



Durability-Related Transport Properties of Limestone Calcined Clay Cement Mortars

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Abstract— This study investigates the effect of utilizing a combination of limestone and calcined clay as cement replacement on the transport properties that indirectly define the behavior of the matrix throughout its service life, such as total porosity, water absorption due to capillary action, water permeability penetration, chlorine diffusion, and carbonation depth. Mortars were made of blends with different levels of cement replacement (%15, %30, %45) by a substitution ratio of 2:1 (calcined clay: limestone), with adjusted gypsum content in each system composition. Furthermore, the influence of kaolinite content in clays on transport properties was also investigated. Based on the results, all LC³ blends exhibited a significant improvement in terms of total porosity, capillary water absorption, water penetration resistance, and chloride diffusion compared to OPC mortars. Moreover, the higher the replacement level of cement, the higher the resistance to movement or transport of ion species through the mortars. The improvement in the transport properties for LC³ blends can be attributed to its enhanced porosity refinement as well as the enhanced chloride binding capacity. In contrast, a slight improvement in the transport properties was observed with increasing the kaolinite content of the calcined clays. This result indicates that utilizing low-grade calcined clay has no significant effect on the transport properties. The carbonation resistance of the LC³ blends was lower as compared to OPC. Furthermore, increasing the cement substitution rate resulted in a progressive decrease in carbonation resistance. Overall, an increase in cover depth would be required to achieve a similar service life in terms of carbonation as that of the OPC.

Index Terms: LC³, Calcined clay, Limestone, water resistance, chloride diffusion

I. INTRODUCTION

The recent societal shift toward sustainable consumption and growth applied to housing and civil infrastructure systems, necessitates that construction materials be designed and used with the utmost consideration to their durability and long-term performance [1]. Durability remains one of the most significant properties of concrete, specifically for structures that are exposed to extreme environments or

severe conditions [1-3]. Improvements in concrete's durability performance have been widely accepted by the community as a means of reducing an infrastructure's life-cycle cost, especially in terms of maintenance during its service life [4]. The durability of concrete structures is significantly affected by transport properties such as permeation, diffusion, sorption, and absorption, which allow harmful agents such as water, CO₂, chloride, etc. to penetrate the bulk of the concrete and cause deterioration [1-4].

In recent years, many researchers have established that, in addition to lowering costs and mitigating the environmental issues surrounding cement manufacture, the use of supplementary cementitious materials (SCMs) such as fly ash, blast furnace slag or silica fume enhance the durability of cement binders, largely by reinforcing resistance to fluid transport, and thus improves performance. [5, 6]. These effects are primarily due to the formation of secondary hydration products during the pozzolanic reaction and the resulting alteration of the pore system, in which decreased connectivity raises network complexity and decreases permeability [3, 6, 7]. The growing worldwide demand for cement, however, and the declining availability of the aforementioned SCMs has driven the pursuit of alternative mineral additions [8]. In such a situation, the use of metakaolin in combination with limestone in OPC-based systems is reported to obtain increased substitution levels without impacting concrete characteristics [9, 10]. When limestone and metakaolin are combined, in addition to the pozzolanic reaction of metakaolin and limestone reaction with C₃A, there is the synergetic effect between alumina in calcined clay and limestone producing carbo-aluminate phases. Mono-carbo-aluminate hydrate are formed and fill the pore spaces which will increase the strength [9, 11, 12]. The stoichiometric formation of mono-carboaluminate hydrate (an AFm phase) is by reaction of one mole of metakaolin (MK) with one mole of calcium carbonate CaCO₃ (LS) in the presence of water and calcium hydroxide CH to give 1 mol of monocarboaluminate. This corresponds to an addition with a weight ratio of 2:1 MK:LS [9]. This ternary blend of limestone, calcined clay, and clinker is known as LC³, and it is on its way to proving its potential in the cement industry by solving the twin issues of CO₂ emissions and natural resource conservation. However, the use of this

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Table 1: Chemical composition and Physical properties of the materials

Chemical composition %	Cement	Limestone	MKA	MKB	MKC	Phase composition % - cement		
CaO	63.09	54.53	0.117	0.102	0.172	C ₃ S	58.14	
SiO ₂	19.88	2.729	54.775	71.557	74.33	C ₂ S	13.22	
Al ₂ O ₃	5.37	1.641	41.033	26.025	21.589	C ₃ A	9.4	
Fe ₂ O ₃	2.86	0.064	1.48	1.255	1.009	C ₄ AF	8.7	
MgO	1.52	0.598	0.349	0.108	0.244			
Na ₂ O	0.01	-	-	-	-	ASTM C618 requirements for pozzolanic material		
K ₂ O	0.95	-	-	-	-		MKA	MKB
Cl	0.018	0.092	-	-	-	Total of SiO ₂ , Al ₂ O ₃ and Fe ₂ O ₃ > 70%	97.29	98.84
SO ₃	2.59	0.026	0.033	0.013	0.017	SO ₃ < 4%	0.033	0.013
TiO ₂	0.31	0.004	-	0.679	0.956	LOI < 10%	0.91	0.84
MnO	0.041	0.008	0.021	0.025	0.025			
P ₂ O ₅	0.18	0.065	0.053	0.052	0.06			
Loss on ignition	2.54	41.26	0.91	0.84	0.96	Gypsum	> 85% purity	
Physical property								
Specific gravity	3.158	2.690	2.510	2.570	2.606			
Blaine's (m ² /kg)	364	581	1318	962	994			

system to produce durable concrete depends on the durability-related transport properties attained in the systems, which needs to be investigated further. Especially since exploration of the LC³ in Libya is still in its infancy.

This study focuses on the transport properties that indirectly define the behavior of LC³-blends throughout their service life. A detailed assessment has been conducted of the transport properties such as total porosity, water absorption due to capillary action, water permeability penetration, chloride resistance, and carbonation resistance. Mortars are made of blends with different levels of clinker replacement (%15, %30, %45) by substitution ratio of 2:1 (calcined clay: limestone). In addition, three types of calcined clays are used in order to investigate the influence of the grade of calcined clays on the transport properties of LC³-blends.

II. EXPERIMENTAL WORK

A. MATERIALS PROPERTIES

- **CEMENT**

The cement used in this study is Portland cement (CEM I 42.5N), manufactured by Al-Fattaih Cement Factory and conforming to the Libyan standard which mainly based on the European EN 197-1. The physical and chemical properties and phase composition of the cement used are given in Table 1.

- **LIMESTONE**

Limestone used in this study It was supplied by Al-Fattaih Cement Factory. Limestone is ground by using Los-Angeles aberration machine, this process converts lumps Limestone to powder form. after grinding, Limestone powder is collected from passing 90-micron sieve. Knowing, limestone powder is used in mixtures

without thermal treatment. The physical and chemical properties of the limestone used are given in Table 1.

- **GYP SUM**

The gypsum (>85% purity) used in this study was supplied from Al-Fattaih Cement Factory. The addition of gypsum was chosen in order to prevent the system being under-sulfated for the blends.

- **CALCINED CLAY**

three different clays of varying kaolinite contents sourced from south region of Libya were used in this study. The clay produce from these places were crushed and calcined at 800 °C for 2 h. After the calcination process, the calcined clay was cooled then milled to pass on 90-micron sieve. The details of mineral clay are shown in Table 2, it was taken from a previous study by a research group from Sebha University [13]. The physical and chemical properties of calcined clays used are given in Table 1. The chemical composition of the calcined clays was compared with ASTM C 618 for its suitability to use it as a pozzolan.

Table 2: Location of clays and clays mineral

Symbol of site	Location	Kaolinite content (%)
MKA	10 km from Sebha	86
MKB	10 km from Temenhint_Sebha	55
MKC	Tarout_Wadi Shatti	48

- **AGGREGATES**

Fine aggregates used to produce the mortar cement in this study. The fine aggregate was natural sand, and its gradient falls outside the gradient limits specified in ASTM C778, but has been modified to conform to the

standard sand specified in ASTM C778. The particle size distribution of fine aggregates measured by sieving test is given in Figure 1. The physical properties of fine aggregates are given in Table 3.

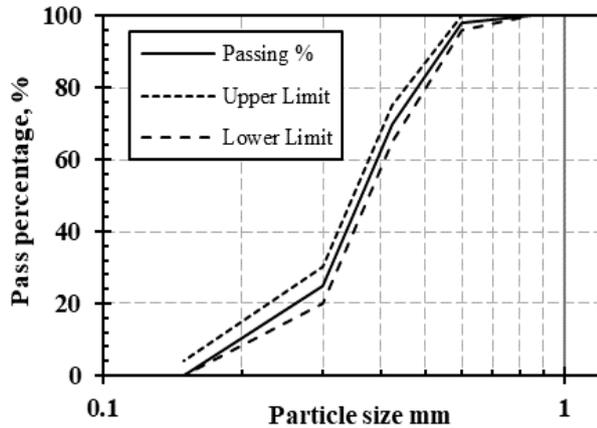


Figure 1: Particle size distribution of Fine aggregate

Table 3: physical property of Fine aggregates

Specific Gravity	Absorption (%)	Moisture Content (%)	Fineness Modulus
2.73	2	0.56	2.07

B. MIX PROPORTIONS

The Mix design for the blended cement samples consisted of looking at changes in two parameters in the sample. The first variable is the percentage of clinker replacement by calcined clay and limestone (with fixed ratio 2:1) at 15, 30 and 45%. Consequently, LC³-80, LC³-65 and LC³-50 correspond to blended cements with approximately 80%, 65% and 50% of clinker content. In addition, the cement in this study was directly used as a part of clinker. Therefore, the addition of gypsum was chosen in order to prevent the system being under-sulfated for each systems composition. The second

variable is the type of calcined clays used depending on the kaolinite contents in each clay type. Three type of calcined clays are used with various kaolinite contents at 86%, 55% and 48%, respectively. Therefore, ten standard mortar mixtures were prepared and used in this study according to ASTM C109, the proportions of materials for the standard mortar, one part of cement to 2.75 parts of graded standard sand by weight with a water-binder ratio of 0.485 as shown in Table 4.

C. TEST METHODS

• WATER ACCESSIBLE POROSITY

The porosity test was performed using the vacuum saturation method shown in Figure 2, as recommended by RILEM (1994). Three 50-mm cube specimens were tested for each test period. Testing period were 7, 28 and 90 days. The hardened cube specimens were oven-dried at 105°C until a consistent weight was achieved. The heated specimens were then allowed to cool before being weighed. The specimens were subjected to vacuum condition for 2 hours. After vacuuming, the specimens were immersed in de-aired water for 24 hours. The specimens were then taken out, wiped, and weighed in air; their weights in water were also recorded. Then, the porosity is calculated.



Figure 2: Vacuum saturation apparatus setup

Table 4: The proportions of materials for the standard mortars

Mixture ID	Cement		Calcined clay	Limestone	Gypsum addition	W/Cm*	S/Cm**	
	Clinker	Anhydrite						
OPC	94	6	-	-	-	0.485	2.75	
LC ³ -80 (86)	AI	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -80 (55)	BI	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -80 (48)	CI	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -65 (86)	AII	65	4.15	19.33	9.67	1.85	0.485	2.75
LC ³ -65 (55)	BII	65	4.15	19.33	9.67	1.85	0.485	2.75
LC ³ -65 (48)	CII	65	4.15	19.33	9.67	1.85	0.485	2.75
LC ³ -50 (86)	AIII	50	3.19	29.33	14.67	2.81	0.485	2.75
LC ³ -50 (55)	BIII	50	3.19	29.33	14.67	2.81	0.485	2.75
LC ³ -50 (48)	CIII	50	3.19	29.33	14.67	2.81	0.485	2.75

W/Cm* - Water to cementitious ratio S/Cm** - Sand to cementitious ratio

- **CAPILLARY WATER ABSORPTION**

The sorptivity test (rate of absorption) of blends cement mortars was performed according to ASTM C1403. Two 50-mm cube sample were tested for each test period. Testing period were 7 and 90 days. The hardened cube sample were oven-dried at 60°C until a consistent weight was achieved. The temperature of 60°C was chosen because it allows the evaporation of the free porous water without affecting the microstructure of the samples [14]. The consistent mass is obtained for all samples between 72 and 120 hours of drying. Before weighting the samples, the dried mortar samples are covered with Pacifix silicon on four side flanges. Also, the cube sample dimensions were recorded. The specimen was placed in a container on supports and filled with water (Figure 3). The water level was maintained at 1-3 mm above the bottom of the specimen. The initial water absorption was measured by weighing the samples for up to 5 hours. Subsequently, a reading was taken once a day up to 7 days.

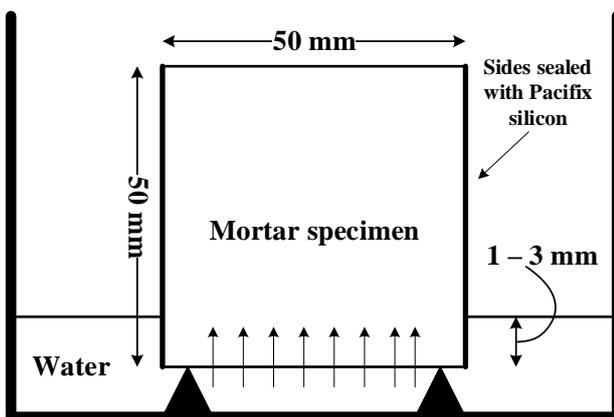


Figure 3: Schematic of rate of absorption Test

- **WATER PERMEABILITY PENETRATION**

The water penetration resistance of blends mortar samples was performed according to European standard (EN 12390-8) for determining the depth of penetration of water and permeability coefficient of each blend mortars. Figure 4 depicts the water permeability apparatus used for this test. According to this standard, the concrete sample surface must be in contact with water under 5 bar pressure for 72 hours. For each mortar mix, three Ø70 × 140 mm cylinders were cured for 28 days. It should be noted that the size of the samples is not in accordance with European Standard, and therefore, this test does not determine the true values of penetration depth, but is for the purpose of only comparing the studied mixtures. At testing day, the cylinders sample were oven-dried at 60°C until constant weight. Before being placed in the permeability testing machine, the weight of the samples was measured. They were then placed in testing machine for 72 hours. When the test was completed, the samples were weighed again to determine their mass difference. The sample is then split into two, and the depth of water penetration is measured. Three values were recorded for each sample, and the average is expressed as the water

penetration depth. Thereafter, the permeability coefficient is calculated as follows:

$$K (m/s) = \frac{Q L}{A T H}$$

Where Q is the quantity of water in millimeters percolating over the period of test, L is the thickness of sample (m), A is the area of the sample (m²), T is the time in seconds over which Q is measured and H is the pressure head (m).



Figure 4: water permeability apparatus setup

- **CHLORIDE PENETRATION**

The chloride penetration resistance of blends mortar samples was investigated according to ASTM C 1556 using Ø70 × 70 mm cylinder specimens. After curing the samples in water to 28 days, all the sides of the sample were coated with waterproof Silicone in a way that only one side of the sample (the upper surface) is free for chloride penetration. thereafter, the sample was placed in a vacuum desiccator for 2 hours and then the water stopcock was opened to cover the specimen for 22 hours. After that, the specimens were taken out from the vacuum desiccator and placed in a container with 5% sodium chloride solution (with fixed the volume of the solution for all samples) for 60 day without renewal of solution (Figure 5). When the test was completed, the samples was taken out from the container and then split into two halves. the two halves were used to determine the chlorides ion along the depth with interval (0-1, 1-3, 3-5, 5-8, 8-12, 12-16, 16-20, 20-25 and 25-30 mm) as recommended by ASTM C1556 using the water-soluble method described in [15]. The obtained samples were ground to pass through 75 µm sieve. After that, the dust samples were oven-dried at 105°C for 2 h in an oven. About 2g of each dried sample was dispersed with 50 mL (80 °C) warm dis-tilled water in plastic container. a magnetic stirrer was used for 10 min to guarantee that the sample and dis-tilled water were well-mixed. After that, the sample was kept in plastic container for 24 h to dissolve the water-soluble chloride from the samples. The solution was then filtered using a dry Whatman filter

paper number 40 into a flask. The filtrate was analyzed for chloride ions by a titration of a 0.1 M silver nitrate solution with 5% potassium chromate as an indicator, with the pH adjusted during the measurements by dilute sulfuric acid in order to keep pH at 9. Thereafter, the chloride diffusion coefficient D_{app} determining mathematically by fitting to experimentally determined chloride profile concentrations using the equation called the "error function" of Fick's second law [16].

$$C_{(x,t)} = C_s \left[1 - \operatorname{erf} \left(\frac{x}{\sqrt{4 D_{app} t}} \right) \right]$$

Where $C(x,t)$ is concentration of Cl^- at any depth x , C_s is the surface concentration, erf is the Gaussian error function obtained from computer, x is the distance from the exposed surface, D_{app} is the chloride diffusion coefficient and t is the exposure time.



Figure 5: The experimental setup for chloride penetration test

• CARBONATION RESISTANCE

The carbonation performance of blends mortars samples was investigated on $40 \times 40 \times 160$ mm prism specimens. After 28 days of curing condition, the mortar bars were exposed to a natural atmosphere condition with natural concentration of CO_2 for 180 day (Figure 6). After 180 days of CO_2 exposure, the samples were split into two halves and the fresh surface was sprayed with a 1wt.% phenolphthalein indicator solution to determine the carbonation depth of blend mortars samples.



Figure 6: Exposure to natural carbonation (laboratory environment)

III. RESULTS AND DISCUSSION

A. WATER ACCESSIBLE POROSITY

The results of porosity of LC^3 blends and OPC cement mortars cured at three distinct ages are shown in Figure 7. It is clear that the porosity values for all the LC^3 blends were lower than those of the OPC at all ages. It can also be observed that the higher the replacement level of cement with limestone and calcined clay, the lower is the water-permeable absorption especially at later ages. The decrease in the porosity value of LC^3 blends could be attributed to a reduction in the total volume of capillary pores as well as increased tortuosity. The hydration products formed by the pozzolanic reaction of metakaolin in LC^3 blends are deposited in the pores and contribute to minimize the capillary porosity of the system in which the refinement of pore structure caused by pozzolanic reaction of metakaolin has been widely reported in the literature.

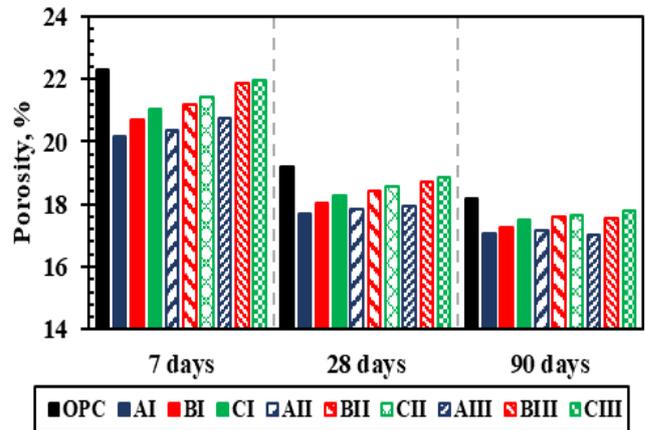


Figure 7: Porosity of OPC and LC^3 blends mortars at different ages

The correlation between compressive strength and porosity of LC^3 blends mortar are shown in Figure 8. It can be seen that with the correlation coefficient of $R^2 = 0.87$, the LC^3 blends mortars followed a trend similar to that of OPC mortar.

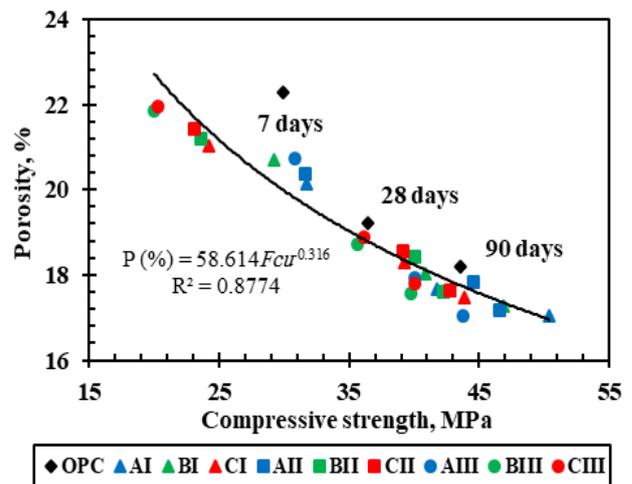


Figure 8: Relationship between the compressive strength and porosity of OPC and LC^3 blends mortars

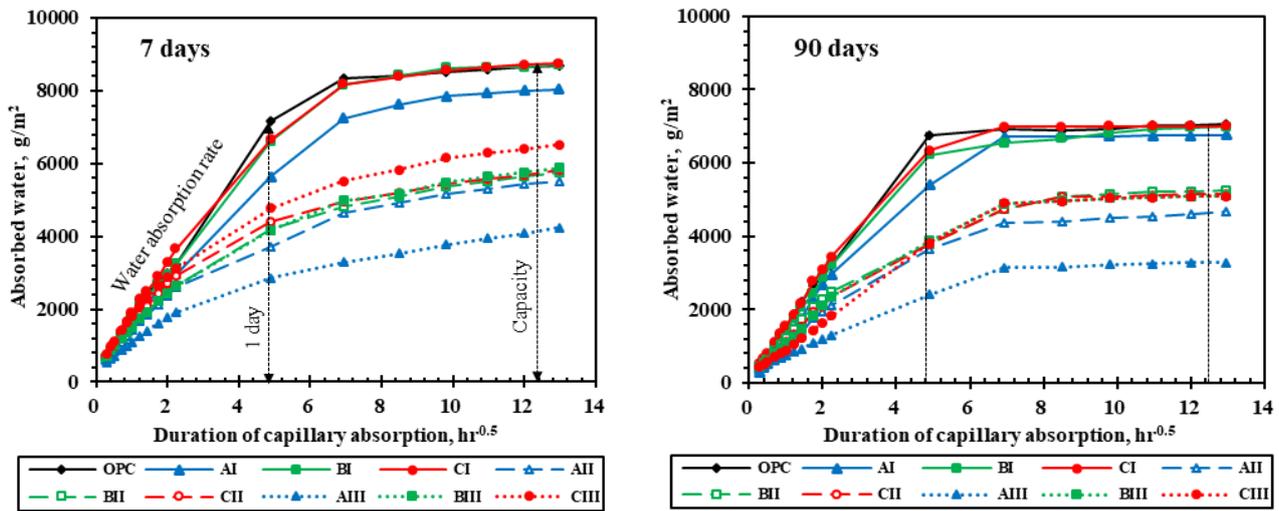


Figure 9: Capillary water absorption of OPC and LC³ blends mortars at 7 days (Left) and 90 days (Right)

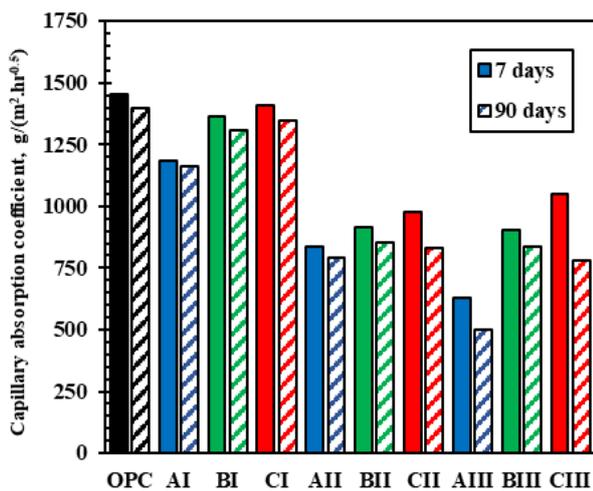


Figure 10: Capillary absorption coefficient of OPC and LC³ blends mortars after 24 hours

B. CAPILLARY WATER ABSORPTION

Figure 9 shows the capillary water absorption curve of LC³ blends and OPC cement mortars determined after 7 and 90 days of curing. Figure 10 shows the values of the water absorption coefficient by fitting a straight line to the water uptake of the sample in 24 hours. The water absorption rate and the capacity of absorbed water decreased for all mortars studied with increasing age. All the LC³ blends exhibited lower water absorption rates and capacities than the OPC at all ages. It can also be observed that, the reduction in water absorption rate and capacity was inversely proportional to the increase in cement substitution by limestone and calcined clay. The reduction in values of the water absorption rate and absorbed water capacity of LC³ blends could be attributed to a decrease in the total volume of capillary pores and the significant refinement of pore connectivity. The capillary pores present in the concrete and their connectivity are the main governing factors in the water absorption rate in concrete. Previous studies [12, 17, 18] have shown that, the number of pores corresponding to smaller size increases in LC³ blends due to the pozzolanic reaction of calcined clay and the ongoing formation of

secondary hydration products (i.e., AFm phases) which fill the voids, making the pore structure denser and less connected. These results are consistent with the results presented in Figure 9 and Figure 10.

However, it is worth mentioning that the LC³ blends containing fairly pure metakaolin (86% kaolinite) showed lower water absorption rates than blends containing clays with 55 and 48% kaolinite. This implies that kaolinite content in LC³ blends may increase the refinement of pore connectivity, which agree with the findings by H.Maraghech [17].

C. WATER PERMEABILITY PENETRATION

The results of the maximum and average water penetration depth (mm), and water permeability coefficient ($\times 10^{-9}$ m/s) of the studied mortars determined after 28 days of curing are presented in Figure 11. It was observed that all the LC³ blends mortars exhibited lower water permeability than the OPC. This effect was more pronounced as the calcined clay and limestone content increased. Generally speaking, the permeability of water is related to the capillary pores present in the concrete and their connectivity. This shows that the most porous composites have the highest water permeability coefficients. These findings are consistent with the results of capillary water absorption given in the previous section. However, it should be noted that the improvement in water penetration resistance increased with increasing the kaolinite content of the clays, but in general, there is not a huge gain achieved from using fairly pure calcined clay (86% kaolinite), compared with LC³ blends containing clays with 55 and 48% kaolinite, especially in mixtures with a high substitution level.

D. CHLORIDE PENETRATION

The total chloride content profiles of the mortar samples ponded in 5% NaCl solution after 2 months of exposure are illustrated in Figure 12. It can be seen that the penetration depth as well as the chloride content at the surface decreased with increasing the level of cement replacement with limestone and calcined clay. This indicates that porosity refinement is of greater importance with respect to the impact of limestone and calcined clay

on the transport of ions. Furthermore, LC³-50 blends show the best resistance to chloride penetration. The chloride content of these systems decreased rapidly throughout the mortar depth and was negligible after 12 mm from the exposure surface after ponding for 2 months. On the other hand, the OPC mortars presented a gradual decline in chloride content from the exposure surface to a depth of up to 25 mm.

The apparent chloride diffusion coefficients of LC³ blends and OPC cement mortars are presented in Figure 12. The diffusion coefficients of chloride ions were obtained by using non-linear regression analysis and the method of least squares of Fick's second law of diffusion according to ASTM C1556. The apparent chloride diffusion coefficient decreased as the level of cement replacement with limestone and calcined clay increased.

In contrast, a slight improvement in the apparent diffusion coefficient was observe with increasing the kaolinite content of the calcined clays. This result indicates that utilizing low-grade calcined clay has no significant effect on the apparent chloride diffusion coefficient. The significant low of chloride diffusion coefficient for LC³ might be interpreted as a result of refinement of porosity resulted from the pozzolanic reaction. In addition, limestone and the high alumina content in calcined clay increases the high capacity of chloride binding, which bond to the chloride ions to produce Friedel's salt compared to OPC, this was confirmed in Previous investigations [17, 19].

E. CARBONATION RESISTANCE

Figure 13 illustrates the carbonation depths of the mortar samples after 6 months of exposure to natural carbonation, using a colorimetric pH indicator test. The results show that all LC³ blends generally display lower

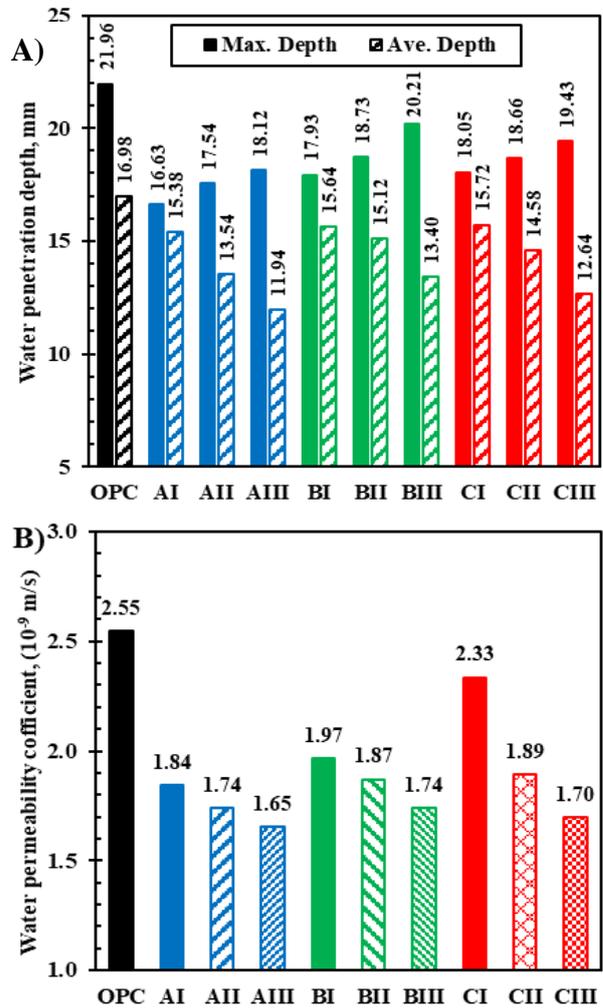


Figure 11: A) Maximum water penetration depth and B) Water permeability coefficient of OPC and LC³ blends mortars at 28 days

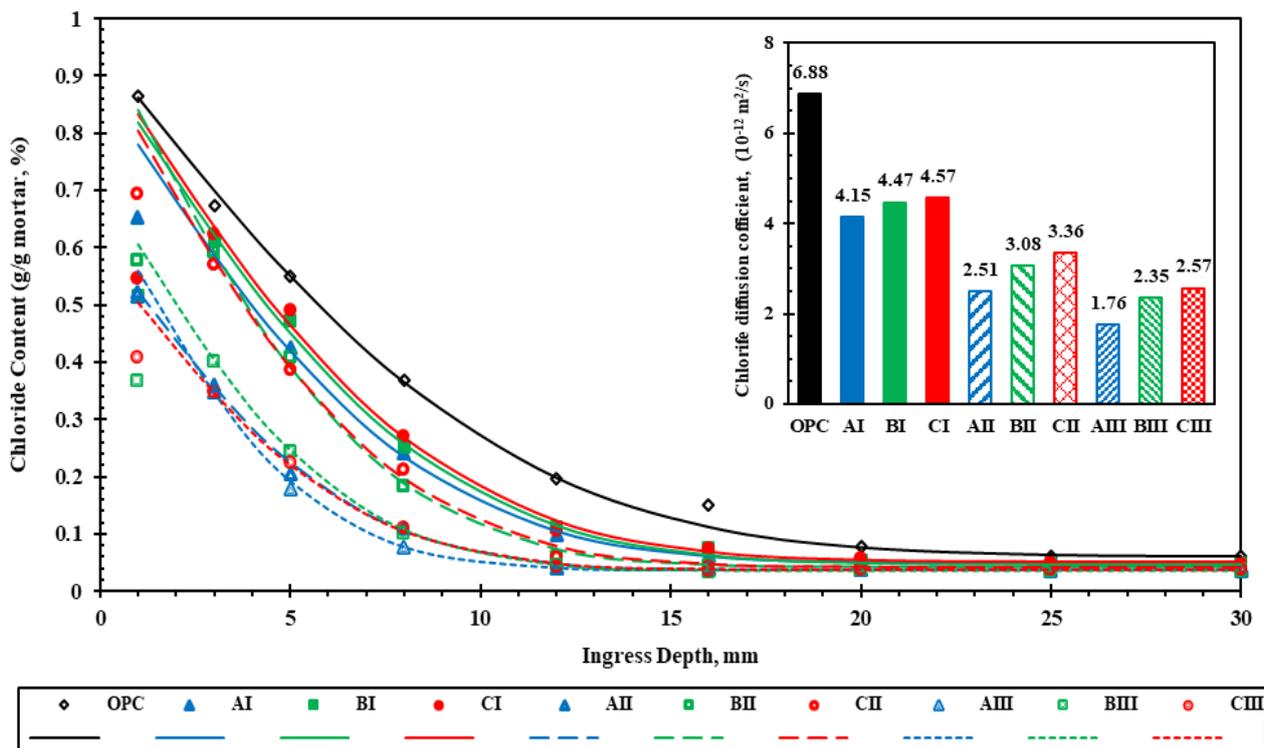


Figure 12: Total chloride content profiles and chloride diffusion coefficients in OPC and LC³ mortars

resistance against carbonation attack compared to OPC. In general, the carbonation resistance of cement containing SCMs is generally lower as compared to OPC due to lower total alkalinity [20]. Therefore, increasing the cement substitution rate resulted in a progressive decrease in carbonation resistance. Resistance against carbonation is not dominated by porosity and pore connectivity of the mortar, which are refined in LC³ blends compared to the OPC, as previously discussed. Carbonation is mainly influenced by the chemical composition of the blended matrix. The lower carbonation resistance of LC³ blends can be attributed to a lack of calcium hydroxide content due to the lower amount of clinker in the blends as well as the consumption of calcium hydroxide by the pozzolanic reactions. As a result, the extent of chemical reactions that reduce CO₂ diffusion is reduced in LC³ blends compared to OPC. Which leads to an increase in the diffusion of CO₂. However, an increase in cover depth would be required to achieve a similar service life as that of the OPC.

IV. CONCLUSIONS

Based on the results of this study, the following conclusions were drawn:

1. All LC³ systems showed significantly improved results in terms of total porosity, capillary water absorption and water penetration resistance compared to OPC. Moreover, the higher the replacement of cement by limestone and calcined clay, the better the promotion of porosity refinement.
2. The apparent chloride diffusion coefficient decreased as the level of cement replacement with limestone and calcined clay increased. The improvement in chloride resistance for LC³ blends can be attributed to its enhanced chloride binding capacity as well as the enhanced porosity refinement.
3. A slight improvement in the transport properties of LC³ blends was observed with increasing the kaolinite content of the calcined clays. This result indicates that utilizing low-grade calcined clay has no significant effect on the transport properties of LC³ blends, especially in blends with a high substitution level.
4. The carbonation resistance of the LC³ blends was lower as compared to OPC. Furthermore, increasing the cement substitution rate resulted in a progressive decrease in carbonation resistance. However, at 15% replacement, the carbonation resistance of the LC³ blend was found to nearly equal or slightly lower than the OPC. Overall, an increase in cover depth would be required to achieve a similar service life as that of the OPC, especially in blends with a high substitution level.

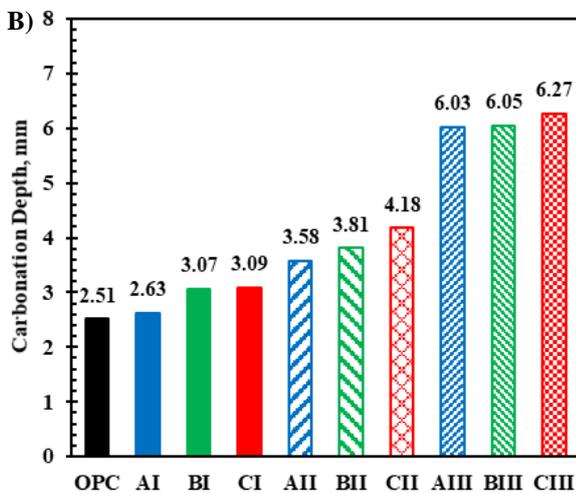


Figure 13: A) Variation in the carbonation front by colorimetric pH indicator and B) Carbonation depths (mm) of OPC and LC³ blends mortars for 6 months of exposure to natural carbonation

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