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Fresh properties of cement pastes/mortars incorporating waste glass powder and cullet

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Abstract: This work was aimed at studying the fresh properties of cement-based mixtures containing waste glass powder (WGP) and waste glass cullet (WGC) obtained from crushed post-consumer beverage bottles. The experimental results show the setting time of the WGP modified pastes were prolonged due to the lower rate of hydration. Irrespective of the addition WGP, a good correlation between the early hydration characteristics and the stiffening or flowability can be established. However, during the first 30 minutes of hydration, the fresh properties of the WGP modified mortars were predominantly dependent on the particle size and the morphology of the WGP investigated.

Keywords: Fresh property; Waste glass powder (WGP); Heat of Hydration; Stiffening; Flowability

1 Introduction

Waste glass has become an important component in the municipal solid waste (MSW) stream. According to the Hong Kong government [1], the amount of waste glass disposed of at landfills was about 300 tons per day in 2014. Due to the low commercial value and the lack of a glass manufacturing industry in Hong Kong, the recovery rate of waste glass is less than 10%. For this reason, it is very essential to find ways that can increase the recycling rate of waste glass and reduce the amount of waste glass requiring disposal at landfills effectively.

In Hong Kong, due to the lack of sources of pozzolanic materials (such as coal fly ash, granulated blast furnace slag or silica fume), the use of glass powder as a partial replacement for cement in concrete can be attractive. This would not only reduce cement consumption (lower the production cost and the emission of CO_2) and the amount of waste requiring disposal, but also reduce the reliance on the external sources of pozzolanic materials.

Generally, there are two main forms for the applications of waste glass in concrete: one is using the waste glass cullet (WGC) as aggregates, the other is using waste glass powder (WGP) as a supplementary cementitious material (SCM). As reported, the use of WGC as aggregates to produce concrete paving blocks has been successfully applied in Hong Kong [2], and it has been demonstrated that it is also feasible to utilize the WGC to prepare self-compacting concrete [3] and mortars [4]. However, a major concern with the use of glass aggregates in cement-based construction is the deleterious alkali-silica reaction (ASR) between the silica-rich glass and the alkali in the concrete or mortars [5]. Also, a high replacement level (more than 20%) of waste glass as aggregates in concrete could lead to a reduction of strength [6][7]. Therefore, Pasetto and Baldo [8] recently designed a controlled low-strength road sub-base containing waste glass, fly ash and other recycled materials and stated that the fly ash used probably contributed to preventing the ASR damage. Furthermore, WGC was further grinded to become WGP to prevent the ASR problem as WGP with proper particle size possesses pozzolanic activities and can be used as a cement replacement [9-11]. Shao et al. [9] and Shi et al. [10] also showed that the addition of finer WGP had beneficial effects in inhibiting the ASR and enhancing the strength. Therefore, combined use of WGC and WGP is considered to have good application values.

Traditional SCMs, namely fly ash, granulated blast furnace slag, metakaolin and silica fume, are used to improve the strength and durability performance of concrete [12-16]. Also, several related studies reported that these SCMs have significant effects on the fresh properties of concrete [16-20]. According to earlier publications, the inclusion of glass aggregate in concrete mixes is able to improve the flowability [21][22] or reduce superplasticizer dosages [4]. But, as the percentage of recycled glass cullet in concrete increased, severe bleeding and segregation were observed due to the smooth surface and non-absorbent nature of glass cullet [23]. Whether the excessive use of glass materials in concrete result in a less cohesive mix may be a concern. Therefore, it is necessary to study the influence of WGP as cement replacement on the fresh properties of cement mixes, especially when the natural aggregates are fully substituted by the WGC.

From economic and operational considerations during practical production, this study first focused on studying the setting time and hydration process of cement pastes containing WGP with different fineness, ground by using a ball milling approach for specified periods (0.5h, 1h, 2h, 4h respectively). On this basis, the early stiffening behavior and flowability of architectural mortars incorporating both WGP and WGC were further investigated in this study.

2 Experimental programs

2.1 Materials

Cementitious materials

White ordinary Portland cement (WPC) was used in this study to provide a better aesthetic effect of the produced architectural mortars. Besides, WGP of four different fineness, grinded from the WGC by using a laboratory ball milling machine for 0.5h, 1h, 2h and 4h respectively (namely as WGP-0.5h, WGP-1h, WGP-2h, WGP-4h), were adopted to partially replace the WPC. The detailed chemical compositions of the WGP and WPC (TAIHEIYO Cement Corp., Japan) are listed in Table 1.

As regards the particle size distribution, a comparison of different types of WGP with the WPC are presented in Fig. 1. The scanning electron micrographs of WGP-2h (glass cullet milled for 2h) particles are shown in Fig. 2, which exhibit a smooth surface texture and irregular shape.

Table 1
Chemical composition of WPC and WGP (ms %).

	WPC	WGP
SiO_2	21.36	73.5
Al_2O_3	5.27	0.73
Fe_2O_3	0.2	0.38
CaO	67.49	10.48
MgO	1.14	1.25
K_2O	0.077	0.69
Na_2O	0.048	12.74
TiO_2	0.14	0.087
SO_3	2.6	-
Loss in ignition	1.58	-

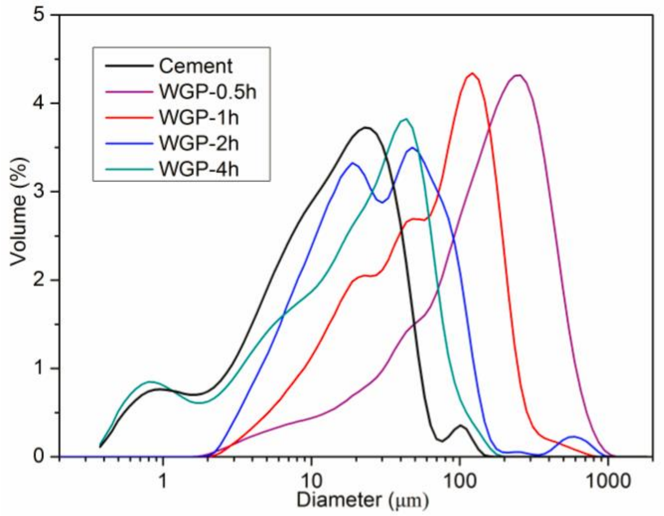
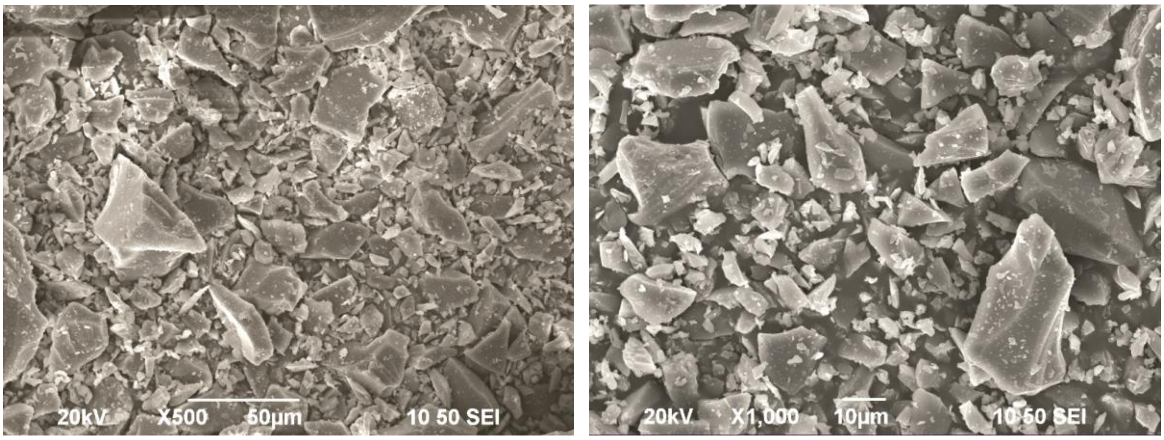


Fig. 1 Particle size distributions of cement and WGP



(a) (b)

Fig. 2 Morphology of WGP-2h

Aggregates

WGC with a mixed colour, obtained from a local glass recycling plant, was derived from crushing of collected post-consumer beverage bottles. The washed glass cullet was oven-dried for a minimum of 24h at 105 ℃ before use. The gradation and density of WGC used in this study is shown in Table 2.

Table 2

Grading and density of WGC

Sieve size (mm)	10	5	2.36	1.18	0.6	0.3	0.15	Relative density (g/cm^3)
WGC (% passing)	100	99.7	73.1	44.4	20.8	6.1	1.2	2.49

2.2 Mix design

According to the previous study for the production of architectural mortar, the binder-to-aggregate and water-to-binder (*w*/*b*) ratios were adopted at 1:2 and 0.4, respectively. A superplasticizer (SP) ADVA-109 was employed to achieve the desired workability. Additionally, the use of SP was at 0.6% by weight of the cementitious material. The mix proportions of the mortars are listed in Table 3. It should be noted that the control mortar (M-Control) without WGP was prepared as a reference mixture and the other four groups were proportioned with 20% of cement replaced by WGP obtained separately after 0.5h, 1h, 2h, 4h of grinding (namely as M-WGP0.5h, M-WGP1h, M-WGP2h, M-WGP4h).

Table 3

Mix proportions of different mixtures.

Mix	WPC:WGP:WGC	SP (%)	*w*/*b*
M-Control	1:0:2	0.6	0.4
M-WGP	0.8:0.2:2	0.6	0.4

2.3 Test methods

2.3.1 Setting time

The setting time of pastes with and without WGP was assessed in accordance with BS EN 196-3: 2005+Al: 2008 [24]. In order to be comparable with the *w*/*b* of the mortars, the *w*/*b* of the pastes was also brought to 0.4. The pastes prepared with and without WGP were named P-Control, P-WGP0.5h, P-WGP1h, P-WGP2h, P-WGP4h, respectively.

2.3.2 Heat of hydration

The heat hydration test was utilized to investigate the incorporation of WGP on the early hydration of cement. During the test, 20% of WGP was used to replace WPC. The cement and WGP were firstly mixed in an insulated container thoroughly, then 60 grams of this mixture were mixed with 24 grams of water (w/b = 0.4) for 2 minutes in the container used for the heat of hydration test. Having completed the above steps, the container was sealed and then placed into the isothermal calorimeter (Calmetrix I-CAL). The instrument was set to a constant temperature of 20 ℃. After 48h, the measurement was stopped and the data were exported and analyzed.

2.3.3 Stiffening

The purpose of this test was to determine the degree of the early stiffening of the cement mortars. The cement mortars containing different particle sizes of WGP as SCM and WGC as aggregates were prepared and tested according to ASTM C359 [25] As the early hydration of mortar was low, the testing time was extended to up to 30 mins. The method uses a modified Vicat apparatus to measure the resistance to penetration at 5 min, 8 min, 11 min, 15 min, 20 min, 25 min, and 30 min after the initial mixing. Every test value reported is the average of two measurements. The details of the mix proportions are shown in Table 3.

2.3.4 Flowability

The flowability of the mortar was determined according to BS EN1015 [26]. Table 3 shows the mix proportions. The flow value was measured by means of a mini-slump flow cone with a 100 mm internal diameter on a 250 mm flow table disc. The mould was first filled with the fresh mortar, and was then raised vertically to spread out the mortar on the disc by jolting the flow table 15 times at a constant frequency. Two perpendicular spread diameters of the mortar before and after jolting were measured and recorded.

3 Results and discussion

3.1 Particle size

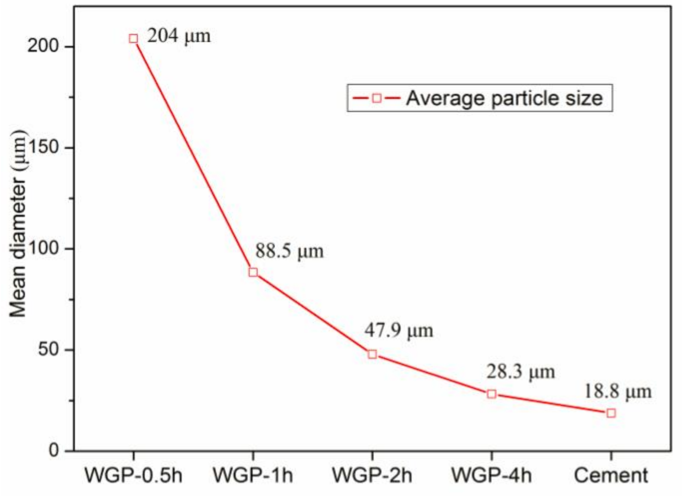


Fig. 3 Mean diameters of WGP and cement

Apparently, it can be seen from Fig. 3 that as the milling time increased, the average particle size of WGP decreased. Compared to the mean diameters of cement (18.8 μm), the particle sizes of WGP were larger, especially for the WGP-0.5h (204 μm). However, there was a drastic reduction in size when the milling time was increased from 0.5h to 1h. Then, the average particle size only reduced relatively slowly as milling time was further increased, which indicates the grinding efficiency was greatly reduced after 1h.

3.2 Setting time

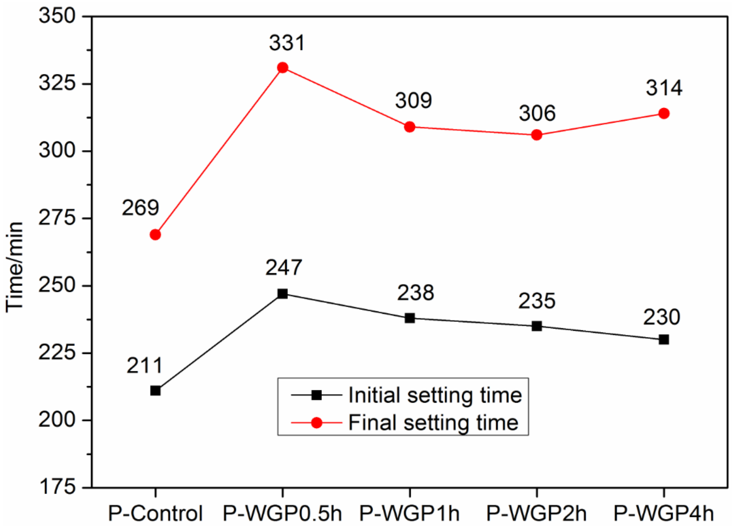


Fig. 4 Setting time of cement pastes with and without WGP

Fig. 4 shows the influence of the WGP sizes on the setting time of the pastes. The results reveal that longer initial and final setting times were recorded for the mixes prepared with the WGP regardless of the particle size of the powder. Noticeably, it can be seen that the WGP ground for 0.5h had a significant influence on the setting time. Compared to that of pure cement, it delayed the initial and final setting time by around 17% and 23% respectively. This may be caused by the larger particle size and the smooth surface of the glass particles, which rendered more water available for the initial hydrolysis and an increase of the effective water-to-cement (*w*/*c*) ratio. As known, cement pastes with a higher *w*/*c* ratio, take longer for the cement hydration products to form a rigid structure [27], thus the initial set and final set times were extended. The results also showed the setting time did not decrease significantly even when the waste glass was ground for more than 1h. These phenomena should be attributed to the similar average particle size of these WGPs compared to that of WGP-0.5h.

3.3 Heat of hydration

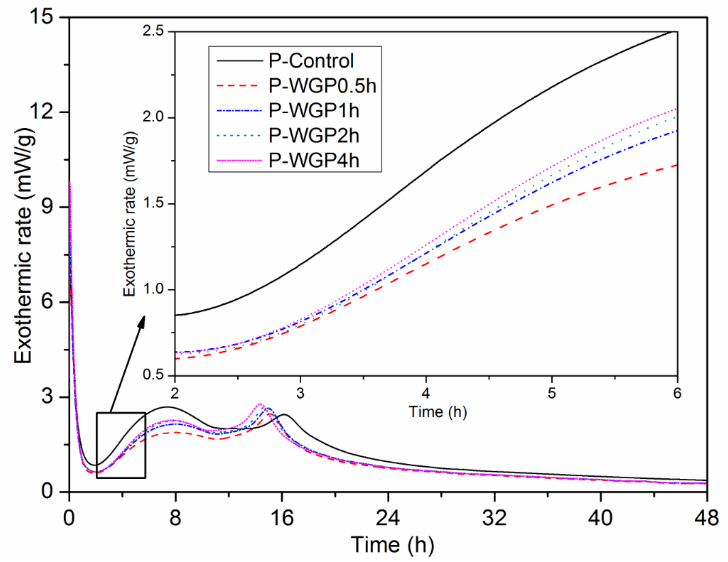


Fig. 5 Heat evolution rate of cement pastes with and without WGP

Based on a previous published literature [28], for the Portland cement, the acceleration period of heat evolution usually begins at approximately 2h after mixing with water and ends approximately 6h later. This period is consistent with the setting time of cement. Therefore, it is of interest to explore the relationship between the hydration process and the setting time when different fineness of WGP are used as cement replacements.

Fig. 5 shows the heat evolution curves of the cement pastes that were prepared with 20% replacement by different sizes of WGP. Regardless of the particle size of WGP, it can be easily noticed that the heat evolution rates of the WGP modified pastes were lower than that of the pure cement. This demonstrates that the WGP has a lower reactivity at the early stages of hydration. The heat evolution rate of WGP-0.5h mix was the lowest among the five mixtures probably due to its coarser particle size (lower surface area). However, the heat evolution rates of pastes with WGP-2h and WGP-4h were only slightly higher than that of paste containing WGP-1h. This suggests that when the particle sizes of the WGP were reduced to less than 88 μm (refer to Fig. 3), the contribution to hydration by the WGP was less apparent. It is consistent with the results of the setting time measured, which showed similar setting time although the fineness of WGP increased.

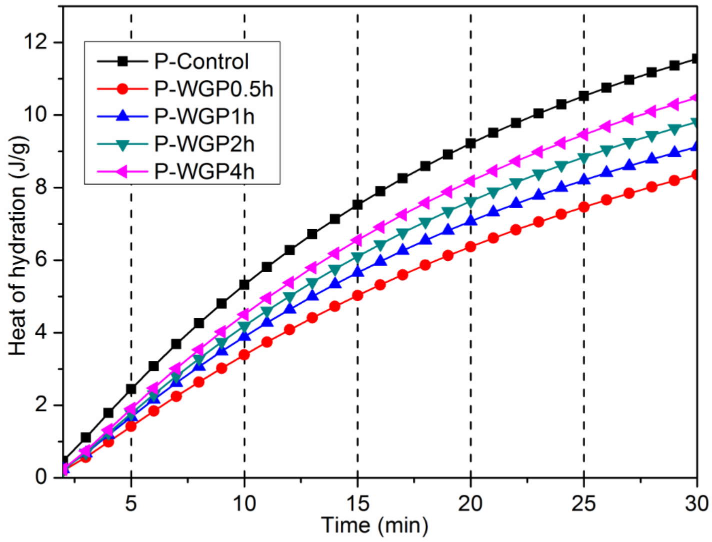


Fig. 6 Hydration heat of WGP blended pastes within first 30 mins

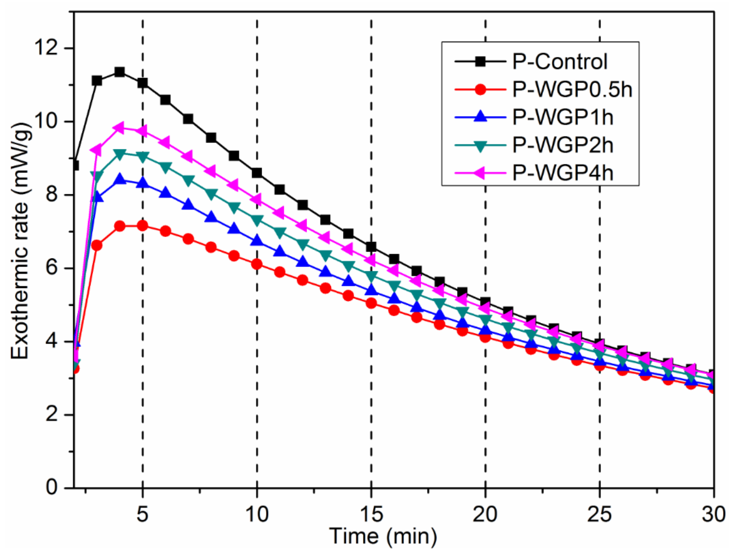


Fig. 7 Exothermic rate of WGP blended pastes within first 30 mins

A close examinations of the hydration within the first 30 mins are shown in Figs 6 and 7. Fig. 6 presents the influence of WGP with different fineness on the initial hydration heat. The curves for the pastes both with and without WGP exhibited an increase in the cumulative total heat output during the first 30 mins. This is mainly resulted from the hydration of cement, since most of the reactions occurred during the hydration are exothermic. When the WGP was used to replace cement, the heat of hydration decreased due to the dilution effect. It is further confirmed by Fig. 7 that the heat evolution rate decreased consistently for all the mixes. It can be also noted that the heat evolution rate increased with decreasing particle size of the WGP, although the total heat of hydration was still very low compared to the total normal heat output (300-500 J/g) [28]. In Fig. 8 there is a rapid evolution of the heat culminating in a peak within 5 mins. Generally, the first few mins of hydration is referred to as the initial reaction of hydration between the free water and tricalcium aluminate (C_3A) as well as tricalcium silicate (C_3S) phases [29][30]. In particular, it is worth pointing out that a low rate of hydration heat is the characteristic for the subsequent dormant period when the rate of reaction slows down dramatically after a short burst of rapid reaction. The fact of slowdown in reaction has been discussed by several researchers [31-33].

3.4 Stiffening

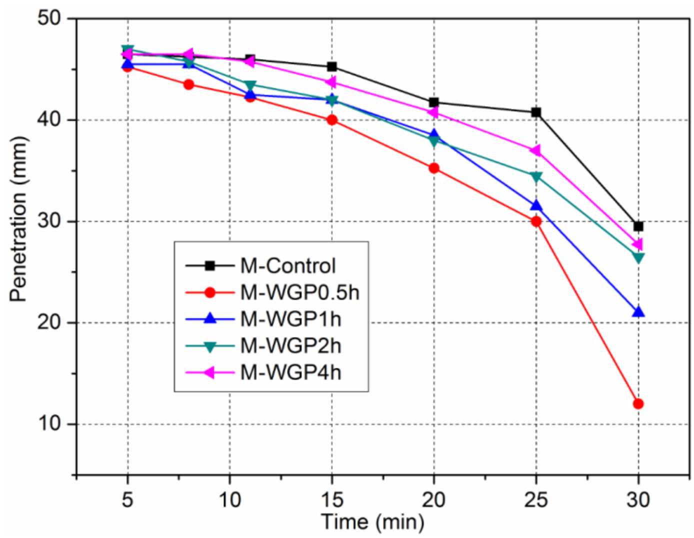


Fig. 8 Effect of WGP with different fineness on stiffening of mortar

Both the setting and the hardening of the cement paste are the result of a series of simultaneous and consecutive reactions between water and the constituents of the cement. The stiffening test was used to determine the rate of strengthening of the WGP blended mortars (Fig. 8). The results show that the penetration of all the mortars decreased as time increased. The primary source of the stiffening was due to hydration of cement. As the hydration proceeded, the cement particles were gradually changed to their hydration products, forming mostly a poorly crystallized solid (so called C-S-H gel). Then, with the increase in volume of C-S-H within the boundaries of specimen, interlocking laths were developed resulting in a reduction in the overall porosity of mortar.

When WGP was used as 20% substitution of cement, an enhancement in the stiffening rate was observed. This result seems to be contrary to the results of the setting time test where a retarding effect of WGP was observed. The primary reason for this is: in this study, setting time was largely hydration dependent, while the stiffening was highly dependent on the particle characteristics due to the slow early hydration of WGP. During the course of the hydration reaction, setting took place during the acceleration period. Meanwhile, the minerals in a cement grain began to hydrate rapidly, reaching the maximum rate at the end of the acceleration period, which corresponded with the second peak of heat evolution curve (see Fig. 5). Therefore, the setting of the cement paste was generally attributed to the formation of C-S-H [29][30]. However, the stiffening (happened within 30 mins) occurred before the early dormant period. During this early stage of the hydration process, there was a rapid evolution of the heat culminating in a peak within a few mins, but the total heat evolved during this period was very low relative to that of the acceleration period, hence hydration had only a minor contribution to the early stiffening of the mortar. Therefore, it implies that the stiffening in this stage depended primarily on the physical properties, presumably the particle characteristics.

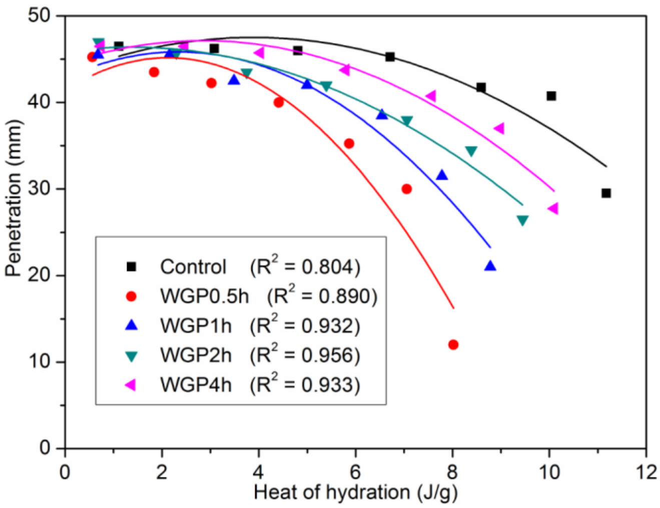
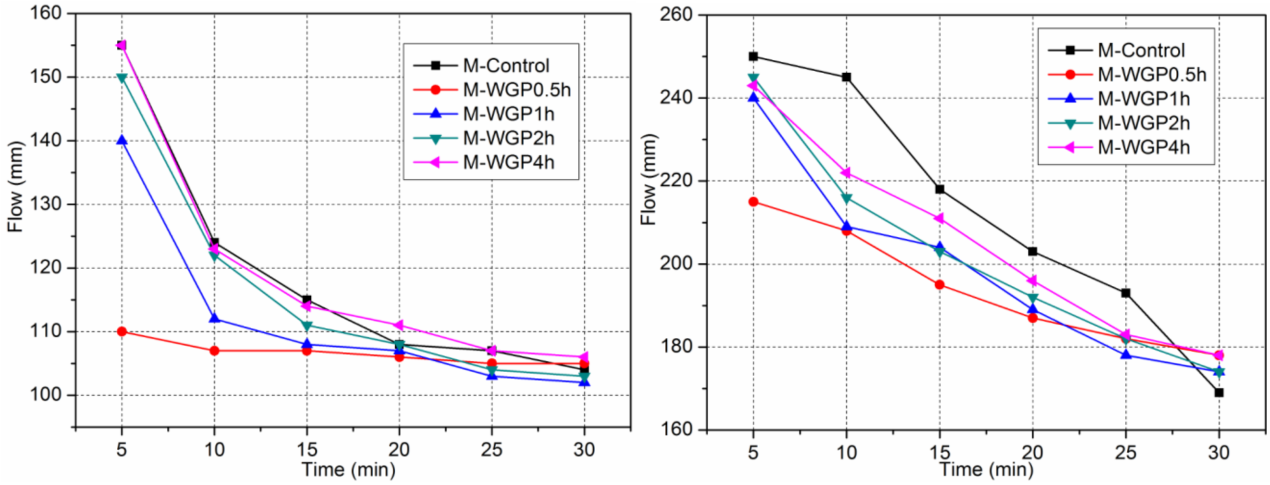


Fig. 9 Relationship between hydration characteristic

In order to clearly understand the effect of hydration on the stiffening of the mortar containing the WGP, the correlation was established (Fig. 9) between the heat of hydration and the needle penetration of the stiffening test of the blended mixtures within 30 mins. Five curves were fitted based on the results of five groups of test. It is obvious that the heat output of WGP-0.5h mixture was the lowest, but it had the highest resistance to penetration rather than the control mortar (pure cement with the highest heat output). This demonstrates that the stiffening of the mortar with the WGP was not mainly controlled by the hydration of cement. Therefore, it is suggested that the effect of stiffening at early ages was predominantly controlled by the physical properties of the cementitious materials, such as particle size distribution, surface area, surface morphology.

As mentioned before, the particle sizes of WGP used were larger than that of cement. In addition, the WGP was irregular in shape (as shown in Fig. 2) although the mean diameter was about 50 μm. This irregular shape provided interlocking between the powder particles to increase the resistance to penetration during the stiffening time test. Another observation is that the resistance to penetration decreased as the particle size of WGP decreased, which is consistent with the hypothesis that the interlocking of the bigger irregular shape particles provided better resistance to penetration. When the particle size of the WGP was similar to that of the cement grains, the stiffening time of the blended mortar was close to that of the control mortar.

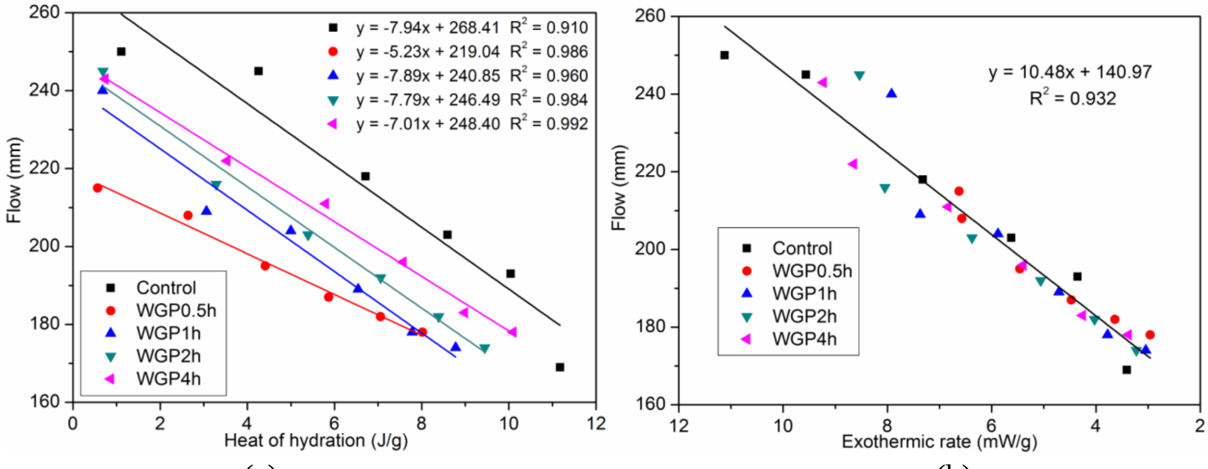
3.5 Flowability



(a) before jolting (b) after jolting

Fig. 10 Flow of mortars prepared with different fineness of WGP

The results of flow table test for the mortars are presented in Fig. 10. The test was run for a period of 30 mins similar to that of the stiffening time test. The results show regardless of the WGP fineness, the flow values gradually decreased with time. It is mainly due to the process of cement hydration. A closer examination of Fig. 10a shows that compared to the initial flow value, a sharp drop occurred at 10 mins after the dry cementitious materials were first mixed with water. This is due to the initial hydration reaction was about to end within 10 mins (based on the results of the rate of heat evolution, see Fig. 7). And it is obvious that the flow values of all the groups without jolting were similar after 20 mins. In comparison, the flowability of the mixes after 15 times of jolting (Fig. 10b) was consistent with that of the results of stiffening time. The WGP-0.5h mortar had the lowest flowability, while the reference mortar had the best flowability. The phenomenon can be attributed to the larger and irregular particles of the WGP, which would hinder the movement of the glass mortars.



(a) (b)

Fig. 11 Relationship between hydration characteristics and flowability of WGP mortar after jolting

Fig. 11a shows the relationship between the heat of hydration and the flow values. There were good correlations between the heat of hydration and flow values, indicating that the hydration process had some impacts on the flow of the mortar. It is confirmed from Fig. 11b, which shows that the flow values decreased with the decrease of the heat evolution rate. This is attributed to the fact a network structure gradually formed along with the hydration process.

However, as shown in Fig. 10b and Fig. 11a, larger particle sized WGP resulted in a reduction of the flow. This is mainly because, in the presence of the WGP, the fineness of the particle could act in a similar way as the filling effect in controlling the stiffening time, which suggested that, the influence of WGP fineness on the flowability remained a dominant factor. Conversely, the finer WGP might have contributed to improving the flowability compared to the coarser WGP. This phenomenon should be considered from two effects: one is the finer particles can optimize the gradation of the mixture, the other is the filling effect. The latter made the finer particles easier to replace the water which was enclosed in the voids of the flocculated structure. Then the released water can reduce the friction between the particles.

According to the above discussion, one can speculate that the particle size distributions of cement, glass powder and glass cullet play an important role in the hardening of the glass mortar. In addition, it is conceivable that a proper combination of finer and coarser particles may optimize the rheological properties of the mortar similar to the optimization of concrete workability by the grading of aggregates.

4 Concluding remarks

This study was devoted to evaluating the fresh properties of cement-based materials containing WGP as a cement replacement. Use of the WGP resulted in a reduction of heat of hydration within the first 30 mins and an extension of the setting time. However, it can be envisioned that, if the particle size of WGP is reduced to the same level with that of cement, the workability and the stiffening of WGP modified mortar are similar to that of the control mortar. Therefore, in terms of the fresh properties, it is feasible to produce architectural mortars incorporating WGP and WGC. The results are summarized as follows:

1. The use of WGP as a cement replacement in cement paste increased the setting time attributed to the higher effective *w*/*c* ratio as compared with the control mix. This may be due to the smooth surface and negligible water absorption of the glass particles. At the same time, the presence of the relatively less reactive glass particles prevented the contact between the unhydrated cement particles and the water, leading to the weakening of the cement matrix at the early stage of hydration.
2. The result of the heat evolution rate during the acceleration period provided a good explanation for the extended setting time of WGP modified mixes. When WGP was used to replace cement, the heat of hydration decreased due to the dilution effect. However, the heat evolution rate at the early stage increased with the increase of WGP fineness.
3. The 20% substitution of cement by WGP caused an enhancement in the stiffening rate. The primary reason for this is that the stiffening within the first 30 mins was highly dependent on particle size and morphology of WGP. In addition, the stiffening of WGP blended mortar decreased as the particle size of WGP decreased. It was probably due to the reduction of particle size of WGP not only decreased the resistance to penetration but also released more water due to the filling effect, causing a decline of the friction between the WGP and the cement grains.
4. The hydration characteristics of the blended pastes had a good correlation with the flowability of WGP mortar, and the flow decreased with an increase of the heat output and decrease in the heat evolution rate. However, the flowability was largely controlled by the fineness of WGP. An increase of WGP fineness resulted in a reduction in the flow of the mortar with the WGP compared to that of control mortar.

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