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The effect of hard coal fly ash substitution for cement on properties of high sulphide tailings cemented paste backfill

Introduction

Cemented paste backfill method (CPB) is preferred for the storage of tailings resulting from enrichment. CPB is expressed as a successful mixture of fine-sized ore enrichment tailings (75–85% ratios by weight), binder (3–9% ratios by weight), and added water to ensure the desired fluidity and solid ratio (70–80%) (Ercikdi et al. 2013; Kesimal et al. 2005; Yilmaz and Guresci 2017; Yılmaz et al. 2017).

Cement is one of CPB's main materials. Obviously, one of the main costs of the paste backfill plant is cement (Naylor et al. 1997; Grice 1998; Belem et al. 2000; De Souza et al. 2003; Fall and Benzaazoua 2003; Ercikdi et al. 2017; Adiguzel et al. 2022; Bascetin et al. 2023; Esmailzadeh et al. 2023). It is therefore very important to select the optimum type

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and dosage of binder to ensure both the desired strength and stability and to minimize the operating costs of the paste backfill plant. Therefore, various researchers have tried to reduce the binding cost by using pozzolanic minerals (marble powder, zeolite, fly ash, pumice, silica fume, coal, blast furnace slag, etc.) instead of cement to reduce the cement costs (Benzaazoua et al. 2002, 2004; Klein and Simon 2006; Tariq and Nehdi 2007; Ercikdi et al. 2009a, 2010a, 2010b, 2015; Fall et al. 2010; Cihangir et al. 2011, 2012, 2015; Yılmaz et al. 2018; Demir Şahin et al. 2020; Yıldız 2020, 2024; Tuylu 2021, 2022a; Adrianto and Pfister 2022; Chen et al. 2022; Eker and Bascetin 2022a, 2022b; Demir et al. 2024; Yıldız et al. 2024; Yıldız and Tombal-Kara 2024).

One of the pozzolanic materials used instead of Portland cement (PC) is fly ash. Fly ash (FA) is a by-product of the thermal power generation process and consists mainly of silt-sized, spherical, and amorphous ferroaluminosilicate minerals (Salé et al. 1997). According to the Canadian Standards Association (CSA) (CAN/CSA-A3000-03, 2003) ashes are classified as high lime Type CH (CaO > 20%), medium lime Type CI (8% < CaO ≤ 20%), and low lime Type F ash (CaO ≤ 8%).

Depending on the chemical composition of the burnt coal, ASTM C 618 (Shon and Kim 2013) classifies fly ash as either Class C or Class F. The sum of the three main oxides, such as SiO_2, Al_2O_3, and Fe_2O_3, is required to be 50% for at least C class fly ash and 70% for class F fly ash. Class F fly ash is also referred to as low limestone fly ash. Anthracite and bituminous coals have a CaO content of less than 10%. So far, many researchers have investigated using fly ash instead of PC (Weaver and ve Luka 1970; Manca et al. 1983; Ravindra 1986; Yu and Counter 1988; Udd and Annor 1993; Amaratunga and Hein 1997; Benzaazoua et al. 1999; Hassani et al. 2001; Değirmenci and Okucu 2007; Godbout et al. 2007; Hassani et al. 2007; Jiang et al. 2019; Jiang et al. 2020a; Cavusoglu et al. 2021b; Kasap et al. 2022; Sari et al. 2022, 2023; Tuylu 2022a; Eker et al. 2023; Demir Şahin and Eker 2024).

Partial replacement of PC with Class C FA has been shown to be cost-effective in the production of high modulus pastes, but is not suitable for paste strength (Amaratunga and Hein 1997). As a result of mineralogical and chemical analyses, the presence of sulfur in tailings caused the melting of the calcic phases of cement hydrates and supported the formation of inflatable phases, causing the deterioration of cemented paste backfill material (Benzaazoua et al. 1999). Calcium aluminate compounds are formed during the formation of calcium aluminate compounds, which makes the binder the only suitable option for ensuring the long-term stability of the paste backfill containing high sulfate. However, the strength gain is better during long-term curing time when cement is used in combination with class C fly ash (Hassani et al. 2001). Besides, there was an increase among samples used in 50% ratios of fly ash in the value of UCS compared to the ones in which PC was only used (Hassani et al. 2007). In paste backfill mixes with high binder content, using fly ash containing low lime is not appropriate (Ramlochan et al. 2004).

Type C fly ash was commonly used to replace cement in CPB mixes (Aldhafeeri and Fall 2017; Ghorbanpour and Yu 2020; Behera et al. 2020; Cui et al. 2020; Ercikdi et al. 2009a; Gorakhki and Bareither 2017, 2018; Jiang et al. 2019; Sun et al. 2018, 2019; Yilmaz 2018;

Yılmaz et al. 2020; Zhang et al. 2017; Zhao et al. 2019; Zhao et al. 2019; Zhou et al. 2019). Ercikdi et al. (Ercikdi et al. 2009a) used 10–30% ratios of type C fly ash as a substitute for cement. However, there was an increase in CPB samples until the 56th day, followed by a decrease. The strength continued to increase until day 90 in the study by Ghorbanpour and Yu (Ghorbanpour and Yu 2020). However, none of the FA-contributed samples exceeded the UCS strength of the reference sample (FA-free sample). Besides, there were decreases in UCS strength with the increases in the proportion of the FA substitution (Cihangir et al. 2015; Değirmenci and Okucu 2007; Ramlochan et al. 2004). In the Klein and Simon (Klein and Simon 2006) study, they noted that when the FA was added, the stiffness decreased in its samples in addition to the UCS decline. In their study, Zhao et al. (Zhao et al. 2019) say that fly ash substitute increases strength and hardness. Zhou et al. (Zhou et al. 2019) stated that the effect of increasing the strength of fly ash occurred during the 28-day curing period. Behera et al. (Behera et al. 2020) found that, contrary to other studies, the use of fly ash in CBP has been found to reduce its strength, but it can be substituted for Portland cement (OPC) up to a ratio of 25%. Zhao et al. (Zhao et al. 2019) demonstrated that the tailings improved the bonding interface with the fly ash exchange in the binder by increasing the strength of the paste backfill material. Sun et al. (Sun et al. 2019) discovered that the strength and fluidity of the paste backfill mixes increased as the fly ash content increased.

Paste backfill mixtures using type F fly ash are also available in the literature (Cavusoglu et al. 2021a; Jiang et al. 2019, 2020b; Jiang et al. 2020; Ouattara et al. 2018; Singh et al. 2019; Sun et al. 2019; Yao and Sun 2012). It caused the increased strength in cemented rock filling mixtures using F-type fly ash (Jiang et al. 2019). Singh et al. (Singh et al. 2019) and Wang et al. (Wang et al. 2020) found that using fly ash instead of PC did not increase strength as much as PC. Jiang et al. (Jiang et al. 2020) observed that up to 30% fly ash replacement increased the strength and obtained high values from the strength of the reference sample (FA-free sample). In previous studies, it is seen that F-type fly ash is not used in metallic ore tailings, but it is preferred in paste backfill mixtures where coal tailings are usually used.

While previous studies have investigated the use of fly ash (FA) as a partial substitute for Portland cement (PC) in various cemented paste backfill (CPB) applications, several critical gaps remain. Notably: a) most research has focused on Type C FA, while studies on the mechanical and microstructural performance of CPB with Type F FA – particularly derived from hard coal – are limited, b) the interaction between high-sulfur-content tailings and FA in CPB mixtures, including the effects on long-term durability and secondary mineral formation, has not been extensively examined, c) existing studies often lack detailed analysis of cost-saving potential alongside environmental benefits associated with the substitution of PC by FA in CPB applications.

In this study, FA (10%, 20%, 30%, and 40% ratios by weight) from the Zonguldak-Çatalağzı thermal power plant was used instead of CPB, which was composed of mixing water and Portland cement (CEM I 42,5 R). This Portland cement is 3%, 5% 7%, 9%, and 11% ratios (by weight); thus, the effect of FA was investigated on the mechanical characteristics of CPB. In addition, for the first time in CPB mixes, fly ash from hard coal combustion was used. However, tailings that contain high pyrite (67.82%) in CPB mixtures are used.

1. Materials and methods

1.1. Fly ash

In this study, type F fly ash (FA) was substituted for cement. FA is supplied from ÇATES thermal power plant in Zonguldak-Çatalağzi. Fly ash used within the scope of the study is an ash formed as a result of the use of hard coal in the thermal power plant. For this reason, it has high silica and low CaO content. However, it is one of the ashes with the highest activity index value in mortar samples. These properties provide positive advantages in the use of this fly ash as a cement replacement compared to other ashes. The FA has not undergone crushing or grinding and is provided in a thin form. To minimize the weakening effects of

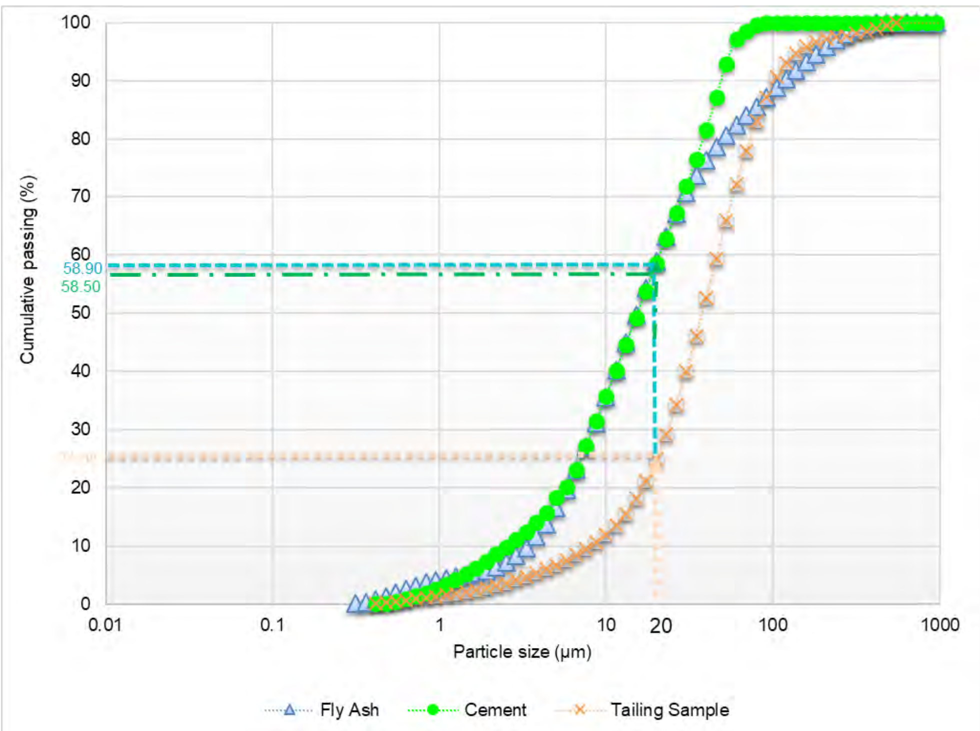

Fig. 1. Materials used particle size distributions

Rys. 1. Rozkład wielkości cząstek użytych materiałów paste backfill, it is preferable to substitute FA. For this purpose, the basic material properties of the FA to be used have been investigated as a priority. The Malvern (Mastersizer Hydro 2000 MU) is used to analyze the particle size distribution of fly ash (Figure 1).

According to the findings presented in Figure 1, the proportion of fly ash with a size value below 20 µm was 45.30%. The specific surface area is 2,220 cm^2/g. Table 1 presents comprehensive information on the mineralogical, physical, and chemical properties of fly ash. According to the XRD difficult pattern of fly ash; there were quartz and mullite as the main phases (Figure 2). Table 1 shows the chemical and physical properties of cement and copper plant tailings.

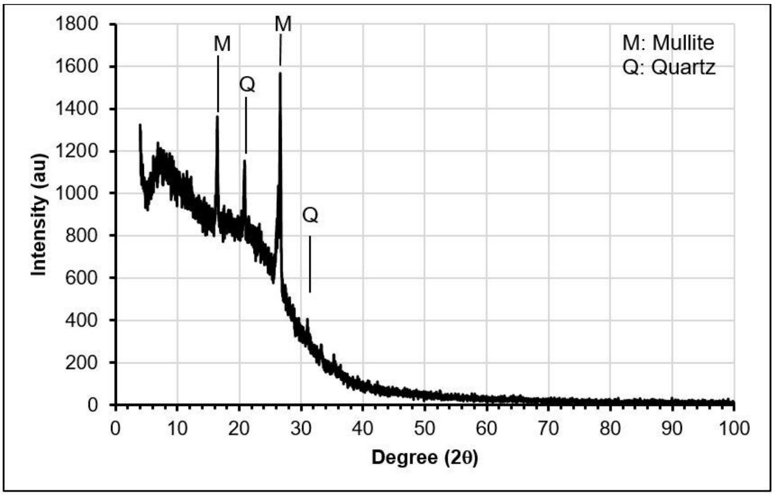

Fig. 2. XRD diffraction pattern of fly ash (Kesimal et al. 2005)

Rys. 2. Wzór dyfrakcyjny XRD popiołu lotnego

1.2. Tailings and binders

The size value of the dimensions of the tailings material, which is below 20 µm, is determined as 24.88% ratios (Figure 1). The mineralogical analysis reveals that the tailings contain a pyrite content of 67.82% ratios. The tailings include calcite (3.34%), chamosite (10.64%), siderite (4.59%), hematite quartz (3.30%), and magnetite (0.96%).

The biggest expense of paste backfill plants is in cement costs. To investigate the potential cost savings of CBP plants, a standard PC was selected to assess the impact of mineral supplements as substitutes for cement in CBP mixtures. Table 1 shows the results of the chemical analysis of the cement employed.

Table 1. The chemical and physical properties of materials

Tabela 1. Właściwości chemiczne i fizyczne materiałów

	Tailings (%)	Cement (%)	Fly ash (%)
Chemical properties			
SiO_2	12.26	19.11	54.08
Al_2O_3	4.08	4.71	26.08
Fe_2O_3	54.28	3.28	6.681
MgO	2.33	1.30	2.676
SO_3	–	3.47	0.735
CaO	1.76	64.09	2.002
Na_2O	0.03	0.26	0.791
K_2O	0.09	0.84	4.537
Free CaO	–	1.66	0.11
Loss of ignition	24.00	2.10	1.36
Physical properties			
Specific gravity (g/cm^3)	3.62	3.15	2.61
Specific surface (cm^2/g)	1,805	3,639	2,220
Mineralogical properties			
Fly ash	*Tailings of copper*		
Quartz – SiO_2	Calcite – $CaCO_3$		
Mullite – $Al_6Si_2O_{13}$	Chamosite – $(Fe^{2+}Mg, Fe^{3+})_5Al(Si_3Al)O_{10}(OH,O)_8$		
Hematite – α-Fe_2O_3	Gypsum – $CaSO_4 \cdot 2H_2O$		
Biotite – $(K(Mg, Fe)_3AlSi_3O_{10}(F, OH)_2)$,	Hematite – α-Fe_2O_3		
	Magnetite – Fe_3O_4		
	Pyrite – FeS_2		
	Quartz – SiO_2		
	Chalybite – $Fe^{2+}CO_3$		

Source: Eker 2019.

1.3. Procedure for the preparation of CPB and uniaxial compressive strength (UCS) tests

Copper tailings, cement, fly ash (FA), and tap water are homogeneously mixed to produce CPB samples. The reference specimens were prepared by mixing the tailings with cement and tap water in weight proportions of 3%, 5%, 7%, 9%, and 11%. Cemented paste backfill

Table 2. Test conditions used to prepare samples of CPB

Tabela 2. Warunki testowe zastosowane do przygotowania próbek CPB

Mixtures	Materials used (wt.%)		Total solids content (wt.%)	w/c	Slump (cm)	Dosage of cement (wt.%)
	fly ash (FA)	cement (C)				
Reference	0	100	81	0.59	18	3
10 wt.% FA	10	90	81	0.46		
20 wt.% FA	20	80	81			
30 wt.% FA	30	70	81			
40 wt.% FA	40	60	81			
Reference	0	100	81	0.54	18	5
10 wt.% FA	10	90	81	0.30		
20 wt.% FA	20	80	81			
30 wt.% FA	30	70	81			
40 wt.% FA	40	60	81			
Reference	0	100	81	0.47	18	7
10 wt.% FA	10	90	81	0.28		
20 wt.% FA	20	80	81			
30 wt.% FA	30	70	81			
40 wt.% FA	40	60	81			
Reference	0	100	81	0.43	18	9
10 wt.% FA	10	90	81	0.29		
20 wt.% FA	20	80	81			
30 wt.% FA	30	70	81			
40 wt.% FA	40	60	81			
Reference	0	100	81	0.57	18	11
10 wt.% FA	10	90	81	0.25		
20 wt.% FA	20	80	81			
30 wt.% FA	30	70	81			
40 wt.% FA	40	60	81			

samples containing 10%, 20%, 30%, and 40% FA substitutes were then prepared for each of the cement quantities used (3–5–7–9–11% ratios). The paste was mixed in a mixer for 10 minutes to produce a uniform paste backfill. A slump value of 18 cm and a solids content of 81% were used to produce the paste backfill mixes. In CPB applications, the value was chosen because the slump was between 15.24 and 25.4 cm, which was the preferred range (Table 2).

Cylindrical sample molds (5 cm in diameter and 10 cm in height) were filled with backfill mixes, which had four holes in the bottom for drainage, and kept for the specified curing times (3, 7, 14, 28, 56, and 90 days) in the curing cabinet, which was set to a temperature of at least 80% of humidity and 25°C. The UCS of paste backfill was determined using the ASTM C 39 (ASTM C39/C39M-18, 2018) standard. Cylindrical samples with dimensions of 50×100 mm were prepared for testing. 3–5–7–9–11% cement ratios reached the specified curing times and the samples, which was obtained by using it instead of cement which is at 10% 20%, 30%, and 40% ratios by weight from fly ash, were broken in an automatically controlled press with a constant loading speed of 1 mm/min with a loading capacity of 50 kN and strength values were calculated separately for three, seven, fourteen, twenty-eight, fifty-six and ninety days daily cures. The cylindrical paste backfill samples have a height/diameter ratio of at least 2. Prior to the experiment, the lower and upper surfaces of the samples were corrected. For each curing time, 3 samples were tested in the experiment and the average of the values was obtained. To determine the quality of paste filling mixtures, 0.15 MPa as the liquefaction risk limit value (Been et al. 2002; Le Roux et al. 2004), 0.7 MPa ≥ for sublevel stopping (Brackebusch 1995; Landriault 1995), and ≥ 4 MPa values in Grice's (Grice 1998) study for specified roof support were identified.

1.4. SEM analysis results

SEM-EDS analyses were conducted using a Scanning Electron Microscope (SEM-EDS) (Jeol JSM-5600 model) device at Istanbul University. For this purpose, the mixtures that provided the highest strength during the 28- and 90-day curing periods were selected from CBP mixtures, and specimens broken in the UCS test were taken. After covering the samples with a gold film, they were photographed at various magnification values (50–9000), using an electron microscope.

1.5. XRD analysis results

The Acme laboratory X-ray diffractometer was used for X-ray diffraction (XRD) analysis. Samples were taken from the fragments and carefully prepared for mineralogical analysis at the end of the 90-day curing period. XRD scans took place between 3 and 80° 2°. XRD analyses were carried out to learn about the formation of secondary minerals (ettringite and gypsum, etc.) that cause the paste backfill to lose its durability.

2. Results

2.1. Analysis of UCS test results

In the context of this study, cement (CEM I 42,5 R) and cement substitutions for fly ash, which were at 3%, 5%, 7%, 9%, and 11% ratios (by weight), were used together. Fixed consistency (18 cm/7.09 inch) putty filling samples were prepared using ratios of 10%, 20%, 30%, and 40% of cement-FA mixture. It is important to consider this option as previous studies in the literature suggest that additional proportions of fly ash can be used to substitutions cement (Aldhafeeri and Fall 2017; Ghorbanpour and Yu 2020; Behera et al. 2020; Cavusoglu et al. 2021a; Cui et al. 2020; Ercikdi et al. 2009a; Gorakhki and Bareither 2017, 2018; Jiang et al. 2019, 2020b; Jiang et al. 2020; Klein and Simon 2006; Ouattara et al. 2018; Singh et al. 2019; Sun et al. 2018, 2019; Yao and Sun 2012; Yilmaz 2018; Yılmaz et al. 2020; Zhao et al. 2019; Zhao et al. 2019; Zhou et al. 2019). The samples were cured in a chamber (25°C and at least 80% humidity) to simulate the underground conditions where they were obtained. The results of the UCS tests carried out at the end of the curing periods are shown in Figures 3–7.

Figure 3 shows that adding fly ash to the paste backfill mixture, which contains 3% cement at different rates, did not increase the compressive strength. In addition, throughout the curing period, it shall not exceed the compressive strength values of the reference specimen. There is evidence that the reference sample is in excess of the liquefaction risk limit (0.15 MPa) in 28 days in all mixture rates. In addition, none of the mix ratios exceeded the desired limit (0.7 MPa) strength during the 28 days of curing (Figure 3).

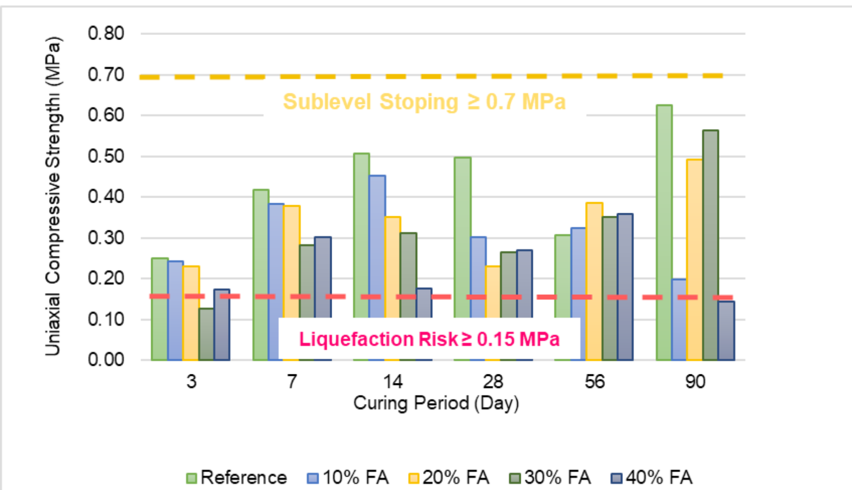

Fig. 3. Effect of fly ash substitutes for 3% CBP samples on UCS

Rys. 3. Wpływ substytutów popiołu lotnego dla próbek 3% CBP na UCS

The addition of various proportions of FA to the CBP mix containing 5% cement increased the UCS, as shown in Figure 4. The compressive strength of the reference sample was exceeded in only 10% of the mixes. It is seen that the reference sample exceeded the liquefaction risk limit (0.15 MPa) in 28 days in all mixture rates. In addition, the desired strength limit (≥ 0.7 MPa) is exceeded by only 10% mixing during the 28-day curing period.

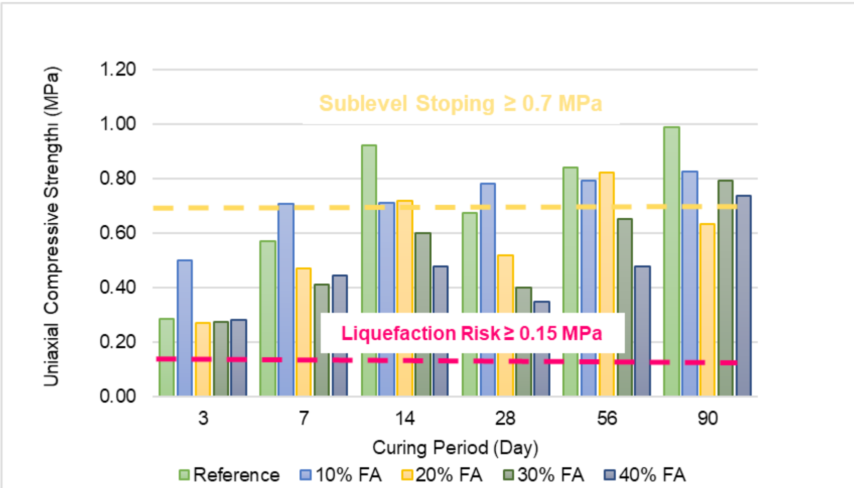

Fig. 4. Effect of fly ash substitutes for 5% CBP samples on UCS

Rys. 4. Wpływ substytutów popiołu lotnego dla próbek 5% CBP na UCS

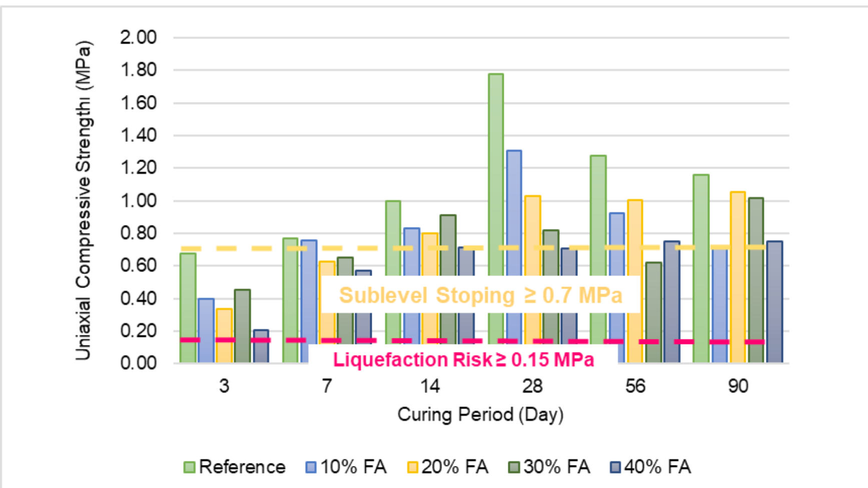

Fig. 5. Effect of fly ash substitutes for 7% CBP samples on UCS

Rys. 5. Wpływ substytutów popiołu lotnego dla próbek 7% CBP na UCS

It can be seen that other mix ratios exceeded this limit during the 56-day and 90-day curing periods. In addition, the mixing rate does not exceed the limit of ≥ 4 MPa.

As shown in Figure 5, the addition of various amounts of FA to the 7% CBP mix resulted in an increase in compressive strength. None of the mixing ratios exceeds the compressive strength value of the reference sample. It is seen that the liquefaction risk limit of ≥0.15 MPa

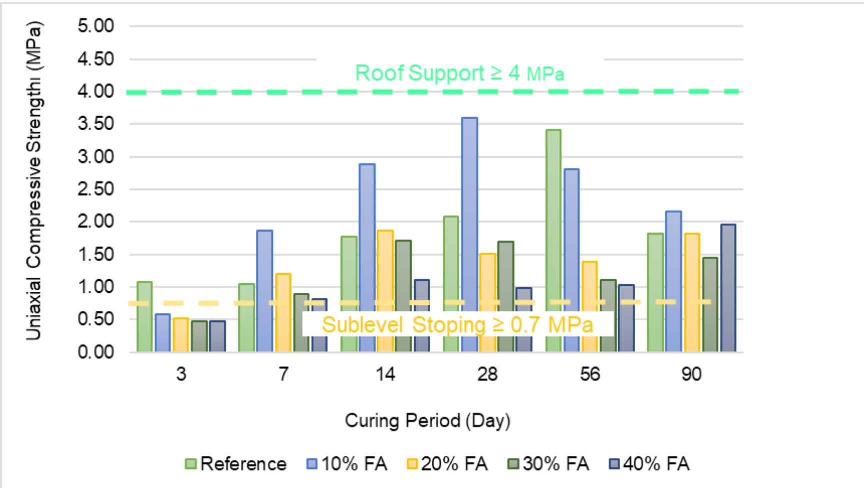

Fig. 6. Effect of fly ash substitutes for 9% CBP samples on UCS

Rys. 6. Wpływ substytutów popiołu lotnego dla próbek 9% CBP na UCS

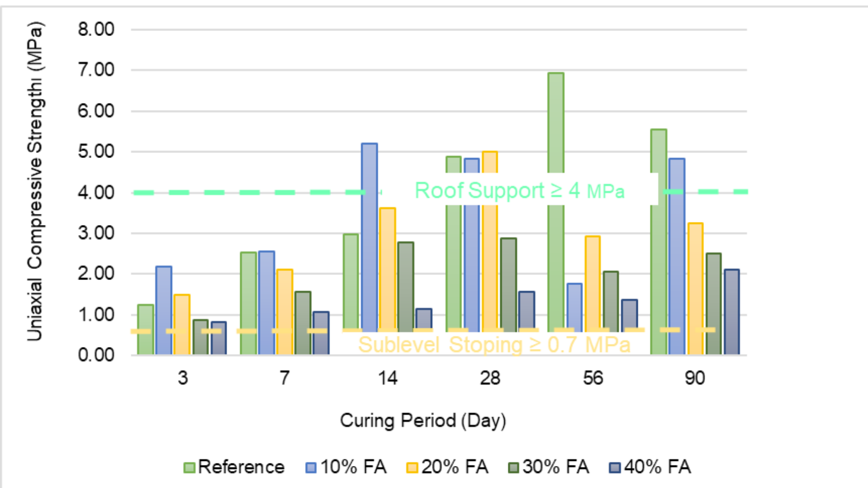

Fig. 7. Effect of fly ash substitutes for 11% CBP samples on UCS

Rys. 7. Wpływ substytutów popiołu lotnego dla próbek 11% CBP na UCS

in 28 days is exceeded in all mixture rates. In addition, during the 28-day curing period, the desired strength limit of ≥ 0.7 MPa was exceeded at all mixing rates. In addition, the mixing rate does not exceed the limit of ≥ 4 MPa.

As shown in Figure 6, the addition of different proportions of FA to a CBP mix containing 9% cement increased the UCS. The mixture ratio of the reference sample exceeding the compressive strength value in 28 days is a 10% ratio. It is seen that the liquefaction risk limit of ≥ 0.15 MPa in 28 days is exceeded in all mixture rates. In addition, the desired ≥ 0.7 MPa strength limit value was exceeded in all mixture rates within the 28-day period of curing. Furthermore, the mix ratio should not exceed the required roof support limit of 4 MPa. With a mixing ratio of 10%, the highest strength value of 3.60 MPa was obtained.

As seen in Figure 6, the addition of different proportions of FA to the CBP mixture containing 11% ratios of cement increased UCS. The mixture ratio of the reference specimen exceeding the compressive strength value in 28 days is 20% ratios. It can be seen that the limit value for the risk of liquefaction of ≥ 0.15 MPa in 28 days is exceeded for all the mixing rates. In addition, the desired strength limit value (0.7 MPa) was exceeded in all mixture rates during the curing period (28-day).

It can also be seen that the desired limit for roof support exceeds the limit of ≥ 4 MPa by 10% and 20% of the mix ratio. 5.01 MPa at 20% of CBP mix ratio was the highest strength value (Figure 7).

FA (10, 20, 30, and 40%) substituted into cement (3, 5, 7, 9, 11%) enhanced the UCS of mixes at all cement proportions. All mixes provide limited liquefaction risk. All mixtures with 7, 9, 11% cement ratios and 5% ratios of cement – 10% ratios of the FA substituted mixture provide only 11% cemented – 10% and 20% ratios of substituted paste backfill mixtures for roof support. In general, the FA substitution was more effective in early strength (up to day 28), and in general, the strength values did not change significantly thereafter.

2.2. SEM analysis results

After the compressive strength value during the 28- and 90-day curing periods, SEM pictures were taken with the help of the SEM-EDS device of reference (R), and FA-substituted samples. SEM images are provided in Figures 8–9.

Figure 8 has a denser structure with the help of gels formed as a result of low gaps and FA additive cement hydration. It is seen that FA is effective in creating a more uniform structure than its thin size and large tailing. It is understood that the reactions of the pozzolanic activity of fly ash began more than 28 days ago. This is due to the formation of hydrated products from pozzolan reactions on the surface of spherical ash particles (Ouattara et al. 2018; Yao and Sun 2012; Zhang et al. 2017). In addition, ettringite formation is also observed, which has a long-term negative effect on strength. There is also the presence of $Ca(OH)_2$ when SEM pictures of FA replaced specimens are tested for 28 days. It consists of minerals with elements Mg, O, Si, Al, S, Fe, and Ca, according to SEM-EDS analysis.

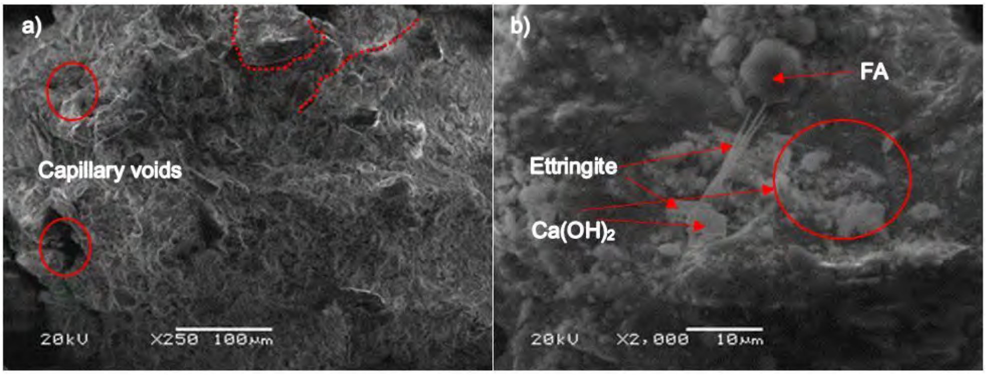

Fig. 8. SEM pictures of the 28 days fly ash replacement specimen

Rys. 8. Zdjęcia SEM próbki po 28 dniach zastępowania popiołu lotnego

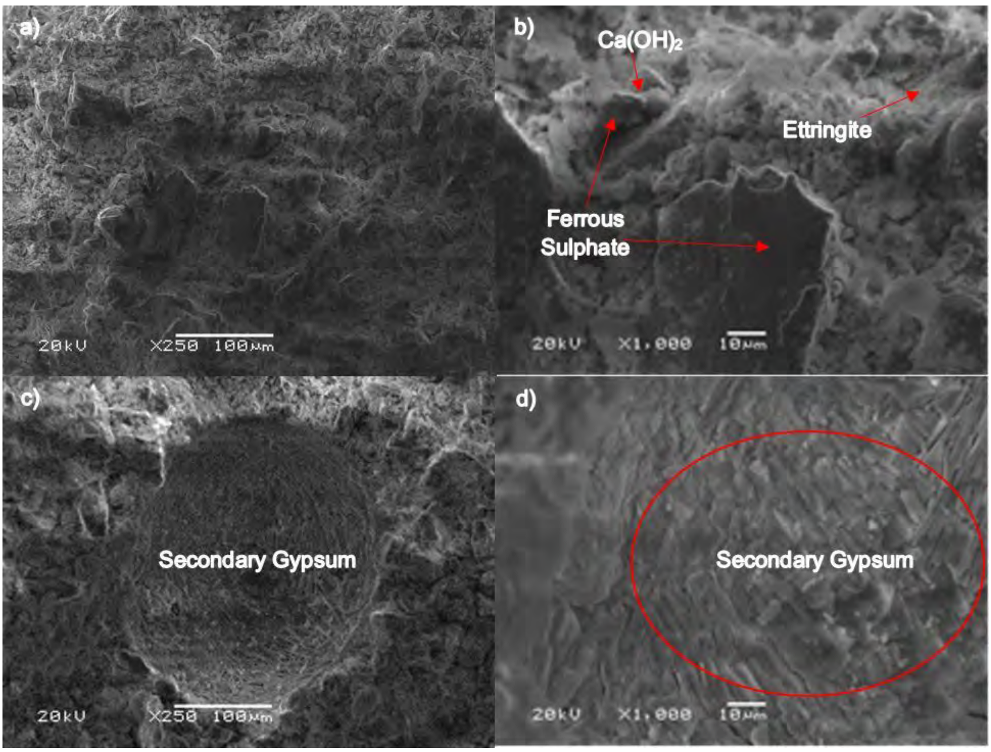

Fig. 9. SEM pictures of the 90 days fly ash replacement specimen

Rys. 9. Zdjęcia SEM próbki po 90 dniach zastępowania popiołu lotnego

As seen in Figure 9, sulfate reacts with $Ca(OH)_2$ (portlandite), one of the hydration products. Then it forms $CaSO_4 \cdot 2H_2O$ (secondary gypsum) and $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$ (ettringite) minerals (Cihangir and Akyol 2018). It has been observed that iron sulfate, ettringite, and secondary gypsum formations have a negative impact on strength. Secondary gypsum and ettringite are seen to fill the gaps in the 28-day images. As a result, the UCS decreases by 5.01 MPa in 28 days and by 3.25 MPa in 90 days. Using SEM-EDS analysis, it can be inferred that the 90-day FA-substituted sample contains minerals that contain Fe, S, Al, Ca, Si, O, and Mg elements when examining SEM images.

2.3. XRD analysis results

Samples were taken from among the samples of mixtures with the best strength (with 11% ratios of cement, 11% ratios of cement, and 20% ratios of the fly ash substitution) among the CBP specimens that broke at the conclusion of the 90-day curing time, was brought to a certain delicacy at first and then analyzed using the help of XRD test device in Acme laboratory. XRD scans took place between 3 and 80° 2°. Bruker's Search-Match software and the International Diffraction Centre Database were used to analyze the XRD scans. XRD analyses are presented in Figure 10. Quantitative phase analysis using the program (Rietveld Topas 4.2) was also carried out (Table 3).

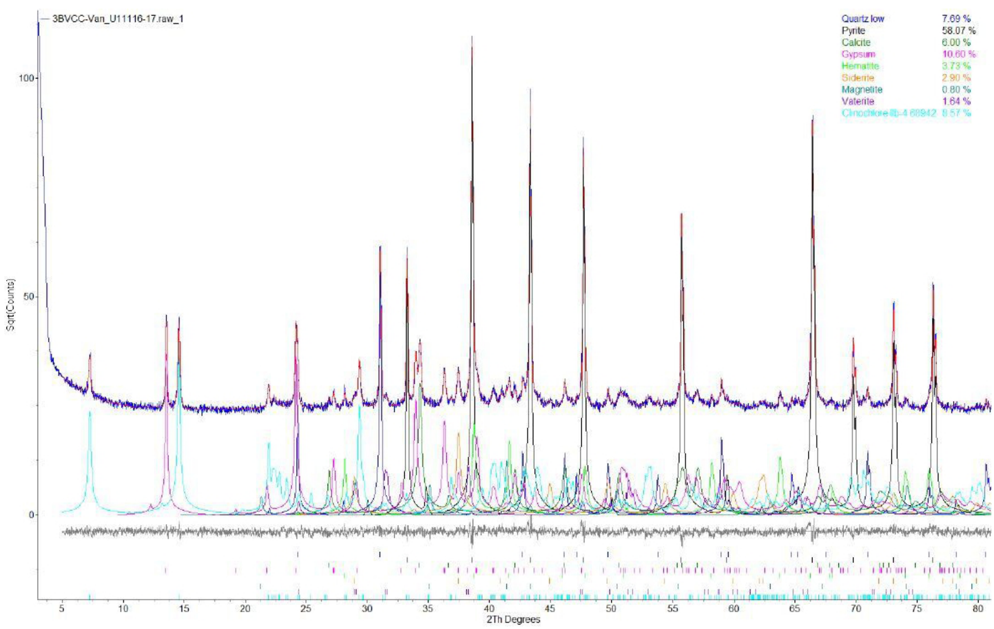

Fig. 10. Mineralogical composition of the fly ash sample

Rys. 10. Skład mineralogiczny próbki popiołu lotnego

Table 3. Quantitative phase analysis results (wt%)

Tabela 3. Wyniki analizy faz ilościowych (% wag.)

Mineral	Formula	Sample of reference (% by weight)	Sample of fly ash-containing (% by weight)
Pyrite	FeS_2	56.1	58.1
Gypsum	$CaSO_4 \cdot 2H_2O$	11.3	10.6
Clinochlore	$(Mg, Fe^{2+})_5Al(Si_3Al)O_{10}(OH)_8$	9.4	8.6
Quartz	SiO_2	8.1	7.7
Calcite	$CaCO_3$	7.5	6.0
Hematite	α-Fe_2O_3	3.3	2.9
Siderite	$Fe^{2+}CO_3$	2.5	3.7
Magnetite	Fe_3O_4	1.1	0.8
Vaterite	$CaCO_3$	0.7	1.6

Quantitative analysis of all R and FA samples submitted for XRD analysis at the end of the 90-day period indicates the presence of 56.1% and 58.1% by weight pyrite, respectively. (Table 3). In addition, reference and FA samples contained 11.3% and 10.6% gypsum by weight, respectively. Previous studies have suggested that secondary gypsum minerals are responsible for the loss of strength observed in these samples (Belem and Benzaazoua 2008; Ercikdi et al. 2009a; Ercikdi et al. 2009c; Fall and Samb 2008; Li and Fall 2016; Yılmaz et al. 2020).

It is known that OH^- ions appeared in CBP specimens, and these ions increased the 12–13 pH value of the medium. Primary ettringite, portlandite, iron sulfate, and C-S-H gels are formed with the onset of the hydration process. C-S-H gels provide strength for CBP specimens. $SO4^{2-}$ (Sulfate) is formed as a result of sulfurous minerals such as pyrite oxidation with oxygen and water, and reacts to portlandite, forming a secondary gypsum. The expansion of these secondary gypsums within specimens causes cracks and disrupts the integrity of CBP. Moreover, the oxidation-induced acidity of H^+ decreases the binding abilities of C-S-H gels by causing CH to dissolve in the environment. (Ercikdi et al. 2009a; Ercikdi et al. 2009c; Fall and Benzaazoua 2005; Hassani et al. 2001; Tariq and Nehdi 2007).

When we examine the mineral composition of R and FA samples, it consists of Pyrite and Gypsum minerals, as well as Clinoxide, Quartz, carbonate minerals Calcite and Vaterite, iron-containing Hematite, Siderite, and Magnetite (Table 3).

3. Discussion

It is a known fact that the strength of the material increases as the amount of binder used in CBP increases. More hydration products form with the increase in cement ratio, and cohesion increases. This reduces porosity and increases the UCS (Cihangir et al. 2012; Ercikdi et al. 2009a; Ercikdi et al. 2014; Fall et al. 2010; Fall and Benzaazoua 2005; Fall and Samb 2008; Ghirian and Fall 2016; Kesimal et al. 2005; Klein and Simon 2006; Pokharel and Fall 2011). However, in terms of the cost of the paste backfill plant, this situation has a disadvantage. The UCS requirement for CBP liquefaction risk is at least 0.15 MPa (Been et al. 2002; Le Roux et al. 2004). In addition, the material must have a minimum strength of 0.7 MPa at the end of the 28-day curing period and a minimum of 4 MPa for use in roof supports to ensure stability (Brackebusch 1995; Grice 1998; Landriault 1995). Therefore, it is expected to provide these strength limit values for the use of mineral additives to be substituted instead of cement in paste backfill mixtures.

Liquefaction limit (≥ 0.15 MPa) was provided in the reference of CBP mixtures prepared at cement ratios (3%, 5%, 7%, 9%, 11%). At the end of the 28-day curing period, it is seen that they provide the desired strength (≥ 0.7 MPa) in cement ratios (7, 9, and 11%). Roof strength limit (≥ 4 MPa) was only 11% cement. When the cement proportion is increased from 7% to 11% ratios, it can be seen that UCS increases by 1.9–5.5 times during all curing periods of CBP samples. There was a marked improvement in the long-term efficiency of CBP specimens as the cement content increased.

Fly ash (FA), substituted as 10%, 20%, 30%, and 40% of cement quantities (3%, 5%, 7%, 9%, 11%), increased UCS in mixes of all cement proportions. Moreover, all mixtures provide the liquefaction risk limit for CBP. Within the 28-day curing period, the desired strength limit value (≥ 0.7 MPa) is provided by all mixtures with 7, 9, 11% cement ratios and 5% ratios of cement – 10% ratios of FA substituted mixture. In addition, the limit value required for using as roof support (≥ 4 MPa) was obtained only in 11% cemented – 10% and 20% ratios of mixtures of the FA substitution.

During the 90-day curing period, the strength limit value (≥ 0.7 MPa) is provided by all mixtures with 7.9, and 11% cement ratios, and 5% of cement – 10% ratios of the FA substituted mixture. Besides, the limit value required for using as roof support (≥ 4 MPa) was obtained only in 11% cemented – 10% and 20% ratios of mixtures of the FA substitution. In general, it was observed that there were increases in strength of the FA substitution (up to the 28th day) and afterward there were either very few decreases in strength values or not much change (Alhomair et al. 2017; Ghorbanpour and Yu 2020; Benzaazoua et al. 1999; Hassani et al. 2001, 2007; Ramlochan et al. 2004; Tuylu 2022b). In this study; although it was stated that paste backfill containing type C-FA gained strength in the long term, it was also found that mixtures containing type F-FA used in this study also gained strength up to 90 days. Furthermore, the decreases occurred due to the increase of FA content added to type C-FA paste backfill mixtures (Behera et al. 2020; Cui et al. 2020; Ercikdi et al. 2009b; Ji et al. 2022; Jiang et al. 2020; Qi et al. 2015; Zhang et al. 2017).

The results from CPB samples containing type F-FA, which was used in this study, showed a similar trend to paste backfill mixtures using type C-FA.

Although Singh et al. (Singh et al. 2019) and Wang et al. (Wang et al. 2020); stated that the use of type F fly ash instead of PC did not increase the strength as much as the PC, this study shows that it increases PC strength. In addition, the studies of Jiang et al. (Jiang et al. 2019) and Jiang et al. (Jiang et al. 2020) support this study, in which the use of type F-FA in cemented rock filling and paste backfill mixtures is obtained with greater strength than FA-free reference samples.

According to the SEM analysis, there are excessive gaps in the reference specimens, and the presence of pyrite minerals in the analysis carried out after 28 days. Gaps in reference specimens were reduced by adding FA to provide a replacement for paste backfill mixes. During these curing periods, ettringite is also formed, which has a negative effect on long-term strength and is often seen in iron sulfate. FA samples have a denser, homogeneous structure. It is thought that the tailings material is large and the gaps between them have a smaller grain size than these substances. And FA grains fill this gap. From the results of the 90-day analysis, it can be seen that the loss of strength of the CBP specimens and the causes of capillary cracking are due to the presence of secondary gypsum, ettringite, and iron sulphate. Secondary gypsum mineral causes resistance loss in paste backfill applications (Belem and Benzaazoua 2008; Ercikdi et al. 2009a; Ercikdi et al. 2009c; Fall and Benzaazoua 2005; Fall and Samb 2008; Ghirian and Fall 2014; Li and Fall 2016). Pyrite forms acid and sulfate by oxidation in the presence of water and oxygen. Acid attack occurs because of the oxidation of sulphur tailings, and the structure of the resulting C-S-H bonds is broken. The acid released by oxidising causes C-S-H and $Ca(OH)_2$, portlandite (hydration products) to lose their binding characteristics ($pH \leq 9$), reducing filler strength and durability (Belem and Benzaazoua 2008; Benzaazoua et al. 1999; Cihangir et al. 2011, 2012; Ercikdi et al. 2009a, 2009c; Hassani et al. 2001; Tariq and Nehdi 2007).

Due to cement hydration and the pozzolanic effect of substituted materials, the pores fill up, and their diameter decreases (Fall and Samb 2008). This is because of the increasing development of small pores as a result of binding hydration (Ghirian and Fall 2014). In addition, there is evidence that the tailings from the CBP mixes have had an impact on the environment, are large, and will have a better mix of strength and durability than the high fines (≤ 20 μ) replacement materials (Adrianto and Pfister 2022; Aldhafeeri and Fall 2017; Naylor et al 1997). FA substituted samples, which were obtained with high strength, are thought to best fill the gaps between grains in the paste mixture due to their high content of thin grains below 20 μm (58.9%).

The study examined beneficiation tailings with high sulphur content. This characteristic overlaps with many other copper mine tailings around the world. For example, tailings with similar chemical properties can achieve similar positive results from the use of fly ash (FA) instead of Portland cement (PC) (Benzaazoua et al. 1999; Kesimal et al. 2005). The strong mechanical strength values reported in the study can also be used for the stability of other copper mine tailings that have oxidation problems due to high sulfur content

(Hassani et al. 2001). Furthermore, the study demonstrates the economic and environmental benefits of using FA in the management of mine tailings. Similarly, the potential contribution of FA in tailings from the processing of other metallic ores, such as gold and zinc ore, can be investigated (Fall et al. 2010; Ramlochan et al. 2004). In particular, blends using FA with low lime content (class F) can be optimized for tailings with different mineralogical properties (Cihangir et al. 2012; Jiang et al. 2020a). However, the findings of the study can be related to CPB performance in different climatic regions. For example, chemical and mechanical strengths can be compared between tropical regions with high humidity and arid regions (Fall and Samb 2008; Ghirian and Fall 2014). Furthermore, the regional availability of fly ash and cement can also be evaluated in terms of environmental and economic impacts (Eker and Bascetin 2022a).

The study highlights the potential of FA utilization to reduce carbon emissions. These results can be used to help the mining industry adapt to global initiatives supporting environmental sustainability (Gorakhki and Bareither 2018; Sun et al. 2019). By referring to experiences from different mining sectors and international literature, applications of FA utilization in other countries can be evaluated (Hassani et al. 2007; Zhao et al. 2019; Zhao et al. 2019).

Conclusion

CPB mixtures were created by mixing ore tailings from a copper mine near Kastamonu in Turkey with water and Portland cement at 3%, 5%, 7%, 9%, and 11% ratios by weight. Besides, the FA, which is obtained by using the hard coal in a thermal power plant instead of PC, has been added to CPB mixtures in 10%, 20%, 30%, and 40% ratios by weight. Then, the mechanical effects of the FA were revealed according to the results of UCS, SEM, and XRD analyses at the end of the 3-, 7-, 14-, 28-, 56-, and 90-day curing periods of CPB samples.

In the substituted FA cement mixtures, it is understood that the UCS increases positively. In addition, the strength limit values, which were identified in the literature review, also provided the paste backfill samples containing the FA. Moreover, it was demonstrated by this study that hard coal fly ash (F type) can also be used to help reduce the cost of the paste backfill system. Thus, it was understood that the substituted FA cement mixtures may also be preferred to reduce the negative effects of cement production on the environment. Also, thermal power plants using hard coal can reduce storage costs by giving the FA tailings to paste backfill plants. Finally, it is seen that this study provides significant benefits to the mining sector in terms of business, environment, and cost.

The continuation of this study with the proposed extensions can contribute to:

- developing field-specific guidelines for optimizing the use of industrial by-products in CPB,

- establishing sustainability benchmarks for the mining sector by integrating waste reuse strategies into backfill applications,
- advancing the understanding of interactions between binders and diverse tailings types, supporting more efficient and environmentally friendly CPB designs.

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THE EFFECT OF HARD COAL FLY ASH SUBSTITUTION FOR CEMENT ON PROPERTIES OF HIGH SULPHIDE TAILINGS CEMENTED PASTE BACKFILL

Keywords

copper tailings, high sulphur content, hard coal fly ash, cement, cemented paste backfill (CPB)

Abstract

Cemented paste backfill (CPB) is a method used to fill the gaps in the underground production method. The main component of CPB is cement, which costs a high price. Therefore, some studies have continued in the search for materials to replace cement. In this context, the mechanical behavior of the material obtained following the substitution of ore preparation plant tailings from Kastamonu-Küre copper mine instead of cement using thermal power plant fly ash (FA) in certain proportions by weight was investigated. The results indicate that substituting FA in all cement proportions increases the compressive strength of the CBP mixtures. Up to 20% of the amount and cost of cement

has been saved by using FA instead of portland cement (PC). Thus, there is an opportunity for a reduction in the amount of CO_2 emissions, a greenhouse gas, from the cement production process. Besides, an economic income was provided to the enterprise by reusing coal thermal power plant waste. As a result, significant benefits have been provided to the mining sector in terms of operation, environment, and cost.

WPŁYW ZASTĄPIENIA CEMENTU POPIOŁEM LOTNYM Z WĘGLA KAMIENNEGO NA WŁAŚCIWOŚCI CEMENTOWANEJ PASTY WYPEŁNIAJĄCEJ ODPADY WYSOKOSIARKOWE

Słowa kluczowe

odpady miedziowe, wysoka zawartość siarki, popiół lotny z węgla kamiennego, cement, cementowane wypełnienie pastowe (CPB)

Streszczenie

Wypełnianie cementową pastą (CPB) to metoda stosowana do wypełniania szczelin w podziemnej metodzie wydobycia. Głównym składnikiem CPB jest cement, który charakteryzuje się wysoką ceną. W związku z tym kontynuowano badania nad poszukiwaniem materiałów, które mogłyby go zastąpić. W tym kontekście zbadano zachowanie mechaniczne materiału uzyskanego w wyniku zastąpienia odpadów z zakładu przeróbki rudy w kopalni miedzi Kastamonu-Küre zamiast cementu popiołem lotnym (FA) z elektrowni cieplnej w określonych proporcjach wagowych. Wyniki wskazują, że zastąpienie FA we wszystkich proporcjach cementu zwiększa wytrzymałość na ściskanie mieszanek CBP. Dzięki zastosowaniu FA zamiast cementu portlandzkiego (PC) zaoszczędzono do 20% ilości i kosztów cementu. Stwarza to możliwość zmniejszenia emisji CO_2, gazu cieplarnianego, powstającego w procesie produkcji cementu. Ponadto ponowne wykorzystanie odpadów z elektrowni węglowej przyniosło przedsiębiorstwu korzyści ekonomiczne. W rezultacie sektor górniczy odniósł znaczące korzyści pod względem działalności, środowiska i kosztów.