MICROSTRUCTURE OF CEMENT-BASES COMPOSITES CONTAINING ROCK WOOL PARTICLES

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Abstract

Rock wool is an inorganic fibrous substance produced by steam blasting and cooling molten glass. Like other industry by-products, rock wool particles can be used as cementitious materials or ultra fine fillers in cement-based composites. This study investigated the microstructure of cement-based composites with addition of various rock wool particles in mortar specimens. Compressive strength, rapid chloride penetration test (RCPT), X-ray diffraction analysis (XRD), thermo gravimetric analysis (TGA) and scanning electronic microscopy observation (SEM) were conducted to evaluate the properties of cement-based composites. Test results indicated that inclusion of rock wool particles had higher compressive strength than control specimens. Rock wool specimens also had lower chloride ion penetration based on the results of RCPT. In addition, less Ca(OH)₂ was found in rock wool specimens and rock wool specimens had dense internal structure as illustrated by SEM observation.

Key words: durability, X-ray diffraction analysis, thermo gravimetric analysis, waste treatment
1. **INTRODUCTION**

Rock wool is an inorganic fibrous substance produced by steam blasting and cooling molten glass. Rock wool is frequently used for acoustic insulation, fire protection, cement reinforcement, pipe insulation, and even as synthetic soil for growing plants [1]. Taiwan generates more than 100 million tons of rock wool particles annually [2]. Over the last few decades, the inventory of solid insulation material particles composed of rock wool in the nuclear power plants has reached to a certain extent of heavy loads. The rock wool particles installed in the storages have a dramatic increase in the past decade [3]. How to reduce the quantities of the rock wool particles in storage is also an important issue.

This rock wool waste is loose and bulky, and requires a large space to be stockpiled or landfilled. Traditional landfill or stockpile methods are not environment-friendly solutions, and it is very difficult for these disposal processes to meet Environmental Protection Agency regulations [4,5]. The substitute methods now used are melting, compressed, briquetted and reprocessed [6,7]. However, this melting process consumed a lot of energy to heat the particles and has proven to be uneconomical. Besides, the pervious study indicated that the rock wools were used to be economic components to substitute glass fibers and synthetic polymers in insulating composites [8]. Another method was used as the fiber-modified asphalts containing rock wools [9]. And it also seems to be a complex method for treating the rock wool particles.

Like other industry by-products, such as fly ash, silica fume, and ground granulated blastfurnace slag (GGBS), rock wool particles can be reused and recycled to avoid environmental problems in civil constructions for many years [10-12]. Moreover, these industrial by-products usually used in concrete are partial replacement for aggregates or cementitious materials, depending on the chemical composition and grain size of the by-product. The study in Cheng provides that the composition of rock wool waste is similar to other pozzolan materials and the rock wool particles can be used as a suitable substitute for coarse and fine aggregates [13]. The use of rock wool particles also enhances the mechanical properties and durability. Furthermore, it also suggests that the rock wool particles can be considered as a supplementary cementitious material.

The pervious study has reported that the cement-based composites containing pozzolanic materials could react with water in the alkali environment and then with calcium hydroxide to form cement hydration product through pozzolanic reaction to form extra C-S-H gel in the paste [14]. The denser microstructures and lower porosity could form due to the pozzolanic reaction as illustrated by the microstructure techniques, such as scanning electronic microscopy (SEM) observation, X-ray diffraction (XRD) analysis and thermo gravimetric analysis (TGA) [15-16]. The pastes had lower calcium hydroxide and higher C-S-H content than the Portland cement pastes and then formed the better performance on strength and permeability of cement-based composites. This study is aimed to evaluate the effect of rock wool particles on the microstructural characterizations of cement-based composites. The rock wool particles with various rock wool particles (0%, 10%, 20%, 30% and 40% by weight) were considered as partial replacement of cement in the mixtures. The SEM observation, XRD analysis and TGA test were conducted to evaluate the variations of hydrations and microstructures. In addition, the compressive strength and rapid chloride penetration test (RCPT) on different percentages of rock wool particles of the mortar specimens were also reported and confirmed.

2. **EXPERIMENT**

2.1 **Materials**

Rock wool particles obtained from thermal insulation materials were crushed and ground. Table 1 lists the chemical composition of rock wool particles and various pozzolanic materials. The SEM image in Fig. 1 shows that rock wool particle has a cylindrical shape, with an average diameter of 3.5 μm. In addition, the surface area of rock wool particles is 206 m²/kg, which is lower than other pozzolanic materials and cement.

2.2 **Mix proportion**

Type I Portland cement conforming to ASTM C150-11 was used in all mixes. Table 2 shows the mix proportions of mortar specimens. The water/binder ratios (w/b) were 0.34 and 0.55 and the sand/binder...
ratios were kept a constant of 2.75. Rock wool particles (10%, 20%, 30% and 40% by weight) were added to different mixes to partially replace the cement. The fine aggregates had a fineness modulus of 2.51. The absorption of fine aggregates was 0.63% and its relative density at the saturated surface dry (SSD) condition was 2.49. Mixture was achieved using the high-range water-reducing admixture (superplasticizer). The particle size of rock wool particles was less than 75 μm.

Table 1: Chemical composition of various recycled materials and Portland cement

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Rock wool</th>
<th>Fly ash</th>
<th>GGBS</th>
<th>Silica fume</th>
<th>Cement</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂ (wt.%)</td>
<td>38.7</td>
<td>54.0</td>
<td>33.5</td>
<td>91.5</td>
<td>21.2</td>
</tr>
<tr>
<td>Al₂O₃ (wt.%)</td>
<td>18.6</td>
<td>24.0</td>
<td>9.0</td>
<td>0.2</td>
<td>5.4</td>
</tr>
<tr>
<td>Fe₂O₃ (wt.%)</td>
<td>5.3</td>
<td>8.0</td>
<td>3.6</td>
<td>0.7</td>
<td>3.2</td>
</tr>
<tr>
<td>CaO (wt.%)</td>
<td>20.9</td>
<td>2.0</td>
<td>43.8</td>
<td>0.4</td>
<td>63.8</td>
</tr>
<tr>
<td>MgO (wt.%)</td>
<td>7.0</td>
<td>1.3</td>
<td>2.7</td>
<td>1.5</td>
<td>2.0</td>
</tr>
<tr>
<td>K₂O+Na₂O (wt.%)</td>
<td>2.0</td>
<td>0.9</td>
<td>0.6</td>
<td>1.9</td>
<td>0.8</td>
</tr>
<tr>
<td>Other (wt.%)</td>
<td>7.5</td>
<td>9.8</td>
<td>6.8</td>
<td>3.8</td>
<td>3.6</td>
</tr>
<tr>
<td>Surface area (m²/kg)</td>
<td>206</td>
<td>420</td>
<td>415</td>
<td>22500</td>
<td>364</td>
</tr>
</tbody>
</table>

Figure 1: SEM observation of rock wool particles

Table 2: Mix proportions (kg/m³)

<table>
<thead>
<tr>
<th>Mix no.</th>
<th>w/b</th>
<th>Rock wool content (wt. %)</th>
<th>Water</th>
<th>Cement</th>
<th>Rock wool</th>
<th>Fine aggregate</th>
<th>SP</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.34</td>
<td>0 %</td>
<td>191.8</td>
<td>564.1</td>
<td>0.0</td>
<td>1551.2</td>
<td>5.0</td>
</tr>
<tr>
<td>AR10</td>
<td></td>
<td>10 %</td>
<td>191.6</td>
<td>507.7</td>
<td>56.4</td>
<td>1549.7</td>
<td>5.0</td>
</tr>
<tr>
<td>AR20</td>
<td></td>
<td>20 %</td>
<td>191.4</td>
<td>451.3</td>
<td>112.8</td>
<td>1548.1</td>
<td>5.0</td>
</tr>
<tr>
<td>AR30</td>
<td></td>
<td>30 %</td>
<td>191.2</td>
<td>394.9</td>
<td>169.2</td>
<td>1546.6</td>
<td>5.0</td>
</tr>
<tr>
<td>AR40</td>
<td></td>
<td>40 %</td>
<td>191.0</td>
<td>338.5</td>
<td>225.6</td>
<td>1545.0</td>
<td>5.0</td>
</tr>
<tr>
<td>B</td>
<td>0.55</td>
<td>0 %</td>
<td>278.8</td>
<td>506.9</td>
<td>0.0</td>
<td>1394.0</td>
<td>0.0</td>
</tr>
<tr>
<td>BR10</td>
<td></td>
<td>10 %</td>
<td>278.5</td>
<td>456.2</td>
<td>50.7</td>
<td>1392.7</td>
<td>0.0</td>
</tr>
<tr>
<td>BR20</td>
<td></td>
<td>20 %</td>
<td>278.3</td>
<td>405.5</td>
<td>101.4</td>
<td>1391.5</td>
<td>0.0</td>
</tr>
<tr>
<td>BR30</td>
<td></td>
<td>30 %</td>
<td>278.0</td>
<td>354.8</td>
<td>152.1</td>
<td>1390.2</td>
<td>0.0</td>
</tr>
<tr>
<td>BR40</td>
<td></td>
<td>40 %</td>
<td>277.8</td>
<td>304.1</td>
<td>202.8</td>
<td>1388.9</td>
<td>0.0</td>
</tr>
</tbody>
</table>
2.3 Specimens
The experiments in this study cast a total of 200 specimens for 10 different mixes. All the specimens were cured in saturated limewater until testing. For each mix, 50 x 50 x 50 mm cube specimens were prepared to test the compressive strength. 100 x 50 mm circular specimens were used for the RCPT. In addition, the specimens sliced from the mortar specimens of 10 x 10 x 10 mm were prepared for the SEM observation and the mortar powders of 3 g were prepared for the XRD and TGA test.

2.4 Testing methods
In accordance with ASTM C109-11, compressive strength tests were performed at 7, 28, and 91 days. The RCPT was prepared following the ASTM C1202-10 at the age of 91 days. In addition, the microstructure of the mortar specimen was assessed by SEM according to ASTM C856-11.

XRD and TGA were performed on the specimens at the age of 91 days. The chemical compounds of the matrix were determined by using XRD. The patterns were taken using a Cu-Kα radiation between 10° to 50° in 2θ with a scanning speed of 1°/min. TGA of the calcium hydroxide was conducted using a heating rate of 10 °C/min and a heating range from 25 °C to 995 °C under nitrogen. The amount of calcium hydroxide (CH) was calculated as follows:

\[ W_{CH} = \frac{4.1(W_{440} - W_{580}) + 1.68(W_{580} - W_{995})}{W_{995}} \times 100\% \]  

where \( W_{CH} \) is the amount of CH (%); \( W_{440} \) is the sample weights at 440 °C (g); and 4.11 and 1.68 are the conversion factor for the molar ratio derived CO2 and H2O to CH, respectively.

3. RESULTS AND DISCUSSION

3.1 XRD analysis
The X-ray diffractograms for hydrated pastes of the specimens with and without rock wool particles at age of 91 days are illustrated in Fig. 2. These graphs reveal that the main peaks corresponded to quartz and calcium hydroxide formed in the cement pastes. The qualitative similarity in the hydrations between control specimens and rock wool specimens was existed. There were few changes in formation of pozzolanic reaction products and no new hydrations were formed with incorporating rock wool particles. In addition, the residual portlandite were present as the rock wool particles increased. The specimens containing rock wool particles generated the pozzolanic reaction to consume the calcium hydroxide values and also led to the formation of more calcium silicate hydrate. This result is consistent with the previous study, which indicated that pozzolanic reaction may occur in fine rock wool particles from the pozzolanic strength activity index test [13].

![Figure 2: XRD patterns of the specimens at the age of 91 days](image-url)
3.2 TGA results

The TGA results of cement-based composites with and without rock wool particles at the age of 28 days and 91 days for each mixes are shown in Figs. 3 to 4, respectively. The TGA curves are represented as the quantitative mass loss of paste products, as shown in four mass loss transitions similar to the previous studies [17-18]. The weight loss between 25°C and 105°C, 105°C and 440°C, 440°C and 580°C, 580°C and 995°C corresponded to the decomposition of water from the capillary and gel pores, the decomposition of water from the hydrated phases, the dehydroxylation of water from calcium hydroxide, the decomposition of calcium carbonate, respectively. The weight losses of the phases from the TGA results are summarized in Table 3. At the age of 28 days, the weight loss decreased as the rock wool particles increased from the temperature range between 105 and 440. The increased rock wool particle replacements led to decrease in the use of the cement, resulting in the lower weight loss of rock wool specimens than that of the control specimens. However, the rock wool specimens had higher weight losses at the age of 91 days than those of 28 days. The results clearly indicated that the increases were generated from the pozzolanic reaction products. It also confirmed that the rock wool particles were benefit to the formation of pozzolanic reaction.

![Graph](image)

(a) 28 days                                                                 (b) 91 days

Figure 3: Weight loss versus temperature curves from the TGA results (w/b=0.34)

![Graph](image)

(a) 28 days                                                                 (b) 91 days

Figure 4: Weight loss versus temperature curves from the TGA results (w/b=0.55)

The CH contents calculated from the Table 3 and the equation (1) for each mixes are illustrated in Fig. 5. At the age of 28 days for each mixes, there was a decrease in CH content with the increasing rock wool content at 10% and 20% compared to that of the control specimens. But there was an increase in CH content with the increasing rock wool content at 30% and 40%. It could be explained that the addition of rock wool content might have resulted in quantities of CH and it required for the cement particles and
time to react. At the age of 91 days for each mix, the CH content decreased with the increase in the inclusion of rock wool excepting for the rock wool content at 20% in the lower w/b ratio. The increased rock wool replacements led to decreases in weight loss of CH, suggesting that the addition of rock wool particles had a significant influence on the CH consumption in cement-based composites. Consequently, it was evidently explained that more of calcium silicate hydrate gels can be detected in both mixes with rock wool particles and this was seen increasing with rock wool content. And it also showed the pozzolanic activity on the rock wool specimens.

Table 3: Calculated weight loss of the pastes from TGA results

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Age (days)</th>
<th>A</th>
<th>AR10</th>
<th>AR20</th>
<th>AR30</th>
<th>AR40</th>
<th>B</th>
<th>BR10</th>
<th>BR20</th>
<th>BR30</th>
<th>BR40</th>
</tr>
</thead>
<tbody>
<tr>
<td>105~440</td>
<td>28</td>
<td>5.2</td>
<td>5.1</td>
<td>4.5</td>
<td>4.0</td>
<td>3.9</td>
<td>6.1</td>
<td>5.1</td>
<td>4.6</td>
<td>4.2</td>
<td>3.7</td>
</tr>
<tr>
<td></td>
<td>91</td>
<td>4.7</td>
<td>5.5</td>
<td>5.1</td>
<td>4.5</td>
<td>4.5</td>
<td>4.5</td>
<td>5.8</td>
<td>5.7</td>
<td>6.2</td>
<td>5.1</td>
</tr>
<tr>
<td>440~580</td>
<td>28</td>
<td>1.6</td>
<td>1.5</td>
<td>1.2</td>
<td>1.6</td>
<td>1.4</td>
<td>1.8</td>
<td>1.5</td>
<td>1.4</td>
<td>1.5</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>91</td>
<td>1.6</td>
<td>1.6</td>
<td>1.8</td>
<td>1.3</td>
<td>1.1</td>
<td>1.9</td>
<td>1.7</td>
<td>1.4</td>
<td>1.6</td>
<td>1.3</td>
</tr>
<tr>
<td>580~995</td>
<td>28</td>
<td>1.7</td>
<td>1.6</td>
<td>1.2</td>
<td>3.9</td>
<td>3.3</td>
<td>3.3</td>
<td>3.3</td>
<td>4.3</td>
<td>4.0</td>
<td>4.2</td>
</tr>
<tr>
<td></td>
<td>91</td>
<td>4.2</td>
<td>3.8</td>
<td>4.6</td>
<td>1.3</td>
<td>1.5</td>
<td>7.1</td>
<td>2.0</td>
<td>2.8</td>
<td>1.7</td>
<td>1.6</td>
</tr>
</tbody>
</table>

Figure 5: CH content histograms

3.3 Compressive strength and total charge-passed

The compressive strength histograms at the age of 28 and 91 days are illustrated in Fig. 6. At the age of 28 days, the compressive strength decreased with an increase of the rock wool particles. Compared to the age of 91 days for lower w/b ratio, the compressive strength increased with an increase of the rock wool particles. And it got a contrary result for higher w/b ratio. Because the pozzolanic activity of rock wool particles was less than that of cement, the initial reaction rate was lower. The rock wool particles contained more SiO₂ and Al₂O₃, prompting a secondary hydration reaction with the cement hydration product CH, to produce C-S-H and C-A-H, increase the compactness of the slag concrete, and enhance its compressive strength. The rock wool particles in the composites helped to consume the CH and form a denser microstructure, and then cause an improvement in strength, which is consistent with the results of TGA testing. In addition, the specimens containing 30% rock wool particles had higher compressive strength than those containing 10% and 20% rock wool particles at the age of 28 days. The enhancement may be due to the particle parking effect from the finer rock wool particles.

The total charge-passed histograms at the age of 91 days are illustrated in Fig. 7. The apparent total charge-passed was reduced on increasing the rock wool particle content and on decreasing the w/b ratio. Rock wool particles mixed into the composites improved the compactness through pozzolanic reactivity.
and pore filling effects. The specimens with 20%, 30%, and 40% rock wool particles showed significantly lower permeability than the control specimens, which was less than 2000 coulombs. The pozzolanic reaction between calcium hydroxide and reactive silica in rock wool particles were formed the denser internal structures, which is also is consistent with the results of TGA and compressive strength testing.

![Figure 6: Compressive strength histograms](image)

![Figure 7: Total charge-passed histograms](image)

### 3.4 SEM observation

The pozzolanic reaction between calcium hydroxide and reactive silica in rock wool particles and the denser internal structures are formed as illustrated by SEM observations in Fig. 8. The addition of rock wool particles may cause smaller sizes of capillary pores and rock wool particles may decrease the pore interconnectivity because the fiber-shape rock wool particles could bridge cracks and arrest capillary pores. The SEM observations of AR20 specimens at the age of 28 days and 91 days are illustrated in Fig. 9. Rock wool particles in the composites helped to form a denser microstructure and cause improvement in strength and permeability, which is consistent with the results of TGA, compressive strength and total charge-passed. In addition, the inclusion of rock wool particles in composites formed a denser microstructure when the CH consumption increases. (The CH content between A and AR20, B and BR20 specimens were 1.98 % and 2.18 % at the age of 28 days, respectively, and BR20 specimens were 3.75% at 91 days.) The hydrations were also observed on the surface of the rock wool particle. Thus, the addition of rock wool particles leads to reduction of permeable voids and improvement of the durability.

The SEM observations of AR20 and AR40 specimens at the age of 91 days are illustrated in Fig. 10. It was found that a great number of large capillary pores were found in AR40 specimens. The capillary pores in AR20 specimens were less than those in AR40 specimens. The capillary pores could be filled up with pozzolanic reaction products and the hydrations were formed in the surface of the rock wool particle. Not only the pores became finer but also the change in the cement hydrations of the mortar surface led to reduce in the mobility of chloride ions or other ions. In addition, the microstructure of AR20 specimens was very compact and few needle-shaped ettringite or plated shaped calcium hydroxide was observed.
The compacter and denser microstructure led to the lower porosity, which resulting the reduction in absorption and total charge-passed. From those SEM observations, the inclusion of rock wool particles in cement-based composites was benefit to enhance the pozzolanic reaction and reduce the content of calcium hydroxide.

4. CONCLUSIONS

- X-ray diffraction analysis showed that the quartz and calcium hydroxide were more significant formed in the paste containing rock wool wastes than the control specimens. There were few changes in formation of pozzolanic reaction products and no new hydrations were formed with incorporating rock wool particles.
The results of the thermo-gravimetric analysis showed that the decomposition of water from the capillary and gel pores was decreased and the calcium hydroxide consumptions were increased as the rock wool particles increased. It was evidently explained that more of calcium silicate hydrate gels can be detected and showed the pozzolanic activity on the rock wool specimens.

Due to the particle parking effect and secondary hydration reaction of rock wool particles, the compressive strength and total charge-passed increased as the content of rock wool particles increased.

The initial reaction rate (at the age of 28 days) of pozzolanic reaction was lower. At the age of 91 days, the compressive strength significant increased as the rock wool particles increased for lower w/b ratio.

Microscopic analysis with a scanning electron microscope confirmed that the inclusion of rock wool particles in cement-based composites was benefit to enhance the pozzolanic reaction and reduce the content of calcium hydroxide. And the rock wool particles were attributed to compact the microstructure and form a denser microstructural changes of the hardened paste.

ACKNOWLEDGEMENTS

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