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Pozzolanic reaction of the waste glass sludge incorporating precipitation additives

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Abstract. The waste glass sludge is a waste produced in the glass industry. It is in a dust form and disposed with water. In the disposal process, various cohesive agents are incorporated in order to precipitate the glass particles efficiently. In this paper, we investigate the pozzolanic reaction of the waste glass sludge incorporating precipitation additives experimentally. The consumption of calcium hydroxide, the setting time and the compressive strength and the pore structure were tested for two different types of the waste glass sludge depending on whether precipitation additives were used. It was found that the waste glass sludge incorporating the precipitation additives had a higher pozzolanic potential than the reference waste glass sludge without precipitation additives.

Keywords: waste glass sludge; precipitation additives; cohesive agent; coagulant aid; caustic soda; alkali activation; pozzolanic reaction;

1. Introduction

Various kinds of industrial wastes or by-products, such as silica-fume, fly-ash, blast furnace slag, etc., have been investigated as pozzolanic admixtures for various application in the construction industry (Yehia *et al*. 2015, Rashad *et al*. 2014, Divsholi *et al*. 2014, Bondar *et al*. 2014). The potential of a waste as a pozzolanic material can be checked by three crucial requirements: high silica content, amorphous crystalline structure and large surface area (Shao *et al*. 2000). According to ASTM C618-15 (2015) the sum of silicon dioxide, aluminum oxide and iron oxide should be greater than 70% for a class C pozzolan. Because of the high content of silica, glass wastes can also be considered as a pozzolanic admixture in the construction industry. Some of the literatures are available in this matter (Bazant *et al*. 2000, Shao *et al*. 2000, Schwarz *et al*.

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2008, Kim *et al*. 2014, Kim *et al*. 2015, etc).

The research for the use of waste glass for the construction industry goes back to the early 1970s, mainly to develop lightweight concretes by using glass aggregates (Malisch *et al*. 1970, Richards *et al*. 1971, Bynum *et al*. 1972, Johnston. 1974). In particular, Johnston (1974) reported that concretes mixed with waste glass aggregates were damaged by the alkali-silica reaction (ASR). After Johnston's work, many researchers investigated the alkali-silica reaction of glass aggregates mixed in concretes. Meyer *et al*. (1996a, b) tried different sizes of glass particles and found that if waste glass was used in the form of fine particles, there was no harmful effect to the strength of the concrete with waste glass. Bazant *et al*. (2000) developed a chemofracture mechanics theory to explain the size effect of the glass particles. Now it is understood that the fineness of glass particles is the one of the key parameters for their use as a concrete admixture.

Shao *et al*. (2000) used fine glass powder produced from wasted fluorescent lamps as a cement replacement, in which the average size of the glass particles was less than 38 μm. He reported that the strength of the concrete with a certain size of fine glass particles became very similar to that of the reference plain concrete in 90 days. Shi *et al*. (2005) also reported a similar result. They tested three different fineness of 262, 467 and 582 m^2/kg, in which 20% of the cement was replaced by the glass powders. The strength with the glass powder with the fineness of 467 m^2/kg was the same as the strength of the reference plain mortar.

All the researches do not produce the same result. Shayan *et al*. (2004) reported that the strength of a concrete with 10% of glass powder as a cement replacement was only 87% of the reference concrete at 28 days, in which the fineness was as very high as 800 m^2/kg. They explained that the low strength development might be because of the slow rate the pozzolanic reaction of the glass powder.

There are many different sources of waste glass such as container glasses, bottle and jar glasses, and plate glasses, etc. Of course their chemical composition would depend on the sources of the waste glass. The pozzolanic reaction of the glass powder may be influenced by the difference of the chemical composition. Although the pozzolanic reaction of glasses with different colors are found in the literature (Jin *et al*. 2000, Park *et al*. 2006, Dhir *et al*. 2009), the comparative research for the different sources of waste glass is limited.

Recently, Zi and coworkers investigated the pozzolanic reactions of two different sources of waste glass: the ball-milled glass powder produced from bottle and plate glass and the dried waste glass sludge powder (Korea Expressway Corporation Research Institute 2012, Kim *et al*. 2014, Kim *et al*. 2015). Although the fineness of the two glass powder was the same, they reported that the strength development of the concrete with the waste glass sludge powder was faster than the other although the chemical compositions of the two glass powders were similar to each other. They also reported beneficial effects of the waste glass sludge on the resistance to ASR and chloride ion penetration combined with freezing-thawing action.

A potential reason of the difference between the ball-milled glass powder and the waste glass sludge can be found in the disposal process of the waste glass sludge. Small glass particles produced in various manufacturing processes are disposed with waste water. In this disposal process, precipitation additives are incorporated in order to precipitate the glass particles efficiently within the mixture of glass particles and the waste water. The precipitated sludge of the mixture is called the waste glass sludge. Typically, these precipitation additives consist of cohesive agents and a coagulant aid. Polymer and aluminium sulfate, $Al_2(SO_4)_3$ are used as the cohesive agents in the glass industry. If the cohesive agents are incorporated into the waste water, the water becomes a strongly acidic solution. Therefore, a coagulant aid, caustic soda NaOH, is added to

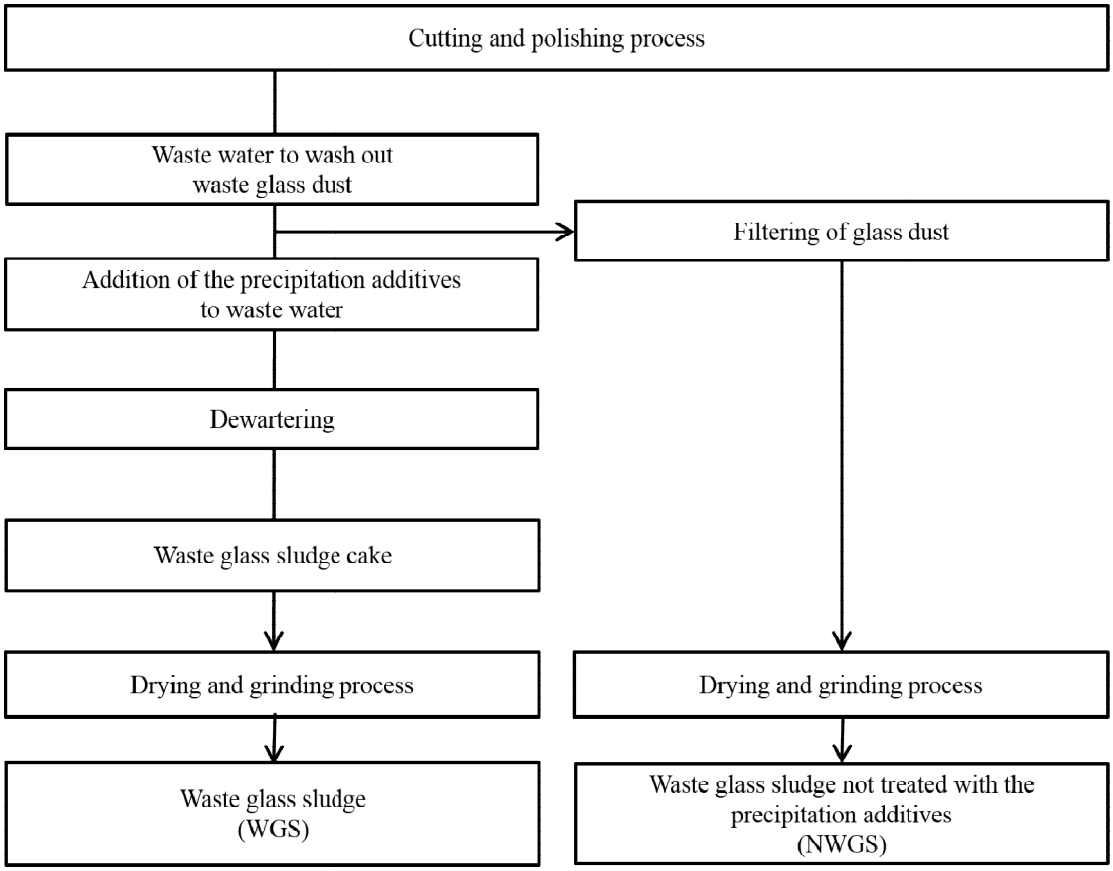

Fig. 1 WGS and NWGS formed in the manufacturing process of glass products

control the pH of the waste water. Note that the caustic soda is one of the popularly used alkali activators for pozzolanic reaction.

2. Materials and experimental programs

2.1 Preparation of WGS and NWGS

Fig. 1 shows the typical manufacturing process in the glass industry. A significant amount of glass dust is produced in the cutting and polishing process of glass products. The glass dust is washed out with water and disposed as the waste water. To expedite the precipitation of the glass dust or particles in the waste water, the cohesive agent is added. A coagulant aid such as caustic soda is added to control the pH. At the final step of the waste water treatment, the water is removed by a filtering process to form the sludge cake of the waste glass shown in Fig. 2(a). The powder for this experiment was obtained after drying the cake at 100℃ for twelve hours and grinding it. Note that because the water content in a grinding process should be less than 20%, the drying process is necessary (Kim *et al*. 2015). The sludge cake was ground by the Raymond

Fig. 2 Morphology of the waste glass sludge (Kim *et al*. 2015): (a) sludge cake and (b) powder

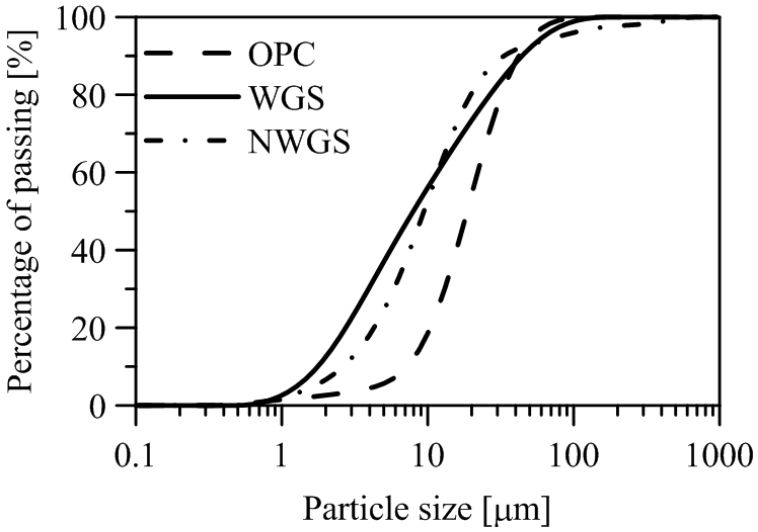

Fig. 3 Particle size distributions of the OPC, WGS and NWGS

milling process as shown in Fig. 2(b).

Both WGS and NWGS for this experiment were prepared by the same process except the use of the precipitation additives, i.e., the cohesive agent and the coagulant aid. In the case of NWGS, none of cohesive agent and coagulant aid was used. Their distributions of the particle sizes are shown in Fig. 3. The distribution of the OPC used in this experiment is also shown in the figure for a comparison. WGS and NWGS had finer distributions than the OPC. The mass median diameters (D_{50}) of WGS and NWGS were 7.95, 9.40 μm, respectively. However, D_{90} of WGS and NWGS were 44.23, 33.19 μm, respectively.

2.2 Chemical composition of the materials

Table 1 Chemical composition of OPC, WGS and NWGS used in this experiment

Contents [%]	OPC	WGS	NWGS
SiO_2	20.72	68.2	66.47
Al_2O_3	4.73	10.1	0.88
Fe_2O_3	3.72	0.24	0.18
CaO	61.94	9.9	9.06
MgO	3.07	2.94	3.27
SO_3	2.31	0.37	0.27
Na_2O	0.01	7.2	12.45
K_2O	1	0.23	0.21

A Portland cement complied with specification KS L 5201 (2006) was used as the reference material. This cement belongs to the category of Type 1 Portland cement according to ASTM C C150/C150M-15 (2015). The chemical compositions of the three binders of OPC, WGS and NWGS determined by the X-Ray fluorescence (XRF) are presented in Table 1. The total amount of the major chemical components SiO_2, Al_2O_3 and Fe_2O_3 was 78.54% in the case of WGS and 67.53% the NWGS. Both of them satisfy the requirement for a pozzolanic material class C specified in ASTM C618-15 (2015).

There was a noticeable difference in the content of Al_2O_3 of the two materials, i.e., 10.1 and 0.88% for WGS and NWGS, respectively. This was due to the aluminum sulfate included in the cohesive agent for the precipitation of glass dust. Also, Na_2O contents of WGS and NWGS were different, i.e., 7.2 and 12.45%, respectively. According to the (Shi *et al*. 2009, Du *et al*. 2013), Na^+ are the first dissolved from glass when the OH^- in the pore solution attacks the glass surface. Therefore, the Na^+ of glass dust might be dissolved into the waste water when the caustic soda was added.

As mentioned before, the precipitation additives are added to the waste water before the dewatering process. The chemical components shown in the table are for the mixture of the glass dust and the residual of the additives after the dewatering process. In the case of the factory where WGS in this experiment was obtained, 0.07 kg of a polymer, 25 kg of aluminum sulfate and 6.25 kg of caustic soda were added per fifty tons of the waste water. Of course, NWGS was free of the additives.

2.3 Experimental programs

2.3.1 Setting time of cement-based pastes

The setting time was measured by using the Vicat needle on two cement-based pastes incorporating WGS and NWGS, respectively, according to KS L 5108 (2007) equivalent to ASTM C191-13 (2013). The Vicat needle was dropped into the cement paste casted in a round steel-mold. The hydration degrees of the paste could be estimated by the penetration depth of the Vicat needle to the surface of the paste at the time of the test. The penetration depth would decreases gradually as the hydration process is matured. The moment of the initial setting is defined as the time when the penetration depth reaches 4 ± 1 mm. The maximum penetration depth for the final setting is 0.5 mm.

2.3.2 Pozzolanic reaction tests by using calcium hydroxide-based pastes

Table 2 Mix proportions of the CH-based pastes for the mineralogical and thermal analysis

Mix	Water [g]	Cement [g]	WGS [g]	NWGS [g]	CH [g]
OPC	400	200	-	-	200
WGS	400	-	200	-	200
NWGS	400	-	-	200	200

Table 3 Mix proportion of the mortars for the compressive strength and the mercury intrusion pressure test

Mix	Water [g]	Cement [g]	WGS [g]	NWGS [g]	Sand [g]
OPC	1125	2250	-	-	6750
WGS	1125	1800	450	-	6750
NWGS	1125	1800	-	450	6750

The pozzolanic reaction was investigated by using the X-Ray diffraction test (XRD), and the differential thermal and thermogravimetric analysis (DT-TGA). The aim of these tests was to assess the remaining amount of calcium hydroxide (CH) at different curing ages, such as 7, 28 and 91 days. Because in the pozzolanic reaction the CH is converted to the additional amount of the calcium silicate hydrates (C-S-H), the degree of the pozzolanic reaction can be evaluated by the consumption rate of the CH (Chindaprasirt *et al*. 2007, Mindess *et al*. 2003, Perraki *et al*. 2003, Altwair *et al*. 2011, Kim *et al*, 2015). To focus on the pozzolanic reaction of the two materials, the CH-based pastes were prepared. The CH, WGS or NWGS and water were mixed. The mix proportions are shown in Table 2.

The specimens made of the CH-based pastes were cured in a thermo-hygrostat. The temperature and the humidity were controlled to be 25ºC and 50%, respectively. The specimens were immersed in acetone for twenty-four hours after the specified curing ages in order to stop the hydration process. And then, they were ground to powder, the maximum particle size of which is 75 μm.

The rate of CH consumption of each specimen was measured by the XRD analysis. The XRD patterns were obtained by using a DB ADVANCE of Bruker-AXS-GmbH, in which the scanning range is from 5 to 80º at a step size of 0.02º. In order to quantify the amount of the CH, the DT-TGA tests were carried out in an inert atmosphere with nitrogen. 12-14 mg of each sample was placed in a DSC2010 of TA Instrument. It was heated in a range of temperature from 25 to 1000 ℃ with a heating rate of 10℃/min.

2.3.3 Compressive strength of mortars

The compressive strength of mortars incorporating WGS and NWGS were investigated. The mortars for the test were prepared according to (KS L ISO 679. 2006) equivalent to (ASTM C311/C311-13. 2013). The mix proportions are shown in Table 3. The water to cement ratio and cement to sand ratio were 0.5 and 0.33, respectively. 20% of the cement was replaced by WGS and NWGS. The strengths were measured by using three specimens for each mix at 7, 28, 56 and 91 days.

2.3.4 Pore size distribution of the mortars

Pore structures of the mortars incorporating WGS and NWGS were investigated by the

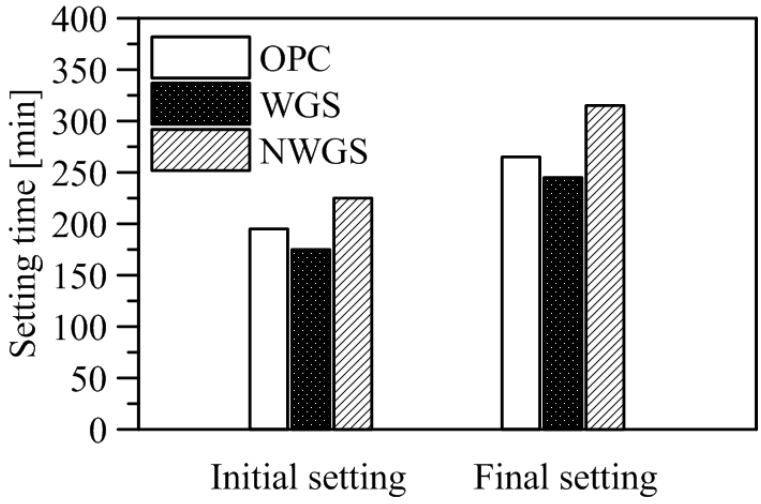

Fig. 4 The initial setting time and the final setting time

mercury intrusion pressure (MIP) test. After the compressive strength test explained in Sec. 2.3.4, the fragments of the specimens were sieved to collect only 2-3 mm size of pieces. The collected fragments were immersed in acetone solution for twenty-four hours to stop the hydration process. Before the MIP test, the fragments were dried in a thermo-hygrostat at 60℃ for twenty-four hours. The maximum injection pressure was 3,000 psi (204 MPa) and the range of the pore sizes was 3-360,000 nm with the contact angle of 130 º.

3. Results

3.1 Setting time of the cement pastes

The initial and final setting times of the cement-based pastes are shown in Fig. 4. Both initial and final setting time of the paste with WGS were faster than the paste made of OPC. Similar results were reported in the researches for alkali-activated slags and pozzolans in which NaOH was used as the alkali-activator (Shi *et al*. 2006, Allahverdi *et al*. 2006). The initial and final setting times of the paste with NWGS were delayed by 50 and 70 minutes, respectively.

3.2 Pozzolanic reaction tests by using the CH-based pastes

3.2.1 Mineralogical analysis by using the XRD test

The CH peaks of each CH-based paste are shown in Fig. 5 for three different curing ages. The peak intensities corresponding to CH were detected at around 18, 34 and 47º (2θ). Lower CH peak intensities were detected clearly for the paste with WGS than other pastes at 7 days. On the other hand, the CH peak intensities of the paste with NWGS were decreased slightly until 91 days of curing ages. This indicates that the pozzolanic reaction of WGS is more active than NWGS.

3.2.2 Thermal analysis by using the DT-TGA test

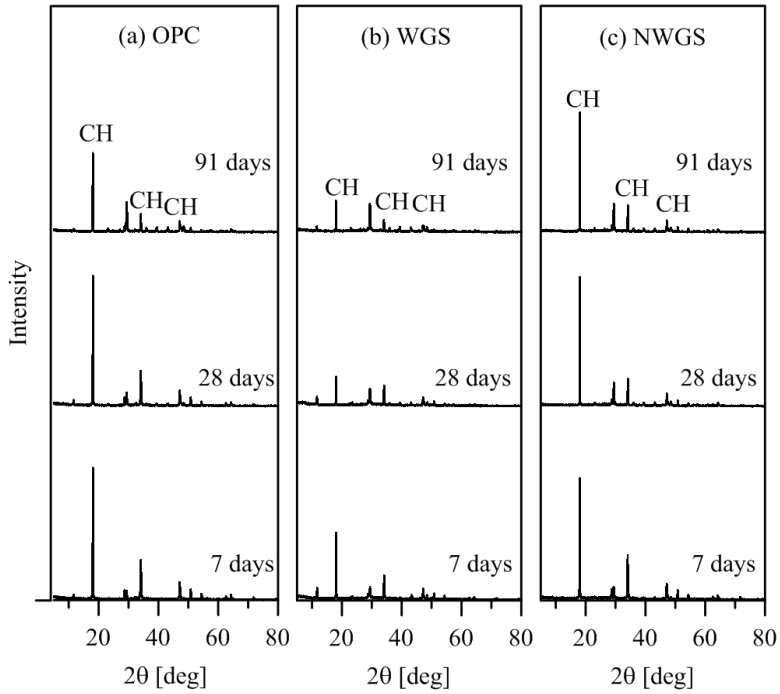

Fig. 5 Result of the X-Ray diffraction test for the CH-based pastes: (a) OPC, (b) WGS and (c) NWGS

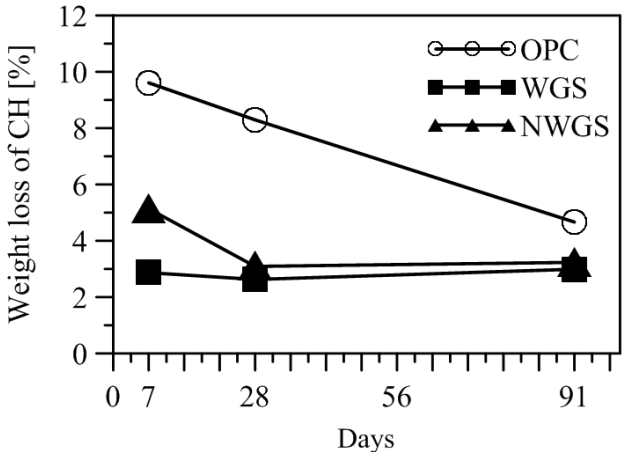

Fig. 6 Weight loss of the CH-based pastes with OPC, WGS and NWGS obtained from the hermogravimetric analysis

The activity of the pozzolanic reaction can be estimated by the change of CH because CH is converted to C-S-H by the pozzolanic reaction. The amount of CH at an age was estimated by the weight loss of the CH in the temperature range corresponding to the decomposition of the CH as shown in Fig. 6. Note that the decomposition of the CH is at around 420 to 480℃ (Almeida *et al*. 2006, and Pelisser *et al*. 2012).

It was found that the amount of CH of the paste with WGS was much less than the paste with

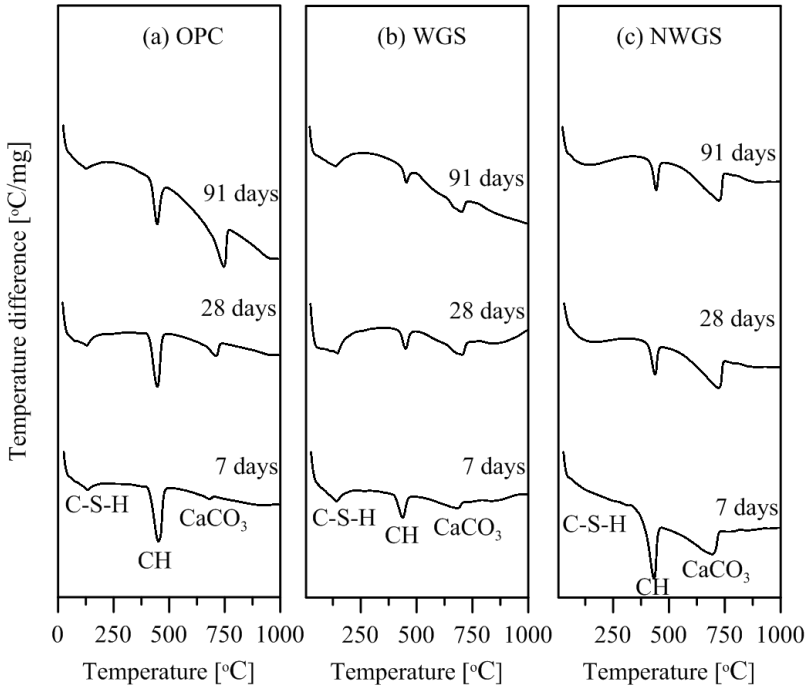

Fig. 7 Differential thermal analysis of CH-based pastes: (a) OPC, (b) WGS and (c) NWGS

NWGS even at 7 days, which indicates that the pozzolanic reaction of WGS became active before the age of 7 days. The change of CH of the NWGS paste was noticeable in the period of 7 to 28 days. There was no noticeable decrease of CH for both WGS and NWGS pastes beyond 28 days.

A similar trend was obtained from the result of the differential thermal analysis shown in Fig. 7. The endothermic peaks of C-S-H, CH and $CaCO_3$ were detected at around 140, 430 and 680℃, respectively, as shown in Fig. 7. The CH-peaks of all the pastes were decreasing as the pastes were cured for longer period. The CH-peak of the WGS paste was much lower than the NWGS paste at 7 days which also supports the fast consumption of CH at its early ages.

The conversion of CH to C-S-H could be confirmed by the C-S-H peak. Because C-S-H would be formed in the pozzolanic reaction by the consumption of CH, a C-S-H peak must be detected if any pozzolanic reaction is expected. There were both CH-peak and C-S-H peak in the case of the WGS paste at 7 days while the C-S-H peak of the NWGS paste became noticeable after 28 days.

There was an interesting study for the effect of an alkali-activation of glass powder by Maraghechi *et al*. (2014). They also tested the CH-based paste to investigate the consumption of CH. In their research, they used 1M of NaOH solution as the mix water to activate the glass powder in their experiment. They reported that the pozzolanic reaction of the glass powder was accelerated significantly with the use of the NaOH solution. Although any alkali activator was not used apparently in our experiment, a fast pozzolanic reaction was found in the case of WGS while the reaction of NWGS was slow. It seems that the caustic soda, NaOH, added to control the pH of the waste water during the manufacturing process in the glass industry activates the glass dust contained in the waste water.

3.3 Compressive strength of mortars

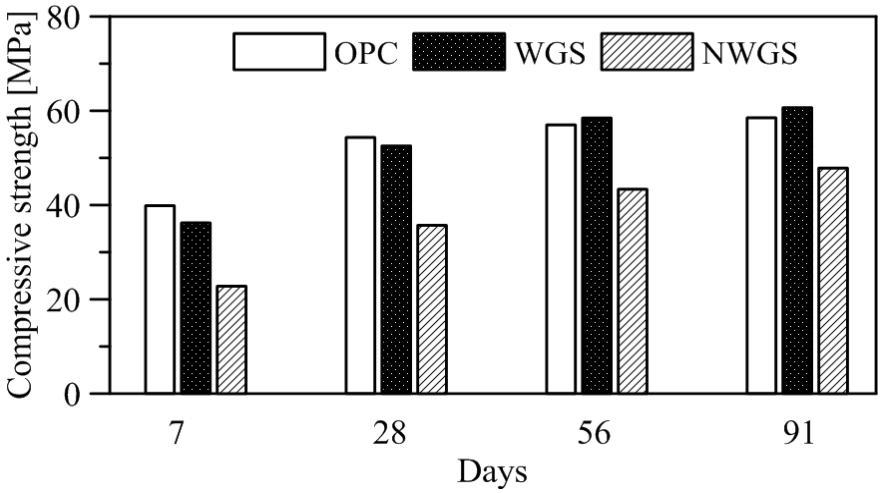

Fig. 8 Compressive strength of OPC, WGS and NWGS mortar specimens

Table 4 Standard deviation and coefficient of variation of compressive strength measurements of OPC, WGS and NWGS mortar specimens

Days	OPC			WGS			NWGS		
	Mean value [MPa]	STD	COV	Mean value [MPa]	STD	COV	Mean value [MPa]	STD	COV
7	39.87	0.09	0.00	36.20	0.45	0.02	22.78	0.15	0.01
28	54.33	0.52	0.01	52.55	0.95	0.00	55.70	0.47	0.01
56	57.00	1.56	0.03	58.46	0.27	0.03	43.35	0.98	0.02
91	58.51	0.94	0.02	60.66	1.98	0.03	47.84	1.40	0.03

The compressive strengths of the mortar specimens are shown in Fig. 8. Also, the mean value, standard deviations (STD) and coefficient of variations (COV) of the compressive strength are presented in Table 4. At all the ages of 7, 28, 56 and 91days, the compressive strength of NWGS was less than OPC specimens. The strength of NWGS mortar was only 57% of the reference strength at 7 days, and 82% at 91 days. The strengths of OPC mortars at the same ages were used as the reference strengths. On the other hand, the compressive strength of WGS mortar is already 91% of the reference strength. It was almost same as the reference strength at 28 days. The long-term strength of WGS mortar was greater than the reference strength. The strength at 91 days was 104% of the reference strength. This result also supports that the faster pozzolanic reaction of WGS than NWGS.

3.4 Pore structures of mortars

The log differential and cumulative intrusions of mercury with respect to the pore sizes are shown at 7 and 28 days in Figs. 9 and 10. The volume of pores greater than 0.1 μm decreased at 28 days for all the mortars under consideration. Overall, the pores of NWGS mortar were larger than OPC and NWS mortars. The pore size distribution of WGS mortar is very close to that of OPC mortar at both 7 and 28 days as shown in Fig. 10.

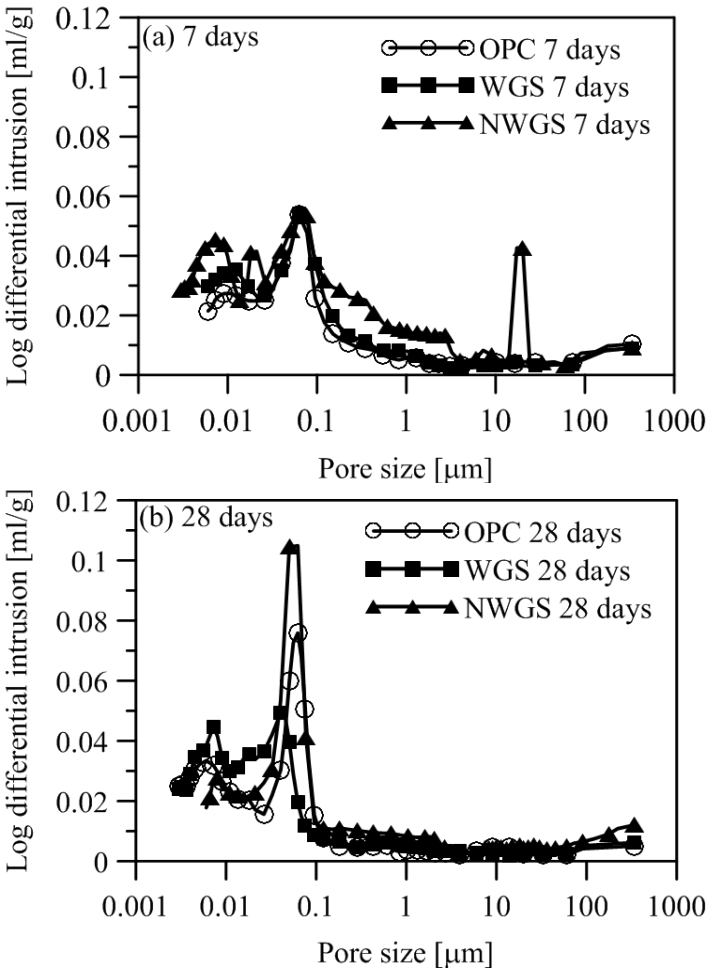

Fig. 9 Log differential intrusion with respect to pore sizes at (a) 7 and (b) 28 days

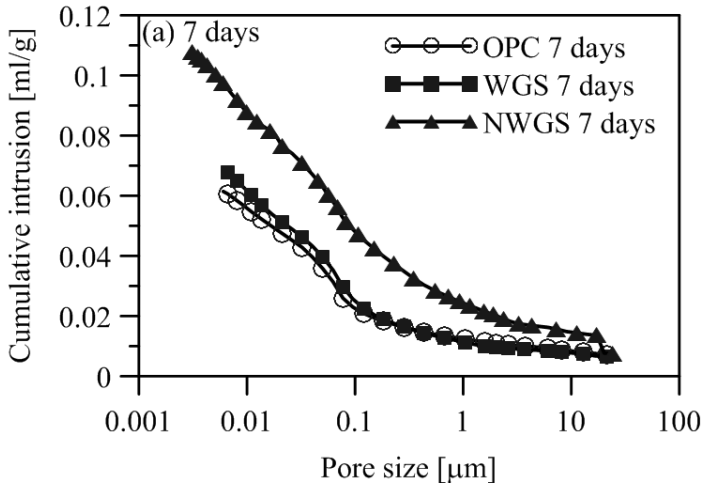

Fig. 10 Cumulative intrusion with respect to pore sizes at (a) 7 and (b) 28 days

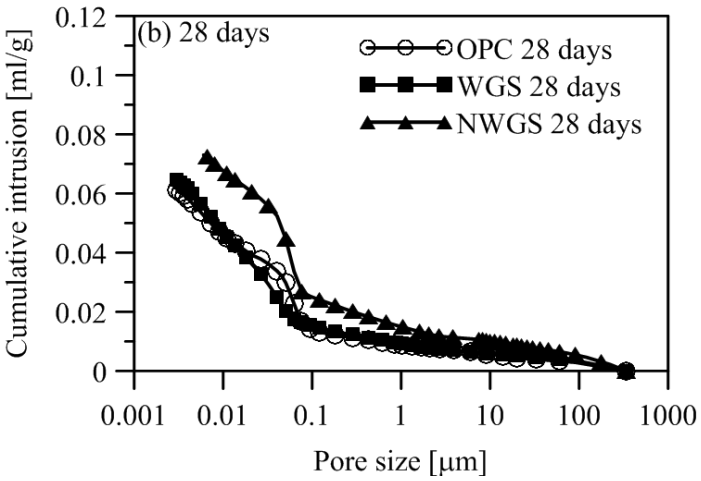

Fig. 10 Continued

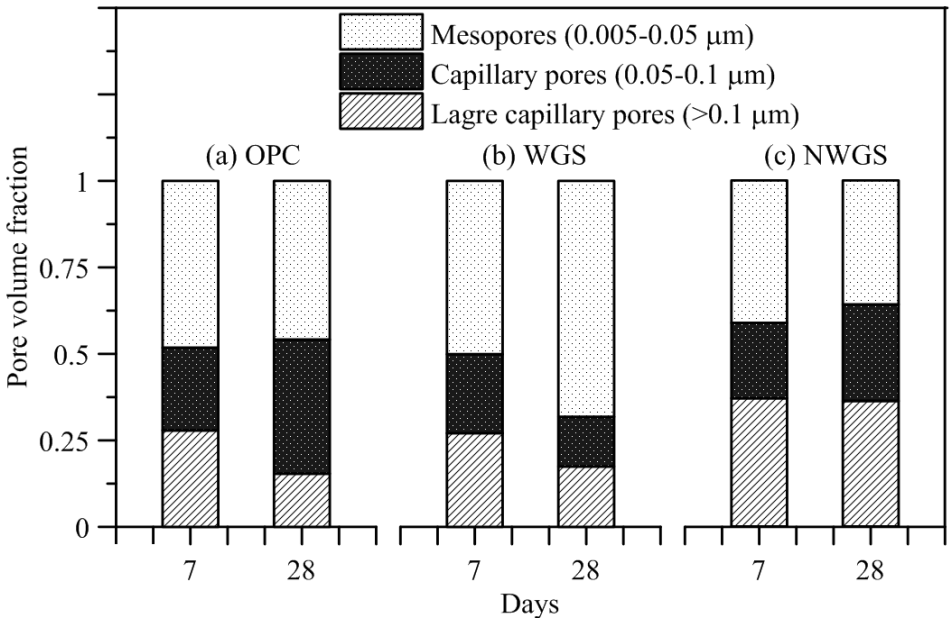

Fig. 11 Volume fractions of meso, capillary and large pores of mortars: (a) OPC, (b) WGS and (c) NWGS

The volume occupied by pores can be classified into three different categories depending on their sizes (Zeng *et al*. 2012): meso pores (0.005-0.05 μm), capillary pores (0.05-0.1 μm) and large pores (> 0.1 μm). The volume fracture of the pore volume by the classification is given in Fig. 11. The pore volume of the capillary and large sizes pores of WGS mortar decreased as the age changed from 7 days to 28 days; the volume fraction of the meso pores increased in the same period. Note that the deposition of C-S-H reduces the volume fraction of the capillary and large pores (Espinosa *et al*. 2006, Kontoleontos *et al*. 2012, Björnström *et al*. 2004, Berra *et al*. 2012). Because of the increased C-S-H, the more meso pores were detected in WGS mortar.

4. Conclusions

In this research, a potential application of the waste glass sludge for a partial replacement of cement was studied. The rich content of silica of the glass sludge and its high specific surface area formed naturally in the manufacturing process of glass products. Two different types of the waste glass sludge were considered depending on the use of the precipitation additives. The conclusions of this research are as follows

• The waste glass sludge obtained from the waste water treated with the cohesive agents and the caustic soda used as the precipitation additives was superior in the pozzolanic reaction to the reference sludge for which no additives were added. It seems that the caustic soda activated the glass particles in the waste glass sludge although it was used to control the pH of the waste water.

• The setting time of the cement mortar containing the waste glass sludge was shorter than the mortar with the reference sludge. The pozzolanic reaction of the waste glass sludge was also confirmed by the XRD and DT-TGA tests. The calcium hydroxide in the specimens containing the waste glass sludge was converted to C-S-H in the early ages of hydration process.

• This positive aspect of the waste glass sludge also influences the compressive strength of the mortar for which 20% of the cement was replaced by the waste glass sludge beneficially. Its compressive strength at 7 days is 91% of the reference mortar made of fully cement. Its strength got higher than that of the reference mortar after 56 days.

• The distribution of the pore sizes of the specimens containing the waste glass sludge was very similar to the specimens made of only the ordinary Portland cement. The pores greater than capillary pores were decreased significantly by the pozzolanic reaction in the early ages of hydration process.

Acknowledgments

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