The Korean Journal of Ceramics, **6**[2] 120-123 (2000)

Consolidation of Quartz Powder by the Geopolymer Technique

Ko Ikeda and Yoshinori Nakamura

Department of Advanced Materials Science and Engineering, Yamaguchi University, Ube 755-8611, Japan
(Received September 23, 1998)

The geopolymer technique may be a future-oriented technology for saving energies and resources. This technique requires 3 fundamental elements so-called filler, hardener and geopolymer liquor being generally an alkaline silicate solution. Quartz powder, water quenched granulated blast furnace slag and sodium silicate solution prepared from $Na_2O{\cdot}2SiO_2$ were chosen for these three elements. After mixing these starting materials in appropriate proportions, monoliths were prepared by casting at room temperature. Strength tests showed the following results for 28d age specimens: 7.9-12.7 MPa in flexural strength and 20.2-32.8 MPa in compressive strength, depending on geopolymer liquor/solid ratio, P/S and fineness of the quartz powders used.

Key words: Geopolymer, Quartz powder, Sodium silicate solution, Water glass, Blast furnace slag

I. Introduction

Sodium silicate solutions generally called water glasses have been applied to refractory bricks as repairing binders.[1] In 1982, Davidovits obtained a patent concerning "geopolymer binders".[2] The author studied alkaline silicate binders from the aspect of polycondensation of $[SiO_4]^{4-}$-complexes and prepared monoliths by reacting strong alkaline solutions with both reactive silica and metakaoline at 80 °C(the high temperature process). He described that no contraction occurred during the solidification of slurry casts and potassium hydroxide is recommended rather than sodium hydroxide for the strong alkaline source. Analogous process can be realized by reacting mineral powders with alkaline silicate solutions.[3-9] Sometimes strong alkaline solution and reactive silica are mixed in order to react them at high temperatures. Most advantage of this technique is that this process works at room temperature, even when the strong alkaline solution and the reactive silica are lacking. However, most disadvantage of this process is large contraction of slurry casts occurring during solidification (the room temperature process). The room temperature process requires generally three essential materials called filler, hardener and geopolymer liquor. The filler is mineral powders, and fly ash and other wastes are also applicable.[6-9] The hardener is mixed to accelerate hardening, when applied fillers are inactive, i.e., hardly soluble to geopolymer liquor. Malladrite(Na_2SiF_6) and water quenched granulated blast furnace slag are conveniently used for this purpose as well as metakaoline.[5-9] The geopolymer liquor is alkaline silicate solutions. In this paper geopolymer monoliths will be prepared from typically inactive quartz powders by the room temperature process and mechanical strength of monoliths as well as contraction of monoliths will be studied.

II. Experimental

Pure quartz powder of JIS 1st grade commercially available from Katayama Chemicals Co. was primarily used for the filler (Q1). This was secondly pulverized in a pot mill with water and completely dried (Q2). In addition impure quartz powder associated with some kaolin from Namera Mine located in Yamaguchi Prefecture was also used (Q3).

Table 1. Physico-chemical Characteristics of Source Materials

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	CaO	MgO	K_2O	Na_2O	P_2O_5	SO_3	LOI	Total
Esment	33.30	1.81	13.44	0.66	0.58	40.74	7.46	0.45	0.45	0.03	1.40	-	100.32
Quartz(Q3)	89.00	0.41	6.87	0.62	–	0.13	0.34	0.32	0.12	–	–	2.29	99.73
Quartz(Q1,Q2)				---------- 99.5 percent purity guaranteed. ----------									
Geopolymer liquor	SiO_2	Na_2O	H_2O	S.G.									
	18.6	8.8	72.6	1.27									
Specific surface area	Esment	Quartz(Q1)	Quartz(Q2)	Quartz(Q3)									
Blaine, cm^2/g	4000	4900	6400	5700									

Q1 and Q2: pure quartz. Q3: impure quartz containing 17.4 % potential kaolin.

Table 2. Mixing Proportions of Source Materials by Weight

	Q1-35	Q1-40	Q1-45	Q1-50
Quartz(Q1)	9	9	9	9
Esment	1	1	1	1
P/S wt ratio	0.35	0.40	0.45	0.50
	Q2-45	Q2-50		
Quartz(Q2)	-	-	9	9
Esment	-	-	1	1
P/S wt ratio	0.45	0.50		
	Q3-35	Q3-40		
Quartz(Q3)	9	9	-	-
Esment	1	1	-	-
P/S wt ratio	0.35	0.40		

P/S: (Geopolymer liquor)/(solid) wt ratio, S including quartz and Esment

They all belong to inactive fillers and independently used in preparatory mixing. Water quenched granulated blast furnace slag commercially available from Shin-nittetsu Chemicals Co., "Esment" was used for the hardener in constant amount to assist hardening of slurry casts. Sodium-based water glass corresponding to JIS variety No.1 ($Na_2O{\cdot}2SiO_2$) was used as the geopolymer liquor. Characteristics of these source materials were tabulated in Table 1.

Monolithic materials were prepared by hand-mixing of the three source materials in a plastic beaker and cast into a plastic mold having 95×60×19 mm dimension and kept standing in ordinary air for 2-3d at room temperature. After demolding, specimens were kept drying in air at room temperature and prismatic specimens were cut and polished into 10×10×60 mm dimension according to monolith age, that was 7, 14 and 28d. Then, 3-point flexural (25 mm span) and compressive strengths were measured for 3 test-pieces at constant cross-head speed, 0.016 mm/s and averaged strength was obtained. On the other hand, linear contraction of monoliths was measured by contact measuring of a pair of slide calipers before cutting. Mixing conditions are tabulated in Table 2.

III. Results and Discussion

Strength

As seen in Fig. 1, monolith strength of Q1-series is generally increasing with advancement of materials age and with decreasing P/S ratio. Increasing flexural strength is marked with advancement of age and maximal and minimal flexural strengths reached at 28d age were 9.4 and 7.9 MPa for 0.35 and 0.50 P/S, respectively. Corresponding compressive strengths were 26.3 and 20.2 MPa, respectively. We could not prepare specimens having less than 0.35 P/S due to hard kneading.

Monolith strength of Q2-series is represented in Fig. 2. Although we could not prepare specimens with lower P/S ratio, relatively high strength was obtained in specimens with 0.45 and 0.50 P/S, showing generally around 10 MPa in flexural strength and around 30 MPa or more in compressive strength. A little bit decreasing of flexural strength

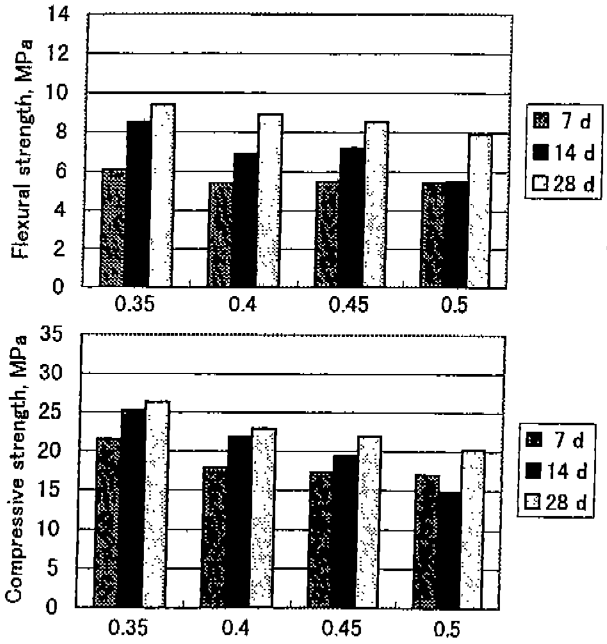

Fig. 1. Strength development of Q1-series monoliths with age and P/S ratio.

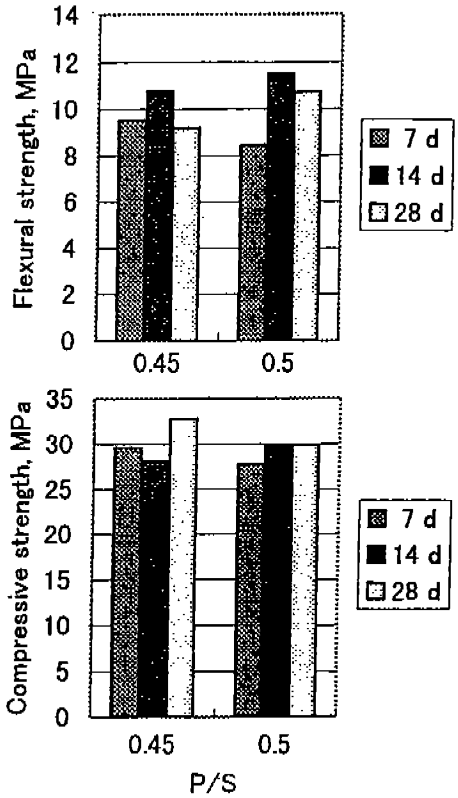

Fig. 2. Strength development of Q2-series monoliths with age and P/S ratio.

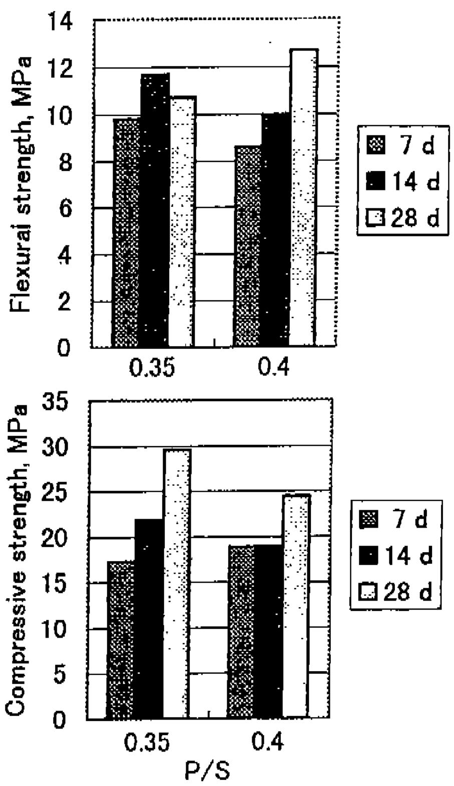

Fig. 3. Strength development of Q3-series monoliths with age and P/S ratio.

may be due to becoming more brittle of specimens with advancement of age. The compressive strength, 32.8 MPa obtained for 28d age specimens with 0.45 P/S was the maximal throughout present study.

Monolith strength of Q3-series is represented in Fig. 3. Preparation of specimens with higher P/S was omitted. Maximal strength was reached for specimens at 28d age, 12.7 MPa flexural strength for specimens with 0.40 P/S and 29.6 MPa compressive strength for specimens with 0.35 P/S, respectively. Increasing of compressive strength for specimens with 0.35 P/S was remarkable with advancement of age. Kaolin contamination may be favorable for improvement of strength, especially for flexural strength.

Comparing the specific surface area between Q1-quartz and Q2-quartz, it can be suggested that higher specific surface area is favorable for strength. However, higher specific surface area is unfavorable in consuming more geopolymer liquor. This may be improved by introducing some kaolin powder into pure quartz powder.

Contraction

Linear contraction of Q1-series is represented in Fig. 4. It was obvious that the contraction became increasing with increasing P/S ratio. Roughly speaking, the contraction change was nearly constant for low P/S specimens with age. However, abrupt change of contraction was observed in case of 0.50 P/S at 28 d age, exceeding 3 %.

Linear contraction of Q2-series is represented in Fig. 5.

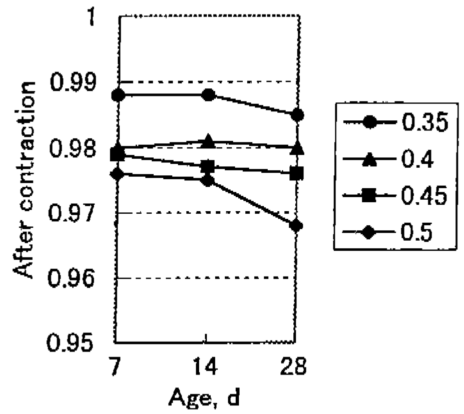

Fig. 4. Contraction of Q1-series monoliths with age and P/S ratio.

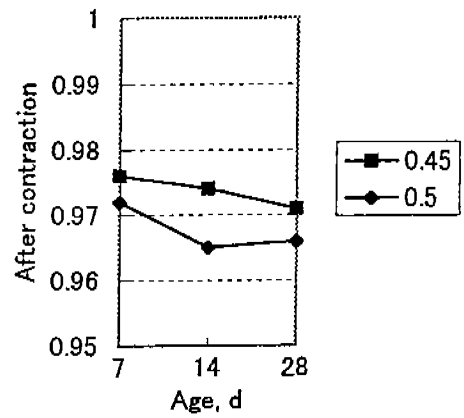

Fig. 5. Contraction of Q2-series monoliths with age and P/S ratio.

Larger contraction was observed for this series due to larger P/S ratio applied. When compared with that of Q1-series, contraction was relatively larger in Q2-series in which specific surface area of the quartz powder was larger than that of Q1-series, consuming much geopolymer liquor.

Linear contraction of Q3-series is represented in Fig. 6. The contraction of this series was quite different from those of Q1- and Q2-series. Abrupt increasing of contraction was

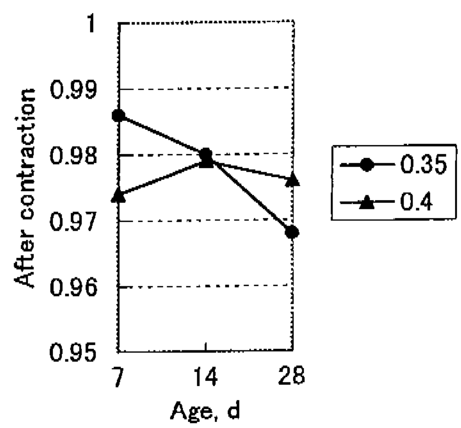

Fig. 6. Contraction of Q3-series monoliths with age and P/S ratio.

seen for specimens with 0.35 P/S, while decreasing contraction was seen for specimens with 0.40 P/S. This may be the cause of contaminated kaolin which is considered to be sometimes advantageous and disadvantageous, depending on P/S ratio.

IV. Summary and Conclusion

Mixing sodium silicate solution with quartz powder under the aid of water quenched granulated blast furnace slag as a hardener, monolithic materials can easily be prepared at room temperature. The solidified materials exhibit white in color and may have a potential of practical application to building and residential materials. Following items were found as conclusion.

1. When using relatively coarse quartz powder, monoliths can be prepared in a wide range of P/S ratio and 0.35 P/S was reached as a minimal ratio, showing 9.4 MPa flexural strength and 26.3 MPa compressive strength with this ratio at 28d age.

2. When using relatively fine quartz powder, applicable P/S ratio moved to higher ratio. However, decreasing of strength was not encountered due to strong contraction, resulting 10.7 MPa flexural strength and 32.8 MPa compressive strength as maximal values at 28d age.

3. Contamination of kaolin is generally favorable. Despite relatively fine powder, this promotes easy mixing of source materials in lower P/S range, resulting 12.7 MPa flexural strength and 29.6 MPa compressive strength as maximal values at 28d age.

4. Around 1 to 3 percent contraction was observed, depending on materials age and P/S ratio. Smaller P/S ratio is generally recommended to prevent much contraction. However, sometimes much contraction is favorable, resulting higher strength due to densification of monoliths.

Acknowledgement

Many thanks are due to Messrs. S. Kumagaya and B. Seo for the courtesy of quartz powders and blast furnace slag and to Mr. T. Koga for the assistance of this study.

References

1. E. Yorita, "Silicate Bonded Castable Refractories," *TAIKABUTU OVERSEAS*, **9**, 29-31(1989).
2. J. Davidovits, "Mineral Polymers and Methods of Making Them," *US Patent*, No. 4349386 (1982).
3. D. M. Roy, "New Strong Cement Materials: Chemically Bonded Ceramics," *Science*, **235**, 651-658 (1987).
4. A. Palomo, M. T. Macias, M. T. Blanco and F. Puertas, "Physical, Chemical and Mechanical Characterization of Geopolymers," *Proc. 9th Int. Cong. Chem. Cem., New Delhi*, **5**, 505-511, (1992).
5. S. C. Foerster, T. Graule and L. J. Gauckler, "Strength and Toughness of Reinforced Chemically Bonded Ceramics," *Ceram. Trans.*, **40**, 247-256 (1994).
6. K. Ikeda and Y. Nakamura, "Consolidation of Fly Ash Powder by the Geopolymer Technique," Conference Papers and Abstracts, PacRim2, Cairns, Symp., **18**, 227-231, 1999.
7. K. Ikeda, "Preparation of Fly Ash Monoliths Consolidated with a Sodium Silicate Binder at Ambient Temperature,"*Cement and Concrete Research*, **27**, 657-663 (1997).
8. K. Ikeda "Consolidation of Mineral Powders by the Geopolymer Binder Technique for Materials Use (in Jan.)," *Shigen-to-Sozai*, **114**, 497-500 (1998).
9. K. Ikeda, T. Nunohiro and N. Iizuka, "Consolidation of Silica Sand Slime with a Geopolymer Binder at Room Temperature and the Strength of the Monoliths," *Chem. Papers*, **52**, 214-217 (1998).