

Optimization of Fly Ash Geopolymer Concrete Mixtures in a Seawater Environment

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Synopsis: Fly ash geopolymer concrete has many constituents and different mixture proportions based on the characteristics of local materials. The optimization of mixture proportions, which is subject to some performance constraints, can be a time consuming task. The objective of this research is to optimize the parameters of fly ash geopolymer concrete using Taguchi orthogonal design to produce concrete that meets the durability requirements in an aggressive environment. In this research, the compressive strength, wetting-drying cycle and capillary pores measurement were investigated by measuring the effects of aggregate content, alkaline solutions/fly ash ratio, ratio of sodium silicate to NaOH and curing condition. For optimization, a three-level factorial experimental design for each parameter was employed. Based on the experimental results, the optimized conditions of a fly ash geopolymer concrete mixture for aggressive environments were proposed.

Keywords: concrete mixture, fly ash, geopolymer, seawater, Taguchi

1. Introduction

Geopolymer concrete is a new potential construction material. It is not only beneficial for the environment, but also as an alternative of a sustainable material for construction. Geopolymer material is obtained from a reaction of source material like fly ash, slag, kaolin with alkaline activators, that produce aluminosilicate rather than calcium silicate hydrate bonds for structural integrity. Earlier research shown that strength properties of the geopolymer concrete were comparable to the strength of OPC concrete (1-3). In fact, the durability of this material in the long term is actually an interesting area to be explored for its technical viability.

Geopolymer has a higher resistance in some aggressive environments than the OPC concrete. This concrete can withstand a high temperature up to 800°C (4). Geopolymer concrete has also been reported to having a good chemical stability in sulfuric acid and sulfate environments (5-7). Low calcium fly ash geopolymer, in particular, can resist up to 10% sulphuric acid because it has low calcium content in the mixture. It was found that in a sulfate environment, there was no significant weight loss of this type of concrete. A seawater environment is a type of aggressive environment that contains chloride and sulfate ions. Both ions can attack and destroy a normal concrete severely. Furthermore, a tidal zone in a marine environment provides an extreme condition of wetting and drying. When the concrete is exposed to the wetting by seawater and drying by air, the concrete can deteriorate more quickly than fully submerged zones or in normal atmospheric zones. It was found that the fly ash geopolymer concrete has good resistance to seawater (8). There was no weight loss because of the seawater and the strength increased with time. Metakaolin geopolymer immersed in seawater has a fluctuated flexural strength with time (9). It was also found that the porosity of the concrete immersed in seawater remained lower after 270 days, than that immersed in deionized water and sulphuric acid. Recent findings have shown that the fly ash geopolymer has a low chloride diffusion coefficient (10). Since a chloride diffusion coefficient depends on the permeability of the concrete itself, the variability on the raw materials and mixture proportions used in that study should be highlighted.

The durability of the geopolymer concrete not only depends on its strength, but also the microstructure of the concrete. Concrete with a small number of pores, a dense microstructure and has low permeability is considered durable. Some researchers have found that strength is not the sole indicator of geopolymer concrete durability. For example, fly ash geopolymer concrete with strength around 35-65 MPa has water absorption values of 3-5% (7, 11, 12). Those water absorption values were typical for the fly ash geopolymer cured for 12-24 hours at a temperature of 60-70°C. Those values classify the geopolymer concrete as a concrete with average permeability (13).

Durability of fly ash geopolymer concrete can be influenced by many factors. It was found that the ratio of sodium silicate to NaOH is important in a strength development. The optimum ratio of 2.5 produced strength of 40 MPa (1). Regarding to durability, this ratio can influence a final product of

geopolymer. Curing is a main important parameter for both strength and durability of geopolymer concrete. Fly ash geopolymer concrete needs to be cured in a high temperature to accelerate a reaction of geopolymerisation (14). Curing duration, temperature and type of curing have been investigated by various researchers. Curing for at least 24 hours at 60°C can produce a concrete with strength of 30-90 MPa (1). Other findings showed that curing for only 12 hours at 70°C was significant to obtaining the good concrete (16). High temperatures may damage the microstructure, since the concrete becomes more porous even though it has a high strength. Then the optimum high temperature curing was found important to develop concrete that has low porosity.

The brief literature review above shows that the ratio of sodium silicate/NaOH and curing condition are some important parameters affecting the durability of geopolymer concrete. In addition to the above parameters, alkaline/fly ash ratio and aggregate content, which are also important for tailoring durable concrete, were also investigated. To obtain a durable concrete that meets the requirement in an aggressive environment, there is a need to understand the effects of these individual factors and the interactions between them. This paper presents the optimization of geopolymer concrete by using Taguchi orthogonal design for durability and some of its properties.

2. A study of geopolymer concrete mixture optimization

2.1 Taguchi experimental design

In conventional experimental design, one factor at a time is a common approach. This means one is kept varied and the other factors are kept constant. This approach is costly and time consuming, particularly in testing all parameters. For a material like the geopolymer concrete with its many related variables, it is important to find the most efficient way to obtain an optimum mix design for a certain application.

Taguchi experimental design is a popular statistical method for quality engineering. This method is used in the production area to achieve optimum results from a variety of good combinations. It can save on the cost and time of doing many trials to try all possible combinations. In concrete technology, the Taguchi orthogonal array was found suitable for designing different mixtures with different effects and constituents (17). In this method, every combination of levels appears in the same repetitions. This approach creates the opportunity for all variables to be tested to obtain the optimum mixture. Table 1 shows a combination of OA₉ (3⁴) orthogonal array that can produce nine trial mixes (18).

Table 1. OA₉ (3⁴) orthogonal array

Trial	Factor A	Factor B	Factor C	Factor D
T1	1	1	1	1
T2	1	2	2	2
T3	1	3	3	3
T4	2	1	2	3
T5	2	2	3	1
T6	2	3	1	2
T7	3	1	3	2
T8	3	2	1	3
T9	3	3	2	1

Four key parameters that can have a significant influence on achieving a durable geopolymer concrete were selected. The experimental levels of the variables (maximum and minimum), aggregate content, alkaline solution to fly ash ratio, sodium silicate to NaOH ratio and curing method are defined. Table 2 shows the factors and levels used in this research.

Table 2. Factors and levels

Factor	Level 1	Level 2	Level 3
A: aggregate content (kg/m ³)	1800	1848	1896
B: alkaline solution/fly ash ratio	0.30	0.35	0.40
C: sodium silicate/sodium hydroxide ratio	1.5	2	2.5
D: curing method	24 hours 60 degree Celsius	12 hours 70 degree Celsius	24 hours 75 degree Celsius

The choice for each factor and level was based on intensive literature reviews of factors affecting the durability of geopolymer concrete. Aggregate content was a new parameter introduced to study the effect of aggregate content on geopolymer concrete durability. Since the aggregate comprises of 75-80% of the concrete, then the amounts of 75%, 77% and 79% by concrete unit weight were adopted. The alkaline solution/fly ash ratio was a significant factor in improving the properties of the geopolymer concrete. The ratio of 0.3-0.4 was used based on the previous finding (19). A reduction in the alkaline solution/fly ash ratio can yield low water absorption values. The ratio of sodium silicate to sodium hydroxide was taken in the range of 1.5-2.5 to optimize the alkaline solution used in this research. Three curing methods were considered. The curing method of 24h-60°C was considered from Hardjito (1). The shorter curing time 12h-70°C was adopted from van Jaarsveld (16) and the elevated temperature curing at 75°C for 24 hours was used according to Shindunata (14).

2.2 Materials and mixture proportioning

The materials used in this study were fly ash, NaOH, sodium silicate, locally available aggregates and superplasticizer. Low calcium fly ash from Collie Power Station was used as the main source material. The chemical composition of the fly ash can be seen in Table 3. Sodium hydroxide with 98% purity was diluted with water to get a 14M solution. The sodium silicate type D with a modulus silicate of 2 ($\text{SiO}_2 = 29/4\%$, $\text{Na}_2\text{O} = 14.7\%$, water = 55.9% by mass solution) was used as one of the alkaline activators. To improve the workability, naphthalene sulphonate-based superplasticizer, with a dosage of 1.5% by mass of the fly ash, was included in the mixture. Aggregates used in this research were crushed granite coarse aggregate and a local sand dune fine aggregate. Coarse aggregate consists of a combination of 7, 10 and 20 mm particle size. The combinations of the aggregates meet a grading requirements for combined aggregates according to British Standard BS 882: 1992 (20) Those aggregates were prepared in a saturated surface dry condition to ensure no excess water in the mixes.

Table 3. Chemical composition of fly ash (XRF analysis)

Elements	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	Na_2O	P_2O_5	SO_3	LOI
% by mass	50.3	26.3	13.6	2.27	0.55	1.44	0.36	1.58	0.32	0.54

A total of nine concrete mixes were cast for this research according to the orthogonal array arrangement (Table 4). This table was used to obtain optimum mix proportions in this study. Each mix has a different variable but every combination of levels appears for the same number of times.

Table 4. Mixture proportions of geopolymer concrete

Trial	Factor			
	Aggregate content (kg/m^3)	Alkaline/fly ash ratio	Ratio of Sodium Silicate/NaOH	Curing condition
T1	1752	0.3	1.5	24h 60°C
T2	1752	0.35	2	12h 70°C
T3	1752	0.4	2.5	24h 75°C
T4	1800	0.3	2	24h 75°C
T5	1800	0.35	2.5	24h 60°C
T6	1800	0.4	1.5	12h 70°C
T7	1848	0.3	2.5	12h 70°C
T8	1848	0.35	1.5	24h 75°C
T9	1848	0.4	2	24h 60°C

Table 5 shows the mixture proportion detail of geopolymer concrete with $\text{H}_2\text{O}/\text{Na}_2\text{O}$ ratio was kept constant at 12.50 to obtain workable mixes (14). The proportions were determined by a basic calculation of geopolymer concrete mixture (15). Geopolymer concrete was made by mixing aggregates with alkaline solutions, superplasticizer and water. To measure the workability, a conventional slump test was carried out. All mixes were cast in 100x200mm cylinders and steam cured using different curing methods according to the parameters in Table 1. After that, those specimens were air cured in the curing room with temperature of 23-24°C until testing time. For all mixes, compressive strength tests were carried out at 1, 7 and 28 days of the age of concrete. Those mixes were also tested for accelerated wetting-drying, sorptivity and water absorption. The accelerated wet-dry cycle in seawater was performed to simulate the combined wetting and drying conditions of seawater in a marine environment (21). At the age of 7 days, those specimens were

immersed in a 6.54% NaCl solution for 24 hours. Then, the specimens were weighed and put in the oven with temperature of 80°C to undergo a drying process for another 24 hours. The same procedure was repeated for 10 cycles (20 days). At the end of the test, those samples were observed visually and tested for compressive strength to check on the change in compressive strength.

Table 5. Proportions of geopolymer concrete mixtures used in the study

Mix	T1	T2	T3	T4	T5	T6	T7	T8	T9
Fly ash (kg/m ³)	498.46	480.00	462.86	461.54	444.44	428.57	424.62	408.89	394.29
Aggregates (kg/m ³)	1752	1752	1752	1800	1800	1800	1800	1800	1848
NaOH 14M	59.82	56.00	52.90	46.15	44.44	68.57	36.40	57.24	52.57
Sodium silicate	89.72	112.00	132.24	92.31	111.11	102.86	90.99	85.87	105.14
Superplasticizer	6.1	6.1	6.1	6.1	6.1	6.1	6.1	6.1	6.1
Added water	26.47	23.65	21.23	18.61	18.55	28.51	15.97	24.46	21.47
SiO ₂ /Na ₂ O	0.85	0.97	1.05	0.86	0.96	0.92	0.85	0.82	0.93
SiO ₂ /Al ₂ O ₃	3.76	3.86	3.96	3.79	3.89	3.87	3.82	3.81	3.92
Na ₂ O/Al ₂ O ₃	0.42	0.48	0.54	0.46	0.51	0.51	0.49	0.50	0.56
H ₂ O/Na ₂ O (design)	12.50	12.50	12.50	12.50	12.50	12.50	12.50	12.50	12.50
H ₂ O/Na ₂ O (actual)	12.50	12.36	12.28	11.71	11.76	13.13	11.18	12.45	12.26

The sorptivity test was carried out based on a Determination of Sorptivity test by GHD Group (formerly Taywood Engineering Limited). In this method, cylinders with height of 100mm and diameter 100mm were cut and dried at the oven with a temperature of 105°C until constant mass before use. Those specimens were immersed in shallow water and the weight of each sample was taken periodically. The weight gain of the specimen at set time intervals of 5, 10, 30 minutes, 1, 2, 3 and 4 hours was measured. The water temperature used was 20°C. The water absorption and AVPV test was carried out based on ASTM C642-06, Standard Test Method for Density, Absorption and Voids in Hardened Concrete. In the water absorption test, a cylindrical specimen is cut into slices of a thickness of 50mm. The result is the average value of the absorption data for the slices. The specimens are dried in an oven for not less than 24 hours at temperature a 100-110°C and cooled to a temperature of 23±2°C. Then the specimens are immersed in water at 23±2°C for not less than 48 hours. Next, the mass of specimens is measured after the surface is dried with a towel. In the VPV test, the specimens are boiled for 5.5±0.5 hours and allowed to cool while still immersed in the water. Then the mass is determined after the specimens are surface dried and weighed in water. Those specimens were tested at the age of 28 days.

2.4 Results and Discussions

Table 6 shows the measured slump and the resulted strength. The slump values of all mixes were in a range of 140-270 mm. Geopolymer mixes are usually cohesive and tacky. The strength for all mixes varied depending on their composition and curing condition. Low water content specimens produced higher strength than the mixes with high water content. A reduction of the water to sodium ratio can increase the strength but at the same time reduce the workability and difficult to be compacted. Those mixes T3, T4, and T7 tend to show a high early strength. The difference between one day and 28 days curing was not significant for them. Both mix T6 and T9 show the lowest compressive strength due to high water content in the mix and insufficient binder content.

Table 6. Slump values and compressive strength of geopolymer concrete

Trial Mix	Combination	Slump (mm)	Compressive strength (MPa)		
			1 day	7 days	28 days
T1	A1B1C1D1	260	37.81	39.52	39.93
T2	A1B2C2D2	260	34.56	35.31	37.09
T3	A1B3C3D3	270	49.67	49.89	49.64
T4	A2B1C2D3	245	41.92	40.93	42.51
T5	A2B2C3D1	250	32.45	37.55	38.69
T6	A2B3C1D2	270	25.17	27.16	28.64
T7	A3B1C3D2	140	54.10	52.29	54.89
T8	A3B2C1D3	250	32.40	34.53	35.73
T9	A3B3C2D1	250	25.86	29.29	29.71

The average contribution of each level of a factor in Table 1 to the final strength was calculated by adding the strength of mixtures corresponding to this level and dividing the sum by repeating the number of times for this level. This method was used to determine the effect of each variable to the strength of the mixes. The plotted trend for the observed compressive strength can be seen in Figure 1. In this study, the proposed best mix is the one with the highest strength. It was found that the amount of sodium silicate/NaOH ratio had the most significant effect among the four factors. An increase in the amount of the sodium silicate produces a high strength concrete, especially at early ages. Judging from all factors that influence the strength development of the geopolymer concrete, it can be suggested that the best combination is the new mixture A1B1C3D3. This combination has an aggregate content of 1752 kg/m^3 , alkaline solution/fly ash ratio of 0.3, sodium silicate/NaOH ratio of 2.5 and curing method at temperature of 75°C for 24 hours.

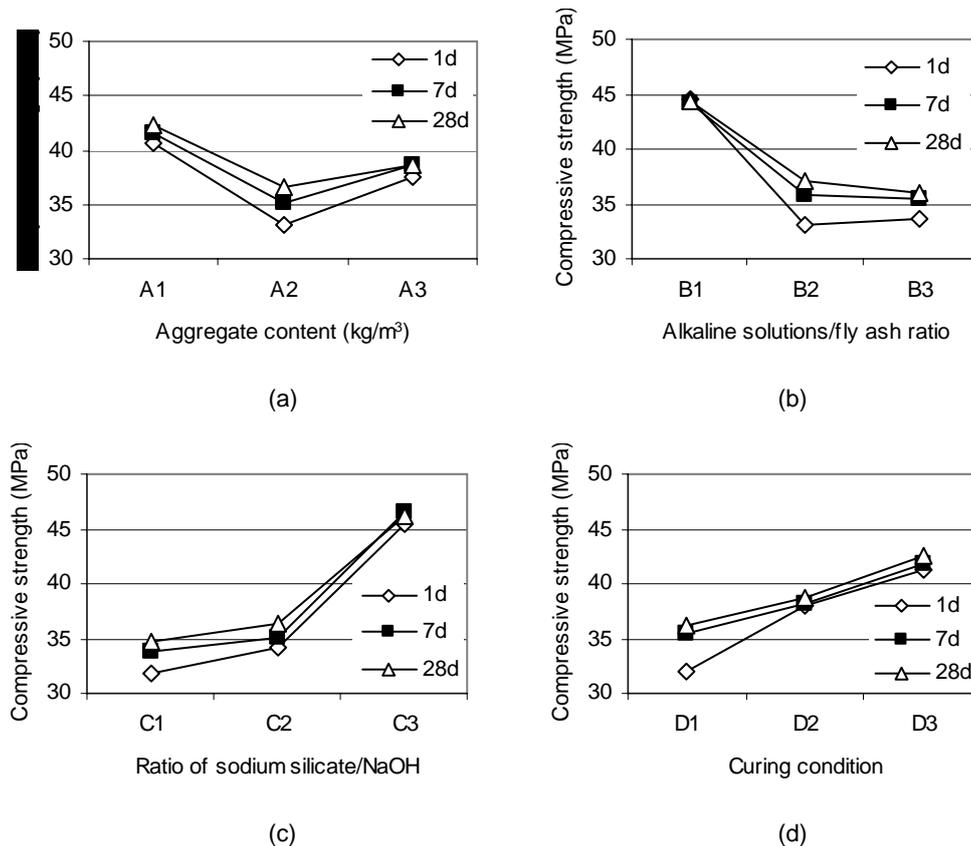


Figure 1. Relationship between compressive strength and variables at each value: (a) aggregate content, (b) alkaline solutions/fly ash ratio, (c) ratio of sodium silicate/NaOH, (d) curing condition

Table 7. Test results of trial mixes

Trial Mix	Combination	Compressive strength after wetting-drying (MPa)	Change in compressive strength (%)	Mass loss during drying process (%)	Total weight change (%)	Sorptivity ($\text{mm}/\text{min}^{0.5}$)	VPV (%)
T1	A1B1C1D1	52.62	31.78	2.65	101.51	0.1324	8.86
T2	A1B2C2D2	50.44	35.99	2.78	101.79	0.1344	9.54
T3	A1B3C3D3	59.48	19.82	2.80	101.14	0.1174	9.87
T4	A2B1C2D3	55.48	30.51	2.55	100.45	0.1034	8.33
T5	A2B2C3D1	47.87	23.73	2.59	101.54	0.1280	9.09
T6	A2B3C1D2	38.20	33.38	3.14	101.57	0.1806	9.95
T7	A3B1C3D2	69.81	27.18	1.97	101.04	0.0805	7.42
T8	A3B2C1D3	42.11	17.86	2.76	100.69	0.1538	8.96
T9	A3B3C2D1	37.92	27.63	2.92	101.73	0.1561	10.60

Table 7 shows the results from further tests to obtain the best mix for durability in a seawater environment. The wetting-drying cycle in salt water and oven drying, sorptivity, and VPV to indicate a

closed porosity were used in this study. It can be seen from the table that all mixes achieved high compressive strength after the 10 cycles of wetting-drying in salt water. The change in the compressive strength is in the range of 17-35%. This behaviour shows that a drying cycle incorporating a high temperature of 80°C might contribute to a further reaction of geopolymerization. The further reaction can increase the geopolymer strength. After a wet-dry cycle, the mass loss was determined. The mass loss was in the range of 1.97-3.14%, which is considered small for a concrete tested in an aggressive environment. It was found that the sorptivity value related to the strength of geopolymer concrete. For high strength mixes, the sorptivity values tend to be lower, despite their high closed porosity (VPV) percentage. It can be seen from the Table 7, that mix T7 has the best properties for durability in a salt water environment. This mix has the highest compressive strength before and after the cycle, the smallest mass loss, the lowest sorptivity and VPV. Those properties indicated the concrete can have a good performance in a seawater environment.

The effects of changing each component of the mixture while holding all other components in a constant ratio are shown in Figure 2-4. The same method of determining strength was applied to obtain the figures. For each test, the average contribution of each level of a factor in Table 7 to the final mass loss, sorptivity, and VPV were calculated. These values were obtained by adding those indicated values of mixtures corresponding to the level and dividing the the sum by the number of a repeated action at this level.

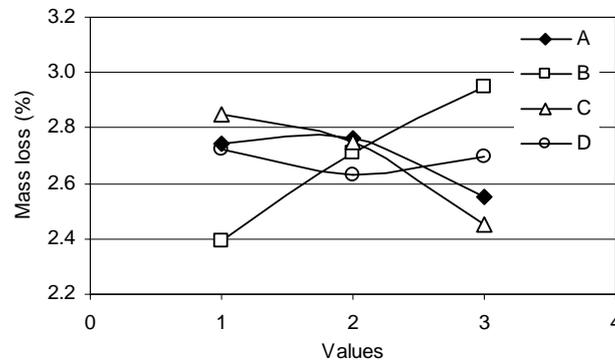


Figure 2. The effect of variables on the mass loss

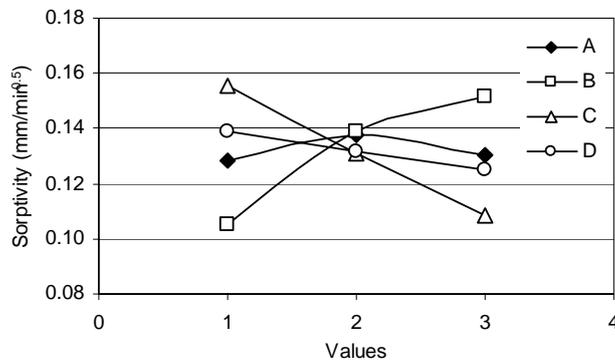


Figure 3. The effect of variables on the sorptivity

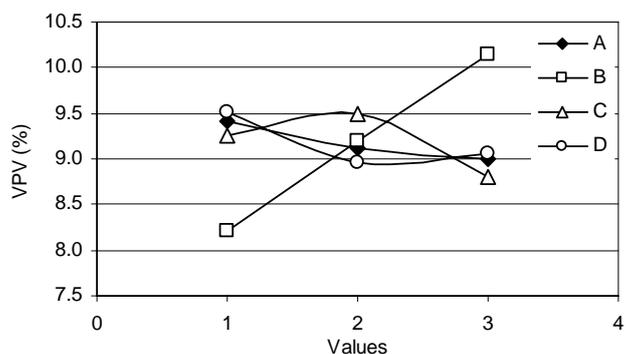


Figure 4. The effect of variables on the VPV

The effect of variables on the mass loss, sorptivity and VPV can be explained as follows. From Figure 2 it can be seen that the optimal combination to getting a low value of mass loss is A3B1C3D2. This combination coincidentally had a similar combination to mix T7. Mix T7 has the smallest mass loss of those mixes. Next, from Figure 3, the low value of sorptivity can be obtained using a combination of mix A1B1C3D3. It was found that the best combination for sorptivity had a similar proportion to those values proposed in determining high strength concrete. Figure 4 shows the effects of variables to concrete closed porosity, in term of VPV percentage. From the figure, the optimal combination to gaining low VPV values is A3B1C3D2, which has a similar combination to mix T7. This means mix T7 can serve as a durable mix composition in seawater, since the mass loss and VPV are low.

A new set of optimum mixtures is shown in Table 8 for further evaluation. Mix T7 has been chosen for its desirable properties in strength and durability. It also has a shorter curing duration which is efficient for geopolymer concrete manufacturing. Mix T10 was yielded from a new combination A1B1C3D3 to obtain high strength concrete with low sorptivity. This mix needed to be evaluated to meet durability standard of concrete in a marine environment. Mix T4 has a moderate strength but good workability and durability after mix T7. By reducing the H_2O/Na_2O ratio, a new high strength mix can be produced.

Table 8. Proposed optimum mixture proportion of fly ash geopolymer concrete

Mix	Combination	Component			
		Aggregate content (kg/m^3)	Alkaline/fly ash ratio	Ratio of Sodium Silicate/NaOH	Curing condition
T7	A3B1C3D2	1848	0.3	2.5	12h 70 ⁰ C
T10	A1B1C3D3	1752	0.3	2.5	24h 75 ⁰ C
T4	A2B1C2D3	1800	0.3	2.0	24h 75 ⁰ C

2.5 An accelerated wetting-drying cycle

A study of the wetting-drying cycle of geopolymer concrete to represent a marine environment has been carried out for this purpose. The test consists of immersion of the specimens in salt water with a concentration of 6.54% for 24 hours followed by drying for 24 hours in the oven at a temperature of 80⁰C. It is interesting to note that after 10 cycles of this test, the geopolymer concrete showed no signs of cracking because of thermal shock or strength degradation.

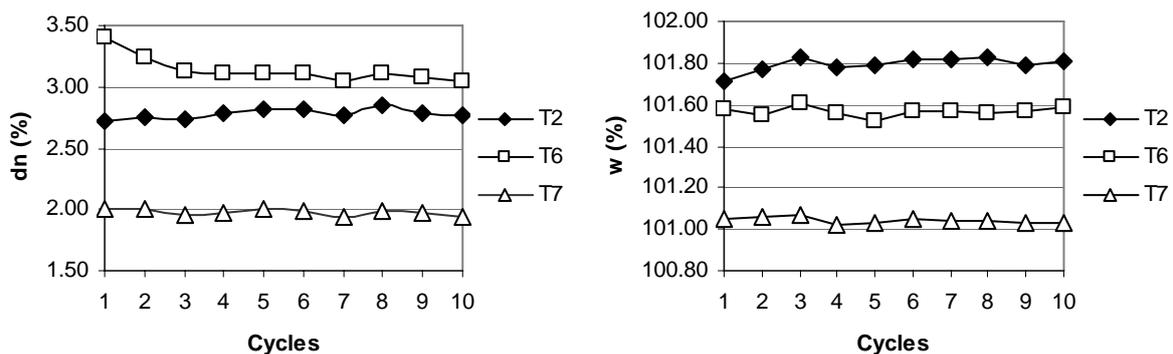


Figure 5. Weight loss during drying process (dn) and total weight change (w) of specimens with curing method of 12 hours at 70⁰C

In general, those mixes cured in a curing regime of 12 hours at 70⁰C showed a constant change in weight loss during the drying process and a total weight change (Figure 5). Mix T7 had the lowest weight loss and total weight change. High strength concrete and a low level amount of pores might have contributed to this behaviour. In Figure 6, the geopolymer concrete mixes cured with a curing method of 24 hours at 75⁰C showed a slight declining trend for all mixes.

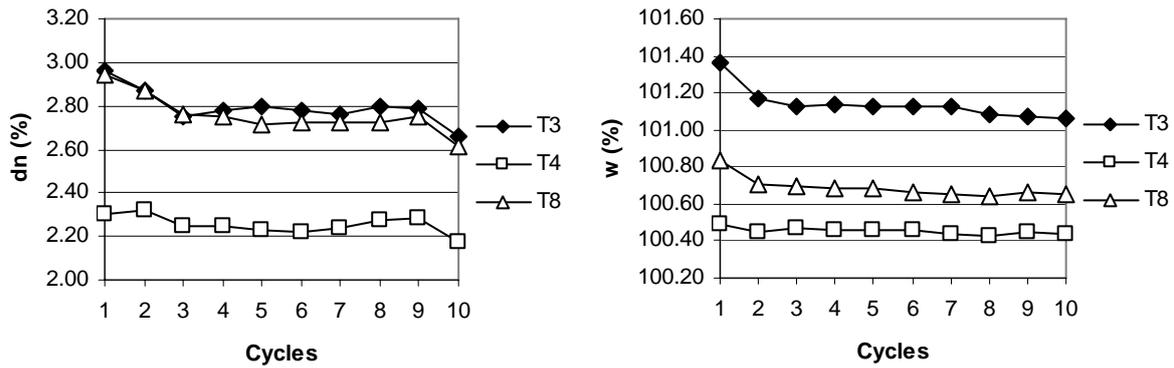


Figure 6. Weight loss during drying process (dn) and total weight change (w) of specimens with curing regime of 24 hours at 75°C

A fluctuating trend of mass loss for geopolymer concrete cured for 24 hours at 60°C was shown in Figure 7. Although mix T5 has the highest strength among those mixes, it showed the lowest mass loss and a fluctuated total weight change. Despite their fluctuating figures, there was a declining trend of those concrete mass losses. Perhaps such a curing method contributes to this behaviour. From this finding, it can be seen that the curing method was also a significant parameter for the durability of fly ash geopolymer concrete.

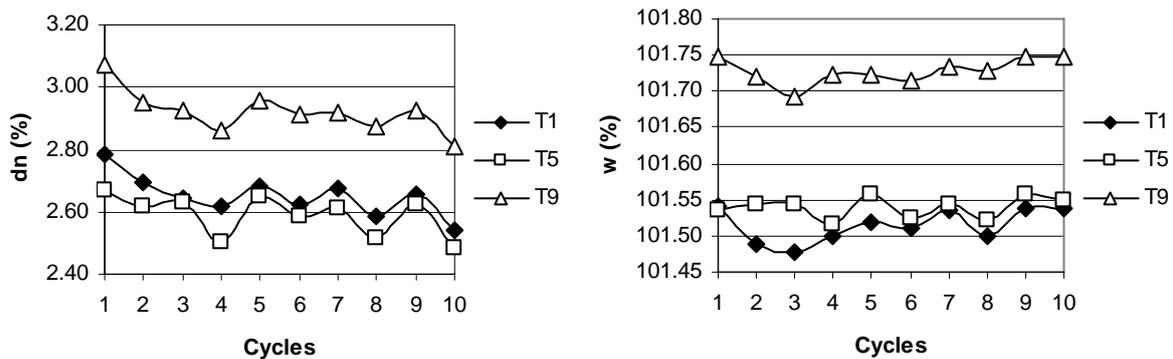


Figure 7. Weight loss during drying process (dn) and total weight change (w) of specimens with curing regime of 24 hours at 60°C

3. Conclusions

The effects of aggregate content, alkaline/fly ash ratio, ratio of sodium silicate/NaOH and curing condition to obtain a durable mixture for a seawater environment were studied. The Taguchi orthogonal array method was found to be well suited to optimising of fly ash geopolymer concrete mixtures. This method can reduce the number of trial batches needed to achieve a durable geopolymer concrete with special requirements. One mix has been proposed for further investigation due to its durable characteristics, and two other mixes for their possible durable properties. In addition, a study of the wetting-drying cycle in 6.54% salt water and oven drying at 80°C has been carried out to investigate geopolymer behaviour in such extreme conditions. It was found that the curing method was the most important parameter for a durable fly ash geopolymer concrete.

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