

Capillary Imbibition of Concrete Containing Cold Bonded Fly Ash-Based Lightweight Aggregate in a Salt Environment

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Abstract

Cold bonded lightweight aggregate generated from fly ash (FA LWA) has become an interesting approach to increase the consumption of fly ash and prevent over-mining of natural aggregate. However, using the cold-bonded hardening method could increase the number of open pores in LWA, making the concrete containing this LWA face an increased risk of degradation due to water ingress. Thus, in this research, the effect of using FA LWA in concrete on the mechanical and transport properties in a salt environment is being investigated. Three aggregate replacement ratios of 0, 50, and 100% were applied, while the salt environment was mimicked with NaCl and Na₂SO₄ solutions. The results show that the optimum replacement rate of FA LWA was 50%. Regarding the transport properties, exposing concrete to a salt environment (NaCl or Na₂SO₄) proved to slow down the capillary imbibition rate slightly.

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INTRODUCTION

According to the Ministry of Energy and Mineral Resources report in 2022, Indonesia has 230 PLTUs (steam power plants) spread throughout the country. With the high number of PLTUs in Indonesia, the availability of fly ash as a by-product of PLTUs is also very high. However, this high ability of fly ash products is not accompanied by high fly ash consumption. The domestic fly ash consumption rate is reported to only reach 12% per year [1]. If there is no effort to increase the consumption of fly ash, fly ash will only become a landfill.

Much research has been conducted on the use of fly ash as a construction material [2–5]. However, most researchers only focus on using fly ash as a substitute for cement in making concrete. Whereas cement only has a maximum portion of 25% in the concrete matrix. One of the efforts to increase the consumption of fly ash is to transform this material into a lightweight artificial aggregate (LWA) that can be used as a substitute for aggregates that account for almost 75% of the concrete matrix. Research on the utilization of fly ash as artificial lightweight aggregate by the granulation method and using cement as the main binder has been conducted by several researchers [6–10]. However, most researchers only focus on investigating the mechanical properties changed due to the addition of LWA, while limited reports could be found on the effect of introducing LWA into the transport properties of resulting concrete [7,11–14].

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One aspect that should become a consideration when LWA is incorporated into the concrete matrix is to see how the existence of this porous material induces the deterioration of the concrete due to water penetration [15,16]. Most literature stated that the water penetrates the concrete in a short period through capillary mechanisms rather than diffusion, as concrete is filled with connected pores [17–19]. Thus, measuring the capillary imbibition of concrete becomes a critical stage to know how fast concrete deterioration due to water penetration or diffusion could occur. In previous findings, the capillary imbibition of water in long-term observation could cause swelling that could lead to severe degradation of the concrete [20,21]. In various concrete infrastructure that is exposed to constant water, such as sewage, seawall, wastewater treatment, or foundation, understanding the capillary imbibition of concrete is essential to prevent degradation [22].

On the other hand, several studies have reported that introducing LWA into the concrete could improve the durability of the resulting concrete due to the ability of LWA to act as an internal curing agent that could improve the ITZ between paste and aggregate [23–27]. Although several studies have already studied the durability of concrete containing LWA, only a few research studies have studied the effect of LWA on the transport properties of concrete measured by capillary imbibition. Moreover, in the field of capillary imbibition of concrete, only a few researchers study the effect of concrete exposed to a salt environment that could accelerate concrete degradation due to chloride penetration.

Based on the need to use as much as possible fly ash in the country and see the possibility of using lightweight aggregate generated from fly ash in the marine environment, in this research, the ability of fresh water and salt solution to penetrate concrete that contains a high volume of lightweight aggregate is investigated. This research will recommend the optimum amount of FA LWA into the concrete that delivers accepted mechanical and physical properties. Moreover, to see the feasibility of employing FA LWA as a coarse aggregate replacement to be accepted in the market, commercial lightweight aggregate named expanded clay lightweight aggregates (EC LWA) will also be incorporated into concrete as a comparison.

MATERIALS AND METHODS

Materials

Fly ash type F from Petrokimia Gresik and cement type 42.5N from *Tiga Roda* were used as the main binder in producing cold-bonded fly ash lightweight aggregate (FA LWA). The chemical composition of fly ash is presented in Table 1.

River sand and crushed stone were used as aggregate in the production of concrete. Based on the sieve analysis test according to SNI 03-2834, the river sand used could be categorized as zone 3, while the maximum particle size of crushed stone was 40 mm. The specific density of sand and crushed stone was 2.5 and 2.6, respectively.

Table 1. Chemical Composition of Fly Ash

Compound	% Weight
SiO ₂	44.49
Al ₂ O ₃	24.86
Fe ₂ O ₃	17.06
CaO	5.7
MgO	2.41
Na ₂ O	0.18
K ₂ O	1.48
TiO ₂	0.92
MnO ₂	0.2
Cr ₂ O ₃	0.01
P ₂ O ₅	0.46
SO ₃	1.91

Methods

The Production and Characterization of FA LWA

The granulation process produced the FA LWA. Beforehand, cement and fly ash with a proportion of 10% cement and 90% fly ash of total binder weight were dry mixed in the mortar mixer until it was homogeneous. The dried mix

binders were then added into the rotated pan granulator, and the water was continuously sprayed until granules formed and fell out of the pan. The solid/liquid ratio between the dried mix binder and water was set to 0.24. The tilting angle of the pan granulator was fixed to 45°, while the rotation speed applied was 60 rpm. The parameter set in this research proved to deliver water absorption around 20% and a bulk density that is accepted by ACI as LWA [9,11]. The fresh granules were then dried at room temperature for 24 hours. The dried granules were then put into a sealed plastic bag for 28 days and stored at room temperature. Finally, the physical appearance of FA LWA, EC LWA, and crushed stone was displayed in Figure 1.

The physical and mechanical properties of the resulting LWA were investigated through the bulk density, the water absorption, and the aggregate impact value (AIV) test. All those tests were conducted on 28-day-old FA LWA, crushed stone, and EC LWA.

The morphology of FA LWA was studied using a Scanning Electron Microscope FEI Inspect S 50. The sample preparation started with impregnating LWA into epoxy resin under vacuum conditions. The impregnated sample was then polished and coated with Au coating. Finally, electrons with an intensity of 20 KVa were shot at the samples at a certain probe level and the topography was captured.

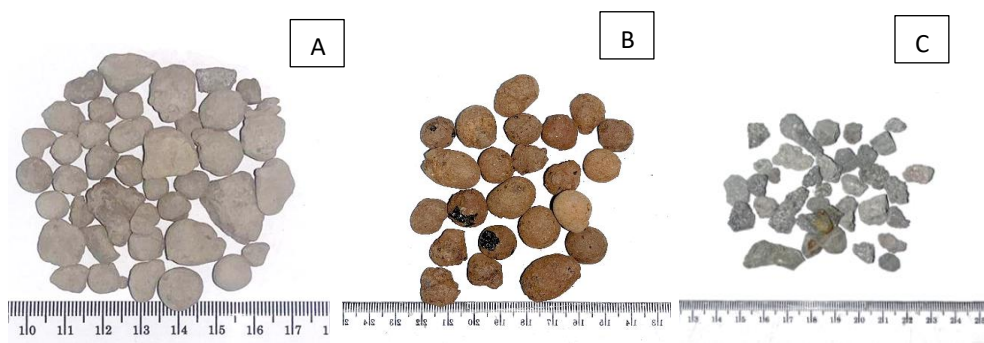


Figure 1. Physical Appearance of (a) FA LWA; (b) EC LWA; (c) Crushed Stone

The Concrete Production

The mixed concrete design followed the guidelines from the British Department of Environment (DoE) method. The water/ cement ratio of 0.6 was applied with a desired strength of 25 MPa. The detailed mix design for producing 1 m³ of concrete is displayed in Table 2. The workability of fresh concrete was conducted with guidance from ASTM C143. Fresh Concrete was cast into a cylinder mold with a diameter of 10 cm and a height of 20 cm. The mold-filled concrete was then cured at room temperature for 24 hours. The next day, the concrete was demolded and immersed in the water bath. The compressive strength and bulk density were then tested at 28 and 56 days, according to ASTM C39/39M. The compressive strength was tested at the age of 56 days to confirm the advance hydration mechanism due to the addition of lightweight aggregate in the system.

Table 2. Mix Design for 1 m³ Concrete

Materials	LWA Content (kg)		
	0%	50%	100%
Cement	350	350	350
Water	210	210	210
Sand	644	644	644
Crushed stone	1196	607.36	-
FA LWA	-	460.20	920.36
EC LWA	-	266.30	532.60

Porosity of Resulting Concrete

The permeable pore was assessed in accordance with ASTM C642. Firstly, the sample was dried in the oven (105 °C) for 24 hours until a consistent mass was achieved, the weight of the dried sample was notated as A. The dried specimens were immersed in water at a temperature of 21°C for not more than 48 hours until reached constant mass and wiped out the surface so the sample reached SSD (saturated surface dry conditions) was notated as B. Place the SSD sample in a container filled with tap water and boiled for 5 hours. After that, allow samples to cool by natural

loss of heat for not less than 14 hours to a final temperature of 20°C. The surface moisture was then removed with a towel. The mass of the sample was then measured and was notated as C. Sample C was then weighed in the water and was notated as D.

The volume of permeable pores was then calculated by following Equation 1. Where g_2 is the mass of the apparent density and g_1 is the dry density of the specimen. The apparent density was calculated by following Equation 2, while the dry density was calculated by following Equation 3.

$$\text{Vol. of permeable pore (\%)} = \frac{g_2 - g_1}{g_2} * 100\% \tag{1}$$

$$g_2 = \frac{A}{(A-D)} * \rho_{\text{water}} \tag{2}$$

$$g_1 = \frac{A}{(C-D)} * \rho_{\text{water}} \tag{3}$$

The Capillary Imbibition of Concrete

The capillary imbibition rate was measured on 56-day-old concrete specimens. The procedure to conduct a capillary water test was adapted from EN 13057:2002 about the determination of the resistance of capillary absorption standard. The sample for the capillary imbibition test is a sliced cylinder with a height of 5 cm, which was generated by cutting a cylinder with a height of 20 cm sample into three pieces (Figure 1). The sliced concrete was then dried in the oven (40±2°C) until it reached constant weight. Dried samples were then covered with aluminium tape on their side part to ensure water penetrated into the top of the sample vertically (Figure 2). The covered samples were then subjected to three different solutions: freshwater, sodium chloride 3.5%, and sodium sulfate 0.5%. The one-liter sodium chloride solution with a concentration of 3.5% was made by adding 35 g Sodium chloride into 1000 ml distilled water, while 5 g sodium sulfate was added to 1000 ml distilled water to made 1 liter sodium sulfate 0.5%. The height of water contact was maintained at 3±1 mm. The amount of water uptake was then monitored by weighing the sample at 0, 0.5, 1, 2, 3, 4, 5, 6, 24, 48, and 72 hours after having contact with the desired solution. The capillary water uptake was then presented as a function of fourth root of time ($t^{0.25}$) instead of two-square roots of time ($t^{0.5}$) due to its best linear correlation [28]. The rate of capillary water uptake was calculated by dividing the amount of water uptake by the sample with final observation time (72 hours).

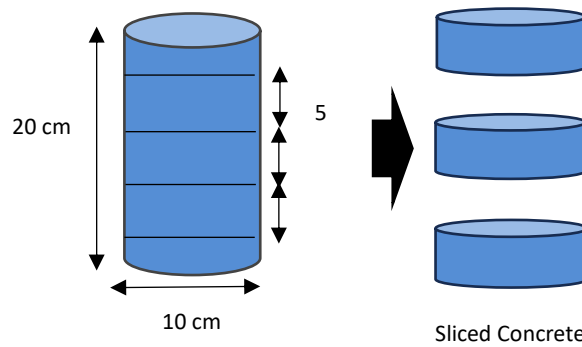


Figure 2. Illustration of Sample Cutting Module for Capillary Imbibition Test

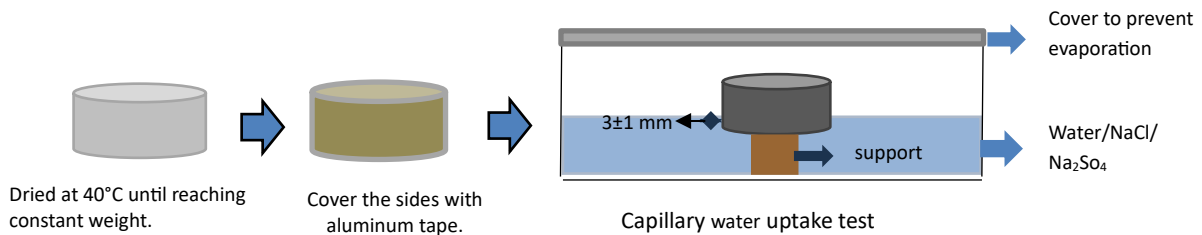


Figure 3. Stage to Perform Capillary Water Uptake Test [29]

RESULTS AND DISCUSSION

The Physical and Mechanical Properties of LWA

Based on the physical and mechanical properties of LWA and natural coarse aggregate (crushed stone) presented in Table 3, it could be seen that the water absorption of both LWAs is higher compared to the crushed stone. In line

with the water absorption result, the bulk and specific density of both LWAs are also lower compared to the crushed stone. Referring to ACI 211.2-98, Standard Practice for Selecting Proportions for Structural Lightweight Concrete, both EC and FA LWA passed the requirement to be categorized as lightweight aggregate, as it has a bulk density of less than 1.04 g/cm^3 . However, based on the BS 812-112:1990 code, the EC and FA LWA could not be used as a pavement application as it has high AIV value.

In conclusion, the properties of FA LWA in this research are equal to similar granular artificial LWA reported in the literature [9,13,14]. Due to the cold-bonded curing method in their production, FA LWA has higher water absorption than EC LWA, which underwent sintering during its production [30]. However, the high-water absorption of cold-bonded LWA was benefited by other researchers as an internal curing agent that prevents early age cracks in concrete [23–26].

Table 3. The Properties of the Natural and Lightweight Aggregate

Test	EC LWA	FA LWA	Crushed Stone
Water absorption (%)	20.65	23.15	1.77
Specific density	1.14	1.90	2.80
Bulk density (g/cm^3)	0.35	0.92	1.20
AIV (%)	52	56.30	19.90

Morphology of Lightweight Aggregates

The morphology of FA and EC LWA observed with SEM is presented in Figure 4. Some residual unreacted fly ash with rounded shape molecules were observed in the FA LWA sample. Still in the same specimens, the hydration product that has a cube shape dominated the morphology of the FA LWA. Pores were observed in random positions with quite small sizes on the surface of FA LWA. On the other hand, the surface of EC LWA was dominated by quite big pores with a size of around $10 \mu\text{m}$. When observing the cross surface of FA LWA, the high-water absorption measured in FA LWA most probably is not only due to the massive number of pores, but also due to the ability of hydration products and unreacted fly ash that still take up high amounts of water. On the other hand, high water absorption monitored in EC LWA most probably occurs due to the high number of open pores on the surface of EC LWA. This result is in line with a previous report, which studied the properties of bottom ash-based LWA, reported that the high-water absorption of bottom ash LWA occurs due to the ability of unreacted bottom ash particles on the surface of bottom ash to absorb water [31].

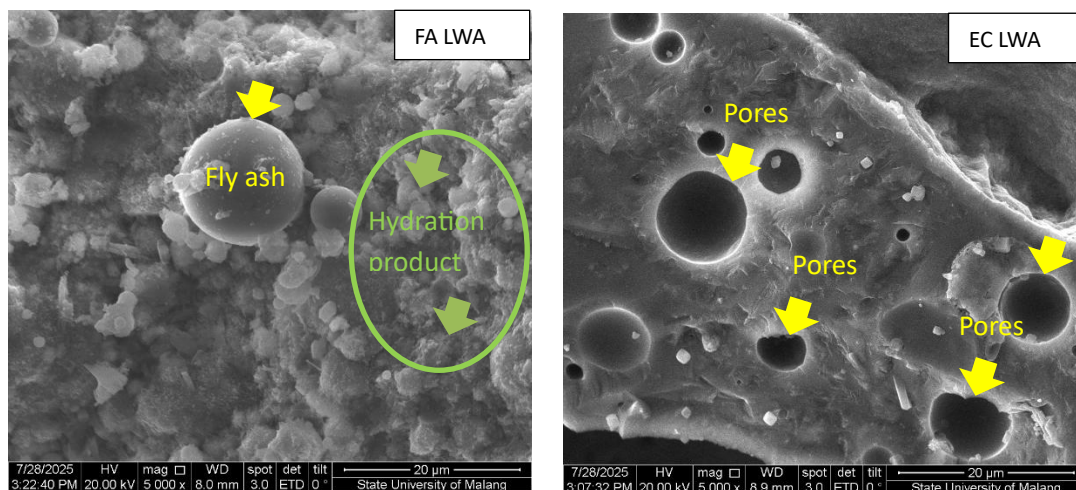


Figure 4. The Cross-surface Morphology of FA LWA and EC LWA observed with SEM

The Workability of Fresh Concrete

The addition of LWA affects the workability of fresh Concrete (Figure 5). The higher the LWA content, the lower the workability of fresh concrete. Similar behaviour was observed whether the natural aggregate was replaced with EC LWA or FA LWA.

Entirely replacing natural coarse aggregate with LWA, decreases the slump value up to 36%, while partially replacing resulting in a 12% slump value decrease. The uncompleted pre-conditioning of LWA to reach the SSD condition

before mixing could cause this condition. Thus, the LWA still slightly absorbs the free water from the concrete mixture. A similar result was also reported by Nahhab and Ketab, who used EC LWA to replace natural aggregates in the production of self-compacting concrete [32]. To avoid this condition, the application of a superplasticizer could be considered for future research.

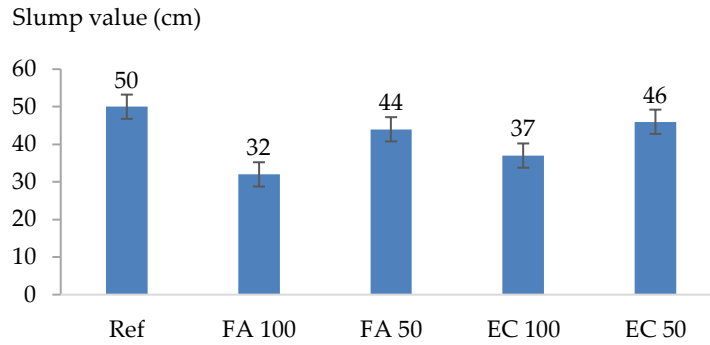


Figure 5. Workability of Fresh Concrete. Error Bars Represent the Standard Deviation (n = 3)

Bulk Density versus Compressive Strength of Concrete

The bulk density of the resulting concrete is inversely proportional to its compressive strength (Figure 6). It is worth mentioning that entirely replacing natural coarse aggregate with FA LWA resulted in acceptable 28-day-old compressive strength (25% strength reduction from the Ref sample). But when EC LWA was used to replace the total amount of natural coarse aggregate with the same testing age, the resulting concrete strength dropped to 70% compared to the reference sample. Indeed, the compressive strength of mortar that contained FA LWA is still acceptable due to its higher bulk density compared to EC LWA mortar.

Based on ASTM C330, only FA 50 and EC 50 concrete pass the requirement to be categorized as lightweight structural concrete, as both have compressive strength higher than 17 MPa. However, when we look at the bulk density required for structural lightweight concrete, only the sample with full replacement of EC (EC 100) meets the requirement. The high bulk density of Indonesian river sand that has almost equal value to the coarse aggregate, might be attributed to the higher bulk density of the resulting concrete.

Overall, the compressive strength of concrete increases when tested at a later age. A noticeable strength increase was observed in concrete containing FA LWA. The strength of the concrete tested at 56 days increased up to 35% compared to the same variation tested at 28 days (Figure 7). It could be that the fly ash at the surface of LWA is still able to react with the free water that might be trapped inside the pores of LWA and form an extra hydration product. From the 56-day-old compressive strength, it can be seen that the strength of EC 50, FA 50, and FA 100 samples is almost equal to the reference sample. Low strength was still monitored when EC LWA was used to entirely replace the concrete's coarse aggregate, most probably due to the low bulk density of the sample.

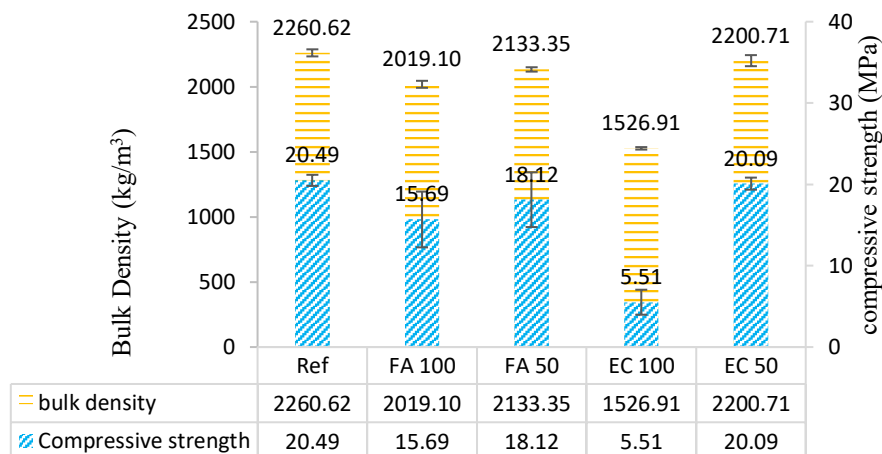


Figure 6. Relationship between Bulk Density and Compressive Strength of Concrete Tested at the Age of 28 Days. Error Bars Represent the Standard Deviation (n = 5)

When we look up to the strength of waste-based LWA concrete in the literature, the strength of concrete containing FA LWA in this research is still in the same range of 17-25 MPa [13,14,33]. Even though the w/c ratio used in this

research is lower than the one in the literature, the mechanical performance of the resulting concrete obtained is still acceptable.

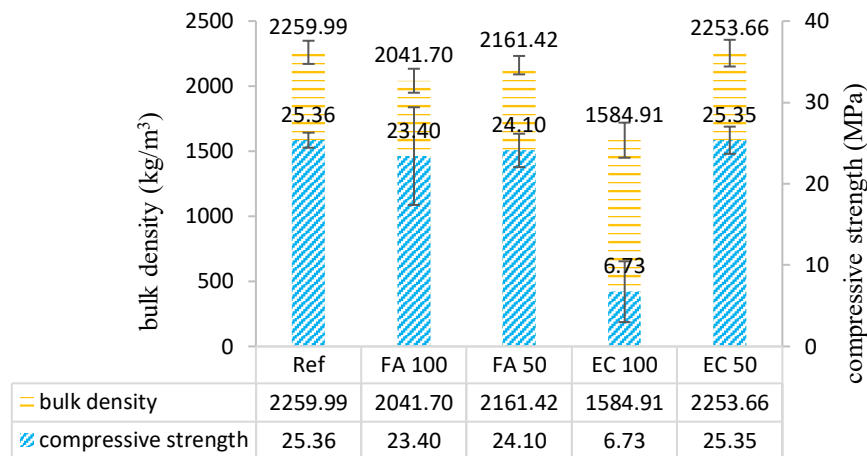


Figure 7. Relationship between Bulk Density and Concrete's Compressive Strength Tested at the Age of 56 Days Old. Error Bars Represent the Standard Deviation (n = 5)

Open Porosity of the Resulting Concrete

Based on the open porosity of resulting concrete, the addition of FA LWA into the mixture has tendency to increase the open porosity of the concrete compared to Ref sample (Figure 8). On the other hand, increasing the amount of EC LWA in the concrete matrix implied to slightly increase of open porosity, but with entirely replacing coarse aggregate with EC LWA resulting to similar open porosity number with Ref sample. The low number of open pores in EC LWA was also reported in the literature. The sintering process during the production of EC LWA was reported to cause the formation of high closed pores and low open pores [12,34]. However, the low number of open pores in EC LWA concrete did not contribute to its mechanical strength (Figures 5 and 6). Even though the percentage of the open pores detected in the EC 100 sample is almost similar to the Ref sample, the strength of EC 100 drops by up to 75% compared to the Ref sample. The wall of the close pores occurred due to sintering process might be too thin, so that it is easy to break when subjected to load.

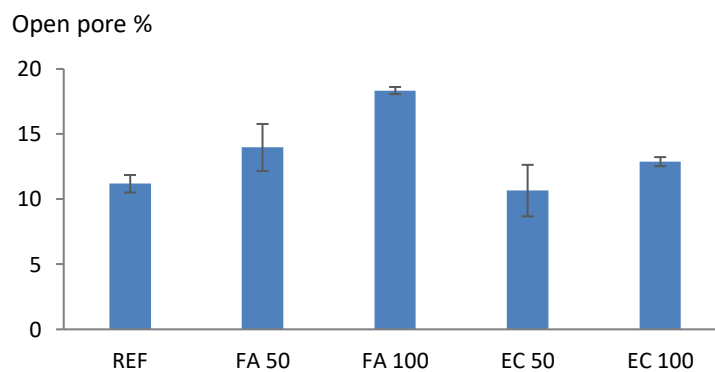


Figure 8. Open Porosity of 56-day-old Concrete. Error Bars Represent the Standard Deviation (n = 3)

Capillary Water Uptake in Fresh Water, Sodium Chloride, and Sodium Sulfate

The capillary water uptake in the function of fourth root of time in various environmental conditions is displayed in Figures 9, 10, and 11, while the capillary imbibition rate of concrete measured at 72 hours are displayed in Figure 12. The R^2 value of all specimens in all environment exposure is less than 0.99, which considered slight low compared to value reported in the literature that used w/c 0.5 [27]. However, a similar R^2 value was reported in the sample that used a 0.6 w/c ratio [28]. It seems that the linear correlation could only be drawn for the first 24 hours of observation because the water uptake significantly slowed down after that period.

In Figure 8, when the samples were exposed to freshwater exposure, the FA 100 sample had the highest water uptake among others. It seems that the surface of FA LWA can absorb water compared to EC LWA. Similar behaviour was observed when pozzolanic material similar to fly ash called Lusi was used as lightweight aggregates [35]. Besides,

introducing LWA into the concrete matrix forms more pores so that more water can penetrate the matrix. It also observed that in most samples, the water reached the top of the sample in 24 hours, as after passing that time, the sample's weight tended to be constant, an indication that the sample was saturated with water.

Compared to the previous report that conducted the capillary water uptake on mortar used a 30% replacement rate and w/c ratio of 0.5, the amount of water that could be taken up by the sample in this research is almost two times higher [27]. This result proved that increasing the w/c ratio has more impact on the capillary sorption of the resulting concrete rather than introducing lightweight aggregate into the concrete mixture. This result is in line with previous findings stated that the w/c ratio, the types of binder, and the fraction of aggregate are reported to be the main aspect that influences the capillary water uptake of concrete [17,32].

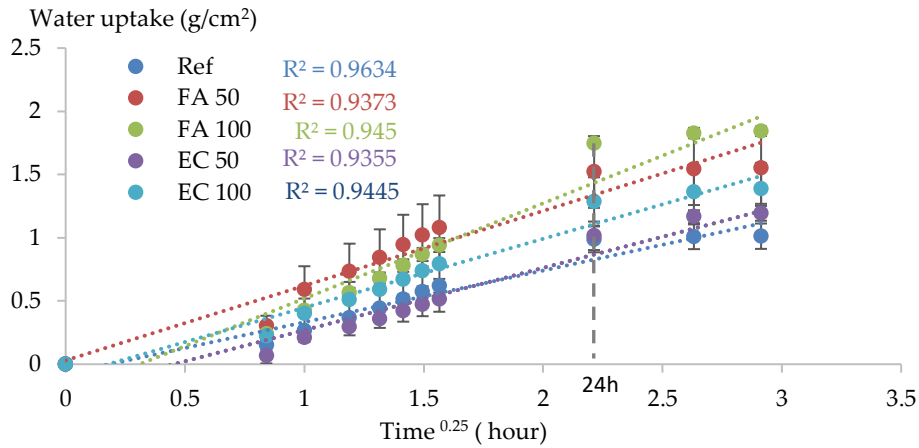


Figure 9. Capillary Imbibition as a Function of the Fourth Root of Time in Freshwater. Error Bars Represent the Standard Deviation (n = 3)

In general, a slightly slower capillary imbibition was observed in a concrete sample that was exposed to sodium chloride solution compared to freshwater (Figures 8 and 9). A sample with an EC LWA replacement rate of 50% (EC 50) has the lowest NaCl uptake. While in the FA LWA sample, no improvement in capillary imbibition was monitored when samples were exposed to sodium chloride solution compared to when samples were exposed to freshwater. The closed pores in EC LWA formed during the sintering process might contribute to the better capillary imbibition behavior of the resulting concrete, as water or liquid could not penetrate these pores. The previous report also observed a slower capillary imbibition rate of mortar samples exposed to a salt environment [18]. However, in that study, the lowest capillary imbibition rates were observed in samples exposed to sodium sulfate solution, while in this research, when the sample was exposed to sodium sulfate, the capillary imbibition obtained was almost similar to the one exposed to fresh water (Figures 8 and 10). The different exposure lengths and pores in a high w/c ratio might cause this slight difference. In most cases, a slower capillary imbibition rate in a salt environment occurs due to the reaction between sodium and cement that induces the formation of ettringite that might block the pores in a concrete matrix [15,19,36,37].

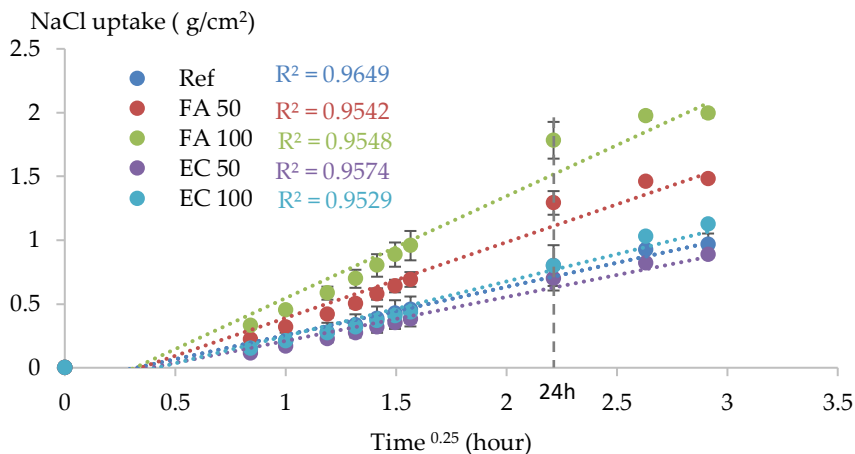


Figure 10. Capillary Imbibition as a Function of the Fourth Root of Time in a Sodium Chloride Solution. Error Bars Represent the Standard Deviation (n = 3)

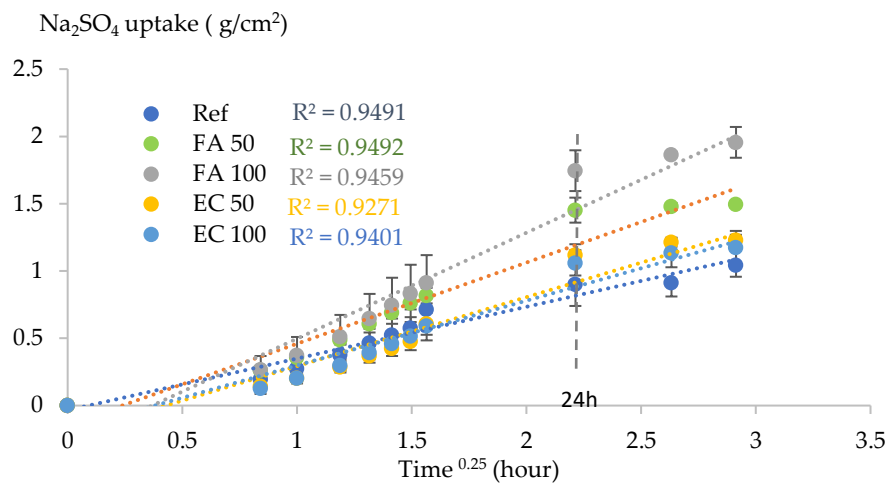


Figure 11. Capillary Imbibition as a Function of the Fourth Root of Time in a Sodium Sulfate Solution. Error Bars Represent the Standard Deviation ($n = 3$)

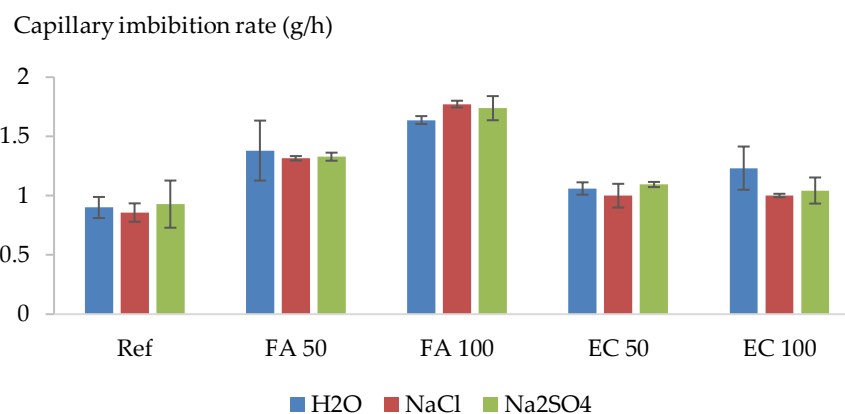


Figure 12. Capillary Imbibition Rate of 56-day-old Concrete in Various Environments. Error Bars Represent the Standard Deviation ($n = 3$)

CONCLUSIONS

Based on the experimental results, the following conclusions could be drawn:

1. The mechanical and physical properties of FA LWA are similar to commercial expanded clay lightweight aggregates EC LWA.
2. The addition of LWA slightly decreased the workability of fresh concrete due to the saturation condition of LWA.
3. Concrete with a 100% FA LWA replacement rate still has an acceptable compressive strength in all testing ages. In the EC LWA sample, with the same replacement rate, the compressive strength of the resulting concrete dropped to 70% from the reference sample.
4. However, replacing all-natural aggregate with FA LWA resulted in a high capillary imbibition rate due to the ability of free fly ash on the surface of FA LWA to absorb water.
5. In general, the capillary imbibition performance of concrete in a salt environments is still acceptable or even better compared to samples exposed to fresh water. The pore-blocking mechanism of salt contributed to this behaviour.
6. Finally, replacing 50% by volume of natural coarse aggregate with FA LWA delivers acceptable mechanical and physical properties. Some improvement should be taken to prevent the surface of FA LWA from absorbing water to decrease the capillary imbibition of the resulting concrete.

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