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The Potential of Seawater in Geopolymer Mixtures – Effect of Alkaline Activator, Seawater, and Steam Curing on the Strength of Geopolymer Paste

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ABSTRACT

The use of seawater in cement-based concrete is debateable because it may increase the hydration rate but significantly decreases the durability. Alternately, seawater can be used as an alkaline activator solution in geopolymer however, very little is currently known about its effects. This study investigated the effect of seawater as alkaline activator mix solution and curing media on the compressive strength of geopolymer paste. The mixtures varied based on the molarity of alkaline activator solution. Alkaline activators were prepared with two solutions: diluted water and seawater. A day after casting, steam curing method was conducted at 65 °C for 2 hours and then immersed in seawater or normal water for 28 days. This study revealed that seawater in alkaline activator reduced the compressive strength by up to 25%. Applying temperature resulted the early age strength nearly comparable to the later age strength. Immersion the paste in seawater increased the strength up to 15%. The X-Ray diffraction analysis shown the presence of chloride on the surface, consequently preserving the compressive strength without any reduction at 28 days of immersion. The Scanning Electron Microscopy analysis inside the geopolymer paste prepared with seawater shown the microstructure of quartz, mullite, hematite, and the presence of chloride spread around resulting the disruption of polymerization. The results indicated that seawater has the potential as an alkaline activator mix solution and curing media, compensated by requirement of higher molarity of NaOH.

Keywords: geopolymer, fly ash, seawater, compressive strength.

INTRODUCTION

The planet's water resources are distributed unevenly, with approximately 71% of the surface covered by water. However, a mere 2.5% of this water is freshwater, and a significant portion of it is inaccessible due to geographical and seasonal limitations. This scarcity of usable freshwater is aggravated by the fact that only about 1.2% is available on the surface, while the majority is locked in glaciers and ice (United States Geological Service, 2014). These constraints on freshwater availability pose significant challenges for various sectors, including human consumption, agriculture, and industrial processes (Rodell et al., 2018).

The critical importance of freshwater, coupled with its limited availability, has led to a global water crisis. Addressing this issue has become a key focus of international sustainability efforts, as evidenced by Sustainable Development Goal (SDG) No. 6, which aims to ensure the availability and sustainable management of water and sanitation for all (UNDP, 2018). Alarming statistics from the WHO indicate that 26% of the world's population lacks access to safe drinking water, highlighting the urgency of finding innovative solutions.

Simultaneously, rapid urbanization and infrastructure development are driving an escalating demand for construction materials, notably concrete. Traditionally, the production of concrete requires substantial amounts of freshwater (Nallaperuma et al., 2023), making it a significant contributor to freshwater depletion. Some of the mega projects in Indonesia, such as National Strategic Project (Republik of Indonesia, 2016) and The new national capital city (IKN) of Indonesia (Republic of Indonesia, 2022), as well as urban expansion further intensify this demand, necessitating a re-evaluation of construction practices to balance developmental needs with responsible water usage. Efforts to mitigate water consumption in construction have led to the development of high-performance concrete with reduced water-cement ratios (Herath et al., 2020; Kumar and Singh, 2020; N. Singh et al., 2019), alongside the use of additives like superplasticizers. On the other hand, efforts to substitute freshwater in the construction sector need to develop, one possibility is the use of seawater. The huge availability of seawater and ease of access make it potential as construction material. Chemical composition of seawater consists of rich chloride accelerate the early strength of concrete, previous study by Xiao et al. (2017) concluded that concrete made with seawater significantly higher at 7-day compressive strength than concrete made with freshwater. A study by Saxena et al. (2023) reported that the potential of seawater in concrete must be followed by using mineral admixtures, retarders, and superplasticizer to lessen the negative effects of chlorides in cement-based concrete.

Additionally, utilizing pozzolanic materials such as fly ash (McCarthy and Dyer, 2019), a by-product of coal combustion, has gained a great concern in Indonesia. In particularly, for electricity generation relies heavily on coal-based energy sources up to 62.5% (PT PLN, 2021), that resulting in a growing volume of fly ash production. Government regulations, represented by PP No. 22 of 2021, have delisted coal ash as hazardous waste material. With this support, Indonesian fly ash has more potential market consumptions. It has been known that the utilization rate of fly ash in 2018 in the United States increased to 50%, while China was 70% and India was 69%. However, by 2018, its utilization was only less than 20% in Indonesia (Ekaputri et al., 2020). So, fly ash in Indonesia urge to applied more in construction caused by its composition, rich in silica and alumina, classified it as a promising pozzolanic material (Davidovits, 1994).

Nowadays, geopolymer concrete has gained attention for its sustainable attributes caused by their composition consists of waste material, no need for cement, and low need of water. Also, geopolymers have demonstrated durability against corrosive agents (Rahman and Al-Ameri, 2022). These advantages make geopolymers as potential material in the aggressive environment considering our needs of concrete that durable in marine environment (Nuruddin et al., 2016; Singh et al., 2015). Previous research has established that geopolymer made by mixing aluminosilicate material and alkaline activator (Cong and Cheng, 2021; Davidovits, 2013; Zerfu and Ekaputri, 2016). Alkaline activator formed from sodium hydroxide solution as solvent agent of silica and alumina from aluminosilicate material (Zhuang et al., 2016) usually made with distilled water. Several researchers reported the use of seawater as alkaline activator solution. Studies such as that conducted by Jun et al. (2021), Ren et al. (2022), Ren et al. (2021), Shi et al. (2019) have shown that the use of seawater as alkaline activator solution decrease the compressive strength less than 20%. This indicates a potential of using seawater as alkaline solution and further research about alkaline activator admixed by seawater to substitute the distilled water.

Factors found to be influencing the performance of geopolymer concrete depend on the curing method. Some researchers (Kovalchuck et al., 2007 and Ekaputri et al., 2017) explored steam curing by applying temperature at geopolymer. Steam curing by applying temperature and humidity have significant role in the development of microstructure and properties of geopolymer concrete caused by accelerates geopolymer reactions (Ekaputri et al, 2017; Kovalchuk et al., 2007). Previous researchers reported by Ren et al. (2022); Shi et al. (2019) have shown that the early age (3 days) strength of fly ash based geopolymer containing seawater with room temperature curing method shown the low strength comparing at later age (28 days). Research to date has not yet determined the influence of steam curing on geopolymer containing seawater.

This paper explores the utilization of seawater as an alternative to freshwater in the mixed of geopolymer paste. The study investigates the effects of varying molarities of sodium hydroxide (NaOH) solutions prepared with seawater on the compressive strength of geopolymer specimens. Moreover, the study explores the impact of curing methods, including moist and steam curing, on the performance of geopolymer concrete when subjected to immersion in seawater. By addressing the potential influence of seawater on geopolymer paste, this research contributes to the broader discourse on sustainable construction practices and water resource management. As the world seeks alternatives to freshwater in construction processes, the use of seawater in geopolymer holds promise as a step towards more responsible water utilization.

MATERIALS AND METHODS

The characterization of the fly ash

In this investigation, fly ash obtained from Petrokimia Gresik, located in East Java, Indonesia, was subjected to analysis. The chemical composition assessment of the fly ash is listed in Table 1 conducted using x-ray fluorescence (XRF) spectrometry, in accordance with the guidelines outlined in ASTM D4326-21. It shows that fly ash is categorized as class F as the total of silica oxide, alumina and ferrite is more than 50% and the calcium content less than 18% based on the ASTM C618-19 (ASTM, 2010).

The preparation of alkaline activator

The alkaline activator used in this study was a combination of sodium silicate/SS (Na_2SiO_3) and sodium hydroxide (NaOH) solutions. Sodium hydroxide (NaOH) solutions were prepared using both distilled water and seawater. The seawater was collected from Lamongan coastal area with the salinity level of 33 ppm containing 38,600 mg/L of chloride and 947.30 mg/L of sulfate. The variation of (NaOH) solutions is illustrated in Figure 1.

The mixing and curing of geopolymer paste

The proportions used were 65% fly ash as the binder and 35% of an alkaline activator. The alkaline activator was formulated by combining SS and NaOH solutions in a ratio of 1:2:3. The mix design is illustrated in Figure 2.

The fly ash and alkaline activator were blended using a mechanical mixer to achieve a homogeneous mixture. Subsequently, the geopolymer pastes were cast into molds with a height of 40 mm and a diameter of 20 mm. The demoulding of the paste was conducted after a minimum of 8 hours from the casting process. Geopolymer paste specimens were cured in moist condition by covering the specimens with wet fabric and steam by applying temperature 65 °C for 2 hours. Curing with oven, steam, wet or moist is the most recommended techniques for geopolymer.

Table 1. Chemical content of fly ash (% by mass)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	MnO_2	P_2O_5	Cr_2O_3	SO_3	LOI
% (by mass)												
58.35	23.51	8.13	4.65	2.19	0.49	1.86	0.99	0.07	0.33	0.02	1.77	1.09

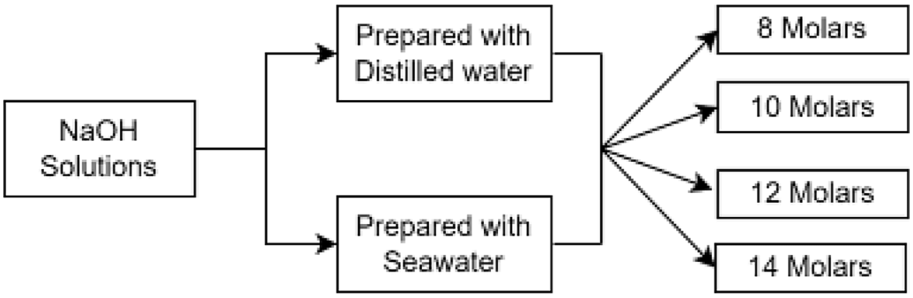


Figure 1. Variation of NaOH solution

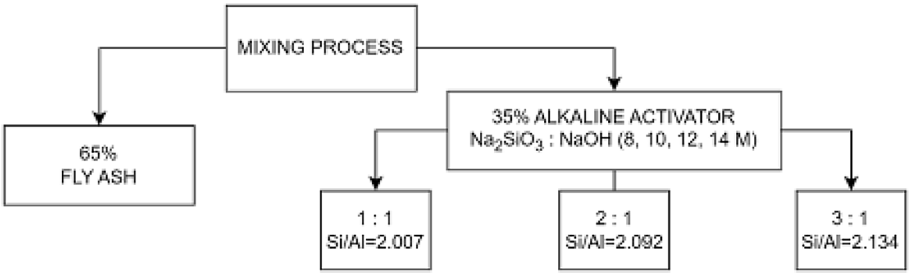


Figure 2. Composition of geopolymer paste

Applying heat to the geopolymer paste contributes to early strength due to the acceleration of geopolymer reaction (Ekaputri et al., 2017; Hardjito et al., 2008). After applying the heat, the specimens were subjected to immerse in normal water and seawater until 28 days. Compressive strength testing was conducted at two different stages: at the early age (3 days) and later age (28 days). The variation of curing process shown in Figure 3 and the prepared samples listed in Table 2.

RESULTS AND DISCUSSIONS

Effect of seawater with visual appearance of geopolymer paste

The impact of seawater on the visual appearance of geopolymer paste was evaluated through visual observation after a 28-days of exposure period, as shown in Figure 4. Geopolymer pastes prepared with both distilled water and seawater and subsequently immersed in normal water (Code as ATN and STN) exhibited the occurrence of efflorescence salts. This phenomenon was attributed to the excess of NaOH reacting with CO_2 present in the normal water resulting to the formation of efflorescence salts. The efflorescence salts, including compounds such as sodium sulfate, sodium chloride, and magnesium sulfate, manifested on the surface of the concrete. These salts were formed due to the reaction between excess sodium and sulphate, or chloride ions present in the surrounding environment (Liu et al., 2014). Distinct conditions occur when the specimen was subjected to seawater curing. As shown in Figure 4b and 4d, a white layer becomes evident on the surface of the paste. This result aligns with the the previous findings that demonstrate the usefulness of the layer in preventing the leaching of necessary reactant elements from geopolymer paste (Giasuddin et al., 2013; Ekaputri et al., 2017).

Effect of steam curing to compressive strength

The influence of applying a temperature of 65 °C for 2 hours as a curing method on

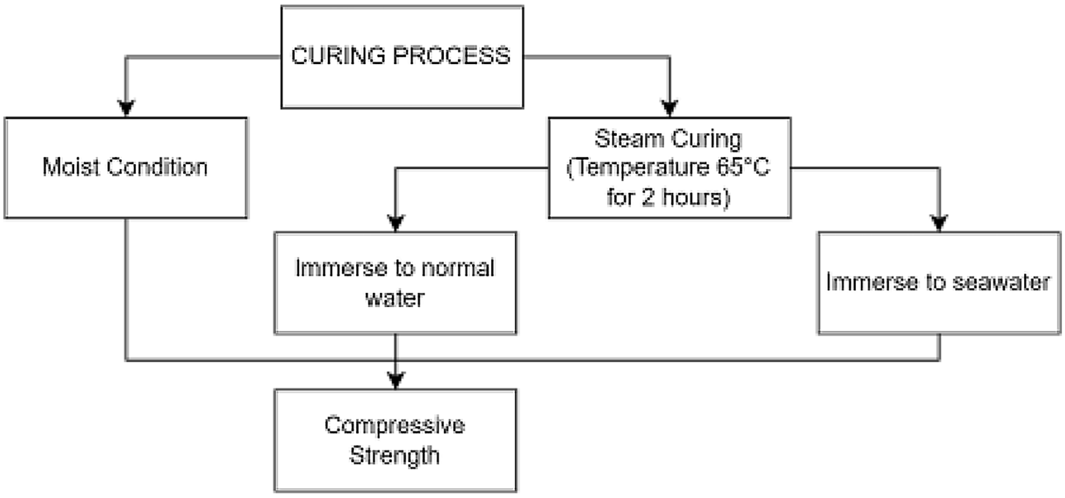


Figure 3. The variation of curing process

Table 2. The prepared samples and curing process

Code	Alkali activator solutions	Curing methods	Curing medias
XAM	Distilled water	Moist Curing	Room temperature
XSM	Seawater		
XATN	Distilled water	Steam curing Temperature 65 °C for 2 hours	Normal water
XSTN	Seawater		
XATS	Distilled water		Seawater
XSTS	Seawater		

Note: *X* – molarity of NaOH Solutions (8 M, 10 M, 12 M, or 14 M).

(a) ATN **(b) ATS**

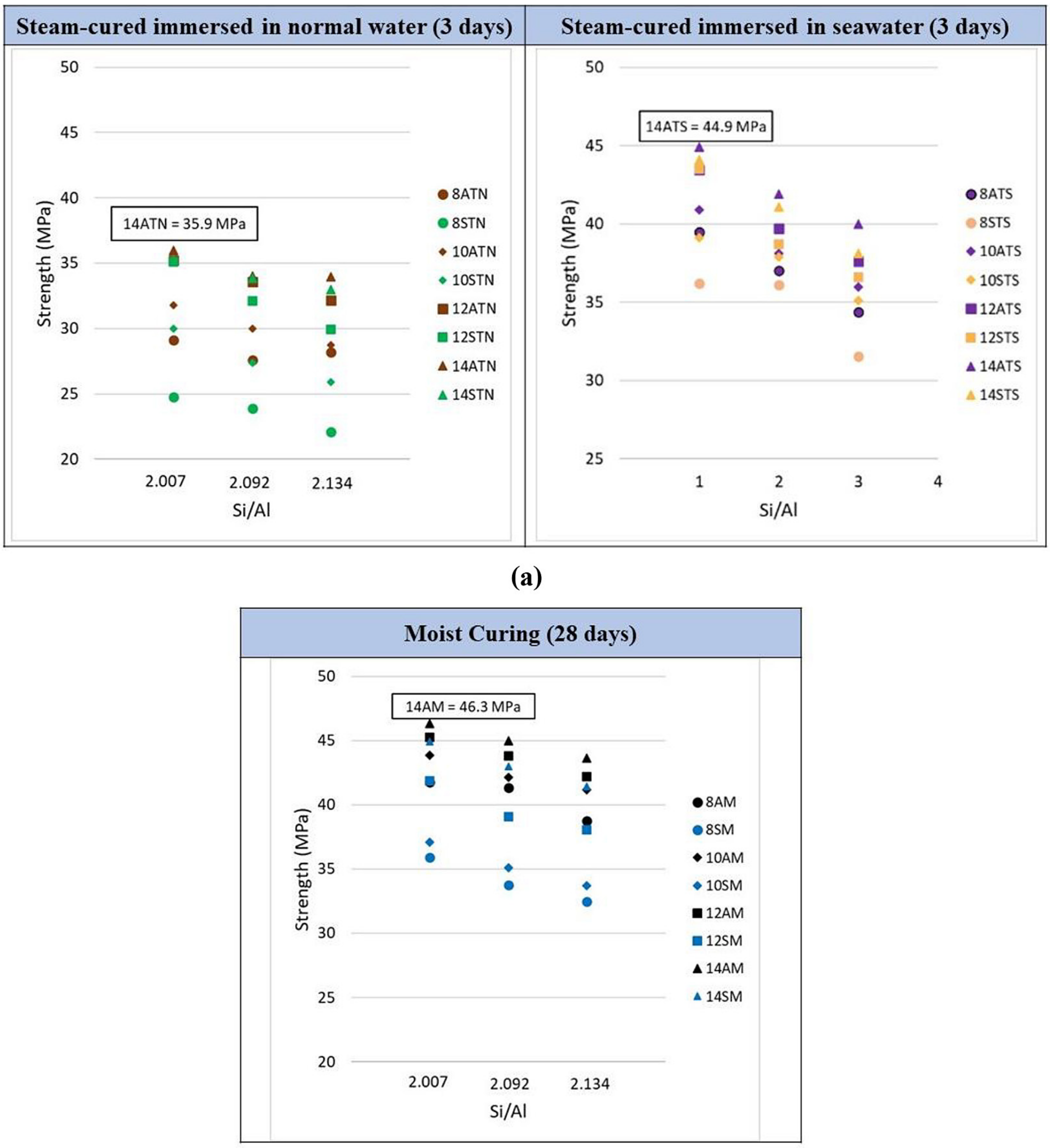
(c) STN **(d) STS**

Figure 4. Visual appearance of geopolymer paste after 28 days immersed in normal water or seawater (a) ATN, (b) ATS, (c) STN, (d) STS

compressive strength is presented in Figure 5. It illustrates that the strength of steam-cured samples at an early stage (3 days) in Figure 5a is comparable to that of the 28 days moist-cured samples in Figure 5b. This is because the application of heat to samples accelerated the polymerization reaction, contributing to early strength in steam-cured samples during the initial stages (Ekaputri et al., 2017). Kovalchuck et al. (2007) highlights the significant role played by temperature and humidity in the formation of microstructure and properties of alkali activated fly ash materials. In agreement with Abdullah et al. (2016) who reported that application of temperature with an oven or microwave can shrink pores thereby increasing strength. The results suggest that utilizing heat through steam curing can accelerate the achievement of significant compressive strength in short time.

Effect of seawater as curing media to compressive strength

The results in Figure 6 reveal that at 28 days, the compressive strength of steam-cured samples immersed in seawater surpasses that of moist cured samples by up to 11%. Moreover, the compressive strength of samples immersed in seawater higher



(a)

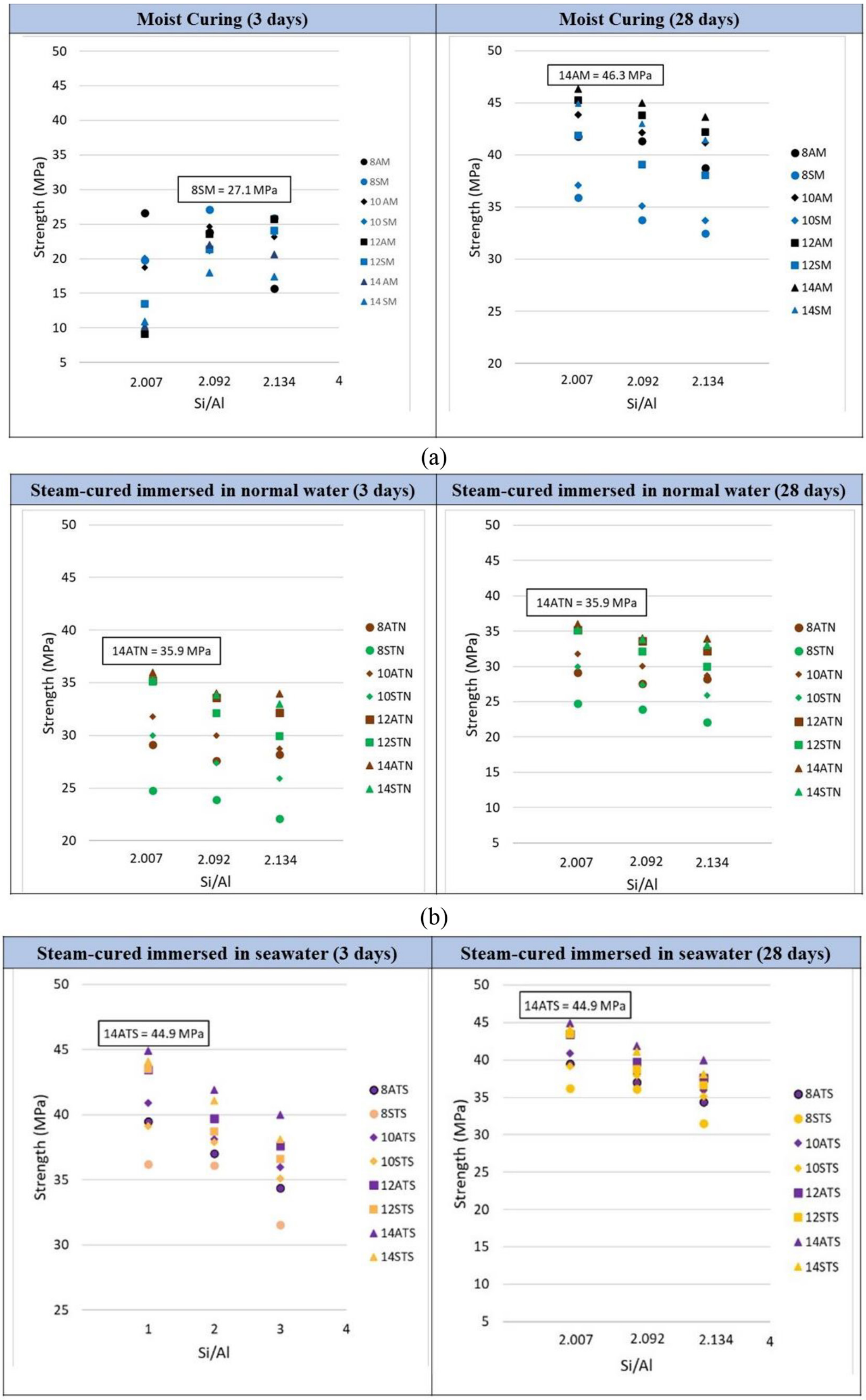
(b)

Figure 5. The effect of steam curing on the compressive strength
(a) steam curing (3 days), (b) moist curing (28 days)

than steam-cured immersed in normal water samples by up to 15%. The results align with Bayuaji et al. (2015), that the geopolymer concrete cured in seawater provides higher compressive strength than freshwater and room temperature. Similarly, Giasuddin et al. (2013) emphasized that curing the samples in seawater prevent the escape of necessary reactant elements from geopolymer sample to the surrounding medium. The Na^+ in salt water is less susceptible to leaching from the geopolymer system. This mechanism produced stable performance on geopolymer concrete that does not occur in cement-based concrete (Halim et al., 2017). The increase in strength observed in the seawater-exposed steam-cured samples is attributed to the formation of a protective layer on the surface, as demonstrated in the Figure 4b and 4d. This layer enhances resistance against leaching. The finding results indicated that geopolymer paste demonstrates remarkable durability in seawater event subjected to aggressive environment (Ekaputri et al., 2017; Rahman and Al-Ameri, 2022; Zhang et al., 2010).

Effect of seawater as alkaline activator to compressive strength

The compressive strength of geopolymer pastes illustrated in Figure 6 shown that both at early stage (3 days) and the later stage (28 days)



(a)

(b)

(c)

Figure 6. The effect of seawater as curing media on the compressive strength (a) moist curing, (b) steam-cured immersed in normal water, (c) steam-cured immersed in seawater

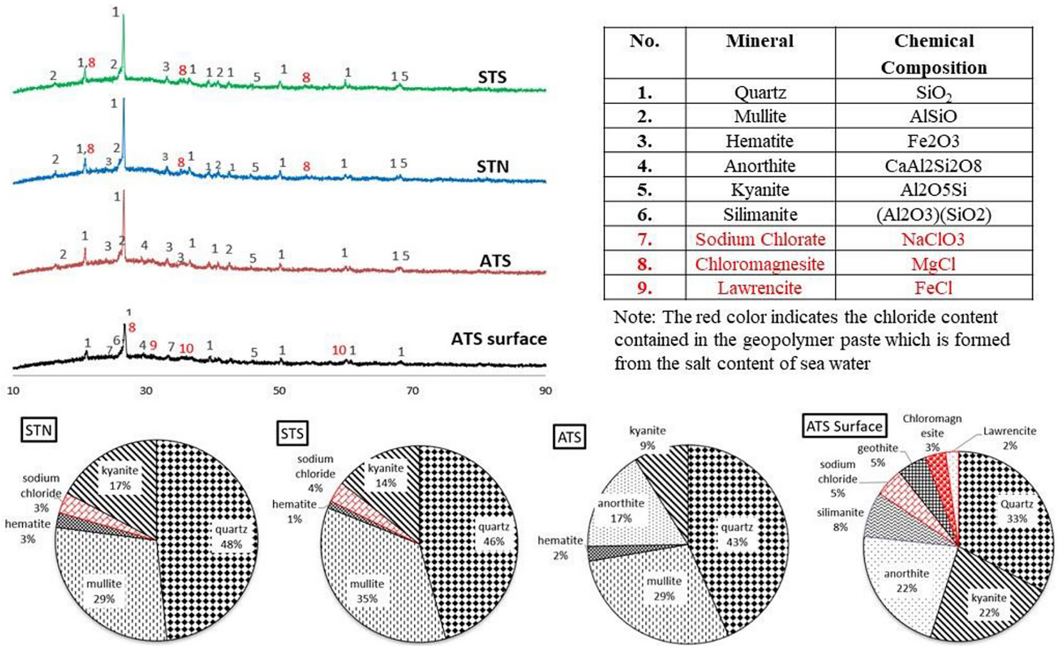
the highest compressive strength is observed in the 14 M specimens, with SS to NaOH ratio of 1. The results demonstrate that the increasing of the SS to NaOH ratio leads to a reduction in strength. The SS to NaOH correspond with the ratio of SiO_2 to Al_2O_3. In agreement with Kovalchuk et al. (2007) stated that compressive strength drops when the SiO_2 to Al_2O_3 ratio rises. These results align with the findings of previous authors who have demonstrated the optimum Si/Al ratio is between 1.5 to 2.5 (Castillo et al., 2021; Liu et al., 2022). It was found that the decreasing in strength with higher silica content contributed to the presence of unreacted material within the specimens.

The variation of molarity of NaOH solutions played significant role at the compressive strength of geopolymer concrete (Chindaprasirt and Chalee, 2014). As illustrated in the Figure 5, higher molarity of NaOH solutions increased compressive strength. The phenomena caused by the increasing of Na^+ concentration promote the acceleration of fly ash dissolution and aids aluminosilicate gel formation, developing tightly bonded structures (Chindaprasirt and Chalee, 2014). Using the seawater as an alkaline solution contributes to a reduction in strength both in early stage (3 days) and later stage (28 days). It is caused by the presence of Na^+ and OH^- in seawater leads to a decrease in compressive strength, possibly due to an excess of Na+ and OH^- in the mixture (Hardjito et al., 2008). Excessive sodium, which remains incompletely involved in the polymerization reaction, assumes the function as a filler and is prone to dissolution or interaction with the surrounding environment. Seawater as the dilutant of alkali decreasing the strength up to 25% at 8M specimens and 10% for 14 M specimens. By this result, utilizing seawater as alkaline solution must be compensated by the higher molarity of NaOH.

X-ray diffraction analysis of geopolymer pastes

The mineral compositions of specimens prepared with distilled water and seawater as NaOH Solution with different methods of curing medium presents at the Figure 7. The XRD specimens was obtained from a paste aged for 28 days with a concentration of 10 molar, the specimen is taken only the inside of the paste (Code: ATS, STN, STS). Additionally, to ascertain the chemical composition of the surface layer, a sample from the outer paste (designated as "ATS surface") was also collected.

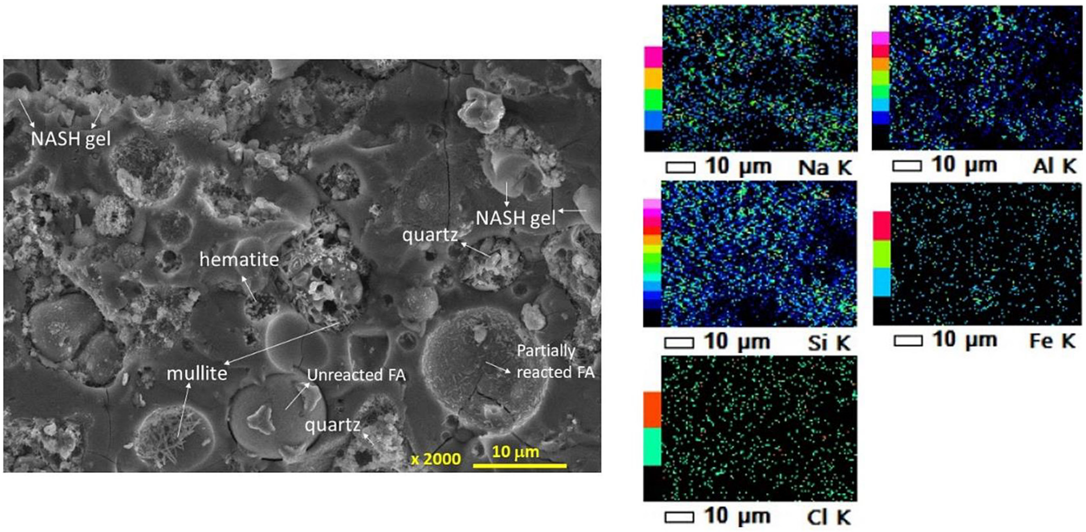
The main component listed as shown in Table 3 in all variation of paste are quartz, mullite, kyanite, and anorthite. The compositions of the XRD patterns indicates the absence of chloride ion inside of pasta but in the surface of pasta the chloride presence in the formation of salt (sodium chloride, chloromagnesite and lawrencite). Chloride ion present on the surface of the paste did not disrupt the polymerization process, as a result did



No.	Mineral	Chemical Composition
1.	Quartz	SiO_2
2.	Mullite	AlSiO
3.	Hematite	Fe2O3
4.	Anorthite	CaAl2Si2O8
5.	Kyanite	Al2O5Si
6.	Silimanite	(Al2O3)(SiO2)
7.	Sodium Chlorate	NaClO3
8.	Chloromagnesite	MgCl
9.	Lawrencite	FeCl

Note: The red color indicates the chloride content contained in the geopolymer paste which is formed from the salt content of sea water

Figure 7. X-ray diffraction of gepolymer paste prepared with 10 M NaOH solutions



(a) **(b)**

Figure 8. (a) SEM picture and (b) EDS of STS sample

not lead to a reduction in compressive strength. The salt formation at the surface prevented leaching of necessary reactant elements from geopolymer paste (Giasuddin et al., 2013). The XRD patterns of specimens prepared with seawater occur sodium chloride in the specimens. Chloride infiltration did not occur in the geopolymer paste after 28 days of immersion.

SEM (scanning electron microscopy) analysis of geopolymer paste

The microstructure by SEM image in magnification 2000x of geopolymer pastes prepared with seawater and subsequently immersed in seawater as curing media (code as STS) shown at the Figure 8. Relating to the XRD of STS sample result, the geopolymer paste in Figure 8a was characterized with the presence of N-A-S-H, quartz, mullite, and hematite. Figure 8b shown the EDS results from STS sample show the presence of Na, Al, and Si as the main elements of geopolymer. In the other hand, there is Fe element attached to the surface of the unreacted fly ash and the absence of sodium elements around the observation point indicates that the fly ash containing Fe is inert/non-reactive. The presence of chloride due to seawater as alkaline activator is spread around the observation area, this lead to disrupt the polymerization process, as a result reduction in compressive strength. EDS mapping at the observation area shows chemical elements are Na (5.09%), Al (9.48%), Si (21.99%), Fe (3.48%), and Cl (0.61%).

CONCLUSIONS

Steam curing at 65 °C for 2 hours shorten the required time to reach the higher compressive strength due to the acceleration geopolymer reaction. The compressive strength of steam-cured samples at an early stage (3 days) was nearly comparable to that of the 28 days moist-cured samples. Seawater as curing media increased the compressive strength up to 15%. It indicates that geopolymer paste is durable in seawater as the aggressive environment. Presence of the salt formation at the surface of specimens prevent the leaching of necessary reactant elements from geopolymer paste. Seawater as alkaline solution decreasing the strength up to 25% at 8 M samples and 10% for 14 M samples at 28 days observation. Based on these result, utilizing seawater as an alkaline solution requires a higher molarity of NaOH. In all variations of the paste, the primary constituents include quartz, mullite, kyanite, and anorthite. Analysis of the XRD patterns confirms the absence of chloride ions inside the geopolymer pasta prepared with normal water after immersion in seawater for 28 days. However, chloride ions are present on the pasta's surface, contributing to the formation of salts such as sodium chloride, chloromagnesite, and lawrencite. The chloride ions on the paste's surface did not disturb the polymerization process, consequently preserving the compressive strength without any reduction. The SEM analysis results inside the geopolymer pasta prepared with seawater showed the microstructure of quartz, mullite, hematite. The presence of chloride spreading around the matrix indicated the disruption of polymerization. Further research with a longer time is required to study the infiltration of chloride to the paste and its effect on the durability of paste. To represent the actual situation, it is necessary for the future research with conditions that are close to actual by changing seawater and normal water as curing medium regularly.

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