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Cite as: AIP Conference Proceedings **1887**, 020063 (2017); <https://doi.org/10.1063/1.5003546>  
Published Online: 29 September 2017

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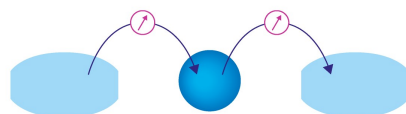
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# Durability of Metakaolin Geopolymers with Various Sodium Silicate/Sodium Hydroxide Ratios against Seawater Exposure

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**Abstract.** This work presents an investigation of the performance of metakaolin geopolymers exposed to the continuous immersion of seawater. The geopolymers were prepared from metakaolin by activating with a mixture of sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and sodium hydroxide (NaOH) solutions and cured at 80°C. The ratios of sodium silicate to sodium hydroxide were varied from 0.20 to 0.32. The result showed that metakaolin geopolymers reduce in strength after immersion in seawater for 28 days. The unexposed samples with highest compressive strength attained greatest strength retention. White deposits were formed on the surface of the geopolymers after the exposure to seawater which was believed due to the depolymerisation process of the geopolymer network. Even so, the metakaolin geopolymers did not substantially change in dimension and remain structurally intact..

## INTRODUCTION

Concrete has been widely used in the building and construction industry with Ordinary Portland Cement (OPC) as its main binder. However, the production and use of OPC has contributed to the global warming with the abundant emission of carbon dioxide (CO<sub>2</sub>) into the atmosphere [1-4]. Even though the application of OPC concrete has been utilised since decades ago, the concern about the resistance towards harsh condition such as marine environment is still at disadvantage where immense deterioration occurred such as strength loss and structural deterioration. A study done by Sotya et al. [5] has shown that OPC concrete shown a strength loss when immersed in seawater for 90 days. In seawater, the aggressive ions, mainly chlorides and sulphates are the major cause for the strength loss and structural cracking.

The world has shown a great interest in sustainability whereby durability of the concrete has become one of the main concerns. As a result, the needs for alternative materials to partially substitute the use and minimise the drawback of OPC concretes become the utmost importance. Geopolymers are a new class of building materials produced by synthesising pozzolanic compounds or aluminosilicate materials with alkaline solution. The geopolymerisation reaction between aluminosilicate materials and alkaline activator produces the polymeric chain of Si-O-Al-O bonds [6-9]. The current biggest contribution of geopolymers was in the construction industry as alternative binders for

concretes [10] which mainly due to low carbon dioxide (CO<sub>2</sub>) emission and also the excellent physical and mechanical properties and good durability [11, 12]. Numerous research in geopolymer has been done towards the effects of manufacturing parameters on physical and mechanical properties while less attention has been paid to the durability study of geopolymer [13-16]. Several numbers of works have been carried out emphasising the strength properties and durability of geopolymers in aggressive environment such as acid solution, sulphate solution and fire [12, 17-20].

Hence, the objective of the current study was to evaluate the physical changes and mechanical properties of the metakaolin geopolymers after exposure to seawater. The metakaolin geopolymers were produced using different sodium silicate-to-sodium hydroxide ratio and its performance after 28 days exposed to seawater was evaluated according to visual observation, change in weight and compressive strength.

## EXPERIMENTAL METHOD

### Materials

Metakaolin supplied by Associated Kaolin Industries Sdn. Bhd., Malaysia, was used as the Si-Al source material. The metakaolin was obtained by calcining the kaolin at 850 °C for 6 h in the furnace. The chemical composition of metakaolin determined by X-ray fluorescence (XRF) spectrometer is shown in Table 1. The particle size distribution of metakaolin was obtained using a Malvern particle size analyser. The mean particle size  $d(50)$  was 9.465  $\mu\text{m}$ . The sodium hydroxide (NaOH) powder was of caustic soda micropearls and 99% purity with the brand name of Formosoda-P, made in Taiwan. The sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) solution was supplied by South Pacific Chemicals Industries Sdn. Bhd., Malaysia, with a chemical composition of 30.1% SiO<sub>2</sub>, 9.4% Na<sub>2</sub>O and 60.5% H<sub>2</sub>O.

**TABLE 1.** Chemical composition of metakaolin as determined by XRF analysis.

Chemical	Wt (%)
	Metakaolin
SiO <sub>2</sub>	55.7
Al <sub>2</sub> O <sub>3</sub>	38.6
Fe <sub>2</sub> O <sub>3</sub>	2.03
TiO <sub>2</sub>	0.78
CuO	0.03
ZrO <sub>2</sub>	0.04
K <sub>2</sub> O	2.43
MnO <sub>2</sub>	0.04
LOI	1.74

### Preparation of Metakaolin Geopolymers

12M NaOH solution was prepared in a volumetric flask using distilled water. The alkaline activator solution was formed by mixing Na<sub>2</sub>SiO<sub>3</sub> and NaOH solutions at a preferred ratio until a clear solution was obtained. The sodium silicate-to-sodium hydroxide ratios (Na<sub>2</sub>SiO<sub>3</sub>/NaOH) used in this study was 0.20, 0.24, 0.28, 0.30 and 0.32 (Table 2). In order to produce geopolymer paste, metakaolin was mixed with the prepared activator solution. Then the paste was poured into steel moulds with a dimension of 50 × 50 × 50 mm. The moulded samples were sealed with a thin film to prevent moisture loss. The samples were cured in the oven at 80 °C for 24 h.

**TABLE 2.** Mixture composition details of metakaolin geopolymer samples.

Sample name	S/L ratio	Na <sub>2</sub> SiO <sub>3</sub> /NaOH	Curing	
			Temperature (°C)	Time (h)
MkG0.20	0.8	0.20	80	24
MkG0.24	0.8	0.24	80	24
MkG0.28	0.8	0.28	80	24
MkG0.30	0.8	0.30	80	24
MkG0.32	0.8	0.32	80	24

### Exposure to Seawater

In order to test the durability of metakaolin geopolymer in seawater, the cured metakaolin geopolymer samples were immersed in the seawater (Fig. 1). An extended drying was required to remove any moisture in the geopolymer samples before exposed to seawater. The samples were put in a tank with a continuous flow of seawater sourced from the North Sea and maintained at room temperature. After 28 days, the samples were removed from the seawater and characterised.



**FIGURE 1.** Immersion of metakaolin geopolymers in seawater.

### Test and Analysis Method

Change in weight of metakaolin geopolymers was calculated by measuring the weight before and after exposure to seawater. Firstly, the weight of the sample was taken after drying process as the initial weight ( $W_1$ ). Then, after immersed in seawater for 28 days, the sample was cleaned out from the residue to obtain the weight after exposure to seawater ( $W_2$ ). Lastly, the weight change was calculated using Eqn. (1).

$$\text{Changes of weight (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

where  $W1$ = Initial weight of the geopolymer sample after drying and  $W2$ = Weight of geopolymer sample after 28 days seawater exposure.

The compressive strength of metakaolin geopolymers before and after 28 days exposure to seawater was measured in accordance to ASTM C109 by using Instron machine series 5569 Mechanical Tester. The testing set-up is shown in Fig. 2.



FIGURE 2. The set up for compressive strength testing.

## RESULTS AND DISCUSSION

### Visual Observation

Figure 3 and 4 show the surface condition of metakaolin geopolymers before and after exposure to seawater, respectively. The seawater showed a minimum effect to the metakaolin geopolymers as the samples were still in good condition without any change in dimensions. The most significant observation was the formation of white deposits on the surface of the sample which relates to the depolymerisation process due to breaking of the Si-O-Al-Si of the geopolymer network [17]. Metakaolin geopolymer samples exposed to seawater showed almost no visual signs of deterioration despite the existing crack (Fig. 3) after the extended drying. As can be seen in Fig. 4, the samples immersed in seawater were in good condition with no change in shape and remained structurally intact.

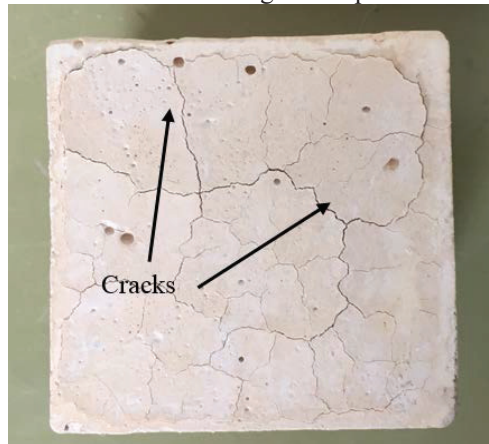
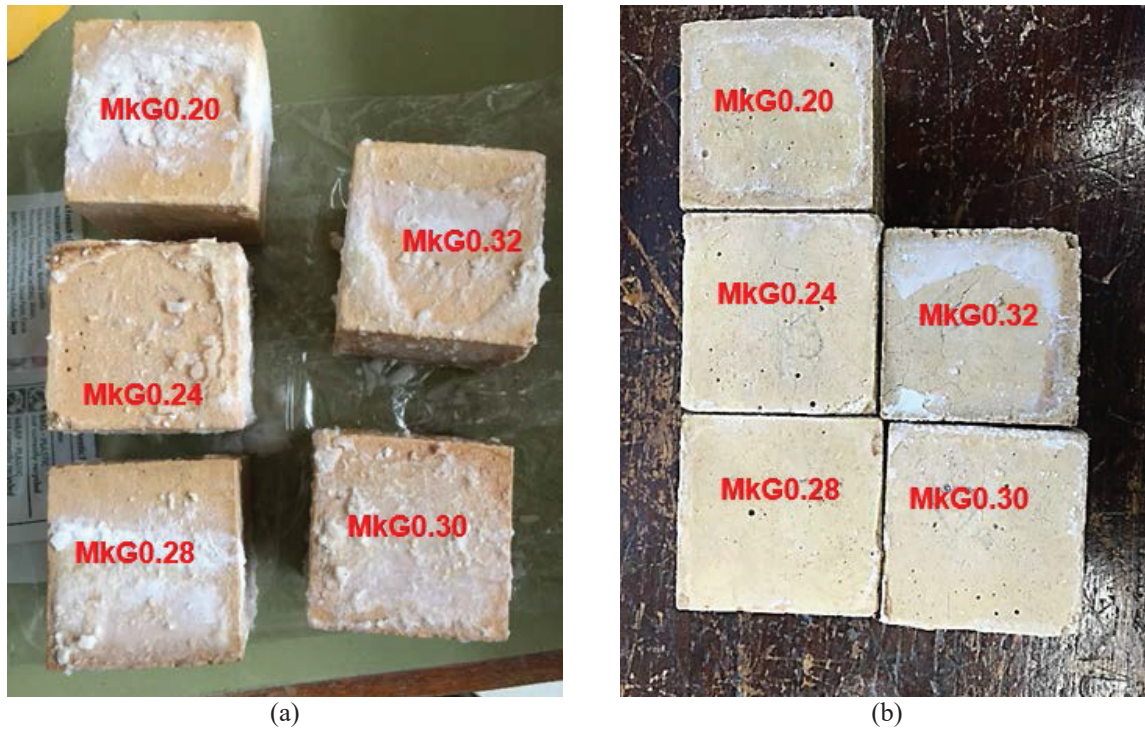


FIGURE 3. The surface condition of metakaolin geopolymers before exposure to seawater.





**FIGURE 4.** The surface condition of the metakaolin geopolymers (a) upon taken out from seawater exposure and (b) after scraping off the white deposits.

### Weight Change

Table 3 shows the percentage weight gain of metakaolin geopolymers after seawater immersion. The weight of metakaolin geopolymers exposed to seawater was compared with the weight before the exposure. All samples experienced an increase in weight. There were white deposits or known as salts appeared on the surface of the sample which probably caused weight gain after the immersion. The MkG0.24 ( $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 0.24) had the highest weight gain of 37.54% while MkG0.30 ( $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 0.30) had the lowest weight gain of 30.28%. The weight gain was in the range of 30.28% to 37.54% which is relatively small. Similar observation was reported by Thokchom et al. [17] where the weight of fly ash geopolymers were increased after immersed in sulphate solution for 24 weeks.

**TABLE 3.** Weight gain of the metakaolin geopolymers after 28 days exposure to seawater.

Sample name	<i>W1</i>	<i>W2</i>	Weight gain (%)
MkG0.20	150.20	204.50	36.15
MkG0.24	154.50	212.50	37.54
MkG0.28	153.70	211.00	37.28
MkG0.30	153.00	208.50	36.27
MkG0.32	151.60	197.50	30.28

## Compressive Strength

Table 4 shows the compressive strength of the metakaolin geopolymers before and after 28 days exposure to seawater. The strength loss is also presented. For unexposed sample, the compressive strength of metakaolin geopolymers increased with the increase in Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio up to 0.30 and dropped significantly when the ratio was increased to 0.32. The compressive strength of the samples were 8.72, 9.60, 10.65 and 14.70 MPa for MkG0.20, MkG0.24, MkG0.28 and MkG0.30, respectively. The maximum strength at high Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio of 0.30 signified that high liquid sodium silicate content is required for geopolymerisation process. However, the ratio was considered as excessive beyond 0.30 because the excess silicate hinders water evaporation and structure formation which affects the geopolymerisation process.

After exposure to seawater, the compressive strength reduced for all samples. The compressive strengths after seawater exposure were 1.82, 2.90, 4.12, 7.14 and 4.20 MPa for MkG0.20, MkG0.24, MkG0.28, MkG0.30 and MkG0.32 respectively. The greatest strength retention was achieved by MkG0.30. This decreasing trend of compressive strength result was in distinct contrast to work done by Sotya et al. [5] whereby the strength of the metakaolin geopolymers did not decrease after immersion in seawater. The loss of strength in this work might be attributed to the appearance of cracks on the samples (Fig. 3) after drying process and before the exposure to seawater. The minor cracks promoted penetration pathways for the seawater to attack the geopolymer network and hence resulted in strength loss. The dissolution of the soluble part of the metakaolin geopolymers in seawater also affected the geopolymer network and consequently decreased the compressive strength [17, 21-23].

**TABLE 4.** Compressive strength of unexposed and exposed metakaolin geopolymers to seawater.

Sample name	Compressive strength (MPa)		Strength loss (%)
	Unexposed	Exposed	
MkG0.20	8.72	1.82	79.13
MkG0.24	9.6	2.9	69.79
MkG0.28	10.65	4.12	61.31
MkG0.30	14.7	7.14	51.43
MkG0.32	14.32	4.2	70.67

## CONCLUSIONS

In this work, the resistance of metakaolin geopolymers toward sea water attack was investigated. The metakaolin geopolymers with different Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratios showed different performance upon seawater exposure. The deteriorative environment of seawater significantly affected the physical and mechanical properties of the geopolymers. The most durable geopolymer samples with Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratios of 0.30 which has the lowest strength loss after the exposure. The decrease in compressive strength after the immersion was related to the cracks existed in the sample which provides an easy pathway for the ions migration in and out of the sample and leads to depolymerisation of the geopolymer network. Regardless of reduced compressive strength, the metakaolin geopolymers were still able to hold the structure intact after 28 days exposed to seawater

## ACKNOWLEDGEMENTS

The author would like to acknowledge Centre of Excellence of Geopolymer and Green Technology (CeGeoGTech) for financial support under 'Partnership for Research in Geopolymer Concrete' (H2020 MSCA RISE 2015 689857)

PRIGeoC) and School of Marines Sciences, at University of Plymouth, United Kingdom for providing access to equipment used in this work.

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