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Geopolymer Mortar Production Using Silica Waste as Raw Material

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ABSTRACT

This research improved the compressive strength using silica waste and pure alumina from waste material for geopolymer mortar production. The basic physical and chemical properties of silica waste were analyzed. The optimum ratio of silica waste to pure alumina and the binding ratio of sodium hydroxide to sodium silicate solution were studied. The mortars were casted in 5*5*5 centimeters cubic shape with curing temperature at 60°C for 24 hours. The geopolymer mortars were tested for compressive strength at 1, 3, 7, 14, 28, 56 and 90 days. The results revealed that the chemical characteristics of silica waste contained silicon dioxide 71%. The leaching tests of heavy metals also indicated that the concentrations of all heavy metals were within the standard set by the Ministry of Industry, Thailand. Therefore, it is possible to utilize silica waste for production of geopolymer mortar for construction. The ratio A (60:20:20), B (70:10:20) and cement mortar control passed the compressive strength standard design at 180 ksc on 3, 7 and 14 days, respectively. The optimum ratio of binder to sodium hydroxide to sodium silicate solution was the ratio B by weight which resulted in the highest compressive strength. In addition, SEM micrographs of the specimens indicated the microstructure which confirms the results obtained by the compressive strength tests.

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1. Introduction

Geopolymer is the new cementatious binder material (Jumrat *et al*., 2011). The geopolymer was developed by Davidovits in 1979. The synthesis of geopolymer takes place by silica alumina sodium hydroxide and sodium silicate (Andini *et al*., 2007 and Nazari *et al*., 2011) and geopolymer is mixed at room temperature and cured in ranging from room temperature to 95 degrees Celsius for about 6 h to 4 days (Jumrat *et al*., 2011). The general chemical formula of geopolymeris $M_n[-(SiO_2)_z - AlO_2]_n.wH_2O$, where M is an alkali cation, z is a number and n is the degree of polymerization. Geopolymeric Structures: poly (sialate) (-Si-) Al-O-), poly (sialate-siloxo) (Si-O-Al-O-Si-O-) (Andini *et al*., 2007)

$$(Si_2O_5, Al_2O_2) + nH_2O \xrightarrow{NaOH,\ KOH} n(OH)_3\text{-}Si\text{-}O\text{-}Al\text{-}(OH)_3$$

$$n(OH)_3\text{-}Si\text{-}O\text{-}Al\text{-}(OH)_3 \xrightarrow{NaOH,\ KOH} (Na, K)(\text{-}\underset{O}{\overset{|}{\underset{|}{Si}}}\text{-}O\text{-}\underset{O}{\overset{|}{\underset{|}{Al}}}\text{-}O)_n$$

(Zheng *et al*., 2010)

The properties of geopolymers are quick compressive strength development and tendency to drastically decrease the mobility of most heavy metal ions contained within the geopolymeric structure (Topcu and Toprak, 2011). The immobilization and solidification of geopolymerization by metal ions are taken into the geopolymer network, metal ions are bound into the structure for charge balancing roles and a precipitate containing heavy metals is physically encapsulated (Zheng *et al*., 2010). The highest volume engineering material in use today is Ordinary Portland Cement (OPC), but Ordinary Portland Cement production contributes 5% of anthropogenic carbon dioxide emission (Tailby and Mackenzia, 2010). Silica wastes are obtained from the depolymerization process of silicone compound recycling, which account for 300-400 tons per year for the world (Sresthaolarn and Chawakitchareon, 2011). Treatment costs of waste disposal and wastes management are expensive, therefore studies are encouraged to utilize used and waste materials, such as silica waste, as part of a mixture of raw materials that can be used productively and consequentially reduce waste pollution. The silica waste yielded characteristics similar to Silica Fume (Sresthaolarn and Chawakitchareon, 2011). The study aims to observe the different mix ratios of raw materials that conform as a mixture of geopolymer mortar, in order to obtain the optimal chemical composition and quality for mortar production.

2. Methodology

2.1 Materials

Silica waste and pure alumina were used as starting materials. Sodium hydroxide solution of 10 M concentration was prepared by dissolving NaOH pellets in distilled water. The study used sodium silicate solution with composition of 8.9 wt. % Na_2O, 28.7 wt. % SiO_2, and 62.5 wt. % H_2O. The sand used for the experiment was washed with water, then dried at temperatures ranging from 103 to 105 degrees Celsius. The sand was then mixed with geopolymer mortar with a fine aggregate ratio of 1 to 2.75. The percentage of sand retained on sieve No. 20 was 15% and sieve No. 30 was 5% (ASTM C778-06).

2.2 Sample preparation

The basic physical and chemical properties of silica waste were analyzed, with pH specific gravity (ASTM C128-07a). The mix design of the geopolymer mortar was prepared for two kinds of binder material (silica waste and pure alumina), which was then compared to the cement mortar controls. The mix design, using the silica waste: pure alumina = 40:20 and 46.7:23.3 by weight, and binder to sodium hydroxide to sodium silicate solution ratio = A (60:20:20) and B (70:10:20) by weight.

2.3 Leaching tests of heavy metals

Heavy metal leaching test was performed using the Waste Extraction Test (WET) in accordance to the Ministry of Industry guidance for disposal of waste or used materials established in 2005 (Ministry of Industry Thailand, 2005). The leachate was analyzed for heavy metals using the inductively coupled plasma (ICP).

2.4 Analysis of size distribution for the mixed materials

Analysis of mixed size distribution for the silica waste was done by Sieve Analysis in accordance with ASTM C136 – 06. The Sieve boxes were sorted by size from larger sizes on the top to the lower sizes at the bottom. The material was then shaken passing the sieve boxes to obtain the different material sizes, which were 100 Mesh, 200 Mesh and 325 Mesh (ASTM C 136-06).

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2.5 Analysis of chemical composition of materials

We analyzed the chemical composition of materials (Silica Waste) by X-ray fluorescence (XRF) and micro structural analysis by scanning electron microscopy (SEM).

2.6 Study of optimum conditions for geopolymer mortar production with silica waste compared to the cement mortar controls

Various proportions of material are mixed with binder to sodium hydroxide to sodium silicate solution ratio. Each cubic sample was 5 x 5 x 5 cm (total 5 sample cubes for each experimental mixture). The geopolymer mortar was cured at 60 degrees Celsius for 24 hours. Compressive strength of geopolymer mortar was tested at 1, 3, 7, 14, 28, 56 and 90 days. The compressive strength of cement mortar was at 180 ksc. The samples were measured for unit weight in accordance to ASTM C109/c 109M-07[12] by weighing the sample, then dividing the volume of the cube specimens. Investigation of the compressive strength was used as the basis for the decision for the experimental samples with different ratios and varying mix design of binder to sodium hydroxide to sodium silicate solution.

2.7 Comparison of heavy metal leaching test

The samples were then tested using the Waste Extraction Test (WET) in accordance to the Ministry of Industry Act 2005 (Ministry of Industry Thailand, 2005). The heavy metals were analyzed using the inductively coupled plasma (ICP).

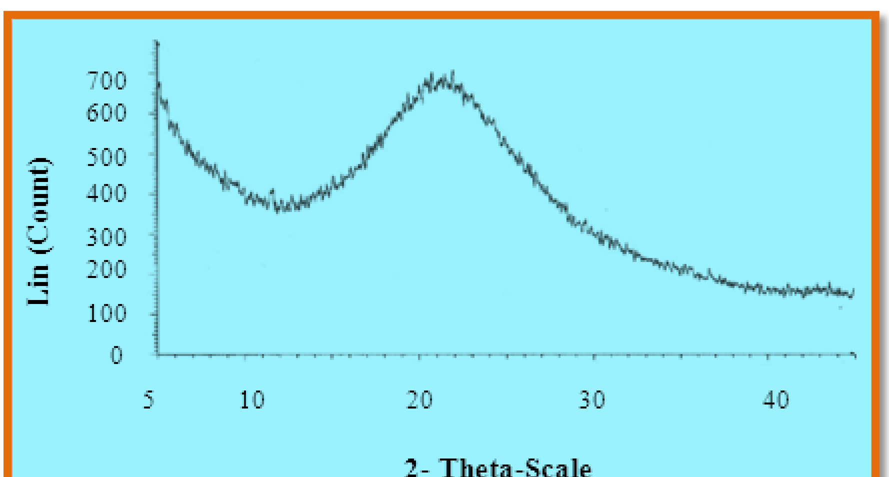

Figure 1: XRD patterns of silica waste.

Table 1: The leaching tests of silica waste, A (60:20:20) and B (70:10:20).

Type	Type of metal (mg/l)			
	Zn	Cr	Pb	Cu
Silica waste	<0.1	1.702	<0.1	<0.1
A(60:20:20)	<0.1	<0.1	<0.1	<0.1
B(70:10:20)	<01	<0.1	<0.1	<0.1
Standard [6]	250	5	5	25

3. Results and discussions

3.1 The basic physical and chemical properties of silica waste

The silica waste (silicone-silicon compounds) was obtained from the recycling plants, which can be advantageous for geopolymer mortar production. Silica waste was gray black in color with specific gravity equal to 2.1 and pH equal to 6.8. The silica waste was in amorphous phase (Figure 1).

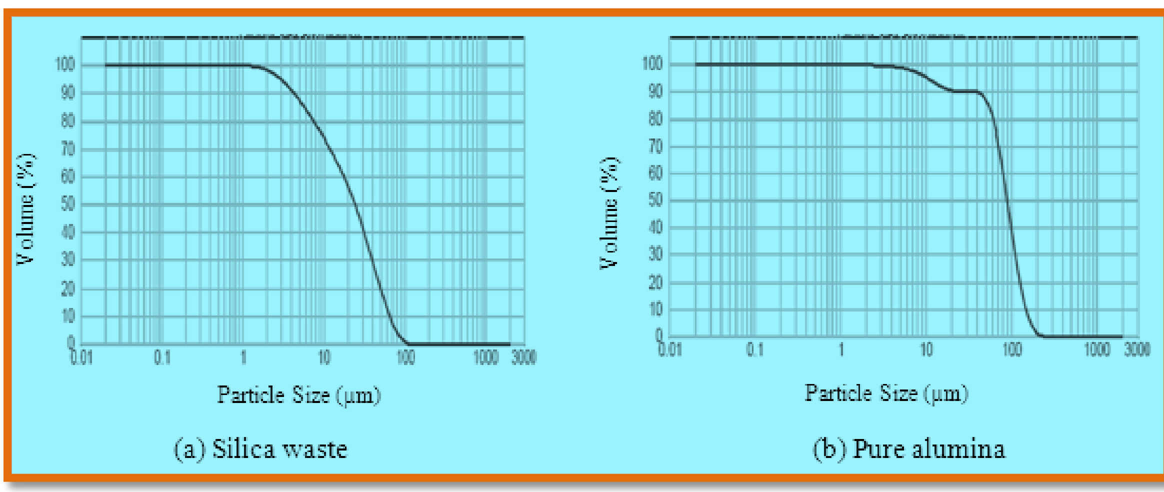

Figure 2: Particle size distribution of (a) Silica waste and (b) Pure alumina.

Table 2: Silica waste and pure alumina particle size distribution.

Samples	Particle size(μm)		
	D(0.1)	D(0.5)	D(0.9)
Silica waste (Not ground) [7]	153.324	288.133	498.317
Silica waste (ground)	4.274	24.461	64.875
Pure alumina (Not ground)	34.751	90.712	149.368

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3.2 Leaching tests of heavy metals

The analysis of heavy metal in leachate obtained from WET reported chromium 1.702 mg/l, lead, copper and zinc less than 0.1 mg/l. The concentrations of all heavy metals were within the standard set by the Ministry of Industry,Thailand. Therefore, it is possible to utilize silica waste for geopolymer mortar production (Table 1).

3.3 The size distribution for the mixed materials

The average particles size of silica waste was 288.133 µm (Sresthaolarn and Chawakitchareon, 2011). Silica waste was grounded in a tube mill until 90% of the particles size smaller than 65 µm. It was no need to process pure alumina as 90% of the particles sizes smaller than 149 µm (Figure 2 and Table 2). To improve the quality of silica waste, particles remaining on sieve No. 325 (aperture of the mesh 45 microns) should not less than 5 percent by weight (ASTM C 618-08).

3.4 The chemical composition of materials

The chemical characteristics of silica waste contains an average of 71.3 percent of silicon dioxide. Silica was main structure in geopolymer (Andini *et al*., 2007). The chemical composition was analyzed by X-ray fluorescence: XRF. The chemical composition of materials used in the experiment compared to different geopolymer material as shown in Table 3.

Table 3: Chemical composition of silica waste compared to different geopolymer materials.

(%) by weight	Type							
	Silica waste	RHBA2	Fly Ash3	PCC-fly ash1	FBC-fly ash1	BFS 4	Kaolin 4	MSWI waste4
SiO2	71.30	84.75	51.5	39.50	21.00	41.10	65.20	26.80
Al2O3	<0.01	0.16	23.63	21.20	8.00	10.70	32.00	12.00
CaO	0.02	2.78	1.74	19.70	42.20	43.50	<0.03	44.70
MgO	<0.01	-	1.84	1.30	0.80	7.60	0.17	3.49
SO3	<0.01	0.60	-	2.70	15.00	-	-	-
Fe2O3	0.08	-	15.30	15.60	6.90	0.41	0.51	2.15
Other oxide	<0.01	-	2.35	-	-	-	-	-
LOI	28.60	3.72	3.32	0.80	1.00	0.31	12.71	24.18

[1]Chindaprasit *et al*., 2011. [2] Songpiriyakil *et al*., 2011.
[3] Temuujin *et al*., 2010. [4] Luna *et al*., 2011.

3.5 The optimum conditions for geopolymer mortar production with silica waste compared to the cement mortar controls

The optimum conditions of geopolymer mortar has binder – silica waste: pure alumina to sodium hydroxide to sodium silicate solution ratio = B (70:10:20) by weight. The samples were tested for the compressive strength at 1, 3, 7, 14, 28, 56 and 90 days (Figure 3). The results indicated that ratio A (60:20:20) passed the compressive strength standard at 180 ksc on 3 day, increased in ranking from compressive strange on 1 to 56 days and decreased on 90 days. The sample of ratio B (70:10:20) passed the compressive strength standard at 180 ksc on 1 day, increase in ranking from compressive strength on 1 to 56 days and decrease on 90 days.

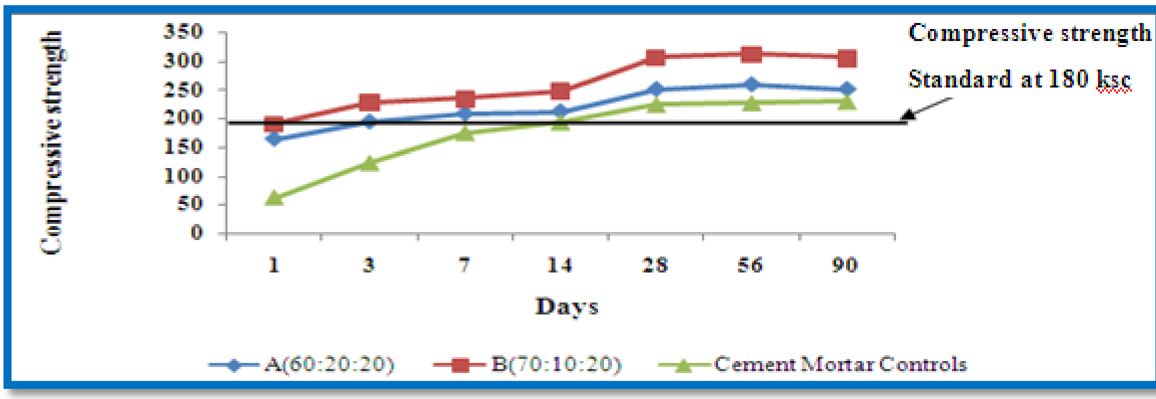

Figure 3: The compressive strength of geopolymer mortar at 1, 3, 7, 14, 28, 56 and 90 days.

The cement mortar control passed the compressive strength standard at 180 ksc on 14 days, and the strength kept on increasing during 90 days. The compressive strengths were swiftly increase during 28 days. Many researchers report the highest compressive strength development on day 28, and compressive strength tends to increase slowly after 90 days. Because aluminosilicate geopolymers gain strength more rapidly than Ordinary Portland cement (OPC) and their ultimate strength can be higher (Klabprasit *et al.*, 2008). There is no Portland cement involved in this cementitious material. The highest compressive strength of the geopolymer mortar ratio A (60:20:20) and ratio B (70:10:20) obtained an average compressive strength of 259 and 312 ksc at 56 days, respectively. It was found that the compressive strengths depend on binder and excellent bonding between the binder sodium hydroxide and sodium silicate (Tailby and Mackenzia, 2010 and Sinsiri *et al.*, 2006). The compressive strengths of geopolymer mortar

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are compared to ready mix concrete mortar (with 10% silica waste addition) shown in Figure 4 (Sresthaolarn and Chawakitchareon, 2011). The results indicated that the compressive strength of geopolymer mortar was more than that of mortar using silica waste replacing 10 percent of cement**.** This result is also supported by the research of Tailby, J. (Tailby and Mackenzia, 2010). Therefore the microscopic examination by electron scanning microscope at 28 days showed that the finer the particle size of the silica waste, the denser the microstructure which confirms by the compressive strength tests shown in Figures 5-7. The microscopic examination by electron scanning microscope at 28 days for the speciment using silica waste replacing 10 percent of cement by weight is shown in Figure 8.

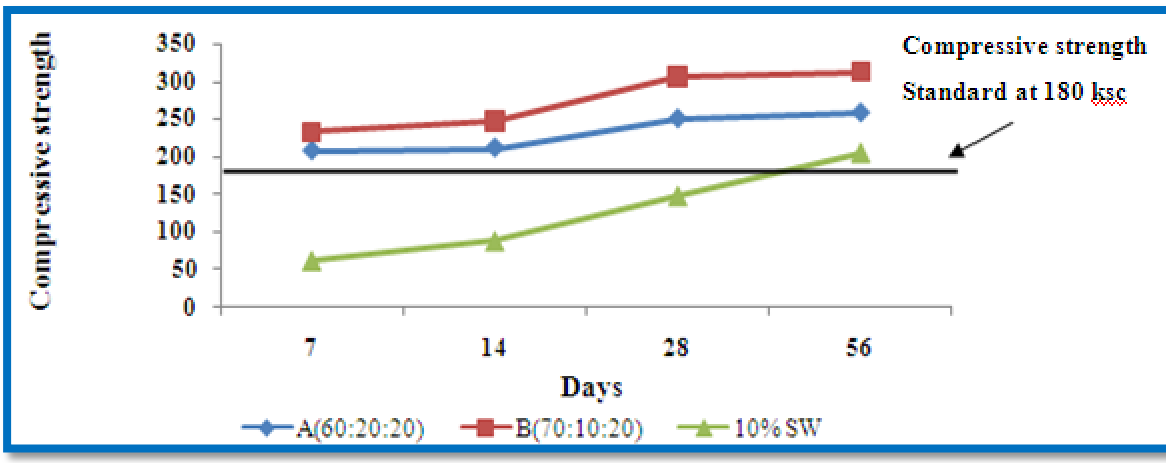

Figure 4: The compressive strength of geopolymer mortar compared to the cement mortar using silica waste replacing 10 percent of cement by weight (Sresthaolarn and Chawakitchareon, 2011). at 7, 14, 28 and 56 days.

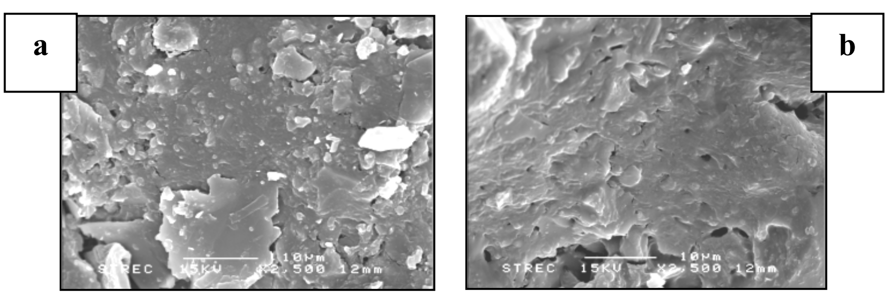

Figure 5: SEM photographs of scanning electron microscope expand 2500 times.: (a) A (60:20:20) and (b) B (70:10:20).

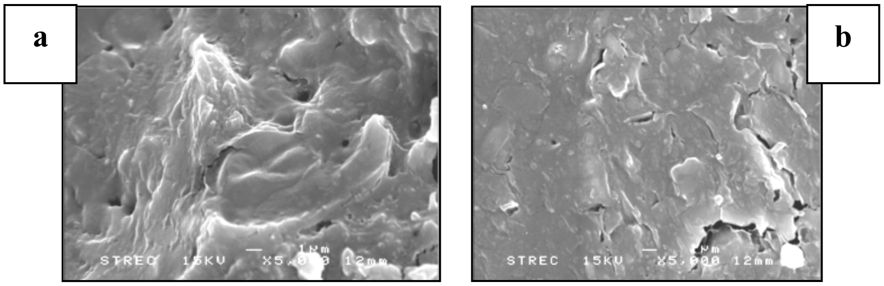

Figure 6: Photographs of scanning electron microscope expand 5000 times. : (a) A (60:20:20) and (b) B (70:10:20).

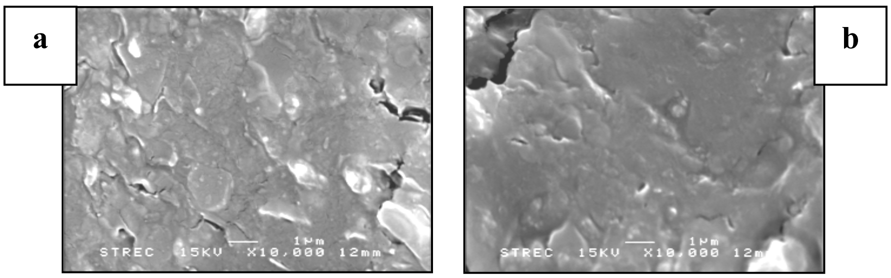

Figure 7: Photographs of scanning electron microscope expand 10,000 times. : (a) A (60:20:20) and (b) B (70:10:20).

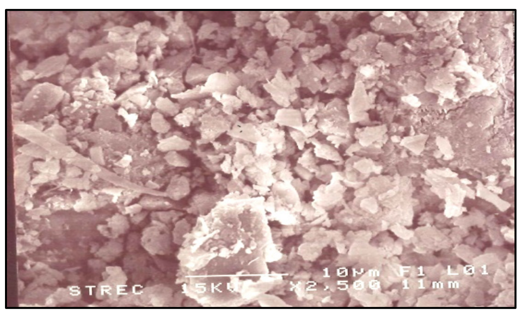

Figure 8: SEM photographs of scanning electron microscope expand 2500 times. The cement mortar using silica waste replacing 10 percent of cement by weight. (Sresthaolarn and Chawakitchareon, 2011).

4. Conclusion

The chemical characteristics of silica waste contained silicon dioxide 71.3 % which similar to pozzolanic material. The optimum conditions of geopolymer mortar which have binder (silica waste: pure alumina) to sodium hydroxide to sodium silicate solution ratio = B (70:10:20) by weight yielded the highest compressive strength equals to 312 ksc at 56 days. The results was obtained from Waste Extraction Test of silica waste, A(60:20:20) and B(70:10:20) indicated that the leachate contains heavy metals, which include lead, copper, zinc and chromium, within the

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acceptable standard set by the Ministry of Industry, Thailand (Ministry of Industry Thailand, 2005).

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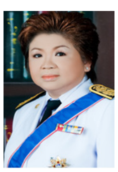

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