Microstructure of New Cement Matrix Phase and Performance of Mortar Incorporating Recycled Fine Aggregate

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MICROSTRUCTURE OF NEW CEMENT MATRIX PHASE 
AND PERFORMANCE OF MORTAR INCORPORATING 
RECYCLED FINE AGGREGATE

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ABSTRACT

In this study, the strength and the carbonation speed of the mortar incorporating recycled fine aggregate were examined. Strength and carbonation speed could be evaluated by total water-cement ratio, which was obtained by summation of water content and total water absorbed in aggregate. Pore volume between 50nm and 2μm in new cement matrix phase was porous and loose when recycled fine aggregate with high water absorption was used in mortar. Performance of mortar incorporating recycled fine aggregate correlated closely with pore volume between 50nm and 2μm in new cement matrix phase. It was suggested that water included in recycled aggregate moved to new cement matrix phase during cement hydration.

Keywords: recycled fine aggregate, pore size distribution, strength, carbonation, interfacial transition zone (ITZ)

INTRODUCTION

Sustainable development is an important factor, which is directly connected with harmonious coexistence of human beings and nature. Large quantities of construction and demolition waste are disposed all over the world. Using these construction and demolition waste is effective for reduction of concrete waste. Moreover, extraction of sea sand is being prohibited from the viewpoint of environmental disruption. Production of alternative aggregate is important for reduction of environmental protection.

Recycled aggregate is produced from crushed concrete, one of the demolition wastes of concrete structures. There have been a number of researches on the use of recycled aggregate in concrete. They were almost concluded that concrete strength decreases when recycled aggregate was used in concrete and the reduction degree was various according to types of aggregates (Hansen and Narud 1983). However, the relationship compressive strength and characteristics of aggregate is not cleared. It is difficult to make constant quality of recycled aggregate. Especially, characteristics (i.e.,
density, water absorption, fineness modulus) of recycled fine aggregate are of great variety. Thus, performance of concrete incorporating recycled aggregate changes variously. It needs urgently to reveal the reduction mechanism of compressive strength.

Ion transportation in concrete also depends on the properties of concrete. Interfacial transition zone (ITZ) is porous region in concrete (Kato and Uomoto 2005). Water, ions and gas penetrate mainly through ITZ (Zimbelmann 1985). It is expected that microstructure of concrete incorporating recycled aggregate will be more porous and loose than that used natural aggregate.

This paper presents the relationship between performance of mortar incorporating recycled fine aggregate and microstructure of new cement matrix phase. Three types of aggregates were used in this study. The focus of this study is pore volume between 50nm and 2μm in new cement matrix on performance of mortar. The effects of the pore volume in new cement matrix phase on the mortar performance are discussed.

**EXPERIMENTAL DETAILS**

**Materials**

The cement used was an ordinary Portland cement, whose specific gravity and specific surface area of cement were 3.16g/cm³ and 3220cm²/g, respectively.

Three fine aggregates were used in this study, including natural aggregate and two recycled aggregate. The natural aggregate was sea sand (SS). The recycled aggregates were sourced from crushed concrete. Two types of crushed concrete were selected for use. One (RI) was identified as a prestressed concrete slab, and another (RK) was identified as prestressed concrete pole. Table 1 shows the physical properties of these aggregates. High water absorption of aggregates can be attributed to adhered mortar. Fine aggregates were used in water saturated surface-dry condition.

<table>
<thead>
<tr>
<th>Type of fine aggregate</th>
<th>Specific density (g/cm³)</th>
<th>Water absorption (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS</td>
<td>2.47</td>
<td>2.70</td>
</tr>
<tr>
<td>RK</td>
<td>2.23</td>
<td>6.05</td>
</tr>
<tr>
<td>RI</td>
<td>2.00</td>
<td>11.05</td>
</tr>
</tbody>
</table>

**Specimen preparation**

Three mortar mixes were prepared in the laboratory using the three types of fine aggregates. Water-cement ratio of 0.4, 0.5 and 0.6 and constant sand-cement ratio of 3.0 were used for all mixes.

40 × 40 × 160mm prism specimens were used for flexural strength test and compressive strength test. 100 × 100 × 400mm prism specimens were used for accelerated carbonation test. Demolding was done 24 hours after casting. The specimens were cured in water at 20°C until the each tests.

**Compressive strength test**

Compressive strength tests were conducted on the 40×40×80mm prisms after flexural test at the age of 7, 28, 91 days.
Accelerated carbonation test

The conditions for the accelerated carbonation test were as follows; temperature of 20°C, a relative humidity of 60%, and a CO₂ concentration of 5%. The test was started at the age of 35days. Between the ages of 28days and 35days, the finished surface, bottom surface and two end surfaces of the prisms were covered with an epoxy resin. This was carried out in order to ensure a one-dimensional penetration direction of carbon dioxide into the interior of the specimens. The carbonation depth was measured on the surface of splitting specimens. An ethanol solution of 1% phenolphthalein was sprayed on the surface and the depth of uncolored portions was measured.

Mercury intrusion porosimetry test

Mercury intrusion porosimetry tests were performed on 5mm cubes at the age of 7, 28 and 91 days. The measurement was conducted three times. The samples were dipped into acetone in order to stop cement hydration and D-dried. Then, samples were stored in desiccators until testing.

EXPERIMENTAL RESULTS

Compressive strength

Fig. 1 shows the compressive strength of the mortar with the different types of aggregates. The mortar incorporating recycled fine aggregate had a lower compressive strength than that incorporating natural aggregate at all ages. At a constant cement water ratio, strength values of mortar with RI were the lowest, and RK follows.

Generally, it is known that the strength of mortar depends on three phases; strength of the cement matrix, the aggregates, and the cement matrix-aggregate interfacial bond. ITZ is the weakest phase in mortar. The development of ITZ bond may be different in the mortar with different types of aggregate (Zimbelmann 1985). The higher strength of the mortar with RK may be attributed to a stronger bond developed at ITZ. Compressive strength of RI-0.4 was lower than that of SS-0.6. This may be because of development of ITZ.

![Fig. 1 Relationship between cement-water ratio and compressive strength](image-url)
Carbonation

Fig. 2 shows the carbonation depth of mortar incorporating the different types of aggregates. Carbonation depth of mortar with recycled aggregates was larger than that with natural aggregate. It is shown that carbonation depth is linearly increased with the square root of accelerated carbonation period. Since the carbonation speed is usually represented through the following equation.

\[ x = k \sqrt{t} \]  

where \( x \) is the depth of concrete penetrated by carbon dioxide and \( t \) is the acceleration period, the plot \( x \) versus \( \sqrt{t} \) is approximately linear and the slope is related to \( k \) value.

Fig. 3 shows the relationship between cement-water ratio and \( k \) of each mortar. At a constant water-cement ratio, carbonation speed was different in mortar incorporating different types of aggregate. Carbonation speed of mortar of RI-0.4 was slower than that of SS-0.6. Development of ITZ may be change according to water-cement ratio and types of aggregates.

Total water ratio

It is known that the higher water absorption of aggregate, the lower compressive strength of mortar incorporating that. Fumoto and Yamada (2004) revealed that there was correlation between strength and total water, a sum of water content and total water absorbed in aggregate. Total water is expressed as follows.

\[ TW = W + s \times \gamma_{ds} \times w_s \]  

where, \( TW \) is the total water content, \( W \) is water content, \( s \) is the fine aggregate volume per unit, \( \gamma_{ds} \) is the density of the fine aggregate, and \( w_s \) is the water absorption of the aggregate.

Fig. 4 shows the relationship between compressive strength and cement-total
water ratio \((C/TW)\). There is correlation between cement-total water ratio and compressive strength. At a constant cement-total water ratio, compressive strength is almost the same value regardless of types of aggregates. This means that the water absorbed in fine aggregate affects compressive strength of mortar. Interfacial bond strength is strong when low water absorption aggregate was used. The difference in compressive strength between the mortars with three types of aggregates is due to the differences in both the strength of the fine aggregate and the microstructural properties of the interfacial transition zone.

Generally, carbonation speed depends on water-cement ratio of mortar. **Fig. 5** shows the relationship between cement-total water ratio \((C/TW)\) and carbonation speed. At a constant total water-cement ratio, carbonation speed is different according to types of aggregates. Carbon dioxide diffuses through ITZ chiefly. Thus, microstructure of cement matrix-aggregate interfaces affects the carbonation speed of the mortar.

![Fig. 4](image_url)  **Fig. 4** Relationship between cement-total water ratio and compressive strength

![Fig. 5](image_url)  **Fig. 5** Relationship between cement-total water ratio and carbonation speed

**Pore size distribution of mortar with recycled fine aggregate**

Pores distributed between 50nm and 2\(\mu\)m can be attributed to pores in RI and interfacial transition zone (Uchikawa et al. 1996). In this study, the pore volume of new cement matrix phase was determined. Pore volume existing in new cement matrix-aggregate interfaces was calculated by subtracting pore volume of the aggregate from that of mortar (Eq. (3)).

\[
V_{itz} = V_m - V_a \times a
\]

where, \(V_{itz}\) is the pore volume between 50nm and 2\(\mu\)m of new cement matrix phase, \(V_m\) is the pore volume between 50nm and 2\(\mu\)m of mortar, \(V_a\) is pore volume between 50nm and 2\(\mu\)m of the fine aggregate, \(a\) is the mass percentage of the fine aggregate in mortar.

The obtained pore volume of mortar incorporating different types of aggregates at 28days is shown as **Fig. 6**. Pore volume increased with increasing water-cement ratio regardless of aggregate types. This result agree the research in past. \(V_{itz}\), that is
pore volume between 50nm and 2μm, of mortar using RI-0.5 was about ten times larger than that of SS-0.5 whereas that of RK-0.5 was six times. This means the microstructure of new cement matrix phase mainly depends on not only water-cement ratio but also types of aggregates. Moreover, the effect of water-cement ratio on \( V_{itz} \) was different according to types of aggregates. \( V_{itz} \) of SS-0.6 was twice as much as that of SS-0.4. \( V_{itz} \) of RI-0.6 was about seven times as much as that of RI-0.4. Thus, types of aggregates influence the pore volume between 50nm and 2μm larger than water-cement ratio.

![Graph showing pore volume of mortar with different types of aggregates at 28 days](image)

**Fig. 6** Pore volume of mortar with different types of aggregates at 28 days

**Evaluation strength and carbonation speed by pore volume of 50nm–2μm**

The relationship between \( V_{itz} \) and compressive strength of mortar is shown in Fig. 7. There is a linear relationship between compressive strength and \( V_{itz} \) on a double logarithmic chart. The regression line equation without the plots applied circle is given by Eq. (4).

\[
\begin{align*}
\lambda_f' &= a \cdot V_{itz} - b \\
&= 93.71 \cdot V_{itz} - 0.3582
\end{align*}
\]

where, \( \lambda_f' \) is the compressive strength of mortar, and \( a \) and \( b \) are the experimental constant (\( a=93.71, b=0.3582 \)).

Compressive strengths of RI-0.4 and RK-0.4 at 91 days were much smaller than that predicted from the regression line between \( V_{itz} \) and compressive strength. This is because strength of the aggregate is weaker than that of new cement matrix phase. Pore volumes of RI and RK were 23.5μl/g and 7.8μl/g. In case that pore volume of new cement matrix phase was much smaller than that of the aggregate, compressive strength depends on not \( V_{itz} \) but strength of the aggregate. Compressive strength of RI-0.6 at the age of 7 days was smaller than that predicted from the regression line, too. Pore volume of RI-0.6 at 7 days in new cement matrix phase was as much as that of interfacial transition zone. Thus, compressive strength depends on interfacial transition zone and new cement matrix phase.

The relationship between \( V_{itz} \) and carbonation speed of mortar is shown in Fig. 8. There is a linear relationship between carbonation speed and \( V_{itz} \) on a single logarithmic graph. Carbonation speed of mortar increased as \( V_{itz} \) increases because carbon dioxide mainly diffuses through interfacial transition zone.
Microstructure of new cement matrix-aggregate interfacial was different when three different types of fine aggregate were used for mortars. The higher water absorption of aggregate used in mortar was, the larger $V_{itz}$ became. Compressive strength and carbonation speed of mortar with different types of aggregates depend on $V_{itz}$ of each mortar. The mechanisms of the difference of microstructure and the relationship with the character of aggregates are discussed as follows.

These experimental results mean the importance of water content of aggregates in mortar. It is anticipated that water absorbed in aggregate seeped into new cement paste phase. This causes the loose and porous zone in new cement matrix-aggregate interfaces. Poon et al. (2004) reported that microstructure of ITZ in the concrete prepared with recycled aggregate was porous and loose with SEM observation.

In cement hydration process, free water, that exists during initial mixing stage is consumed for hardening of cement composites. In self-drying process of new cement matrix phase, water absorbed in aggregate moves into the new cement matrix because the chemical potential of moisture should be constant in the cement matrix-aggregate system. This could be the reason why compressive strengths decreased and carbonation speeds increased when recycled aggregate with high water absorption was used in mortar.

Ideas of total water proposed by Fumoto and Yamada (2004) assume that total water in mortar affects compressive strength. All the water could not move into cement matrix-aggregate interfaces. At 91days, in fact, mortar incorporating recycled fine aggregate with water-cement ratio of 0.4 was denser than that incorporating natural aggregate.

In case that ITZ bond develops significantly, strength of mortar hardly depends on pore volume between 50nm and 2μm in new cement matrix. This is because ITZ hardly exist in mortar and strength of new cement matrix phase is much stronger than that of aggregate.

Microstructure of new cement matrix phase in mortar incorporating recycled fine aggregate appeared to be important factor in evaluating properties of hardened mortar.
CONCLUSION

This paper reported the relationship between microstructure of new cement matrix phase and performance of mortar incorporating recycled fine aggregate. The results of this study were summarized as follows.

(1) Compressive strength of mortar incorporating recycled fine aggregate was lower than that incorporating natural aggregate. The higher water absorption of aggregate was, the lower compressive strength was. Carbonation speed of mortar incorporating recycled fine aggregate was larger than that with natural aggregate.

(2) There is correlation between performance of mortar with different types of aggregates and cement-total water ratio. Pore volume between 50nm and 2μm in new cement matrix phase became larger according to increasing total water-cement ratio.

(3) Microstructure of new cement matrix-aggregate interfaces was porous and loose when recycled aggregate with high water absorption was used in mortar. Compressive strength and carbonation speed could be evaluated by pore volume between 50nm and 2μm in new cement matrix phase. Porous structure of new cement paste can be attributed to movement of water absorbed in aggregate.

REFERENCES


