Influence of Air Entraining Agent on Strength and Microstructure Properties of Cemented Paste Backfill

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This work was supported in part by the China’s National Key Research and Development Plan under Grant 2017YFC0602901, and in part by the National Natural Science Foundation of China under Grant 41672298.

ABSTRACT

The conveying distance during the backfill operation has become extensively large with the advancement of mining work. It requires the immediate attention of academic and industrial communities. Adding air entraining agent (AEA) can improve the rheological properties of the filling slurry and facilitate the filling slurry transfer to the long-distance underground stope. The AEA can generate a large number of tiny bubbles, which acted as floating balls and reduced the friction between the particles. In addition, AEA acted as a dispersing and wetting agent. Herein, we have obtained filling material from the Sanning mine (Hubei, China) and utilized sodium abietate (SA) and triterpenoid saponins (SJ), as air entraining agents, to improve the fluidity of filling slurry. The influence of AEA content on mechanical properties, structure and morphology has been analyzed by uniaxial compressive strength (UCS) testing, nuclear magnetic resonance (NMR) and scanning electron microscopy (SEM). The results reveal that the addition of AEA increased the fluidity of filling slurry. Moreover, UCS of the AEA-free cemented paste backfill (CPB) sample, cured for 28 days, was found to be 2.23 MPa. However, the addition of 0.2% AEA (SA and SJ) increased the UCS to 2.31 MPa and 2.35 MPa, respectively. However, when SA and SJ amount was increased to 0.6%, UCS decreased to 2.19 MPa and 2.13 MPa, respectively. Hence, UCS of the CPB initially increased with the addition of AEA, followed by a gradual decrease, which can be ascribed to the pore structure inside CPB. One should note that the presence of AEA increased the number of small pores inside the CPB. The SEM results shows that pore structure of CPB can be optimized by adding an optimal proper amount of AEA.

INDEX TERMS

Air entraining agent, nuclear magnetic resonance, cemented paste backfill, uniaxial compressive strength; scanning electron microscope, rheological behavior.

I. INTRODUCTION

Air entraining agent (AEA) is an admixture, which affects the fluidity and strength of materials. A large number of AEA-induced tiny, uniform and independent bubbles play the role of ball bearings, reduce the friction between aggregate particles and improve the fluidity of filling slurry [1]–[3]. It has been reported that the bubbles form a large number of tiny pores during the consolidation process of the filler, which alters the pore structure and mechanical properties of the concrete [4], [5]. However, the influence of AEA on the uniaxial compressive strength (UCS) of the material has not been explored yet. Moreover, AEA renders different influences on different materials. For instance, Zhu et al. have demonstrated that UCS of concrete is reduced with increasing amount of AEAs [6]. Şahin et al. have studied the effect of saponin on concrete performance and demonstrated that the moderate amount of AEA results in optimal UCS of concrete [7]–[9].

In general, AEA mainly affects the pore structure inside the cemented paste backfill (CPB). In recent years, various studies have been carried out to investigate the relationship between the pore characteristics and macroscopic properties of CPB. For instance, Li et al. have utilized mercury intrusion porosimetry (MIP) and differential scanning calorimeter (DSC) to measure the pore volume and characteristic aperture [10]. However, the MIP technique renders certain limitations during the characterization of pore distribution of cement-based materials. For example, MIP cannot...
measure the pores with smaller diameters, which may result in the inaccurate internal pore size distribution of the studied material [11]. Sun et al. have applied X-ray CT to study the relationship between UCS and porosity of CPB [12]. However, the absence of any universal standard for three-dimensional (3D) reconstruction of CT images resulted in a qualitative assessment of the obtained results.

Furthermore, nuclear magnetic resonance (NMR) can determine the porosity, pore structure and fluid content of rocks by analyzing the relaxation time. For instance, Zhang et al. have analyzed the hydration degree of CPB by NMR and demonstrated that polynaphtalene sulfonate has a strong influence on short-duration hydration [13]. Fridjonsson et al. have employed NMR to investigate the internal pore structure of CPB [14].

To sum up, a number of studies have been carried out to investigate the relationship between the pore characteristics and the macroscopic properties. Each of these physical methods has its own advantages.

However, AEA influences the internal microstructure of CPB by creating pores and the effect of microstructural evolution on mechanical properties of CPB has not been investigated yet. Moreover, there are several limitations and shortcomings in testing pores through MIP.

Herein, based on the actual situation of the Sanning mine filling system, the application scheme of different AEAs in CPB has been studied to meet the requirements of long-distance transportation. The mechanical properties and microstructure of filling materials, with different AEAs, have been explored by NMR and scanning electron microscopy (SEM). Finally, the mechanistic insights into the influence of AEA on mechanical properties and microstructure of the CPB have been presented.

II. MATERIALS AND METHODS
A. MATERIALS
1) AGGREGATE
Herein, gangue of phosphorus mine, tailings and fly ash were obtained from Sanning mine in Hubei Province, China (Figure 1). The gangue and tailings were produced by concentrator of Sanning mine.

The apparent density, bulk density and surface water content of aggregates were analyzed according to the GB/T 14685-2011 standard, which describes the standard method to evaluate the performance of ordinary concrete mixtures. The corresponding results are presented in Table 1. According to the GB/T 14685-2011 standard, the particle size distribution (PSD) of the gangue was measured, as shown in Table 2. The gangue contains more coarse fractions and less fine fractions, where ~2.69% and ~96.37% particles were <0.315 mm and <4.75 mm, respectively. PSD of the tailings and fly ash was analyzed by using a MasterSizer 2000 (Malvern Instruments Inc., UK), as shown in Figure. 2.

The median diameter $d_{50}$ of the tailing and fly ash was found to be 12.033 $\mu$m and 14.503 $\mu$m, whereas the non-uniformity coefficient (Cu) was 3.896 and 10.504, respectively. Moreover, the coefficient of curvature (Cc) of tailing and fly ash was 0.798 and 1.417, respectively. According to the theory of soil mechanics, if sand particles satisfy certain conditions, i.e., Cu < 5 and 1 < Cc < 3, it is called well

![FIGURE 1. Physical appearance of as-obtained materials: (a) gangue, (b) tailings and (c) fly ash.](image1)

![TABLE 1. Physical properties of the as-obtained materials.](image2)

<table>
<thead>
<tr>
<th>Class</th>
<th>Apparent density (kg/m³)</th>
<th>Packing density (kg/m³)</th>
<th>Surface moisture content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gangue</td>
<td>2626</td>
<td>1464</td>
<td>0.120</td>
</tr>
<tr>
<td>Tailing</td>
<td>2653</td>
<td>923</td>
<td>0.974</td>
</tr>
<tr>
<td>Fly ash</td>
<td>1990</td>
<td>650</td>
<td>0.049</td>
</tr>
</tbody>
</table>

![FIGURE 2. Particle size distribution of as-obtained tailing and fly ash.](image3)
TABLE 2. Particle size distribution of as-obtained gangue.

<table>
<thead>
<tr>
<th>Particle size (mm)</th>
<th>Incremental Volume (%)</th>
<th>Cumulative Volume (%)</th>
<th>Particle size (mm)</th>
<th>Incremental Volume (%)</th>
<th>Cumulative Volume (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-0.075</td>
<td>0.93</td>
<td>0.93</td>
<td>-1.18--+-0.60</td>
<td>10.83</td>
<td>19.01</td>
</tr>
<tr>
<td>-0.15--+0.075</td>
<td>0.39</td>
<td>1.32</td>
<td>-2.36--+1.18</td>
<td>48.14</td>
<td>67.15</td>
</tr>
<tr>
<td>-0.30--+0.15</td>
<td>1.37</td>
<td>2.69</td>
<td>-4.75--+2.36</td>
<td>29.22</td>
<td>96.37</td>
</tr>
<tr>
<td>-0.60--+0.30</td>
<td>5.49</td>
<td>8.18</td>
<td>-9.5--+4.75</td>
<td>3.63</td>
<td>100</td>
</tr>
</tbody>
</table>

TABLE 3. Mineral composition of the aggregate (%).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hydroxylapatite</th>
<th>Quartz</th>
<th>Hematite</th>
<th>Albite</th>
<th>Plagioclase</th>
<th>Muscovite</th>
<th>Illite</th>
<th>Dolomite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gangue</td>
<td>10.15</td>
<td>6.91</td>
<td>12.75</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>69.65</td>
</tr>
<tr>
<td>Tailing</td>
<td>60.94</td>
<td>2.24</td>
<td>-</td>
<td>8.38</td>
<td>11.42</td>
<td>-</td>
<td>9.76</td>
<td>6.30</td>
</tr>
<tr>
<td>Fly Ash</td>
<td>-</td>
<td>61.55</td>
<td>1.46</td>
<td>15.99</td>
<td>-</td>
<td>20.99</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Cement</td>
<td>3CaO-SiO2</td>
<td>2CaO-SiO2</td>
<td>3CaO-Al2O3</td>
<td>4CaO-Al2O3-Fe2O3</td>
<td>52.8</td>
<td>20.7</td>
<td>11.5</td>
<td>8.8</td>
</tr>
</tbody>
</table>

graded sand [15]. The results indicate that the tailing is poor graded sand, whereas fly ash is a well graded sand. Therefore, the tailing cannot be used as a filler alone and these three materials should be mixed to prepare CPB.

The mineral composition of as-obtained materials was obtained by using X-ray diffraction (XRD) and results are summarized in Table 3. The calcium (Ca) and magnesium (Mg) elements in the gangue are mainly present in the form of carbonates in dolomite, which has a total content of ~ 69.65%. Therefore, dolomite renders a little influence on the cementation activity of the filling body. The content of hydroxylapatite in the tailing was as high as 60.94%, which may influence the cement hydration reaction and reduce the amount of tri-calcium silicate (C₃S). Moreover, the content of hydroxylapatite influences CPB strength. The aluminum (Al) element was mainly present in the form of muscovite and albite, affecting the activity of fly ash.

2) WATER AND CEMENT

Furthermore, the available tap water was used to prepare CPB specimens. The cement was obtained from the Xinxing Cement Factory (Changsha, China), which meets the requirements of national standard GB 175-2007, “Common Portland Cement”. The mineral composition of the cement is provided in Table 2.

3) AIR ENTRAINING AGENT

Herein, two types of AEAs, SA and SJ, were selected to improve the fluidity of filling slurry, where SA is mainly composed of sodium abietate and SJ consists of triterpenoid saponin. Figure 3 shows the physical properties of both AEAs.

Table 4 shows the physical properties of both AEAs. One should note that foaming ability and foam-breaking time are important parameters, defining the overall performance of AEA [16]. The mixing amount of AEA has a certain influence on these parameters. A certain amount of AEA powder was loaded into 100 mL graduated cylinder to prepare different concentrations of solutions. First, the graduated cylinder was shaken and foaming capacity was measured. Then, we have waited for the complete disappearance of the bubbles and recorded the time. Figure 4 shows that the foaming ability of SJ is better than SA, however, the foam-breaking time of SJ is lower than SA.

B. SPECIMEN PREPARATION

Tailing and gangue were selected as filling aggregates, whereas cement and fly ash were used as cementing materials. Three different dosages of each type of AEA have been used. The tailing/cement ratio was set according to the engineering application in Sanning Mine. In order to solve the problem of long-distance transportation, some mines utilize two pipes to transport the filling material. One pipe
The results of the shake-bubble test of two types of AEAs.

FIGURE 4. The results of the shake-bubble test of two types of AEAs.

FIGURE 5. Filling process of sanning mine.

FIGURE 5. Filling process of sanning mine.

TABLE 5. Different mixture proportions of the as-prepared CPB samples.

<table>
<thead>
<tr>
<th>Code</th>
<th>Gangue (kg/m³)</th>
<th>Tailing (kg/m³)</th>
<th>Fly ash (kg/m³)</th>
<th>Cement (kg/m³)</th>
<th>Water (kg/m³)</th>
<th>SA (wt% of cement)</th>
<th>SJ (wt% of cement)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SA1</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>0.2</td>
<td>-</td>
</tr>
<tr>
<td>SA2</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>SA3</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>0.6</td>
<td>-</td>
</tr>
<tr>
<td>SJ1</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>-</td>
<td>0.2</td>
</tr>
<tr>
<td>SJ2</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>-</td>
<td>0.4</td>
</tr>
<tr>
<td>SJ3</td>
<td>1500</td>
<td>400</td>
<td>200</td>
<td>150</td>
<td>562</td>
<td>-</td>
<td>0.6</td>
</tr>
</tbody>
</table>

Note: wt% represents solid mass percentage.

C. EXPERIMENTAL METHODS

1) SLUMP TEST

The slump test of the freshly filling slurry was carried out according to GB/T 50080-20 standard, which describes the standard procedure to assess the performance of ordinary concrete mixtures.

2) NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

After the curing time of 7 days, the CPB sample was taken out and immediately wrapped with a cling film to prevent the water loss. Then, the NMR spectrum of the CPB sample was directly collected. One should note that the sample has not been vacuumed and filled with water due to the fragile nature of the CPB sample. After the curing time of 28 days, the sample was vacuumed by using a saturated vacuum device. Then, the CPB sample was taken out and wrapped with a cling film for NMR analysis. NMR spectra were collected using MiniMR-60 magnetic resonance imaging (MRI) system (Shanghai Newmai Co. Ltd., China). NMR is based on the response of nuclei to the applied magnetic field. NMR spectra were used to assess the porous content in the slurry and backfill. The T2 spectrum distribution, which is a non-destructive characterization technique, was used to analyze the pore size distribution [17], [18]. The experimental parameters were as follow: receiver bandwidth (SW) = 100 kHz; number of sampling points (TD) = 4502; digital gain (DRG1) = 3; analog gain (RG1) = 20 db; cumulative sampling (NS) = 64; 90° pulse application time (P1) = 11.52 μs; 180° pulse application time (P2) = 22.48 μs; number of echo cycles (NECH) = 3000; waiting time (Tw) = 4800 ms; coil waiting time (RFD) = 0.25 ms; and number of times (NS) = 64.

3) UNIAXIAL COMPRESSIVE STRENGTH

The uniaxial compressive strength (UCS) is a critical parameter, which determines the quality of the backfill. The UCS of CPB samples, cured for 7 and 28 days, was measured according to ASTM D2166/D2166M-16 standard by using a computer-controlled full automatic pressure testing machine (WDW-2000) with a maximum capacity of 200 KN. The specimens were loaded at a constant displacement rate of 1 mm/min. Three independently obtained UCS values of each sample were averaged out to report the UCS of a certain sample.

4) MICROSTRUCTURAL ANALYSIS

After the UCS test, a specimen of 1 mm² was sectioned from the center of each CPB sample and dehydrated with absolute ethanol to stop the hydration. Then, scanning electron microscopy (SEM, TESCAN MIRA3) was used to obtain the surface morphology. A conductive film was deposited on the transports non-cemented filling material and the other pipe transports cemented material. Figure 5 shows the filling process. Therefore, the filler slurry used in the slump test was not added with cement and its concentration was set at 80%. The cement-containing sample was used in other experiments. The detailed mixture ratios are summarized in Table 5.

To prepare CPB specimens, raw materials were weighed according to the designed ratio and well-mixed in a cylindrical mold (50 mm × 100 mm). The bottom of this mold can guarantee the purpose of drainage. We prepared 42 CPB samples (three for UCS and three for NMR) for experimental testing. The specimens were cured in a humidity chamber at 20°C, with a relative humidity of 99%, for different durations (7 and 28 days) to attain the prescribed age. Then, the physicochemical properties of the as-prepared CPB samples were analyzed.
surface of the specimen to overcome the charging and obtain high-quality SEM images.

III. RESULTS

A. FLOWABILITY OF CPB

The yield stress of the filling material is inversely proportional to the slump. Therefore, the degree of yield stress can be characterized by a slump. According to the mine information, the slump height of filling slurry need to reach 22cm. Figure 6 shows the experimental results of the slump of the filling slurry under different conditions. It can be readily observed that the slump of the filling slurry is positively related to the amount of AEA. The AEA content of 0.2% increased the slump height of SJ- and SA-added filling slurry to 24.6 cm and 23.9cm, which is 11.8% and 8.6% higher than AEA-free filling slurry. Compared with the filling slurry with sodium rosin, when the mixing amount of AEA was 0.2%, 0.4% and 0.6%, the slump of the filling slurry with SJ increased by 2.93%, 5.76% and 8.37%, respectively. The results reveal that both AEAs can increase the fluidity of the filling slurry, however, SJ rendered optimal performance.

B. UCS RESULTS

The change in uniaxial UCS of the CPB samples, with different concentrations of AEA, is presented in Figure 7. After the curing time of 7 days, UCS of SA- and SJ-added CPB ranged from 0.89-1.41 MPa and 1.04-1.39 MPa, respectively, which increased to 2.05-2.31 MPa and 2.13-2.35 MPa after the curing time of 28 days. According to the mine information, the uniaxial compressive strength of each group of specimens at the curing times of 7d and 28d need to reach 0.7MPa and 1.4MPa. It is worth mentioning that the obtained UCS values meet the requirements of the mine. Moreover, when the mixing amount of AEA was 0.2%, the UCS of CPB sample, cured for 7 and 28 days, attained an optimal value. However, when AEA content exceeded 0.2%, a negative correlation between strength and AEA amount has been observed.

C. NMR RESULTS

NMR can effectively detect the distribution of bound water in the CPB. McDonald et al. [19] have argued that the physically bound water is a part of the solid product, which fills the space between pores. It has been reported [20] that the cement hydration reaction partially consumes physical bound water and form chemically bound water, which exhibits a short $T_2$ value. However, the Carr-Purcell-Meiboom-Gill (CPMG) experiment could not collect the NMR signal of chemically bound water. The peak area of the acquired $T_2$ spectrum reflects the amount of physically bound water in the sample. $T_2$ values correspond to different conditions of the bound water. Each relaxation peak represents a bound state of water. It has been reported that the shorter $T_2$ value corresponds to inferior water mobility [21], [22]. The filling body with a 7-day curing age is more fragile. Therefore, the sample has not been vacuumed before NMR measurements. The $T_2$ spectral curve can be obtained by $T_2$ inversion software (Figure 8). It can be readily observed that $T_2$ spectra contain three distinct peaks, representing different states of bound water. From left to right, the 1st, 2nd and 3rd peak represent the adsorbed, capillary and free water, respectively. The peak area of the 1st peak accounts for 90% of the total spectral area, indicating the presence of a large amount of adsorbed water in the filling body. Moreover, the area of 1st peak increase with increasing AEA content,
corresponding to an increase in adsorbed water content due to the higher amount of AEA.

Moreover, the content of adsorbed water in SA-added CPB remained higher than SJ-added CPB. One should note that the higher content of AEA influenced the conversion process of physically bound water into chemically bound water. It indicates that the addition of AEA inhibited the hydration reaction of the filling slurry. Under the same AEA content, SA-added filling slurry rendered superior inhibition effect than SJ-added filling slurry.

Based on NMR theory [23], the relationship between the value of relaxation time ($T_2$) and pores can be expressed as:

$$1/T_2 \approx \rho_2 (S/V) = F_s \rho_2/r$$  

(1)

where $T_2$ refers to the total transverse relaxation time (ms), $\rho_2$ represents the transverse surface relaxation strength ($\mu$ms), $S$ denotes the pore surface area (cm$^2$), $V$ corresponds to the pore volume (cm$^3$), $F_s$ refers to a geometrical factor and $r$ represents the pore radius. $F_s$ value of 3 and 2 corresponds to spherical and columnar pores, respectively.

Eq. 1 indicates a direct relationship between pore size and $T_2$ value. Hence, NMR $T_2$ distribution curve can be transformed into the pore size distribution of the filling slurry [24]. Since the internal void structure of CPB is similar to the concrete, $\rho_2$ (12 nm/ms) can be obtained from a previous study [25]. One should note that AEA produces dense micro bubbles, rendering spherical pores in the CPB sample ($F_s = 3$). With these value, Eq. 1 presents a relationship between pore size and $T_2$ value in the filling slurry:

$$r = 36T_2 \times 10^{-9}$$  

(2)

When CPB is filled with water, NMR transverse relaxation time represents the size of the aperture. The larger $T_2$ value corresponds to the larger aperture and vice versa.

Similarly, $T_2$ distribution curves of CPB samples after curing for 28 days have been obtained by $T_2$ inversion software, as shown in Figure 9. Herein, $T_2$ spectra of CPB samples contain three peaks, corresponding to macroporous, mesopore, microporous, respectively. Moreover, the weighted average of the $T_2$ relaxation time of 1st, 2nd and 3rd peak ranged from 0.047-2.012 ms, 2.012-20.822 ms and 20.822-95.185 ms, respectively. The area of the 1st peak is $\sim$90% of the total spectral area, which indicates that the samples are mainly dominated by small pores and the addition of AEA increased the concentration of small pores.

Figure 10 shows the relationship between the porosity and AEA amount. NMR results of the CPB sample, cured for 7 days, represent the internal physical binding water content. As shown in Figure 10, the content of physically bound water inside the CPB increased with increasing AEA content. NMR results of the CPB sample, cured for 28 days, represent the porosity of the filling body. The porosity of the CPB samples increased with increasing AEA content and ranged from 7.39% to 9.31%. Under the same amount of AEA and aging conditions, SA-added CPB samples rendered higher porosity than SJ-added CPB sample.

IV. DISCUSSION AND ANALYSIS

A. FLOWABILITY ANALYSIS

With the increase of AEA content, the foaming amount of the AEA increased, the slump height of the filling slurry became larger and the fluidity of the filling slurry has been significantly improved. The results of the shaking experiment and slump test revealed that the slump height of the filling slurry is consistent with the change of foaming amount. Moreover, the same amount of SJ rendered better foaming performance than SA, which resulted in a higher slump height. One should note that the AEA changed the fluidity of filling slurry by generating a large number of tiny bubbles, which acted as floating balls and reduced the friction between the particles. In addition, AEA acted as a dispersing and wetting agent. Therefore, AEA content remarkably influenced the fluidity of the filling slurry.

B. MECHANICAL PROPERTIES AND MESOSTRUCTURE

The cross-sectional SEM images of CPB samples, cured for 7 and 28 days, are shown in Figure 11. It can be readily observed that the amount of hydration products, including AFt and C-S-H, significantly increased with increasing
curing time. Interestingly, these hydration products can fill the voids between the particles [26], forming a dense network structure and, consequently, increasing the compactness of CPB samples [27].

Furthermore, the internal pore analysis of CPB sample, cured for 7 days, provides the variation of internal pore structure with AEA amount, explaining the initial increase in UCS with AEA content, followed by a gradual decrease. Based on the stereology and maximum inter-class variance method, the SEM image of the CPB sample can be used to quantitatively obtain the cross-sectional pore structure of the filling material. Moreover, the relationship between the porosity and AEA amount can be analyzed [28]. In order to ensure the accuracy of the obtained results, the SEM image should be converted into binary image. The image processing is mainly based on the grayscale segmentation threshold value and the individual pixels of the grayscale image are sequentially binarized. The binarization judgment function can be given as:

\[
  f(i, j) = \begin{cases} 
  0, & f(i, j) \leq T \\
  1, & f(i, j) > T
  \end{cases}
\]  

where \( T \) refers to the threshold grayscale. SEM image can be binarized and converted into a black-and-white image, which is represented by a matrix of black-and-white pixels. Herein, 0 represents the black pixel and 1 represents the white pixel, referring to the pores and particles, respectively. SEM binarized image and grayscale image are compared in Figure 12.

It can be readily observed that AEA-free CPB sample contains a large number of interconnected pores. However, the addition of a small amount of AEA resulted in smaller pores with uniform distribution. Moreover, the pore size increased with increasing AEA content. Based on the previous reports, it can be concluded that the addition of AEA produces a large number of tiny, closed and uniform bubbles, rendering optimal internal pore structure of CPB. The lower AEA content resulted in optimized pore structure, which improved the strength of CPB. However, the higher AEA content increased the porosity of CPB. Since the volume of the filling body is constant, the smaller pores were connected and led to an inferior strength of CPB.

Furthermore, NMR analysis revealed that AEA renders an inhibitory effect on the internal hydration reaction of the CPB. Therefore, the amount of hydration product decreased with increasing AEA content. As mentioned earlier, the hydration products fill the gaps between particles and the decreased content of hydration products affected the pore size and strength of CPB.

NMR results of CPB sample, with a curing age of 28 days, exhibit that the filling porosity of SJ-added CPB sample is lower than SA-added CPB sample. One should note that the addition of AEA mainly changed the internal void structure of the CPB by generating microbubbles. Even though SJ renders a higher foaming ability than SA, the defoaming time of SJ-added CPB sample is shorter than SA-added CPB sample. As a result, some of the bubbles are destroyed and disappeared during the consolidation process of the filling body, altering the porosity and resulting in inferior mechanical strength.

V. CONCLUSION

In summary, we have systematically studied the influence of AEA on mechanical properties and microstructural changes of CPB. The main conclusions of the present study can be summarized as:

1. The foaming amount is directly related to the concentration of both AEAs. Under the same experimental conditions, the foaming amount of SJ was better than SA, however, the defoaming time of SJ is lower than SA. Moreover, the toughness of SA-induced bubbles was better than SJ-induced bubbles. AEA-generated microbubbles act as ball bearings during the slurry conveying process, reducing the friction between the aggregate particles and improving the fluidity of the filling slurry. The slump test exhibited that the slump height increased with increasing AEA content. Moreover, the flowability of SJ-added filling slurry was better than SA-added filling slurry, which can be ascribed to the enhanced fluidity.
Moreover, the presence of AEA inhibited the hydration reaction of the cement during the consolidation process. At the same time, AEA increased the concentration of small pores inside the consolidated CPB by generating ordered and uniform microbubbles. The $T_2$ curve of the CPB sample, cured for 7 days, indicated that the higher AEA content rendered more profound inhibition effect of hydration reaction. $T_2$ curve of the CPB sample, cured for 28 days, confirmed that the concentration of small pores increased with increasing AEA content. Under the same experimental conditions, the porosity of SA-added CPB samples was higher than SJ-added CPB sample.

(3) AEA-added CPB samples, cured for 7 and 28 days, meet the mining requirements. The strength of AEA-added CPB samples initially increased with increasing AEA content, followed by a gradual decrease. The minimum strength of the CPB samples, cured for 7 and 28 days, was found to be 0.89 and 2.05 MPa, respectively. Furthermore, the pore structure of CPB can be optimized by adding an optimal amount of AEA, which is capable of reducing the number of connected holes in CPB and uniformly distribute the pores, rendering improved strength of CPB. SEM analysis of the CPB samples, cured for 7 days, revealed that the lower concentration of AEA induced micro-bubbles, which resulted in uniform distribution of internal voids and significantly reduced the connected holes. Therefore, the optimized pore structure improved the filling strength. However, total porosity of the CPB sample increased with increasing AEA content and the higher concentration of small pores reduced the pore spacing, resulting in re-appearance of the pores. Hence, the strength of CPB decreased when AEA content exceeded a threshold limit.

REFERENCES


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