Influences of Waste Concrete Powder on the Strength Development and Hydration Products of Mortar Containing Fly Ash

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During recycling waste concrete, a large amount of waste concrete powder (WCP) is generated. However, efficient utilization of WCP remains an unresolved issue. This paper investigates the influences of WCP on the properties and hydration products of cement mortar containing fly ash (FA). This study used two different types of WCPs. One was made from an ordinary Portland cement mortar, and the other was derived from a Portland cement mortar. WCP replaced 10%, 20%, and 30% of FA. The water requirement, setting time, flow index, strength, hydration products, and microstructure of FA mortar incorporating WCP were investigated. Test results indicate that the WCP has no significant influence on the performances and hydration products of FA mortar. By adequately combining WCP and FA, the FA mortar with required performances could be reached.

Keywords: Fly ash; Waste concrete powder; mortar; hydration products; strength

1. INTRODUCTION

It is reported that waste concrete is the primary component of total solid waste produced in the world, which creates a severe ecological and environmental problem [1]. Recently, recycling of waste concrete to ensure environmental sustainability has become a global issue [2-5]. Using this waste as a recycled concrete aggregate (RCA) to substitute natural aggregate is of great significance for environmental protection and sustainable development [6–11]. However, these studies mainly focus on the coarse RCA and fine RCA. Only limited literature has reported the fresh and hardened performances of concrete incorporating WCP, which is a by-product of the process of producing RCA [12].

WCP is a fine powder fraction from the demolition waste of old concrete. It takes about 10 ~ 20 % of the old concrete [13]. The utilization of WCP in cementitious materials is beneficial and necessary for environmental protection [14 - 17]. Florea et al. reported the possibility of replacing partial cement in concrete with WCP [18]. They found that WCP to be a useful supplementary cementitious material (SCM), especially when using blended cement. Kim et al. [14], also found that WCP can replace partial ordinary Portland cement in producing self-consolidating concrete. Liu et al. studied WCP recovered by the dust collection system [19]. They found that WCP can be used as a supplementary cementitious material for concrete because of its pozzolanic activity. Prošek et al. suggest that WCP can be incorporated into cementitious materials containing FA or blast furnace slag in large amounts [20].

Fly ash, a solid waste produced by coal-fired power plants, is looked upon as an SCM in cement concrete [21]. The substitution of cement with FA not only reduces energy production but also reduces carbon dioxide emissions during cement production [22]. Besides, FA improves workability, mechanical strength, and durability of cement concrete, but reduces the heat of hydration of cement [23-27]. Nevertheless, the availability of FA is limited. There is an urgent need to use new supplementary cementitious materials, such as WCP to replace FA. Furthermore, considering the variety of WCP sources that would be available, an investigation of the influences of different WCP is required to evaluate these recycling materials.

This study focuses on the feasibility of producing FA mortar incorporating WCP. The influence of WCP on the water requirement, setting time, flow index, and strength development of FA mortar were measured. The microstructure and hydration products of the fly ash mortar with WCP were also investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), differential thermal gravity (DTG) and thermal gravimetric (TG) analyses.

2. MATERIALS AND METHODS

2.1. Materials

Portland cement (P·I 42.5), FA, two kinds of WCPs (WCP-1 and WCP-2), and silica sand were used in this work. FA, WCP-1, and WCP-2 were selected as supplementary cementitious materials. The WCP-1 and WCP-2 were prepared by crushing and grinding the hardened Portland cement mortar and ordinary Portland cement mortar. The hardened cement mortar was produced at a water to cement ratio of 0.5 and cured in a moisture room for 28 days.

The particle size distribution (PSD) of the WCP-1, WCP-2, and FA were measured using a laser diffraction spectroscopy (Malvern Mastersizer model MS 2000). The PSD of WCP-1, WCP-2, and FA is shown in Fig. 1. The d (0.5) of WCP-1, WCP-2, and FA is $18.936 \,\mu\text{m}$, $20.403 \,\mu\text{m}$, and $12.731 \,\mu\text{m}$, respectively. It can be seen from this

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figure that WCP-1 and WCP-2 have larger particles than those of FA.

The chemical composition of WCP-1 and WCP-2 was determined by using an X-ray fluorescence analyzer (PANalytical B.V. model Axios advanced). Test results are listed in Table 1, where the chemical composition of FA is also shown for comparison. It can be seen from this table that the dominant elements of WCP are Si and Ca, representing about 75 % of the total mass, while the main elements of FA are Si and Al, which count for about 80 % of the total mass.



Fig. 1. PSD of WCP and FA by laser diffraction

Mineral constituents of WCP-1 and WCP-2 are analysed by XRD. As shown in Fig. 2, the main mineral constituents of WCP are calcium carbonate, C_2S , calcium hydroxide (CH), and quartz. The C_2S phase is unhydrated cement in the WCP specimens. The existence of unhydrated clinker may contribute to the strength development of cementitious materials. CH is typical in hardened cementitious materials, which indicate the hydrated clinker, and hydration products of cement constitute a significant component of WCP [28, 29].

Table 1. Chemical composition of WCP-1, WCP-2, and FA

Chemical compound	WCP-1, %	WCP-2, %	FA, %
SiO ₂	29.74	29.68	45.29
Al ₂ O ₃	3.17	3.03	22.13
Fe ₂ O ₃	3.77	3.76	12.44
CaO	47.22	46.26	3.37
MgO	1.24	1.03	0.87
Na ₂ O	_	0.13	1.42
K ₂ O	0.54	0.68	1.71
TiO ₂	0.27	0.36	4.03
MnO	0.17	0.13	0.13
P2O5	_	0.14	0.30
SO ₃	1.83	2.34	2.89
CO ₂	11.81	12.19	4.93
Others	0.24	0.63	0.48

Calcite in the XRD pattern comes from the carbonate generated during the grinding, which increases the LOI of

WCP. Quartz is derived from the crushed fine aggregates. Furthermore, it can be observed a weak amorphous phase about 30° in this figure.

2.2. Methods

As shown in Table 2, an experiment was designed to investigate the influences of WCP content and type on the fresh state and hardened state performances of FA mortar. The ratio of FA to binder (the total of cement, FA, and WCP) at a constant value of 0.5. Three different dosages of WCP-1 and WCP-2 were used in the mixes. The ratio of WCP to SCMs (the total weight of FA and WCP) is 0, 10, 20, and 30 percent, respectively. All the rations are by weight. The flow index of FA mortar with WCP was tested according to ASTM C 1437. The mortar was prepared in prismatic molds $(40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}).$ The strength of specimens was measured after 7, 28, and 90 days of storage in a moisture room $(20 \pm 2 \,^{\circ}C)$, RH > 95 %). Vicat tests were performed to investigate the influences of the WCP content on the standard consistency and the setting time of fresh FA pastes.



Fig. 2. XRD pattern of WCP-1 and WCP-2

Table 2. Mixture proportion of specimens

Mixture	PC, g	FA, g	WCP-1, g	WCP-2, g	W, g	Sand, g
M-0	225	225	0	-	225	1350
M-1	225	202.5	22.5	-	225	1350
M-2	225	180	45.0	-	225	1350
M-3	225	157.5	67.5	-	225	1350
M-4	225	202.5	-	22.5	225	1350
M-5	225	180	_	45.0	225	1350
M-6	225	157.5	_	67.5	225	1350

A SEM (JSM-IT300) was used to study the morphology of WCP and FA particles. The morphology of hydration products and the microstructure of the specimens were also observed by SEM. XRD patterns were obtained a D8 Advance Diffractometer (Bruker-AXS). Operating conditions were set at a 40 kV and 30 mA using a Cu Ka X-ray source. An analysis from 5 °C to 6 0°C is carried out at a speed of 0.5°C/min. Both DTG and TG were performed on the specimens using a NETZSCH STA 449F3. The DTG depicts the thermal decompositions of phases in the specimens, while TG simultaneously determines the mass loss of a specimen with the

temperature. The thermal analysis was performed in a nitrogen atmosphere at 20 $^{\circ}$ C per minute from room temperature to 1000 $^{\circ}$ C.

3. RESULTS AND DISCUSSION

3.1. Morphology of WCP particle

Fig. 3 describes the particle morphology of WCP-1, WCP-2, and FA. The particle morphology of WCP is very different from that of FA. WCP particles contain sharp edges and rough surfaces, which will increase the water requirement and thus decrease the flowability of cementitious materials. Furthermore, some cement paste from the original mortar is still attached to the surface of the stone powder particles. The enclosed paste is the main reason for the porous of WCP particles [14, 28, 30], which will increase the sorptivity coefficients and reduce the flowability of cementitious materials. It also can be observed that a lot of small particles are coated on the surface of WCP, which weakens the bond between the cement paste and WCP [31].



Fig. 3. SEM micrographs of WCP and FA: a – WCP-1; b – WCP-2; c – FA

3.2. Water requirement and setting time

Fig. 4 shows the water requirement of cement paste containing FA and WCP, respectively. The water requirement of the specimen increases almost linearly with the increase of WCP content. For instance, replacing 10%, 20%, and 30% FA with WCP-1 increased the water requirement of the specimen 0.5%, 1.4%, and 2.2%, respectively. The results occurred because the surface structure of WCP particles is coarser than that of FA particles. Furthermore, the fine capillary pores in WCP particles increase the sorptivity, thus increase the water requirement of specimens. It is noted that replacing 10% FA with WCP had few adverse influences on the water requirement of specimens. Replacing 10% FA with WCP-1 and WCP-2 only increased the water requirement of the specimen 0.5% and 0.7%, respectively.

The initial and final setting time of specimens is shown in Fig. 5 a and b, respectively. As shown in Fig. 5 a that with the increase of WCP-1 or WCP-2, the initial setting time shows a decreasing tendency. It can be seen from Fig. 5 b, the final setting time decreased with increasing content of WCP. For example, replacing 10 % FA with WCP-1 and WCP-2 decreased the final setting time of specimen 21.0 % and 23.5 %, respectively. This is due to the accelerating effects of WCP on the hydration of cement [32]. The addition of WCP in specimens increases the nuclei for the crystal growth of hydration products, which accelerate the structure formation process.



Fig. 4. Water requirement versus WCP content for specimens



Fig. 5. Initial and final setting time versus WCP content for specimens: a – initial setting time; b – final setting time

3.3. Flowability

Fig. 6 shows the flow index of FA mortar containing WCP. Compared with the control specimen (FA mortar without WCP), replacing 10 % FA with WCP has no significant adverse effect on the flowability of FA mortar.



Fig. 6. Flow index versus WCP content for specimens

Replacing 10 % FA with WCP-1 and WCP-2 decreased flow index, only 0.42 % and 0.61 %, respectively. After that, the flow index of FA mortar rapidly reduced as the WCP content increased. For instance, replacing 20 % and 30 % FA with WCP-1 decreased flow index of 3.60 % and 6.04 %, respectively. This is because the rough surface of WCP particles absorbs part of the water and consequently reduces the flowability of FA mortar [14]. Furthermore, as shown in Fig. 2, the roundness and sphericity of WCP particles are significantly lower than that of the fly ash particles, which also reduces the flow index of FA mortar containing WCP. It was thought that the spherical shape of particles might affect the rheology of cementitious materials [33].

3.4. Strength development

Fig. 7 shows the flexural and compressive strength of specimens containing WCP at 7, 28, and 90 days. It shows that the flexural and compressive strength of all specimens increases with curing ages. For instance, M-1 exhibited flexural and compressive strength of 9.1 MPa and 49.9 MPa at 90 days, respectively, which is higher than that of at 7 and 28 days.

It can be seen from Fig. 1 that most particles of FA are finer than that of WCP. Hence, the microstructure of mortar containing FA may be more compact than that of mortar containing WCP. However, compared with control mortar (M-0), replacing 10 % ~ 20 % FA with WCP did not adversely affect the strength of mortar at early-ages. Both the flexural and compressive strengths of M-1, M-2, M-4, and M-5 are equivalent to that of M-0 at 7 and 28 days, approximately. This may be due to the accelerating effects of WCP. It is reported that the C-S-H in WCP will act as a nucleus for further C-S-H formation, therefore accelerate the hydration reaction of clinker [34, 35]. After 28 days, both the flexural and compressive strengths of M-0 are slightly higher than those of M-1, M-2, M-4, and M-5 due to the secondary reaction of FA. M-3 and M-4 exhibited lower flexural and compressive

strengths than those of M-0 at all curing ages. The lower strength of FA mortar, incorporating 30 % of WCP, can be attributed to the ITZ between the original aggregate and the hydrated cementitious materials [36]. The ITZ will significantly affect the strength of FA mortar containing WCP.



Fig. 7. Strength versus WCP content for specimens at 7, 28, and 90 days: a – flexural strength; b – compressive strength

Mortar incorporating a different type of WCP exhibited equivalent flexural and compressive strength at all ages, which indicates that the type of WCP has no significant effects on the strength of cement mortar.

3.5. XRD analysis

Fig. 8 shows the XRD analysis results for M-0 and M-1 for 7 and 90 days, respectively. The diffraction peaks of quartz, calcite, and CH can be observed in the figure. Due to the presence of silica sand in the specimens, the mineral with the highest peak intensities is quartz. C-S-H cannot be identified in the XRD pattern because of its low crystallinity. Ettringite also cannot be identified on the XRD spectra. As shown in Fig. 8 a, the intensity of CH in the two specimens is almost the same. The same trend was also found in Fig. 8 b. The peak of CH was considered to be the primary indicator of the performances of specimens in this study. Therefore, the hydration products in FA

mortar without WCP and FA mortar with 1 % WCP do not show significant differences both at 7 and 90 days.



Fig. 8. XRD patterns of specimens: a - 7 days; b - 90 days

3.6. DTG-TG analysis

To quantitatively analyse the influences of WCP on the hydration products of specimens, M-0, and M-1 at 7 and 90 days were selected to conduct the DTG-TG analysis.

Fig. 9 gives the DTG and TG curves of M-0 and M-1 for 7 and 90 days, respectively. DTG curves show three main endothermic peaks.

Correspondingly, three rapid weight loss can be observed in the TG curves. Mass loss between 105 °C and 400 °C represents the dehydration of C-S-H, and C-A-H et al., between 400 °C and 500 °C indicate the dehydration of CH. De-carbonation of CaCO₃ occurs at a temperature between 600 °C and 700 °C. The CH content of specimens can be calculated according to Eq. 1 [37]:

$$CH(\%) = WL_{\rm CH}(\%) \cdot MW_{\rm CH}/MW_{\rm H},\tag{1}$$

where MW_{CH} is the mass loss of CH, %; MW_{CH} is the molecular weight of *CH*; MW_{H} is the molecular weight of H₂O.

As expected, the CH content of the two specimens increases with increasing curing ages. M-0 and M-1 increased CH content of 57.6% and 41.9% at 90 days than that of at 7 days, respectively. This indicates an increase in the hydration degree of the cement. This phenomenon is consistent with the strength development shown in Fig. 7.



Fig. 9. DTG-TG curves of specimens: a-7 days; b-90 days

Besides, the CH content of M-0 is slightly higher than that of M-1 at 7 days, which indicates the accelerating effects of WCP on the hydration of clinker at early-ages. The accelerating effects can be attributed to the complicated surface structure of WCP particles, which increases the nuclei for the CH growth. However, the CH content of M-1 is slightly lower than that of M-0 at 90 days. The decrease in CH intensity can be attributed to the reaction of CH with FA at later ages.

3.7. SEM imaging analysis

To observe the microstructure of specimens, M-0 and M-1 were also investigated by SEM. Fig. 10 a and b show SEM micrographs of the FA mortar without WCP and FA mortar with 10 % WCP-1 at 7 days.

Fig. 10 c and d show the SEM micrographs of the FA mortar without WCP and FA mortar with 10 % WCP-1 at 90 days. C-S-H, plate-like portlandite, and needle-like ettringite can be observed in the SEM micrographs. Also, the microstructure of M-1 is slightly looser than that of M-0, both at 7 and 90 days, which may be due to the larger size of WCP particles than that of FA particles.

4. CONCLUSIONS

The feasibility of utilizing WCP in FA mortar was studied. Test results for the fresh and hardened performances of FA mortar as well as hydration products are presented and discussed in this paper. Based on the investigation, the following major conclusions can be drawn: 1. WCP particles contain sharp corners and irregular edges, which is very different from that of FA particles. Besides, old hydration products from the original mortar remain attached to the surface of the stone particles.





d _____ 10μm

- Fig. 10. SEM micrographs of specimens: a-fly ash mortar without WCP-1 at 7 days; b-fly ash mortar with WCP-1 at 7 days; c-fly ash mortar without WCP-1 at 90 days; d-fly ash mortar with WCP-1 at 90 days
- 2. It was demonstrated in this study that WCP could replace up to 10 % FA in mortar without a significant loss of fresh and hardened performances. Further increased WCP content results in the increasing water requirement but decreasing the flow index, setting time, and strength. The increased water requirement and the reduced flow index can be ascribed to the irregular morphology and rough particle surface of WCP. The decreased setting time is due to the accelerating effect of WCP. The decreased mechanical strength results from the ITZ between the aggregate and the hardened cementitious materials in WCP.
- 3. The XRD, TG-DTG, and SEM analysis confirm that replacing 10 % FA with WCP did not significantly influence the hydration products and microstructure of mortar. This can be due to the hydration reaction of unhydrated cement grains in WCPs and the accelerating effects of WCP on the hydration of cement.

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