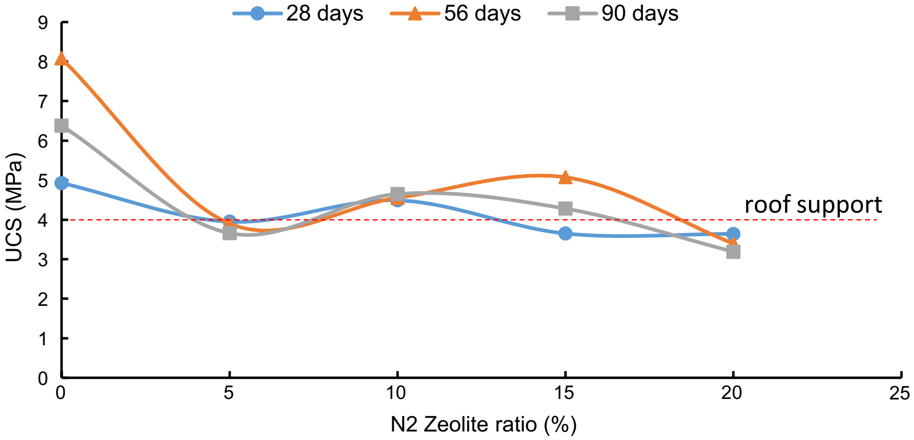


Fig. 7 UCS results as a function of N1 zeolite ratio of 11% cement

Fig. 8 UCS results as a function of N2 zeolite ratio of 11% cement



Effects of zeolite ratio

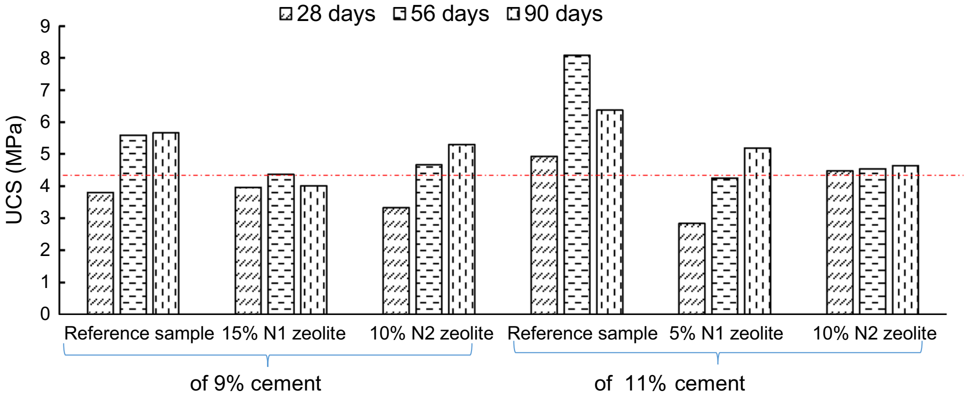
In In this study, to reduce the negative effects of sulfate attacks, natural zeolite was added in two different particle sizes as – 90 µm (N1) and – 180 µm (N2). The UCS results of the zeolite used in the binder content of 9% are given for N1 and N2 in Figs. 5 and 6, respectively.

As seen in Fig. 5, the paste backfills with 5%, 10%, and 15% zeolite provided the required strength for the roof support (4 MPa) at 28 days of curing time. However, increasing the zeolite ratio to 20% caused a significant decrease in the UCS, which posed an obstacle to using it as roof support. Meanwhile, in the case of 56 and 90 days curing time, the substitution of zeolite at 5% decreased the UCS of the paste backfill considerably below the strength threshold. However, the UCS increased with the increase in the zeolite ratio and met the support threshold at 10%. The UCS reached a plateau for 90 days at a 10% zeolite ratio, while it continued to increase for 56 days and reached 5.11 MPa at 15%. Then, it decreased as a function of zeolite and met the UCS of the 90 days near 4.2 MPa, which was still above the roof support threshold. Therefore, it can be inferred that CPB with 10% and 15% can be used for roof support.

For the N2 zeolite, it is seen in Fig. 6 that the strengths on the 28 days are below the strength threshold. On the other hand, the strength of 56 days was above the strength threshold at all zeolite ratios. However, while this situation was similar for 90 days up to 10% zeolite ratio, it showed a significant decrease after 10% and passed under the threshold even at 28 days at 15% and 20%. This decrease in the UCS can be attributed to the sulfate attack.

The UCS results of the zeolite used in the binder content of 11% are given for N1 and N2 in Figs. 7 and 8, respectively. It is clearly seen in Fig. 7 that the UCS values decreased as an effect of the zeolite ratio for all curing days. The UCS of 28 days decreased sharply to 2.9 MPa at 5% and continued to decrease slightly to 2.3 MPa at 10%. After 10%, it reached a plateau. Therefore, it is inferred that the pozzolanic activity in the paste

Fig. 9 Optimum UCS results for different zeolite additive CPB mixtures



backfill is more effective at 5% than the higher zeolite ratios. It is also seen in Fig. 7 that the required strength values could be obtained only at 5% and 20% for 56 and 90 days. It can be concluded from these results that it would be very risky if N1 zeolite substitution were used as roof support instead of cement in 11% binder content.

As seen in Fig. 8, the required strength at 5% zeolite ratio was provided in only 28 days. Meanwhile, it is also seen that the UCS values were above the strength threshold for all curing days at 10%. However, increasing the zeolite ratio to 15% caused a decrease in the UCS below the threshold at 28 days. It is also clear in Fig. 8 that none of the paste backfills can be used as roof support at a 20% zeolite ratio. The optimum UCS results of the experiments of this study are summarized in Fig. 9.

As seen in Fig. 9, 9% cemented paste backfills with 10% N2 zeolite and 11% cemented paste backfills with 5% N1 zeolite caused the UCS of 28 days to remain under the strength threshold, thus they cannot be used as the roof support in the shorter periods of mining activities in terms of safety. On the other hand, the required strength for roof support was provided by all curing days with 9% cemented paste backfills with 15% N1 zeolite and 11% cemented paste backfills with 10% N2 zeolite additive conditions. At 9% cement paste mixture with 15% N1 zeolite additive, the UCS decreased after 56 days due to not being sufficient for pozzolanic effect against sulfate attacks. On the contrary, the increase of the UCS values showed the pozzolanic effect in 11% cement paste mixture with 10% N2 zeolite at the 56 and 90 days. Therefore, it is thought that this phenomenon caused the improvement of the mechanical strength against sulfate attacks depending on the time.

These zeolite ratios of cemented paste mixtures are in accordance with the cement formulation with 10–20% zeolite ratio that is suggested by Burriesci et al. (1985) because of leading to a low free CaO content in the setting of the concrete. In addition, Ikotun (2009) stated that while zeolite addition improved fineness in cement, flexural and compressive strength, the excessive addition of zeolite did not lead to further increase in flexural and compressive strength, and thus it can be ineffective.

Furthermore, in terms of operating costs, the storage of zeolite as a mine waste close to the study area could provide an advantage in terms of raw material supply. To obtain the zeolite supply used for grinding, it must be transported from an approximate distance of 100 km. Moreover, an additional zeolite silo must be integrated into the cement silo located in the paste filling plant.

Meanwhile, the nearest cement production plant is located at the relatively same distance to the study area. Cement, which constitutes approximately 75% of the total paste filling costs (Belem and Benzaazoua 2008), must undergo a series of industrial processes such as high heat treatment, different additives, grinding, and packaging before use, which increases the cost of cement. Eker (2019) stated that the use of zeolite with a particle size of – 125 µm as a substitute to cement up to 20% ratio could reduce the binder costs in the range of 9–35%. Therefore, zeolite is considered a more economical option due to the higher energy costs in cement production.

Conclusion

It is a fact that different pozzolanic materials can provide the required strength as cement substitutions in the CPB method, which can be used for roof support in underground mines. Therefore, in this study, zeolite, obtained as a waste of an open-pit mine located near the study area, was used as a pozzolanic additive. The strength of zeolite-substituted CPB was measured according to the different curing times. As a result of this study, it was revealed that the use of zeolite in different sizes and ratios changed the strength of the CPB depending on the curing time. Moreover, it was understood that the pozzolanic effect improved the mechanical strength against sulfate attacks as an effect of time. In addition, it was shown that the Pb–Zn tailings in the study area could be used as a zeolite-substituted CPB for roof support in the underground mine. The results of this study clearly indicate that the PB–Zn tailings and the zeolite waste rock, which are generally stored on the surface, can be successfully used for backfill.

Acknowledgments The authors wish to thank all who assisted in conducting this work.

Funding No funding was received to assist with the preparation of this manuscript.

Declarations

Conflicts of interests The author has no conflicts of interest to declare that are relevant to the content of this article.

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