

Strength of Geopolymer Cement Curing at Ambient Temperature by Non-Oven Curing Approaches: An Overview

Pitiwat Wattanachai¹, Teewara Suwan^{1,2}

¹Department of Civil Engineering, Faculty of Engineering, Chiangmai University, Thailand

²Center of Excellence for Natural Disaster Management (CENDIM), Chiangmai University, Thailand

teewara.s@cmu.ac.th

Abstract. At the present day, a concept of environmentally friendly construction materials has been intensively studying to reduce the amount of releasing greenhouse gases. Geopolymer is one of the cementitious binders which can be produced by utilising pozzolanic wastes (e.g. fly ash or furnace slag) and also receiving much more attention as a low-CO₂ emission material. However, to achieve excellent mechanical properties, heat curing process is needed to apply to geopolymer cement in a range of temperature around 40 to 90°C. To consume less oven-curing energy and be more convenience in practical work, the study on geopolymer curing at ambient temperature (around 20 to 25°C) is therefore widely investigated. In this paper, a core review of factors and approaches for non-oven curing geopolymer has been summarised. The performance, in term of strength, of each non-oven curing method, is also presented and analysed. The main aim of this review paper is to gather the latest study of ambient temperature curing geopolymer and to enlarge a feasibility of non-oven curing geopolymer development. Also, to extend the directions of research work, some approaches or techniques can be combined or applied to the specific properties for in-field applications and embankment stabilization by using soil-cement column.

1. Introduction

Ordinary Portland Cement (OPC) is one of the most widely used construction materials for concrete making. Its production process is mainly responsible for an enormous amount of carbon dioxide (CO₂) emission to the atmosphere [1]. Therefore, many attempts have been carried out to develop other environmentally friendly materials to achieve the concept of low carbon materials. One among those approaches is to utilise aluminosilicate (pozzolanic) wastes for the production of the OPC-less cementitious binder, which is generally known as Alkaline-activated cement or Geopolymer cement [2]. Geopolymer cement (GP) is usually produced by mixing pozzolanic materials (e.g. fly ash, bottom ash, cement kiln dust, silica fume, rice husk ash or ground granulated blast-furnace slag; GGBFS) with alkaline activators (NaOH and Na₂SiO₃). Heat curing in an oven at above ambient temperature (40 to 90°C for 6 to 48 hours) is then applied to accelerate a geopolymeric reaction and gives strength to the final products. GP sample is afterwards continually cured at ambient temperature until its further handling [3,4]. The properties of heat-cured geopolymer cement have been confirmed to be in the same order or even better than those of OPC in many aspects [5,6]. Nowadays, geopolymer can be produced on a commercial scale as precast reinforced-concrete products, but heat curing (in curing



unit) is still required to achieve good mechanical properties [7-9]. To step over the limitation of using curing units and be more convenient in practical works, the development of geopolymer curing at ambient temperature is, therefore, receiving much more attention [10,11]. This paper provides an overview of the latest alternative curing methods for self-cured geopolymer cement to initiate commercial viability, on-site operation and energy saving aspect.

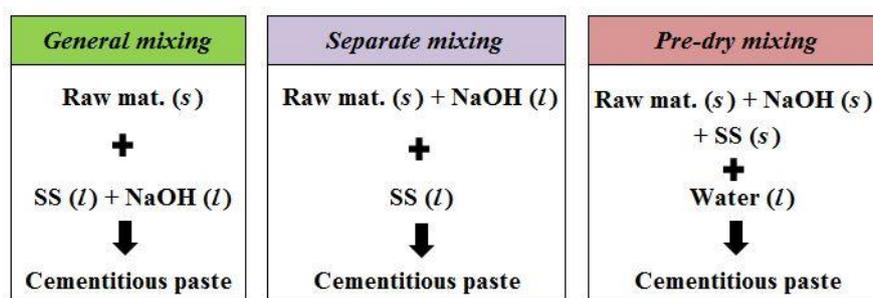
2. Potential factors affecting the strength of geopolymer cement cured in ambient conditions

2.1. Fineness of raw materials

An increase in surface area improves the level of both physical and chemical reactions such e.g. dissolution rate, ions transportation, forming aluminosilicate species which thereby control the initial setting time and geopolymeric gel phase [12]. The experimental data obviously confirmed that the highest strength was achieved by fine fly ash geopolymer, followed by medium-fineness fly ash and coarse fly ash respectively [13]. The similar results were also obtained by using milled fly ash or volcanic rock [14]. The full benefit of those mechanically grinding could be extendedly considered as a viable method for ambient curing with better morphology.

2.2. Mixing sequences and manufacturing procedures

Mixing order is one of the latent factors affecting properties of hardened geopolymer cement. The proper mixing order has been confirmed by many research studies to improve the mechanical properties of any alkaline-activated binder, especially for geopolymer cement [15]. The typical manufacturing process, called General mixing process, starts with the preparation of alkaline activators (e.g. NaOH and Na₂SiO₃). Raw starting materials and those solutions are incorporated and mixed at the same time [16]. A new sequence of adopting the alkaline solution, called Separate mixing process, was also proposed by mixing raw materials with sodium hydroxide solution (NaOH) before adding with sodium silicate solution (Na₂SiO₃). It was found to give better results than other sequences in mechanical properties as NaOH dissolved Si and Al from the origin and then increasingly bonded together in the presence of Na₂SiO₃ [17]. Apart from those two procedures, working with solid activators instead of alkaline solutions was also developed by just mix with called Pre-dry mixing process [18]. The main aim of this dry-mixing approach was primarily focused on the advantage in practical work with adequate strength at the ambient curing temperature (Figure 1).



*S=Solid;l=Liquid;SS=Sodium Silicate;Raw mat.=Raw material

Figure 1. Different manufacturing processes of geopolymer cement

2.3. Type and concentration of alkaline activators

The most widely used alkaline solutions for geopolymer synthesis is a combination of sodium hydroxide and sodium silicate solutions due to its role in dissolve alumina-silicate minerals and initiate formation of geopolymeric gel respectively. A higher concentration gives rise to a stronger ion-pair formation, provides extra dissolution rate and complete faster polycondensation process of particle interface [19]. However, it was reported that too high concentration could lead to an increase of forming coagulated structure and obtain quick setting [20]. A combination of NaOH and Na₂SiO₃

solutions, with the concentration between 8 M to 15 M and between 30 to 50 %w/w respectively, is commonly used for the production of geopolymer cement [21]. However, the concentration level used for ambient-cured geopolymer should be significantly higher than those of typical geopolymer to obtain an extra degree of reaction [22].

2.4. The alternative heat source for curing purposes

Heat curing for geopolymer is typically carried out in electrical ovens, nevertheless, many alternative methods of geopolymer heating were observed for the best possible handling and resultant. For hot climate areas (average temperature around 36 to 42°C), the alternative heat could advocate more degree of geopolymerization to the geopolymer cement, leading to achieving higher strength. It was apparently found that the exposure condition (covered by a transparent plastic sheet and exposed to direct sunlight) was better than in shading due to an alternative heat accumulation inside the samples [23]. Self-internal heat from the massive amount of concrete pouring (huge volume) could be one of the heating sources for geopolymer concrete production. The maximum internal temperature of one cubic yard sample was around 42°C on the first day and reduced slowly to 35°C in next ten days when the ambient temperature was 25 to 30°C. It, therefore, could be applied and counted as a latent heat for geopolymer concrete using in large structure or infrastructure [24]. As aforementioned, the Pre-dry mixing process was primarily focused on the advantage in practical work at the ambient curing temperature. It was found in an internal heat measurement that dry mixing process emitted very high temperature due to the exothermic reaction when hydrating. The Pre-dry mixing method could, therefore, be developed as one of the heat based- self-cured geopolymer cement [25]. Heat from adding high potential energy compounds to geopolymer cement could be possibly counted as one of the alternative heat sources. It was reported that using CaO, Ca(OH)₂ or OPC as additives can shorten the setting time and raise the internal temperature inside the sample [26]. It was also found that, with Ca-based additives geopolymer, the characteristic of heat measurement was close to the general rate of OPC-heat evolution when measured with an isothermal calorimeter (mWatts/g.s or J/g).

2.5. Chemical composition and calcium content in geopolymer mixtures

It was reported that the amount of SiO₂, Al₂O₃ and Na₂O in any form of ratios apparently affects the final properties of hardened geopolymer cement. Therefore, the study of the optimum oxide ratio can be extendedly adjusted to maximize the benefit of geopolymer in ambient curing conditions. SiO₂/Al₂O₃ molar ratio is alternatively used to indicate the presence of Si and Al for geopolymeric bonding. The SiO₂/Al₂O₃ ratio of 2.0 to 3.0 is commonly prepared and resulted in excellent mechanical strength [27]. Na₂O/Al₂O₃ molar ratio and water-to-solid ratio were directly considered to affect the workability and setting time of geopolymer. An increase in sodium oxide (Na₂O) can reduce the setting time due to an efficient dissolution of aluminosilicate materials, leading to additional geopolymerization by the high level of alkalinity [28]. Na₂O/SiO₂ ratio has a role in the acceleration of the chemical shift to complex structures, rising polymeric framework. It was reported that the optimum ratio of Na₂O/SiO₂ to achieve in experiments is around 0.23 to 0.50 [29]. Calcium content in geopolymer mixtures is a key factor to control the setting time. It has been reported that partially added calcium source, such as calcium oxide (CaO), can enhance the mechanical strength of low-calcium geopolymer cement [23]. The similar results of the presence of calcium can also be found in geopolymer synthesis from e.g. high calcium fly ash, bottom ash or GGBFS as prime materials, or even the additional amount of CaO/Ca(OH)₂, cement kiln dust, volcanic ash containing calcium and steel slag, calcium hydroxide (Ca(OH)₂) or even OPC [30]. Calcium mineral could lead to the formation of C-S-H or (C,N)-A-S-H gels within a geopolymeric binder and improve the overall properties significantly and be able to develop for control the setting time of self-cured geopolymer at ambient curing temperature [10].

3. Summary of current knowledge and future research directions for the development of geopolymer cured in ambient conditions

To partially or totally replace the consumption of Portland cement, some of the alternative cementitious materials have been developed, and one among those is geopolymer cement. Most of the geopolymer properties regularly compared with OPC standard, although its formation is entirely different. The main aim of this paper is to summarize the research findings related to geopolymer synthesis on potential factors affecting its properties and characteristics when cured at the ambient temperature which is fineness of materials, manufacturing procedures, alkaline activators, alternative heat source and chemical composition in the geopolymer mixtures. The summary of compressive strength in various scenarios is presented in Table 1. It can be clearly seen that each scenario has its specific condition to achieve the highest performance. More research studies may be launched to the combination of those factors mentioned above for the feasibility of geopolymer production in ambient curing conditions. Most of the recent investigations on geopolymer were on the properties of heat curing process, which is considered as a limitation for in field applications. This study collected and provided the latest research for geopolymer cement curing without oven-heating. The major objective is to elucidate the effects of both chemical and mechanical activation on the mechanical properties of ambient temperature cured geopolymer samples. The combination of some ambient curing techniques would probably lead to skip the oven-curing process and, then be able to perform as low-to-medium strength geopolymer for other specific applications such as embankment stabilization using jet grouting soil-cement column, alternative green materials for pavement or road and infrastructure construction.

Table 1. Compressive strength of geopolymer by prime materials

Prime Materials (% wt)	Additives (% wt)	Compression MPa /Aged ^a	Alkaline Materials	Curing		Sample size	Ref.
				C ^o	Hrs.		
Typical OPC	-	30.3 / 28	-	Ambient		Cube 150 mm ³	[31]
Cement Repair	-	46.1 / 28	-	Ambient		Cube 40 mm ³	[32]
FA class F (90)	-	95.0 / 28	NaOH + Na ₂ SiO ₃	85	20	40×40×160 mm ³	[33]
TMW (90)	Ca(OH) ₂ (10)	75.0 / 56	NaOH + Na ₂ SiO ₃	Ambient		Cube 50 mm ³	[34]
FA (80)	Water Sludge (20)	70.6 / 90	-	Ambient		Cube 38 mm ³	[35]
FA class F (90)	BA (10)	70.0 / 28	KOH + K ₂ SiO ₃	80	24	Cube 50 mm ³	[36]
GGBFS (100)	-	67.0 / 28	NaOH	38	90d ^a	Cube 25 mm ³	[37]
Natural Pozzolan (100)	-	63.0 / 28	NaOH + Na ₂ SiO ₃	Ambient		Cube 20 mm ³	[38]
MK (80)	Steel Slag (20)	44.5 / 28	NaOH + Na ₂ SiO ₃	Ambient		Cube 40 mm ³	[32]
FA (95) class F	Silica fume (5)	35.0 / 28	NaOH + Na ₂ SiO ₃	85	48	Cube 50 mm ³	[39]
Construction waste (70)	MK (20), CaOH ₂ (10)	26.1 / 7	NaOH + Na ₂ SiO ₃	80	24	Cube 25 mm ³	[23]
GBFS (75)	CKD (25)	24.0 / 28	NaOH	38	90d ^a	Cube 25 mm ³	[37]
Waste Paper Sludge (100)	-	17.5 / 28	NaOH + Na ₂ SiO ₃	Ambient		Cube 100 mm ³	[21]

Ferronickel slag (100)	-	15.8/28	NaOH + Na ₂ SiO ₃	60	24	Cube 50 mm ³	[40]
SiltyClay (75)	FA (25)	14.0/28	NaOH + Na ₂ SiO ₃	75	48	Cy. dia. 50x100 mm	[41]

Note: days^a

4. References

- [1] Maholtra, V.M. (2002). Introduction: Sustainable Development and Concrete Technology. *ACI Concr Int*, 24(7).
- [2] Al Bakria, A.M., Kamarudin, H., BinHussain, M., Nizar, I.K., Zarina, Y. and Rafiza, A.R. (2011). The effect of curing temperature on physical and chemical properties of geopolymers. *Physics Procedia*, 22, pp.286-291.
- [3] Chindapasirt, P., Chareerat, T. and Sirivivatnanon, V. (2007). Workability and strength of coarse high calcium fly ash geopolymer. *Cement and Concrete Composites*, 29(3), pp.224-229.
- [4] Wongsuwan S., Paratbhukul S., Dumneunnirun O. and Wattanachai P. (2015). A Study on the Compressive Strength and the Flexural Strength of Geopolymer Containing Ranong China Clay and Rice Husk Ash. Bachelor thesis, Chiangmai University, Thailand.
- [5] Lloyd, N.A. and Rangan, B.V. (2010). Geopolymer concrete: a review of development and opportunities. 35th Conference on Our World in Concrete and Structures, August 2010, pp. 25-27.
- [6] Turner, L.K. and Collins, F.G. (2013). Carbon dioxide equivalent (CO₂-e) emissions: a comparison between geopolymer and OPC cement concrete. *Construction and Building Materials*, 43, pp.125-130.
- [7] Gourley, J.T. and Johnson, G.B. (2005). Developments in geopolymer precast concrete. *World Congress Geopolymer*, Perth, Australia, pp. 139-143.
- [8] Siddiqui K.S (2007). Strength and durability of low calcium fly-ash based geopolymer concrete, Final year Honours dissertation, The University of Western Australia, Perth 2007.
- [9] Cheema, D.S., Lloyd, N.A. and Rangan, B.V. (2009). Durability of geopolymer concrete box culverts-A green alternative. The 34th Conference on Our World in Concrete and Structures, Singapore, August, Vol. 17.
- [10] Suwan, T. and Fan, M. (2014). Influence of OPC replacement and manufacturing procedures on the properties of self-cured geopolymer. *Construction and Building Materials*, 73, pp.551-561.
- [11] Singh, B., Ishwarya, G., Gupta, M. and Bhattacharyya, S.K. (2015). Geopolymer concrete: a review of some recent developments. *Construction and building materials*, 85, pp.78-90.
- [12] Ahmari, S., Ren, X., Toufigh, V. and Zhang, L. (2012). Production of geopolymeric binder from blended waste concrete powder and fly ash. *Construction and Building Materials*, 35, pp.718-729.
- [13] Chindapasirt, P., Chareerat, T., Hatanaka, S. and Cao, T. (2010). High-strength geopolymer using fine high-calcium fly ash. *Journal of Materials in Civil Engineering*, 23(3), pp.264-270.
- [14] Somna, K., Jaturapitakkul, C., Kajitvichyanukul, P. and Chindapasirt, P. (2011). NaOH-activated ground fly ash geopolymer cured at ambient temperature. *Fuel*, 90(6), pp.2118-2124.
- [15] Pacheco-Torgal, F., Castro-Gomes, J. and Jalali, S. (2008). Alkali-activated binders: a review. Part 2. About materials and binders manufacture. *Construction and Building Materials*, 22(7), pp.1315-1322.
- [16] Rattanasak, U. and Chindapasirt, P. (2009). Influence of NaOH solution on the synthesis of fly ash geopolymer. *Minerals Engineering*, 22, pp. 1073-1078.
- [17] Hanjitsuwan, S., Hunpratub, S., Thongbai, P., Maensiri, S., Sata, V. and Chindapasirt, P. (2014). Effects of NaOH concentrations on physical and electrical properties of high calcium fly ash geopolymer paste. *Cement & Concrete Composites*, 45, pp. 9-14.
- [18] Suwan, T., Fan, M. and Braimah, N. (2016). Internal heat liberation and strength development of self-cured geopolymers in ambient curing conditions. *Construction and Building Materials*, 114, pp.297-306.
- [19] Xu, H. and Van Deventer, J.S.J. (2000). The geopolymerisation of aluminosilicate minerals. *International Journal of Mineral Processing*, 59(3), pp.247-266.
- [20] Alonso, S. and Palomo, A. (2001). Alkaline activation of metakaolin and calcium hydroxide mixtures: influence of temperature, activator concentration and solids ratio. *Materials Letters*, 47(1), pp.55-62.
- [21] Anuar, K.A., Ridzuan, A.R.M. and Ismail, S. (2011). Strength characteristics of geopolymer concrete containing recycled concrete aggregate. *International Journal of Civil & Environmental Engineering*, 11(1), pp.59-62.
- [22] Suwan, T. (2016). Development of self-cured geopolymer cement. Doctoral dissertation, Brunel University London.
- [23] Khater, H., 2011. Effect of calcium on geopolymerization of aluminosilicate wastes. *Journal of Materials in Civil Engineering*, 24(1), 92-101.
- [24] Vaidya, S., Diaz, E.I. and Allouche, E.N. (2011). Experimental evaluation of self-cure geopolymer concrete for mass pour applications. *World of coal ash (WOCA) conference*, May 2011, (pp. 9-12). [25] Suwan, T., and Fan, M. (2016). Effect of manufacturing process on the mechanisms and mechanical properties of fly ash-based geopolymer in ambient curing temperature. *Materials and Manufacturing Processes* (2016): 1-7.
- [26] Yip, C.K., Lukey, G.C. and Van Deventer, J.S.J. (2005). The coexistence of geopolymeric gel and calcium silicate hydrate at the early stage of alkaline activation. *Cement and Concrete Research*, 35(9), pp.1688-1697.
- [27] Burciaga - Díaz, O. and Escalante - García, J.I. (2012). Strength and Durability in Acid Media of Alkali Silicate - Activated Metakaolin Geopolymers. *Journal of the American Ceramic Society*, 95(7), pp.2307-2313.
- [28] Ghosh, K. and Ghosh, P. (2012). Effect of Na₂O/Al₂O₃, SiO₂/Al₂O₃ and w/b ratio on setting time and workability of fly ash based geopolymer. *International Journal of Engineering Research and Applications*, 2, pp.2142-2147.
- [29] De Vargas, A.S., Dal Molin, D.C., Vilela, A.C., Da Silva, F.J., Pavão, B. and Veit, H. (2011). The effects of Na₂O / SiO₂ molar ratio, curing temperature and age on compressive strength, morphology and microstructure of alkali-activated fly ash-based geopolymers. *Cement and concrete composites*, 33(6), pp.653-660.
- [30] Suwan, T., Fan, M. and Braimah, N. (2016). Micro-mechanisms and compressive strength of Geopolymer-Portland cementitious system under various curing temperatures. *Materials Chemistry and Physics*, 180, pp.219-225.
- [31] Raijiwala, D. & Patil, H., 2010. Geopolymer concrete: A green concrete. 2010 2nd International Conference on Chemical, Biological

- and Environmental Engineering (ICBEE 2010), 2 to 4 November 2010. Cairo, Egypt, IEEE, 202-206.
- [32] Hu, S., Wang, H., Zhang, G. & Ding, Q., 2008. Bonding and abrasion resistance of geopolymeric repair material made with steel slag. *Cement and Concrete Composites*, 30(3), 239-244.
- [33] Fernández-Jiménez, A., García-Lodeiro, I. & Palomo, A., 2007. Durability of alkaliactivated fly ash cementitious materials. *Journal of Materials Science*, 42(9), 3055-3065.
- [34] Pacheco-Torgal, F., Castro-Gomes, J. & Jalali, S., 2009. Tungsten mine waste geopolymeric binder: Preliminary hydration products investigations. *Construction and Building Materials*, 23(1), 200-209.
- [35] Kongkaew, B., 2007. Sludge-Based Geopolymer. Master Thesis Bangkok: Kasetsart University.
- [36] Hardjito, D. & Fung, S., 2010. Fly ash-based geopolymer mortar incorporating bottom ash. *Modern Applied Science*, 4(1), 44-52.
- [37] Khater, H., 2012. Effect of cement kiln dust on geopolymer composition and its resistance to sulfate attack. *International Journal of Civil and Structural Engineering*, 2(3), 740.
- [38] Allahverdi, A., Mehrpour, K. & Kani, E., 2008. Taftan pozzolan-based geopolymer cement. *IUST International Journal of Engineering Science*, 19(3), 1-5.
- [39] Dutta, D., Thokchom, S., Ghosh, P. & Ghosh, S., 2010. Effect of silica fume additions on porosity of fly ash geopolymers. *Journal of Engineering and Applied Sciences*, 5(10), 7479.
- [40] Komnitsas, K. & Zaharaki, D., 2007. Geopolymerisation: A review and prospects for the minerals industry. *Minerals Engineering*, 20(14), 1261-1277.
- [41] Sukmak, P., Horpibulsuk, S. & Shen, S., 2013. Strength development in clay-fly ash geopolymer. *Construction and Building Materials*, 40, 566-575.