Cement & Concrete Composites 33 (2011) 602–610

Contents lists available at ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Use of reservoir sludge as a partial replacement of metakaolin in the production of geopolymers

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A R T I C L E I N F O

Article history:
Received 12 May 2009
Received in revised form 7 March 2011
Accepted 7 March 2011
Available online 12 March 2011

Keywords:
Reservoir sludge
Geopolymer
Activating solution
Binder
Mortar

A B S T R A C T

Reservoir sludge was first calcined at 850 °C for 6 h and then employed as 50%, 70% and 100% replacement of metakaolin in the production of geopolymers. Various proportions of slag, metakaolin and calcined reservoir sludge (CRS) particles were mixed with different alkaline activating solutions to produce reservoir sludge-based geopolymers (RSBG). Furthermore, the compressive strengths of RSBG geopolymers were measured and compared. Based on the experimental results, proper alkaline activating solutions that produce higher compressive strengths of RSBG geopolymers with various replacement percentages of metakaolin by CRS particles and different water/binder ratios were determined. At the same time, the effects of water/binder ratio, stirring time and curing temperature on the compressive strengths of RSBG geopolymers were investigated. In addition, the flow values and compressive strengths of RSBG mortars were measured and then compared to those of Portland cement mortars to evaluate the efficiency of using RSBG geopolymers as binders in mortars.

1. Introduction

There are more than 40 principal reservoirs in Taiwan constructed for the purposes of agricultural irrigation, public water supply and flood control. Over the past decades, the annual siltation of the principal reservoirs totals about 14 million cubic meters and their effective water storage capacities have been thus reduced. Hence, some evacuation programs of the principal reservoirs are currently being undertaken to ensure their safety and functions. However, the evacuated reservoir sludge is a priority waste in Taiwan due to the increased volume that is produced annually. As compared to municipal sewage sludge, the amounts of toxic substances in reservoir sludge are much lower. Hence, reservoir sludge can be treated as a chemically inert waste and is normally disposed of in landfills. Due to the large volume of reservoir sludge and the limitation of available landfill space, the disposal and reutilization of evacuated reservoir sludge is an important issue. Typically, the evacuated reservoir sludge is first solidified and then burnt to produce bricks. Since the reservoir sludge in Taiwan is mainly composed of smectite clays such as montmorillonite, they can be foamed at higher temperatures in a furnace to produce lightweight aggregates [1]. Also, the reservoir sludge can be organo-modified by a cationic-exchange reaction and further used as a water-proofing agent added in concrete [2]. But, the amounts of reservoir sludge utilized as bricks, lightweight foamed aggregates and water-proofing powders are still small compared to the volume of evacuated reservoir sludge.

Geopolymers, also known as alkali-activated aluminosilicate binders and first developed by Davidovits [3], can be made from a variety of raw materials containing aluminosilicate minerals by mixing with an alkaline activating solution. Metakaolin is most effective as a raw material of geopolymers activated by alkali metal hydroxide and sodium silicate [4,5]. Blast furnace slag used as a raw material is beneficial to the setting and strength development of geopolymers [6–11]. When good setting and high compressive strength are sought, a blend of metakaolin and slag is preferred for the production of geopolymers. Apart from metakaolin and slag, waste materials such as fly ash [11,12], bottom ash from power plants [13] and silt from construction and demolition waste plants [14] can also be used as raw materials for the production of geopolymers. The microstructure and properties of geopolymers depend on the chemical composition of raw materials used, concentration of alkali metal hydroxide and sodium silicate and polycondensation condition such as temperature and time [14]. Moreover, there is a proper ratio of silica to alumina that produces geopolymers of higher strength [15].

Reservoir sludge, used as a raw material of geopolymer, was calcined at various high temperatures ranging from 500 °C to 900 °C for a duration of 6 h to enhance the subsequent alkali-activated reaction and polycondensation and resulting compressive strength [16]. Furthermore, the change of crystalline structure of calcined reservoir sludge at high temperatures was characterized by using FTIR and XRD methods, suggesting that the calcination temperature

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doi:10.1016/j.cemconcomp.2011.03.002

of 850 °C is preferred. In the paper, reservoir sludge particles were first calcined for 6 h at the temperature of 850 °C. Next, various proportions of slag, metakaolin and calcined reservoir sludge (CRS) particles were alkali-activated by mixing with different alkaline activating solutions to produce geopolymers. Three different replacement percentages of metakaolin by CRS particles were considered here: 50%, 70% and 100%. The compressive strengths of reservoir sludge-based geopolymers (RSBG) were then measured and compared. For each replacement percentage, a proper alkaline activating solution that produces the highest compressive strength of the geopolymers is determined. The effects of water/binder ratio, stirring time and curing temperature on the compressive strength of RSBG geopolymers are also discussed. Finally, the flow values and compressive strengths of RSBG mortars containing quartz sands were measured and further compared to those of Portland cement mortars. Consequently, the feasibility of using RSBG geopolymers as binders in the production of mortars is evaluated.

2. Experimental methods

2.1. Materials

The reservoir sludge evacuated from the A-Kung-Tien reservoir in Taiwan is utilized as a partial replacement of metakaolin in the production of geopolymers. The physical and chemical properties of the evacuated reservoir sludge are given in Table 1. From Table 1, it is known that the evacuated reservoir sludge comprises mainly silts and clays with the primary chemical constituents of SiO_2, Al_2O_3 and Fe_2O_3 but little heavy metals. The reservoir sludge was wetted and then screened by a #30 sieve to get rid of some waste such as bricks and plastics. The resulting reservoir sludge particles were dried in an oven, crushed by a pulverizer and further ground using a powder grinder. The ground reservoir sludge particles were calcined for lasting 6 h at the temperature of 850 °C in a furnace. The mean particle size of CRS particles is found to be 50.2 μm while their specific gravity and specific surface area are 2.78 and 3960 cm^2/g, respectively. Metakaolin particles, composed of 44.6% SiO_2 and 37.5% Al_2O_3, are the most effective and widely-used raw material in the production of geopolymers. The mean particle size, specific gravity and specific surface area of metakaolin particles we used here are 8.0 μm, 2.7 and 6750 cm^2/g, respectively. The introduction of blast furnace slag in the production of geopolymers was found to be beneficial to their setting and strength development [6–10]. For the purposes of workability and setting, 30% weight fraction of blast furnace slag particles, with a chemical composition of 41.6% CaO, 33.9% SiO_2 and 13.6% Al_2O_3, were added in any mix proportion of geopolymers. The mean size of the slag particles we used here is 40.3 μm, their specific gravity is 2.9 and their specific surface area is 4180 cm^2/g.

Different proportions of slag, metakaolin and CRS particles were alkali-activated by mixing with various alkaline activating solutions of sodium hydroxide, sodium silicate and water. Here, sodium hydroxide powders and sodium silicate with a chemical composition of 29.1% SiO_2 and 9.5% Na_2O were mixed with distilled water to make the alkaline activating solutions. The amounts of sodium hydroxide, sodium silicate and water used in a mix proportion of a geopolymer are typically denoted by the three parameters of alkali-equivalent content AE%, silicate modulus Ms. and water/binder ratio W/B. The alkali-equivalent content AE% in an alkaline activating solution is the weight fraction of Na_2O to the total sum of slag, metakaolin and CRS particles. The silicate modulus Ms. of an alkaline activating solution is defined as the molar ratio of SiO_2 and Na_2O. The water/binder ratio W/B of a geopolymer is the total weight of water divided by that of solid constituents, namely, the sum of SiO_2, Na_2O, slag, metakaolin and CRS particles.

Table 1
The physical and chemical properties of reservoir sludge particles.

Physical properties		Chemical properties			
		Chemical constituents		Contents of heavy metals (mg/kg)	
Clay content	68.1%	SiO_2	55.0%	Zn	14.460
Silt content	31.9%	Al_2O_3	22.3%	Pb	4.459
Specific gravity	2.78	Fe_2O_3	8.6%	Cr	3.369
Mean particle size	50.2 μm	Others	16.1%	Cu	3.162
Specific surface area	3960 cm^2/g			Cd	0.076

2.2. Reservoir sludge-based geopolymers

A typical geopolymer containing 30% slag and 70% metakaolin, termed as metakaolin-based geopolymer and denoted by MBG, is considered here. When the replacement percentages of metakaolin by CRS particles are 50%, 70% and 100%, the resulting reservoir sludge-based geopolymers are denoted by RSBG50, RSBG70 and RSBG100, respectively. For instance, the weight fractions of slag, metakaolin and CRS particles of a RSBG50 geopolymer are 30%, 35% and 35%, respectively. The weight fractions of slag, metakaolin and CRS particles for MBG, RSBG50, RSBG70 and RSBG100 geopolymers are listed in Table 2.

The amounts of sodium hydroxide, sodium silicate and distilled water to produce the alkaline activating solution with a prescribed set of AE%, Ms. and W/B for a mix proportion of geopolymers containing slag, metakaolin and CRS particles were calculated first. For example, 6.1 g sodium hydroxide, 45 g sodium silicate and 34.1 g distilled water are weighted to prepare the alkaline activating solution when AE% = 9%, Ms. = 1.5, W/B = 0.5 and 100 g particles are prescribed for MBG and RSBG geopolymers. It is known that the dissolution of aluminosilicate minerals, the release of Si and Al ions into an alkaline activating solution and the subsequent polycondensation of a geopolymer are affected significantly by the raw material used and the alkaline activating solution prescribed [17]. Intuitively, the highest compressive strengths of MBG and RSBG geopolymers depend on the amounts of sodium hydroxide, sodium silicate and distilled water added to prepare an alkaline activating solution. In other words, there is a proper set of AE%, Ms. and W/B for a mix proportion of geopolymers containing slag, metakaolin and CRS particles. Hence, various alkaline activating solutions with different ranges of AE%, Ms. and W/B as given in

Table 2
The weight fractions of slag, metakaolin and CRS particles, the ranges of alkaline activating solutions and the W/B ratios used for MBG and RSBG geopolymers.

Geopolymer	Raw material			Alkaline activating solution		W/B
	Slag (%)	Metakaolin (%)	CRS (%)	AE%	Ms	
MBG	30	70	0	9–7	0.5–1.25	0.5–0.40
RSBG50	30	35	35	9–21	0.5–1.0	0.5–0.35
RSBG70	30	21	49	6–15	0.75–1.5	0.5–0.30
RSBG100	30	0	70	6–15	0.75–1.5	0.5–0.25

Table 2 were prepared for the production of MBG, RSBG50, RSBG70 and RSBG100 geopolymers.

Next, the amounts of sodium hydroxide, sodium silicate and distilled water for each mix proportion of geopolymers containing slag, metakaolin and CRS particles were weighted, poured separately into a container, stirred vigorously for a few minutes and then placed at room temperature for one more day to ensure a complete mixture. CRS particles were first added in the container and stirred for 30 min. Then, slag and metakaolin particles were added simultaneously and stirred again for additional 15 min. After complete mixing, the geopolymer slurry was poured into steel molds with a dimension of 3 cm × 3 cm × 3 cm. All geopolymer specimens were removed from steel molds in the next day. The 28-day compressive strengths of MBG and RSBG specimens with a W/B ratio of 0.5 but different alkaline activating solutions were measured and compared to each other to investigate the effects of AE% and Ms. From the experimental measurements, the proper alkaline activating solutions to give higher compressive strengths for MBG, RSBG50, RSBG70 and RSBG100 specimens can be determined.

Since the release of Si and Al ions into an alkaline activating solution is also influenced by the stirring time spent, it can be expected that the compressive strengths of geopolymers vary with respect to different stirring time. Here, MBG and RSBG geopolymers with a W/B ratio of 0.5 were stirred for different times of 45, 90, 180 and 300 min before they were poured into steel molds. The 3-day, 7-day, 28-day and 91-day compressive strengths of MBG and RSBG specimens subjected to different stirring times were measured to investigate the effect of stirring time. In addition, the polycondensation reaction of geopolymers could be affected by the curing temperature they exposed to, leading to different microstructure and compressive strength. Hence, MBG and RSBG specimens were initially cured at high temperatures of 45 °C, 65 °C and 85 °C under a relative humidity of 98% for lasting 2 h and further placed at room temperature for additional 2, 6, 27 and 90 days. The variations of the 3-day, 7-day, 28-day and 91-day compressive strengths of MBG and RSBG specimens exposed to different curing temperatures were recorded and compared to evaluate the effect of curing temperature.

2.3. RSBG mortars

A geopolymeric binder can also be used as a replacement of Portland cement and further mixed with fine aggregates to produce a geopolymeric mortar. Here, quartz sands with a specific gravity of 2.63 and a fineness modulus of 3.07 were mixed with different MBG, RSBG50, RSBG70 and RSBG100 geopolymeric binders. Three different W/B ratios of 0.5, 0.45 and 0.4 for geopolymeric mortars were considered while the ratio of quartz sands to geopolymeric binder was fixed and equal to 2.75. Slag, metakaolin and CRS particles were placed in a container and mixed with the proper alkaline activating solutions experimentally determined in the previous section for MBG, RSBG50, RSBG70 and RSBG100 geopolymers. Further, quartz sands were added in the container and stirred vigorously to produce geopolymeric mortar specimens. After complete mixing, the flow values of the resulting geopolymeric mortar slurries were measured before they were poured into steel molds with a dimension of 5 cm × 5 cm×5 cm. The 7-day, 28-day and 91-day compressive strengths of geopolymeric mortars were measured and then compared to those of Portland cement mortars with a W/B of 0.485 and a ratio of quartz sands to binder of 2.75. The mean particle size, specific gravity and specific surface area of Portland cement grains, composed of 20.6% SiO_2, 4.6% Al_2O_3 and 62.8% CaO, are 22.9 μm, 3.17 and 3460 cm^2/g, respectively.

3. Results and discussion

3.1. Proper AE% and Ms

The 28-day compressive strengths of MBG, RSBG50, RSBG70 and RSBG100 geopolymers with a W/B ratio of 0.5 but different AE% and Ms. are shown in Figs. 1–4, respectively. From the figures, it is noted that the compressive strengths of MBG and RSBG geopolymers alkali-activated by the same Ms. activating solution, increases initially and then decreases after reaching a maximum as AE% is increased, regardless of the replacement percentage of metakaolin by CRS particles. Hence, the proper alkali-equivalent contents AE% of MBG, RSBG50, RSBG70 and RSBG100 geopolymers are found to be 18%, 15%, 12% and 9%, respectively. The compressive strengths of MBG and RSBG geopolymers are still changed with respect to different Ms. even they are alkali-activated by the same proper alkali-equivalent contents. The proper silicate moduli Ms. of MBG, RESG50, RSBG70 and RSBG100 geopolymers that produce the maximum compressive strengths are roughly 0.75, 0.75, 1 and 1, respectively. The variations of compressive strengths of MBG and RSBG geopolymers with respect to various combinations of AE% and Ms. as shown in Figs. 1–4 are consistently

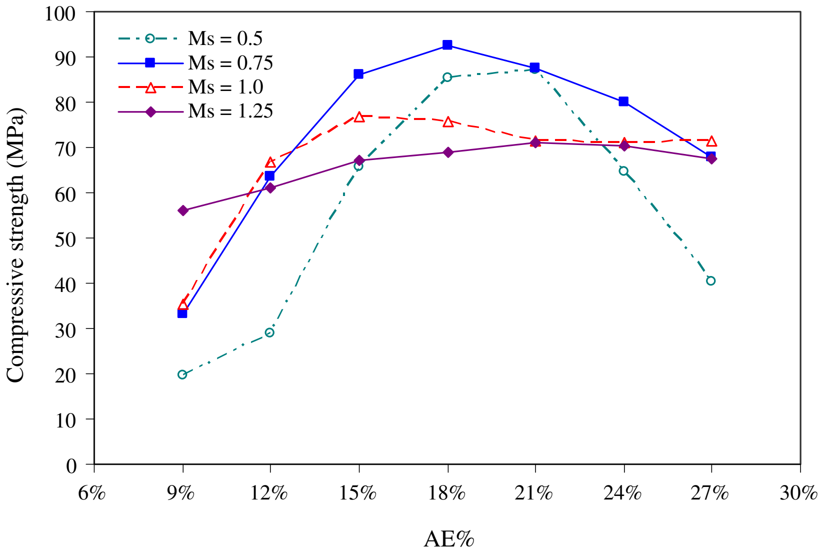

Fig. 1. Variations of the 28-day compressive strengths of MBG geopolymers alkali-activated by different AE% and Ms.

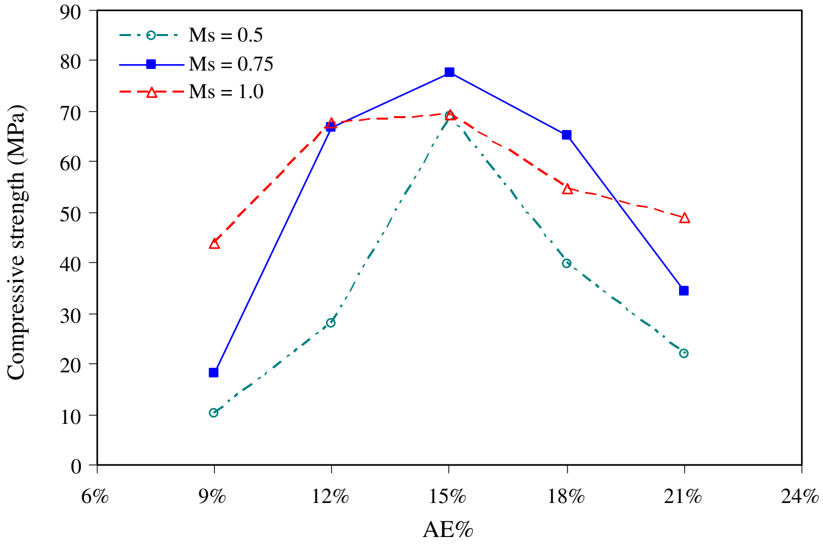

Fig. 2. Variations of the 28-day compressive strengths of RSBG50 geopolymers alkali-activated by different AE% and Ms.

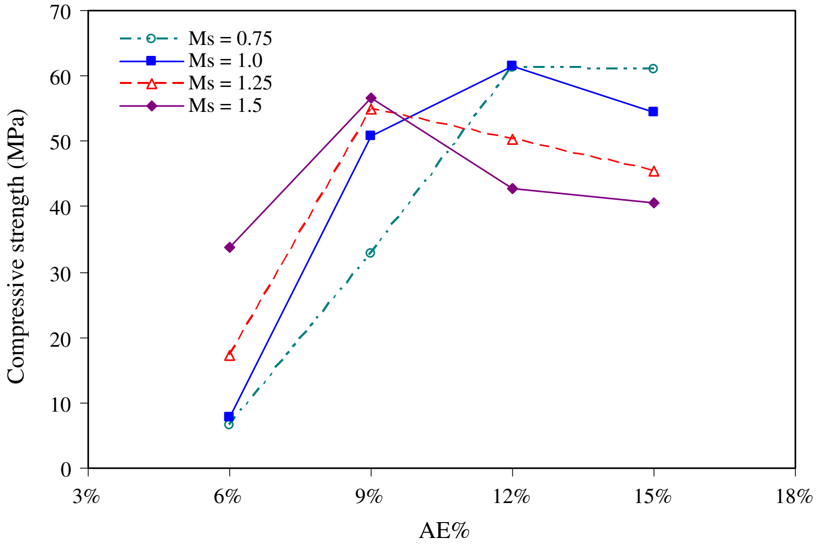

Fig. 3. Variations of the 28-day compressive strengths of RSBG70 geopolymers alkali-activated by different AE% and Ms.

with those of fly ash, slag and metakaolin-based geopolymers [15,18–23].

The dissolution of aluminosilicate minerals, the release of Si and Al ions into an alkaline activating solution and the subsequent polycondensation of RSBG geopolymers are affected significantly by the chemical compositions of raw materials, namely the proportions of metakaolin and CRS particles, and the concentrations of sodium hydroxide and sodium silicate. When the alkali-equivalent content AE% of an alkaline activating solution is low, it is expected that the dissolution of aluminosilicate minerals and the release of Si and Al ions are limited. As a result, the solubility and concentration of geopolymer precursors, aluminosilicate oligomers, are too low to form a dense three-dimensionally Si–O–Al networked microstructure, leading to a smaller compressive strength. The microstructures of RSBG geopolymers become denser and their compressive strengths are higher as AE% is increased slightly. However, the compressive strengths of RSBG geopolymers are reduced when the alkali-equivalent content AE% exceeds their proper value. In that case, the formation of sodium carbonate resulting from the reaction of extra sodium ions with carbon dioxide is responsible for the reduction of compressive strengths of geopolymers [22]. On the other hand, the concentration of geopolymer precursors and their subsequent polycondensation are affected by the Si/Al ratio and the concentration of soluble silica. As the silicate modulus Ms. is increased, the amount of soluble silica and the number of Si–O bonds are increased and thus the compressive strengths of geopolymers become higher [17]. When Ms. exceeds a proper value, the Si/Al ratio becomes larger and the polysialatedisiloxo monomeric unit –Si–O–Al–O–Si–O–Si–O– is more likely to occur, leading to a reduction of compressive strength. Therefore, there are proper silicate moduli that produce the highest compressive strengths of RSBG geopolymers.

The proper values of AE% and Ms. of alkaline activating solutions for MBG and RSBG geopolymers and their highest 28-day compressive strengths are listed in Table 3. It is noted that the proper AE% decreases significantly but the proper Ms. increases slightly as the replacement percentage of metakaolin by CRS particles is increased. As compared to metakaolin, the amounts of reac-

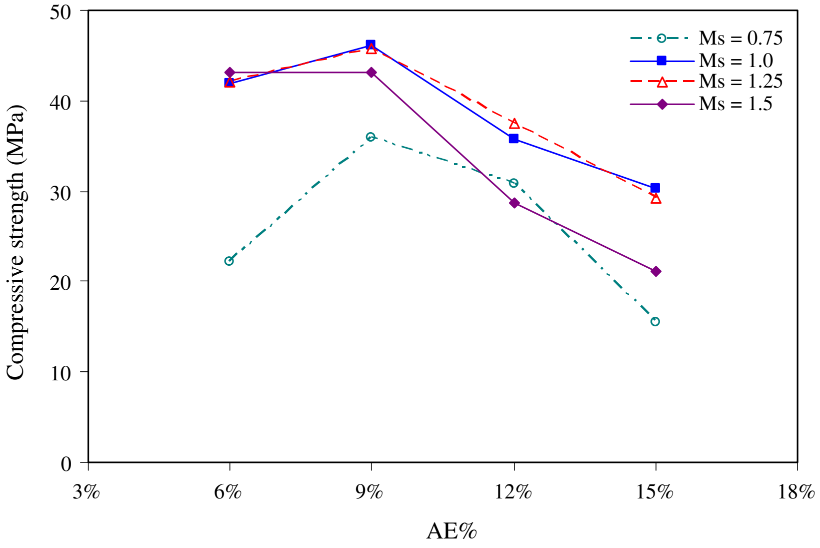

Fig. 4. Variations of the 28-day compressive strengths of RSBG100 geopolymers alkali-activated by different AE% and Ms.

Table 3
The proper values of AE% and Ms. for MBG and RSBG geopolymers and their highest 28-day compressive strengths.

Geopolymer	Proper alkaline activating solution		28-day compressive strength (MPa)
	AE%	Ms	
MBG	18	0.75	92.4
RSBG50	15	0.75	77.7
RSBG70	12	1	61.4
RSBG100	9	1	46.1

tive aluminosilicate minerals and chemical compound Al_2O_3 in calcined reservoir sludge are relatively lower. Under the same W/B, the required alkali-equivalent content for a complete dissolution of Si and Al ions of CRS particles is lower. Undoubtedly, the proper AE% of RSBG geopolymers decreases with the increasing replacement percentage of metakaolin by CRS particles. Similarly, the required amount of soluble silica for the polycondensation of CRS particles that produces the highest compressive strength is lower than that of metakaolin. Due to the reduction of alkali-equivalent content and a roughly fixed Si/Al ratio for a poly-sialatesiloxo geopolymer, the proper Ms. is almost unchanged or increased slightly when the replacement percentage of metakaolin by CRS particles is increased from 0% to 100%.

Also, the highest 28-day compressive strengths of RSBG geopolymers with the same W/B ratio decrease as the replacement percentage of metakaolin by CRS particles is increased. It is found that the highest 28-day compressive strength, 46.1 MPa, of RSBG100 with 30% slag and 70% CRS particles is half that, 92.4 MPa, of MBG with 30% slag and 70% metakaolin. As compared to metakaolin particles, the mean particle size of CRS particles is larger and their specific surface area is smaller. Further, it is expected that CRS particles are less reactive than metakaolin particles and the resulting compressive strength of RSBG is much lower than that of MBG if they have the same W/B ratio. Nevertheless, reservoir sludge-based geopolymers with a compressive strength of 46.1 MPa, which is higher than that requested for normal structural application, can still be achieved if a proper activating solution is used. The compressive strengths of RSBG geopolymers could be enhanced if their W/B ratio is further reduced.

3.2. Effects of W/B ratio

Although low water addition is important to eliminate the microstructural voids and defects of geopolymers, sufficient water is essential to effectively wet all raw materials slag, metakaolin and CRS particles which react with an alkaline activating solution. The lowest W/B ratio allowable for producing RSBG geopolymers decreases with increasing replacement percentage of metakaolin by CRS particles. The lowest allowable W/B ratios of MBG, RSBG50, RSBG70 and RSBG100 geopolymers are 0.4, 0.35, 0.3 and 0.25, respectively. The release of Si and Al ions and subsequent polycondensation of more reactive metakaolin are faster than those of CRS particles when both are mixed with the same alkaline activating solution. Consequently, the workability of MBG geopolymers in a short time after complete mixing becomes worse because the amount of their metakaolin is larger; leading to a higher allowable W/B ratio. The initial and final setting times of MBG and RSBG geopolymers alkali-activated by activating solutions of their proper AE% and Ms. and allowable W/B are: 45 and 75 min. for MBG, 55 and 95 min. for RSBG50, 85 and 135 min. for RSBG70 and 135 and 225 min. for RSBG100. It is noted that the initial and final setting times of RSBG geopolymers lengthen as the replacement percentage of metakaolin by CRS particles is increased.

The ratios of H_2O/Na_2O, SiO_2/Na_2O and SiO_2/Al_2O_3 in geopolymers are the three most important parameters in determining their properties. In evaluating the effect of W/B on the compressive strength of geopolymers, the same AE% and Ms, experimentally determined when W/B = 0.5, are used. Obviously, the ratios of SiO_2/Na_2O and SiO_2/Al_2O_3 are kept unchanged but the ratio of H_2O/Na_2O decrease slightly as W/B is reduced from 0.5 to a lower value. As a result, the proper values of AE% and Ms. are different from those when W/B = 0.5 but their difference is insignificant. Nevertheless, the effect of W/B on the compressive strength of geopolymers can still be investigated by using a particular set of AE% and Ms. The measured 3-day, 7-day, 28-day and 91-day compressive strengths of MBG and RSBG geopolymers activated by alkaline activating solutions of various W/B ratios but the same AE% and Ms. are listed in Table 4. At the same time, the strength developments of MBG, RSBG50, RSBG70 and RSBG100 geopolymers with respect to different W/B ratios are depicted in Figs. 5–8, respectively. Here, the proper values of AE% and Ms. for MBG, RSBG50, RSBG70 and RSBG100 geopolymers as given in Table 3 are used.

Table 4
The compressive strengths of MBG and RSBG geopolymers with different W/B ratios but the same proper values of AE% and Ms. as given in Table 3.

Geopolymer	W/B	Compressive strength (MPa)			
		3-day	7-day	28-day	91-day
MBG	0.55	40.4	54.8	71.6	77.9
	0.5	45.3	72.7	92.4	96.8
	0.45	56.2	79.0	104.6	116.4
	0.4	60.9	84.8	94.2	102.1
RSBG50	0.5	27.5	65.4	77.7	85.5
	0.45	34.0	69.4	89.6	103.6
	0.4	42.3	74.6	95.3	121.7
	0.35	51.1	78.6	96.2	120.7
RSBG70	0.5	24.4	55.2	61.4	66.3
	0.45	32.7	59.4	74.3	82.8
	0.4	37.0	64.8	85.9	101.0
	0.35	46.9	72.7	91.1	116.7
	0.3	54.0	77.8	90.9	105.1
RSBG100	0.5	18.6	28.4	46.1	56.8
	0.45	22.7	33.5	49.0	70.5
	0.4	27.5	40.9	57.8	83.8
	0.35	32.3	51.5	74.1	112.3
	0.3	36.7	58.9	93.5	132.2
	0.25	47.3	71.0	93.8	132.0

From Table 4, it is found that the compressive strengths of metakaolin-based geopolymers, MBG, are superior to those of reservoir sludge-based geopolymers, RSBG100, if they have the same water/binder ratio. When W/B = 0.4, the comparisons of 3-day, 7-day, 28-day and 91-day compressive strengths between MBG, RSBG50, RSBG70 and RSBG100 geopolymers are shown in Fig. 9. From Fig. 9, it is noted that the 3-day and 7-day early compressive strengths of MBG geopolymers are much higher than those of RSBG100 geopolymers. But, the differences of their 28-day and 91-day compressive strengths become less. Obviously, the strength development of reservoir sludge-based geopolymers is different from that of metakaolin-based geopolymers.

In addition, the compressive strengths of MBG, RSBG50, RSBG70 or RSBG100 geopolymers increase significantly at the beginning and then become unchanged or even worse as the W/B ratio is reduced from 0.5 to their lowest allowable value; the minimum W/B ratios of MBG, RSBG50, RSBG70 and RSBG100 geopolymers are 0.4, 0.35, 0.3 and 0.25, respectively. Furthermore, the proper W/B ratios of MBG, RSBG50, RSBG70 and RSBG100 geopolymers that produce the highest compressive strengths are found to be around 0.45, 0.4, 0.35 and 0.3, respectively. If their proper W/B ratios are used, the 28-day and 91-day compressive strengths of RSBG geopolymers are much more close to those of MBG geopolymers. Meanwhile, the 91-day compressive strength, 132.2 MPa, of RSBG100 polymers with a W/B ratio of 0.3 is even better than that, 116.4 MPa, of MBG polymers with a W/B ratio of 0.45.

3.3. Effects of stirring time

The 3-day, 7-day, 28-day and 91-day compressive strengths of RSBG geopolymers, first mixed with activating solutions of W/B = 0.5 and their proper values of AE% and Ms. and then stirred for lasting 45, 90, 180 and 300 min separately, are listed in Table 5. From Table 5, it is found that the compressive strengths of RSBG geopolymers can be enhanced as their stirring time is lengthened. Moreover, the effect of stirring time on the 3-day early compressive strengths of RSBG geopolymers is more significant than that on their 91-day compressive strengths. For example, the 3-day compressive strength of RSBG100 geopolymers subjected to 300 min stirring can be raised up 35%, but their 91-day compressive strength is only 11% higher as compared to those subjected to 45 min stirring. Also, the effect of stirring time on the enhancement of compressive strengths of RSBG geopolymers depends on the replacement percentage of metakaolin by CRS particles. In other words, 45 min stirring is long enough for RSBG50 geopolymers but more than 90 min stirring is apparently beneficial to the compressive strengths of RSBG70 and RSBG100 geopolymers. As compared to metakaolin, the solubility and concentration of geopolymer precursors of less reactive CRS particles in an alkaline activating solution are lower. Hence, more stirring time is needed for RSBG70 and RSBG100 geopolymers containing more CRS particles to provide enough amounts of polymer precursors for their subsequent polycondensation.

3.4. Effects of curing temperature

The compressive strengths of RSBG geopolymers, alkali-activated by activating solutions of W/B = 0.5 and their proper values of AE% and Ms. and cured at different temperatures of 23, 45, 65 and 85 °C for 2 h, are listed in Table 6. The calcium–silicate–hydrate C–S–H gel formed due to the introduction of 30% slag is responsible for the increase of early compressive strengths of metakaolin-based geopolymers when they are cured at higher temperatures [24]. However, the excessive amounts of C–S–H gel

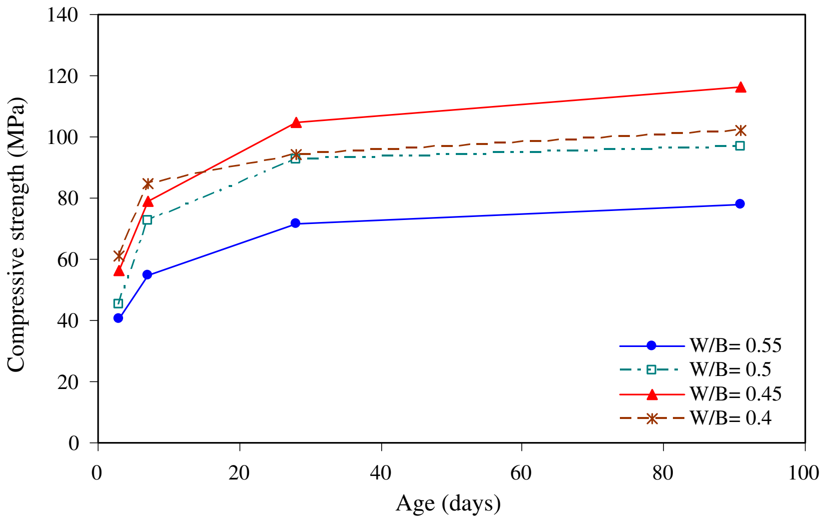

Fig. 5. Compressive strength development of MBG geopolymers alkali-activated by the proper values of AE% = 18% and Ms = 0.75 but different W/B ratios.

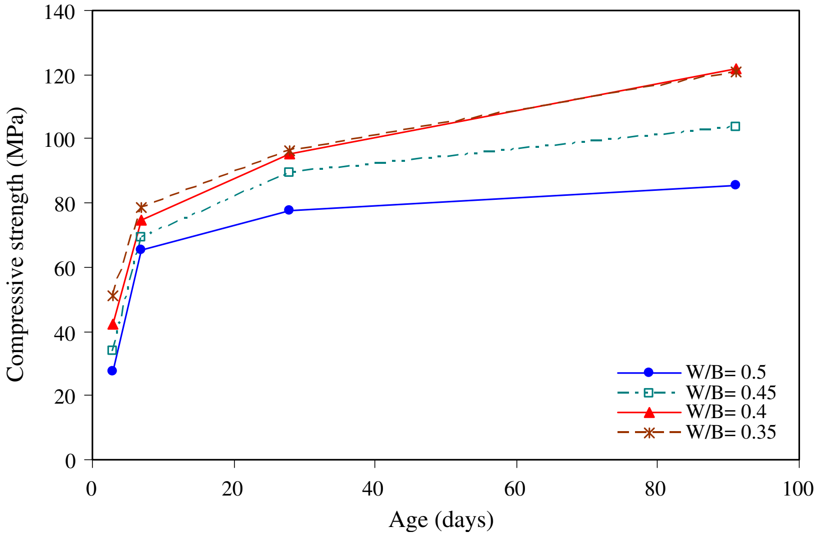

Fig. 6. Compressive strength development of RSBG50 geopolymers alkali-activated by the proper values of AE% = 15% and Ms = 0.75 but different W/B ratios.

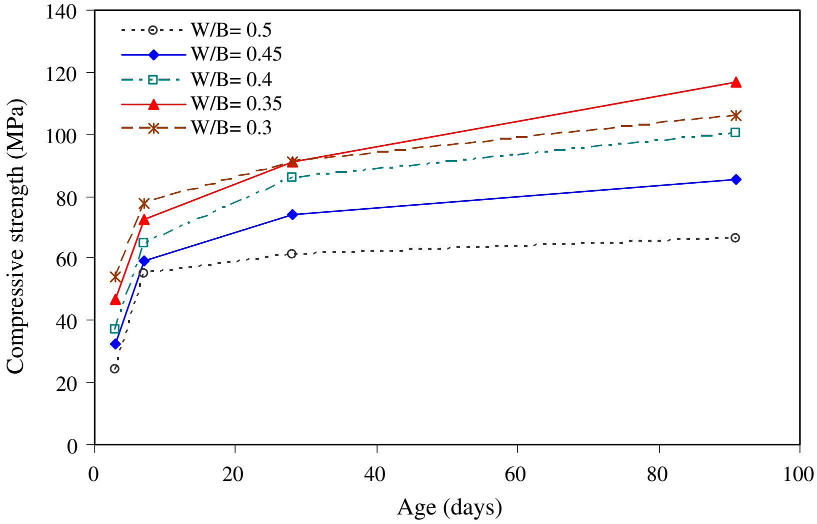

Fig. 7. Compressive strength development of RSBG70 geopolymers alkali-activated by the proper values of AE% = 12% and Ms = 1.0 but different W/B ratios.

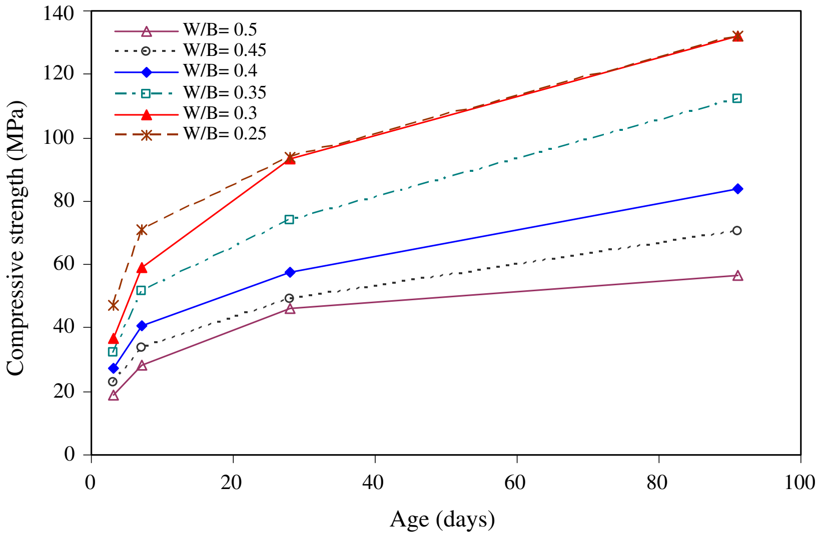

Fig. 8. Compressive strength development of RSBG100 geopolymers alkali-activated by the proper values of AE% = 9% and Ms = 1.0 but different W/B ratios.

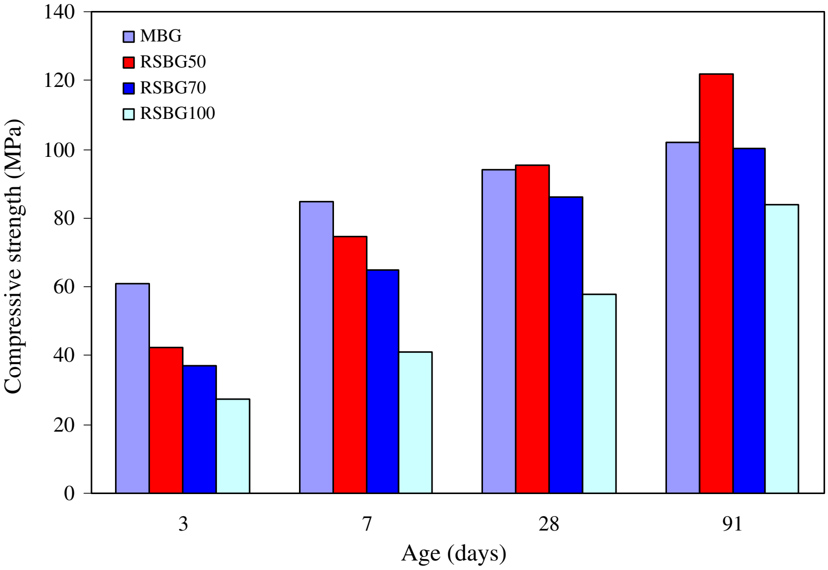

Fig. 9. Comparison of the compressive strengths of MBG and RSBG geopolymers alkali-activated by their proper values of AE% and Ms. and W/B = 0.4.

Table 5
The compressive strengths of RSBG geopolymers subjected to different stirring times of 45, 90, 180 and 300 min.

Geopolymer	Stirring time (min)	Compressive strength (MPa)			
		3-day	7-day	28-day	91-day
RSBG50	45	27.5	65.4	77.7	85.8
	90	39.5	65.1	79.2	88.4
	180	43.0	65.3	83.1	89.3
	300	45.0	66.6	82.2	89.9
RSBG70	45	24.4	55.2	61.4	66.3
	90	32.6	55.1	61.9	71.9
	180	34.2	55.8	64.2	75.0
	300	34.5	55.9	67.0	74.8
RSBG100	45	18.6	28.4	46.1	56.8
	90	22.9	32.5	47.2	63.5
	180	24.4	35.9	49.2	63.9
	300	25.3	37.5	53.5	63.2

Table 6
The compressive strengths of RSBG geopolymers subjected to different curing temperatures of 23, 45, 65 and 85 °C.

Geopolymer	Curing temperature (°C)	Compressive strength (MPa)			
		3-day	7-day	28-day	91-day
RSBG50	23	27.5	65.4	77.7	85.8
	45	28.3	50.7	69.1	81.3
	65	33.6	46.4	62.1	67.8
	85	34.7	39.5	49.9	59.4
RSBG70	23	24.4	55.2	61.4	66.3
	45	23.7	43.4	55.4	65.5
	65	24.6	38.2	49.2	62.7
	85	26.5	32.5	42.8	53.3
RSBG100	23	18.6	28.4	46.1	56.8
	45	16.9	25.9	39.6	56.6
	65	16.3	25.1	36.4	54.5
	85	7.9	17.7	31.8	50.1

produced in a short time at higher temperatures destruct the formation of three-dimensional networked microstructure of subsequent polycondensation of metakaolin, leading to lower late compressive strengths. As expected, curing at higher temperatures is beneficial to the 3-day early compressive strengths of RSBG50 geopolymers but harmful to their 28-day and 91-day compressive strengths. When the replacement percentage of metakaolin by CRS particles is further increased to 100%, curing at higher temperatures is harmful to all compressive strengths, regardless of age. In a short time, the dissolution of aluminosilicate minerals of CRS particles is incomplete and their concentration of geopolymer precursors is still low. But, the polycondensation of limited geopolymer precursors is likely to occur at higher temperatures. As a result, the three-dimensionally networked microstructures of RSBG100 geopolymers are less dense and their 3-day early compressive strengths are reduced.

3.5. Properties of RSBG mortars

To evaluate the feasibility of using reservoir sludge-based geopolymers as binders in mortars, the flow values and compressive strengths of MBG, RSBG50, RSBG70, RSBG100 and Portland cement mortars are measured and listed in Table 7. It is found that the 7-day, 28-day and 91-day compressive strengths of geopolymeric mortars increase but their flow values decrease as the W/B ratio is reduced. The above findings indicate that the effects of W/B ratio on the properties of geopolymeric mortars are similar to those of

Table 7
The compressive strengths and flow values of MBG, RSBG geopolymeric mortars and Portland cement mortars with different W/B ratios.

Mortar	W/B	Compressive strength (MPa)			Flow value (%)
		7-day	28-day	91-day	
MBG mortar	0.5	52.7	70.2	72.7	19.5
	0.45	60.8	84.7	81.1	8
	0.4	67.8	81.4	79.4	0
RSBG50 mortar	0.5	48.5	61.6	69.3	78.25
	0.45	60.1	72.7	77.7	55.25
	0.4	64.4	77.8	82.9	18.25
RSBG70 mortar	0.5	39.2	50.3	53.0	98
	0.45	50.9	61.6	65.0	62.5
	0.4	54.1	65.1	68.4	25.5
RSBG100 mortar	0.5	25.5	37.0	39.3	100.25
	0.45	32.4	43.9	46.8	65.5
	0.4	36.5	49.0	53.3	36.25
Portland cement mortar	0.485	36.6	51.3	53.0	34.5

Portland cement mortars. As compared to Portland cement mortars, the compressive strengths of MBG mortars are higher but their flow values are too low. The workability of geopolymeric mortars can be improved if the larger particle size and less reactive CRS particles are introduced as a partial replacement of metakaolin. From Table 7, it is noted that the flow values of reservoir sludge-based geopolymeric mortars increase but their compressive strengths decrease as the replacement percentage of metakaolin by CRS particles is increased from 0% to 100%. Nevertheless, both compressive strengths and flow values of reservoir sludge-based geopolymeric mortars could be superior to those of Portland cement mortars if a proper alkaline activating solution is used. Even metakaolin is completely replaced by CRS particles, the compressive strengths and workability of RSBG100 geopolymeric mortars are close to those of Portland cement mortars when the alkaline activating solution of W/B = 0.4, AE% = 9% and Ms = 1 is used. Therefore, it can be said that reservoir sludge-based geopolymers can be utilized as binders in the production of mortars with a high compressive strength and a good workability.

4. Conclusions

Calcined reservoir sludge was utilized here as a partial replacement of metakaolin in the production of geopolymers. When the alkali-equivalent content AE% is low, the concentration of geopolymer precursors is too low to form a dense microstructure, leading to a lower compressive strength. The compressive strength becomes higher as AE% is increased slightly but is reduced after reaching a maximum when AE% exceeds its proper value. The concentration of geopolymer precursors and their subsequent polycondensation are affected by the Si/Al ratio and the concentration of soluble silica. As the silicate modulus Ms. is increased, the amount of soluble silica is increased and thus the compressive strength becomes higher. When Ms. exceeds its proper value, the Si/Al ratio becomes larger and the subsequent polycondensation is more likely to occur, leading to a reduction of compressive strength. Based on experimental results, it is found the proper alkali-equivalent content that produces the highest compressive strength decreases significantly but the proper silicate modulus increases slightly as the replacement percentage of metakaolin by CRS particles is increased.

It is also found that the compressive strengths of RSBG geopolymers are reduced with increasing replacement of metakoalin by CRS particles. As compared to metakaolin, the solubility and concentration of geopolymer precursors of less reactive CRS particles in an alkaline activating solution are lower. Hence, more stirring time is needed for RSBG geopolymers to provide sufficient amounts of polymer precursors for their subsequent polycondensation. Reservoir sludge-based geopolymers can be enhanced if they are stirred more than 90 min and then cured at room temperature. Furthermore, RSBG geopolymers can be utilized as binders in the production of mortars with a high compressive strength and a good workability.

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