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Utilization of seawater in high calcium fly ash alkali-activated paste cured at ambient temperature

Athika Wongkvanklom [1a], Patcharapol Posi*[2], Chaiwat Bangsai [2b], Pornnapa Kasemsiri [3c], Piyawat Foytong [3d], Sumrerng Rukzon [4e] and Prinya Chindaprasirt [5f]

[1] *Department of Civil and Environmental Engineering, Faculty of Science and Engineering, Kasetsart University, Chalermphrakiat Sakon Nakhon Province Campus, Sakon Nakhon 47000, Thailand*
[2] *Department of Civil Engineering, Faculty of Engineering, Rajamangala University of Technology Isan, Khon Kaen Campus, Khon Kaen 40000, Thailand*
[3] *Sustainable Infrastructure Research and Development Center, Faculty of Engineering, Khon Kaen University, Khon Kaen 40002, Thailand*
[4] *Department of Civil Engineering, Faculty of Engineering, Rajamangala University of Technology Rattanakosin, Nakhon Pathom, 73170 Thailand*
[5] *Sustainable Infrastructure Research and Development Center, Department of Civil Engineering, Faculty of Engineering, Khon Kaen University, Khon Kaen 40002, Thailand, and Academy of Science, Royal Society of Thailand, Dusit, Bangkok 10300, Thailand*

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Abstract. The influence of seawater upon high calcium fly ash alkali activated paste (AAP) on setting time and strength was studied. The materials included high calcium fly ash, sodium hydroxide (NaOH), sodium silicate (NS) and seawater. Setting time and compressive strength of the mixes were tested. The variables included seawater content, liquid to fly ash ratio, NaOH concentration, and NS/NaOH ratio. All mixes were cured at ambient temperature. Seawater was used instead of distilled water to prepare NaOH solution. The incorporation of seawater resulted in the shortening of setting time and increasing compressive strength. The strength enhancement was highly significant when the seawater is used with low NaOH concentration of 5 molar which is cost attractive. The use of seawater with short setting time and sufficiently high strength AAP is useful in applications such as patch repair of pavement and sidewalk, precast sections and other applications in places such as remote coastal areas and islands where fresh water is scarce.

Keywords: alkali activated paste; high calcium fly ash; seawater; setting time; strength

1. Introduction

There are several steps involved in Portland cement production such as mining, transporting raw materials and firing. Various activities cause impacts on the environment and natural resources. As a result, various problems such as forest degradation, soil erosion, water and air pollution, and carbon dioxide emission are posing problems. To lessen the adverse effect, alternative binders including alkali-activated materials (AAM) were developed to complement and reduce the use of Portland cement. Starting materials from by-products from industries such as fly ash, slag, and bio-mass ash were used (Becerra-Duitama and Rojas-Avellaneda 2022, Chindaprasirt *et al*. 2009, Wongkvanklom *et al*. 2021). Water is an essential component of ecosystems and living things. Although water is a renewable resource, 97.5 percent of the total water on earth is seawater. A major part of water (1.9 percent of water) is in the form of ice cap and glacier and the remaining 0.6 percent is fresh water and support life (Mishra and Dubey 2015). Therefore, a number of research have studied the use of seawater in concrete. For Portland cement concrete, it has been shown that the incorporation of seawater is feasible (Morteza *et al*. 2020, Younis *et al*. 2018). The use of seawater instead of freshwater in cement paste accelerated setting times because the chloride in seawater enhances the hydration of cement (Etxeberria *et al*. 2016, Morteza *et al*. 2020). Due to the fact that sulfate and magnesium are also present in seawater, they also influence the setting time and strength of the product (Zhang *et al*. 2022). The use of seawater in Portland cement concrete is thus feasible with some concern on the adverse effect on the corrosion of steel

*Corresponding author, Associate Professor,
E-mail: Patcharapol.po@rmuti.ac.th
[a] Assistant Professor
E-mail: athika.w@ku.th
[b] Master Student
E-mail: chaiwat.ba@rmuti.ac.th
[c] Associate Professor
E-mail: pornkas@kku.ac.th
[d] Associate Professor
E-mail: Piyafo@kku.ac.th
[e] Associate Professor
E-mail: Sumrerng.ruk@rmutr.ac.th
[f] Professor
E-mail: prinya@kku.ac.th

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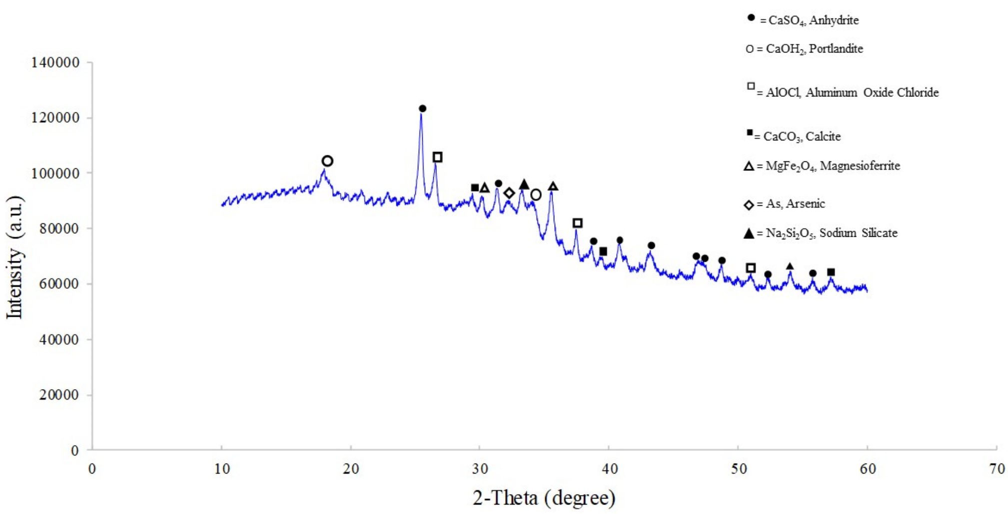


Fig. 1 XRD of fly ash

Table 1 Chemical composition of fly ash

Chemical compositions	Fly ash (%by weight)
SiO_2	36.20
Al_2O_3	15.52
Fe_2O_3	14.25
CaO	22.57
K_2O	1.63
Na_2O	0.33
SO_3	8.90
LOI	0.88

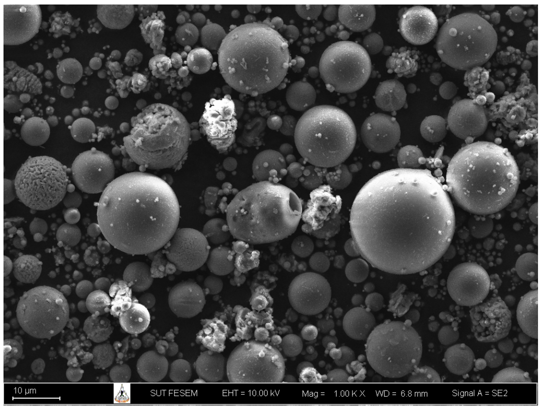


Fig. 2 SEM of fly ash

reinforcement.

In general, good fly ash alkali activated paste AAP is obtained with the use of sodium hydroxide (NaOH) and sodium silicate (NS) as alkali-activators (Chindaprasirt and Cao 2015). The chemical reaction is usually accelerated with temperature curing around 40-75°C (Chindaprasirt *et al*. 2007). It has been shown that seawater could be used in conjunction with sodium silicate in the production of AAM with some reduction in compressive strength (Harmaji 2022, Luhar and Luhar 2020). On the other hand, the incorporation of seawater was also reported to have little effect on the strength of fly ash AAM but a large effect on workability (Lyu *et al*. 2022, Siddique and Jang 2020). For low calcium fly ash, the activation with NaOH and NS results in mainly N-A-S-H gel. For high calcium fly ash, high content of calcium results in the formation of additional C-S-H and C-A-S-H gel. The presence of C-S-H should favor the enhancement of strength development due to the use of seawater as in the case of Portland cement with seawater.

Recently, the use of seawater and sea sand in alkali activated slag concrete (Yang *et al*. 2019), seawater and coral sand in alkali activated low calcium-slag concrete (Tong *et al*. 2021) been studied with encouraging finding on the resistance to chloride ion permeability. There is, however, still a dearth of information on the use of seawater in the alkali activated high calcium fly ash concrete. This work is thus another step towards the use of seawater in alkali activated high calcium fly ash concrete. The results obtained from this research should promote the use of alkali activated material in an even more sustainable way.

2. Experimental detail

2.1 Materials

High calcium fly ash from Mae Moh Power Plant, Lampang Province, Thailand, sodium silicate (NS), sodium hydroxide (NaOH) and seawater were the materials used. The specific gravity of fly ash was 2.55 with 18.6 μm median particle size. Its chemical compositions as shown in Table 1 contained 36.20% SiO_2, 15.52% Al_2O_3, 14.25% Fe_2O_3, and 22.57% CaO, with a LOI of 0.88%. It can be classified as the fly ash Type C according to ASTM C618-22 (ASTM C 618-19 2019). The mineral composition by X-ray diffraction analyzer (XRD) are given in Fig. 1. The microstructural characterization of fly ash by Scanning Electron Microscope (SEM) is given in Fig. 2. The chemical composition of seawater is shown in Table 2. The NS solution with 15.32% Na_2O, and 32.87% SiO_2, and the NaOH solutions at 5, 10, and 15 molars were used.

Table 2 Chemical composition of seawater

Chemical Composition	Seawater (%by weight)
Calcium	1.14
Chloride	57.50
Sulfate	6.70
Magnesium	3.83
Potassium	1.39
Sodium	29.43

Table 3 Weight ratios of AAP mixes

Series	Seawater content (%)	L/FA ratio	NaOH (M)	NS/NaOH ratio
I Seawater content	0,20,40, 60,80,100	0.5	10	1.00
II L/FA	0,100	0.5, 0.6, 0.7	10	1.00
III NaOH concentration	0,100	0.5	5, 10, 15	1.00
IV NS/NaOH	0,100	0.5	10	1:3.2:3, 3:3,3:2

*AAP = alkali activated paste, L/FA = liquid/fly ash ratio, NaOH = sodium hydroxide, NS = sodium silicate, NS/NaOH = sodium silicate/sodium hydroxide ratio.

2.2 Sample preparation

First, fly ash and NaOH solution were mixed in the mixer for 5 minutes. Then the NS solution was added and the mixing was continued for another 5 minutes (Rattanasak and Chindaprasirt 2009). The mixture was cast into 50 mm cubes and then wrapped with plastic wrap. After 24 hours, the samples were demolded and again wrapped and stored at ambient temperature (25-29°C).

2.3 Experimental design

To study the effects of seawater content, the liquid/fly ash ratio (L/FA), NaOH concentration, NS/NaOH ratio were the variables for the study of the AAP cured at ambient temperature (25-29°C). Distilled water was used to eliminate the possible variation in chemicals in tap water. However, the use of tap water will be a common practice in the real world. The experiments were designed into 4 series with the details shown in Table 3.

2.3.1 Series I Seawater content

The distilled water was replaced with seawater at 0, 20, 40, 60, 80, and 100 % for the preparation of sodium hydroxide solution with 10 M NaOH. The NS/NaOH=3:3, L/FA=0.5 were used.

2.3.2 Series II L/FA

In this series, the L/FA of 0.5, 0.6, and 0.7 were used with 10 M NaOH, NS/NaOH=3:3, and seawater only.

2.3.3 Series III NaOH concentration

The 5, 10, and 15 M NaOH with NS/NaOH = 3:3, L/FA = 0.5, and seawater only were used in this series.

2.3.4 Series IV NS/NaOH

The NS/NaOH ratios of 1:3, 2:3, 3:3, and 3:2 were used with 10 M NaOH, L/FA ratio = 0.5, and seawater only.

It should be noted here that in this study, the sequential sets of experiment were used to gauge the effect of four variables viz., seawater content, liquid to fly ash ratio (L/FA), NaOH concentration, and NS/NaOH ratio. This method is simple and gives guided directions of the effect of the individual variable. For additional information, orthogonal or factorial experimental design could be used to obtain the interaction and optimal solution.

2.4 Test details

2.4.1 Compressive strength

The compressive strength of AAP was tested at 28 days as per ASTM C109/C109M-20 (2020).

2.4.2 Setting time

The setting time of fresh AAP was tested using Vicat needle as per ASTM C191-21 (2021).

2.4.3 Chloride content

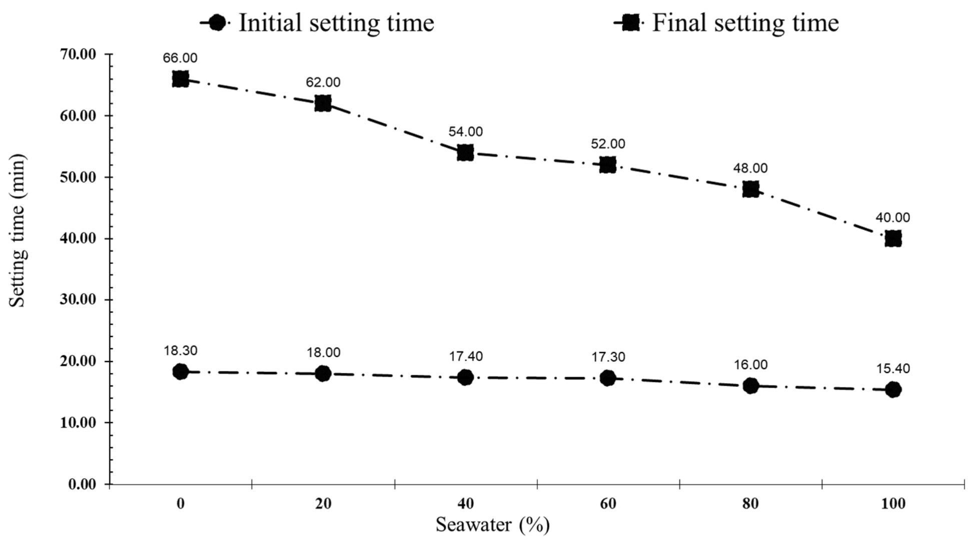
For series I Seawater content, the actual chloride content of each mix was measured in accordance with ASTM C1218/C1218M-20 (2020). The sample was drilled to obtain the paste powder which was analyzed for the chloride content directly.

3. Results and discussions

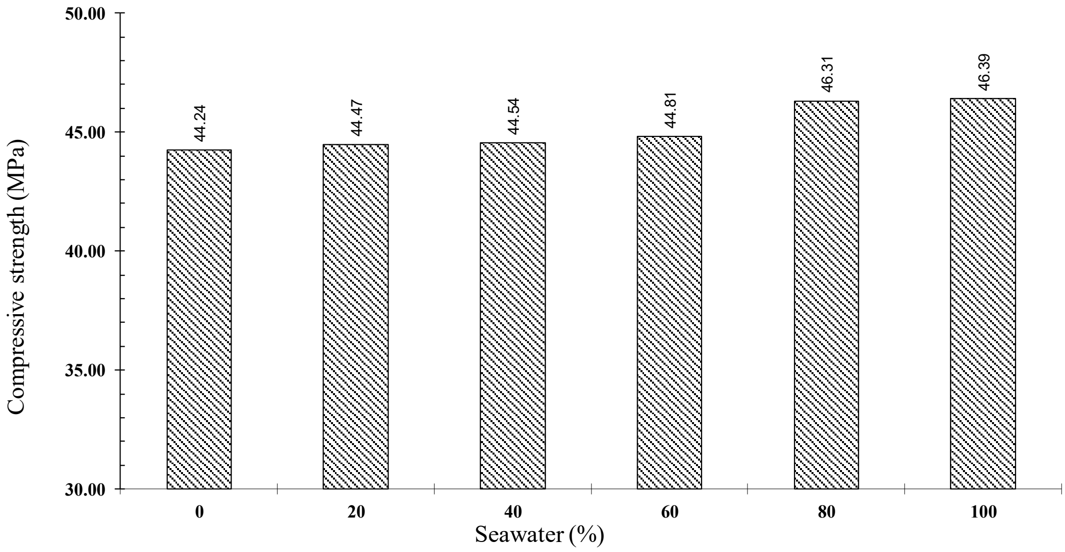
3.1 Series I seawater

The test results of setting times of AAP with various amount of seawater is shown in Fig. 3(a). The use of seawater instead of distilled water in the preparation of NaOH solution had a small effect on initial setting time but final setting time was significantly reduced. The initial and final setting time of AAP with distilled water were 18.30 and 66.00 minutes and reduced to 15.40 and 40.00 minutes, respectively.

Seawater with a high salt content accelerates the alkali activation process making the reaction products work faster (Sun *et al*. 2023). The initial setting time of calcium silicate slag-based AA mortars mixed with seawater was faster than mortars mixed with tap water (Shi *et al*. 2019). It is generally accepted that seawater reduces setting time of ordinary Portland cement (Ghazal *et al*. 2021, Lv *et al*. 2020). Whereas, the ground granulated blast furnace slag-fly ash AAM with seawater also showed the reduction in setting times compared to that with tap water (Yang *et al*. 2019). The CaO content is an important factor contributing to the reaction mechanism of both cement hydration and alkali activation. The other mechanism for the accelerating influence of seawater on cement hydration is the presence of $CaCl_2$. Rattanasak *et al*, (2011) used $CaCl_2$ as an additive



(a) Setting time



(b) Compressive strength

Fig. 3 Series I seawater content: (a) Setting time, (b) Compressive strength at 28 days

in high calcium fly ash AAP and demonstrated the reduction in setting times. $CaCl_2$ induces the formation of calcium silicate hydrates and carbonates of paste. (Ghazal *et al*. 2021). Due to the high CaO content of binder, the AAP in this work can experience a similar reaction.

The compressive strength of AAP as shown in Fig. 3(b) indicated that the use of seawater to replace fresh water in making NaOH solution resulted in a marginal increase in the 28-days strength. The compressive strength of normal AAP was 44.2 MPa and increased to 46.4 MPa with the use of seawater. The presence of sodium, calcium and other elements facilitated additional geopolymerization and increased the strength. The aluminosilicate raw materials dissolved more easily in the increased salt solutions. This would also increase the geopolymerization reaction, facilitate the production of N-A-S-H gels, and provide strength in AAM systems (Lv *et al*. 2020).

Table 4 shows that AAP with seawater and NaOH solution had much higher chloride contents. This was

Table 4 Chloride content of geopolymer in series seawater

Seawater content (%by weight of NaOH)	Chloride (mg/kg)
0	21
20	149
40	285
60	849
80	1,280
100	1,752

consistent with the observation that the setting time tended to accelerate with rising chloride concentration due to seawater containing chloride as indicated in Table 2. The high calcium fly ash has a mass proportion of 22.57% CaO as shown in Table 1. This resulted in increased C-S-H in the AAM (Yousefi Oderji *et al*. 2019) because chlorides in

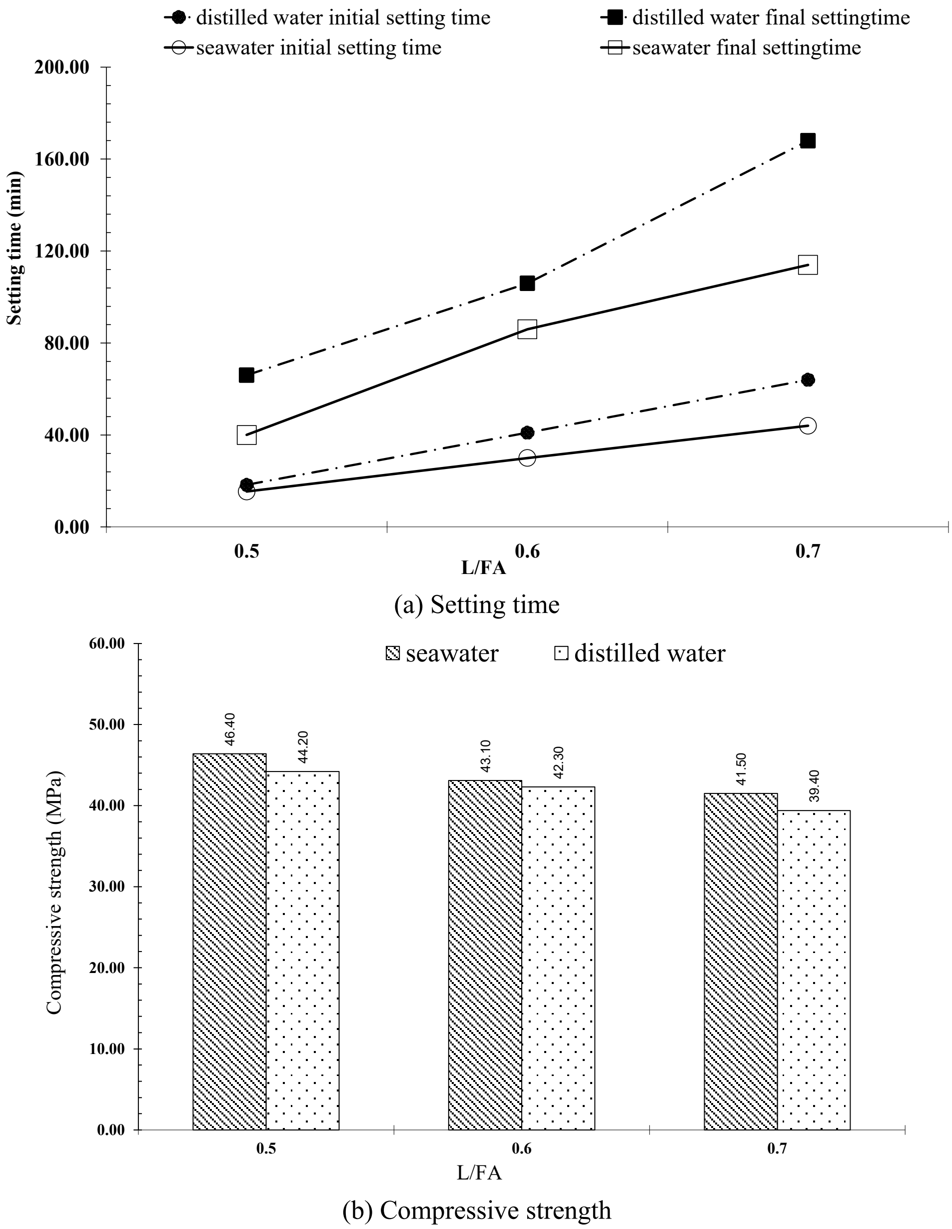
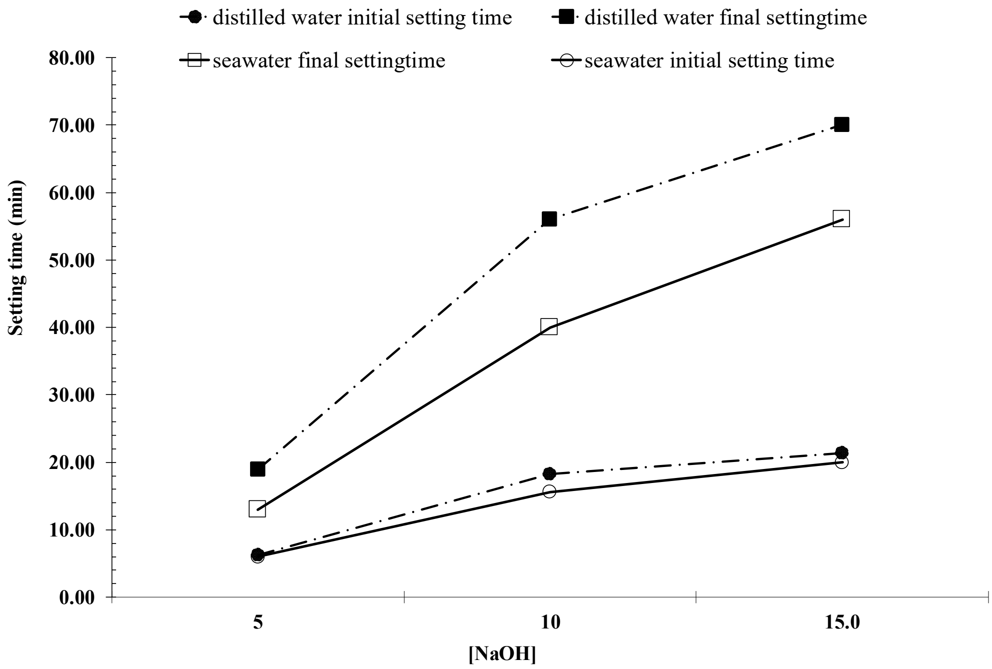


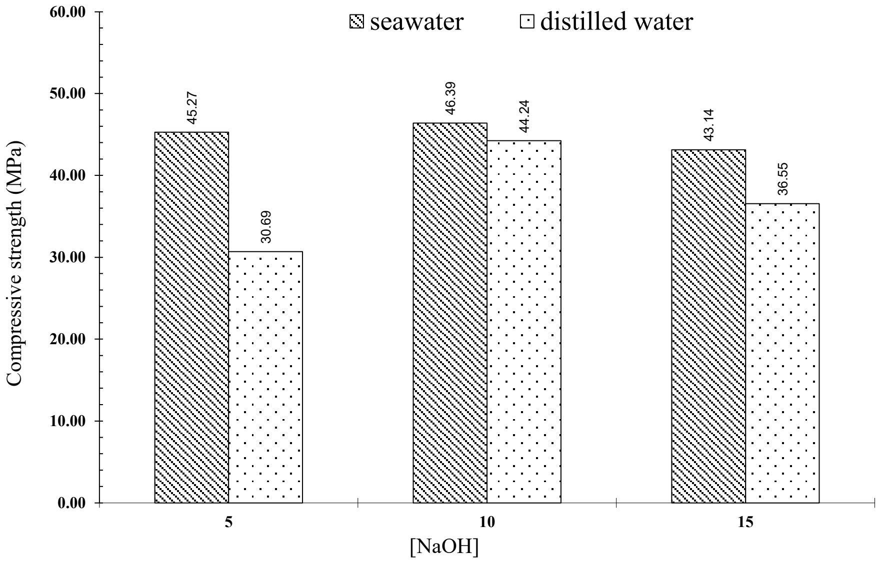
Fig. 4 Series II L/FA: (a) Setting time, (b) Compressive strength at 28 days

seawater accelerate the hydration of cement (Etxeberria *et al*. 2016, Morteza *et al*. 2020, Shi *et al*. 2015). The precipitation of the AFm could also be accelerated and enhanced with the presence of chloride (Sui *et al*. 2023).

The concern is the effect on the durability of reinforced concrete. The corrosion of steel reinforcement is one of the major concerns for reinforced concrete. The corrosion initiates when chloride content reaches the chloride threshold value (Salami *et al*. 2022, Vu *et al*. 2022). For the alkali activated system, the literature on the resistance to chloride corrosion is not conclusive. It was reported that fly ash alkali-activated binder concrete does not perform well in the high chloride environments (Zhang *et al*. 2020) due to the increased pores of size 10-104 nm (Hu *et al*. 2019). On the other hand, Yang *et al*. (2018) reported that the alkali activated slag concrete made with seawater and sea sand has better resistance to chloride ion permeability than Portland cement concrete. Also, the resistance to chloride corrosion attack of alkali activated fly ash-slag concrete was found to be better than OPC concrete (Tennakoon *et al*. 2017). In their review, however, Xue *et al*. (2023) indicated that the chloride induced corrosion of alkali activated low calcium fly ash concrete is questionable and only mixes with fly ash and high slag content showed good resistance to chloride corrosion. Generally, the mixes with high alkalinity, high in alumina content, and high strength with dense microstructure showed increased corrosion resistance of the concrete (Yang *et al*. 2018). The high content of calcium in fly ash helps with strength improvement on the alkali activator concrete. However, more work on this is needed to ascertain that the use of alkali activated high calcium fly ash concrete incorporating seawater would be safe in terms of chloride corrosion.



Setting time



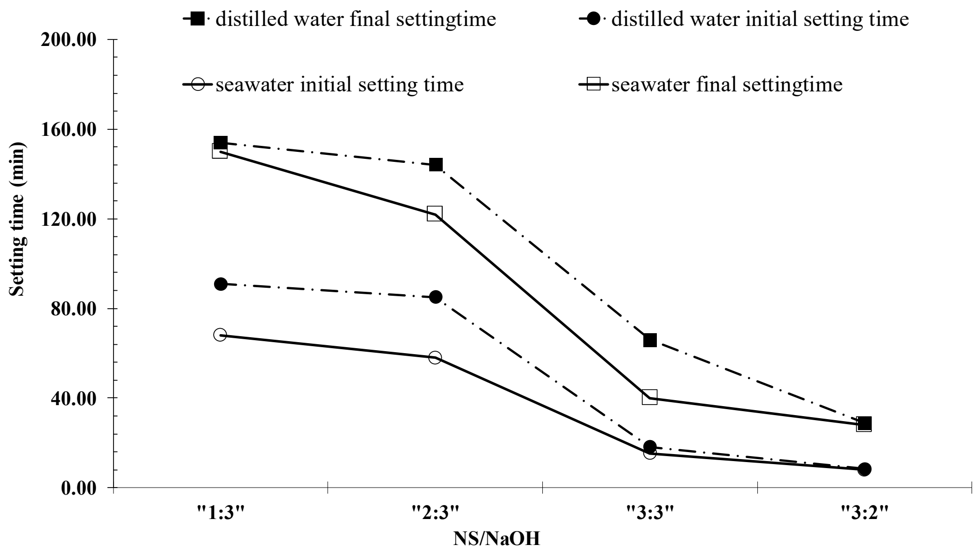
(b) Compressive strength

Fig. 5 Series III [NaOH] concentration: (a) Setting time, (b) Compressive strength at 28 days

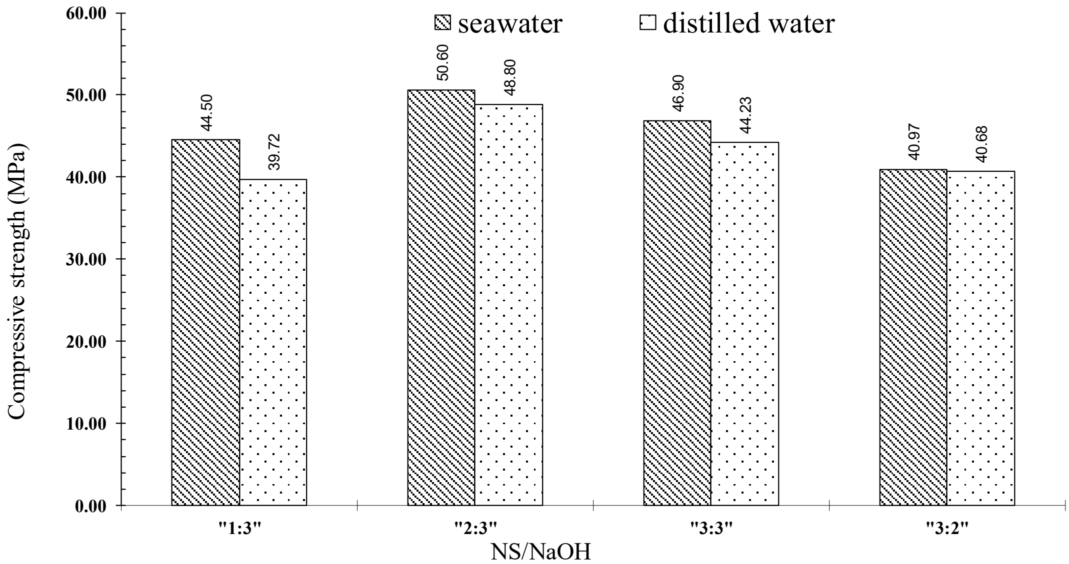
3.2 Series II L/FA

The setting time of AAP as shown in Fig. 4(a) showed that the use of seawater reduced the setting time of AAP for all L/FA ratios. The reduction in initial setting time was more with the high L/FA ratio paste. The initial setting time of 0.5 L/FA paste and distilled water was 18.3 min and slightly reduced to 15.4 min when sea water was used. Whereas the initial setting time of paste with 0.7 L/FA and distilled water reduced to 44.0 min from 64.0 min. However, the incorporation of seawater resulted in significant reductions in final setting time for all L/FA ratios as shown in Fig. 4(a). The increase in setting times due to the increased L/FA ratio was due to the increased solution and associated reduced concentration of leached out silica and alumina. In addition, the increased L/FA ratio increased the distance between the particles and resulted in increased workability (Sathonsaowaphak *et al*. 2009).

The results of compressive strength of AAP at L/FA ratios of 0.50, 0.60, and 0.70 are shown in Fig. 4(b). The strengths were slightly reduced with the increasing L/FA ratio. As discussed earlier, the increased L/FA ratio increased the workability of the paste with the associated reduction in compressive strength of AAM due to the excess liquid content similar to the increase in W/C of concrete (Kharita *et al*. 2010, Posi *et al*. 2013, Rols *et al*. 1999, Wongpa *et al*. 2010). An increase in the liquid content leads to reduced silica and alumina concentrations,



(a) Setting time



(b) Compressive strength

Fig. 6 Series IV NS/NaOH: (a) Setting time, (b) Compressive strength at 28 days

thereby reducing geopolymerization reaction (Rattanasak and Chindaprasirt 2009). The compressive strengths of 44.2, 42.7, and 39.4 MPa for pastes with L/FA ratios of 0.5, 0.6 and 0.7 with distilled water were reduced to 46.4, 43.1, and 41.5 MPa, respectively with the use of seawater.

3.3 Series III NaOH concentration

In this series, the setting times with a change in the sodium hydroxide solution concentration of 5, 10, and 15 molars. The initial setting time was increased with the NaOH concentration due to the leaching out of silica and alumina from fly ash particles in the alkali environment. At high alkali concentration, the leaching out was more and resulted in increased reaction and gel formation (Rattanasak and Chindaprasirt 2009).

In replacing distilled water with seawater, the obtained NaOH solutions from the mixed water gave the same appearance of the solution indicating minimal chemical and physical effects of the seawater replacement. Seawater contains a high concentration of chloride ions (Cl^-), which form stable complexes with sodium ions (Miessler *et al.* 2013). However, the presence of seawater in alkali activated fly ash concrete contributes to increased electrical conductivity, potentially affecting the durability and performance of the geopolymer.

The incorporation of seawater had a very small effect on initial set of AAP. However, the effect on final setting time was large with significantly reduction. The final setting times of paste with distilled water were 19.00, 56.00, and 70.00 minutes, and reduced to 13.00, 40.00, and 56.00 minutes, respectively (Fig. 5(a)).

The compressive strength of paste with distilled water was optimum at 10 M NaOH. The low strength of 5 M NaOH paste was from the low leaching out of the silica and alumina and the resulting low geopolymerization

(Chindaprasirt *et al*. 2007). The relatively low strength with high 15 M NaOH paste was due to the reduced dissolution due the coagulation of silica (Rattanasak and Chindaprasirt 2009, Somna *et al*. 2011). The seawater, however, increased the compressive strength of paste. The increase for 5 M NaOH paste was high and very significant. The compressive strength of the 5 NaOH paste with distilled water was 30.7 MPa and increased to 45.3 MPa (Fig. 5(b)). Test results also showed that the NaOH solution affected the compressive strength. The NH solution ionizes the amorphous silica and alumina from the fly ash outer surface, and polymerization occurs to form a gel that binds the fly ash particles together. The high concentration of sodium hydroxide solution can dissolve the amorphous silica and alumina and increase the AAP viscosity making it difficult to pour into the formwork. The optimal sodium hydroxide solution concentration is in the range of 5-10 molar (Wongkvanklom *et al*. 2021).

The enhancement of strength with low NaOH concentration and seawater is very important. This enables the use of low molarity 5 M NaOH to obtain nearly as high compressive strength as the use of high molarity 10 M NaOH. This would lower the cost of AAP and make the product cost attractive.

3.4 Series IV NS/NaOH

In this Series, the NS/NaOH ratios were varied at 1:3, 2:3, 3:3 and 3:2 and the setting time and compressive strength are given in Fig. 6(a) and 6(b), respectively. The setting times of all AAPs were reduced with the incorporation of seawater and the effect was more with the final setting time and with the high NS/NaOH ratio. The initial setting times for paste with low NS/NaOH of 1:3 and 2:3 showed significant reductions from 95 and 93 min to 70 and 60 min, respectively. For final setting time, pastes with intermediate NS/NaOH of 2:3 and 3:3 showed significant reductions. Normally, the high NS/NaOH ratio provides more reactive silicon and enables geopolymerization to take place resulting in shortening of the formation time (Singh 2018). For seawater incorporation, the effect was more evident with the low NS/NaOH ratio.

For the effect on compressive strength of AAP, seawater increased the compressive strength of paste. The noticeable increase is the paste with low NS/NaOH. For the NS/NaOH paste, the strength increased from 39.7 to 44.5 MPa which is significant. Furthermore, the optimum NS/NaOH for compressive strength was 2:3 and when the NS/NaOH ratio is too high, the mixture's viscosity becomes more and this affected the workability and strength of the mixture (Eiamwijit *et al*. 2015). The compressive strengths of high NS/NaOH AAP were almost the same at around 41 MPa for pastes with both distilled water and with seawater.

3.5 Molar ratio of SiO_2/Na_2O

In general, the molar ratio of SiO_2/Na_2O of the alkali activated concrete has direct influence on the performance especially in terms of strength. The molar ratio of SiO_2/Na_2O of the mix depends on the NaOH and NS content in the alkali activator, and the Na_2O content from the source material. In this study, the Na_2O content of fly ash was rather low at 0.33% resulting in the SiO_2/Na_2O ratios of the mixes in the range of 3.7-5.9. This is comparable to a previously published work on high calcium fly ash with Na_2O content of 1.30%, giving the mixes with SiO_2/Na_2O ratios of 2.1-5.0 with high compressive strength of around 56-66 MPa (Rattanasak and Chindaprasirt 2009). The high strength was also due to the temperature curing at 65°C for 48 h. The compressive strengths obtained in this study were around 40-50 MPa which was very reasonable considering the samples were cured under ambient temperature.

4. Conclusions

The study on the influence of seawater on high calcium fly ash alkali activated paste (AAP) setting time and compressive strength can be summarized as follows. The use of seawater in making NaOH mixture had noticeable effects on setting and strength of the high calcium AAP. The use of seawater shortened the setting time and the effect was more pronounced for mixes with high L/FA ratios and mixes with low NS/NaOH ratios. The increase in salt content in the mixture facilitate the aluminosilicate raw materials to dissolve more easily and increased the geopolymerization, facilitating the production of N-A-S-H gels, and resulted in reduced setting time and increased compressive strength of paste with seawater. The presence of CaO also contributed to the reduced setting time and increased compressive strength. The use of seawater had a significant effect on the improvement of compressive strength for AAP mixes with low NaOH concentration. For mix with 5 M NaOH, the use of seawater improved the compressive strength from 30.7 to 45.3 MPa which is very attractive in terms of using low molarity NaOH as well as reducing the cost of the AAP. Also, the use of seawater is suitable where fresh water is scarce.

The results thus showed that the high calcium fly ash AAP using NaOH solution mixed with seawater has good compressive strength and the fast setting can be useful in applications where rapid strength gain is required. In addition, for the cementitious materials production in coastal areas or remote islands, seawater could be used to produce AAM for construction purposes.

Understanding the behaviour of alkali activated high calcium fly ash geopolymers containing seawater is crucial for optimizing their properties and ensuring their long-term integrity. Further research is needed to explore the potential implications of this phenomenon on the durability and performance of geopolymers in environments exposed to seawater.

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