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LinkedIn Revista Ingeniería de Construcción

Vol. 39
Special Issue 2024
DOI: 10.7764/RIC.00129.21

The influence of Interground limestone fines and metakaolin on the electrical resistivity of portland-limestone concrete

La influencia de la molienda conjunta de finos de piedra caliza y metacaolinita en la resistividad eléctrica del hormigón De cemento Portland con piedra caliza

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Fecha de Recepción: 01/12/2024
Fecha de Aceptación: 12/12/2024
Fecha de Publicación: 30/12/2024
PAG: 1-12

Abstract

The critical climate change has raised concern about the decarbonization of the cement and concrete industry, which is responsible for 8% of global CO2 emissions. Portland limestone cement (PLC), which is made by partially replacing the clinker with up to 15% interground limestone fines (LFs), has been recognized as a viable solution for its feasibility to match the engineering properties of ordinary portland cement (OPC). However, with the necessity of further increasing the LFs contents to meet the desired eco-efficiency, the dilution effect brought by less ultimate hydration products may be detrimental to the long-term performance of reinforced concrete structures, such as chloride-induced corrosion. Thus, this research explores the potential of combining PLC and alumina-rich supplementary cementitious materials (SCMs) to improve the resistivity of the concrete. Concrete specimens were fabricated with PLCs from two sources of three LFs replacement ratios (15, 20, and 25%). MK (8%) is used as the source of alumina. The bulk and surface resistivity results showed that combining PLC with MK can notably improve concrete resistivity even in mixtures with lower amounts of cement. Additionally, compressive strength demonstrated poor correlation with electrical resistivity, which highlights the significance of performance-based design.

Keywords: Portland-limestone cement; metakaolin; chloride ingress; electrical resistivity; decarbonization.

Resumen

El estado crítico del cambio climático ha generado preocupación sobre la descarbonización de la industria del cemento y del hormigón, el cual responsable del 8% de las emisiones globales de CO_2. El cemento Portland con piedra caliza (PLC), fabricado reemplazando parcialmente el clínker con hasta un 15% de finos de piedra caliza intermolida (LFs), ha sido reconocido como una solución viable debido a su capacidad para igualar las propiedades de ingeniería del cemento Portland ordinario (OPC). Sin embargo, debido a la necesidad de aumentar aún más los contenidos de LFs para alcanzar la ecoeficiencia deseada, el efecto de dilución provocado por una menor cantidad de productos de hidratación finales puede ser perjudicial para el rendimiento a largo plazo de estructuras de hormigón armado, como ocurre en la corrosión inducida por cloruros. Por lo tanto, esta investigación explora el potencial de combinar PLC con materiales cementantes suplementarios (SCMs) ricos en alúmina para mejorar la resistividad del hormigón. Se fabricaron especímenes de hormigón con PLC de dos fuentes y tres niveles de reemplazo de LFs (15%, 20% y 25%). La metacaolinita (MK) al 8% se utilizó como fuente de alúmina. Los resultados de resistividad eléctrica, tanto en volumen como en superficie, mostraron que combinar PLC con MK puede mejorar notablemente la resistividad del hormigón incluso en mezclas con cantidades menores de cemento. Además, la resistencia a compresión mostró una baja correlación con la resistividad eléctrica, lo que destaca la importancia de un diseño basado en el rendimiento.

Keywords: Cemento portland con piedra caliza; metacaolinita; penetración de cloruros; resistividad eléctrica; descarbonización.

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DOI: 10.7764/RIC.00129.21

1. Introduction

Portland-limestone cement (PLC) is a Portland cement product with a higher proportion of unprocessed limestone than ordinary Portland cement (OPC). PLC is typically made in two ways: by combining cement and graded LFs during the concrete batching and mixing process or by intergrading cement clinker and limestone during the cement production. Although both methods of cement replacement seem similar, PLC of higher fineness can be achieved by over-grinding the clinker with the limestone, resulting in higher fineness (Kumar et al. 2013).

For many years, the interground limestone fines (LFs) in PLC have been regarded as inert fillers due to their higher fineness than cement particles. The benefits of incorporating LFs are primarily in reaching a more densely packed system (via filling the space between cement grains) and accelerating the early hydration process (via providing nucleation sites of high specific surface area for the precipitation of C-S-H gels). An optimum amount of up to 15% LFs is widely accepted by academia and industry (Cost et al. 2013); (Moon et al. 2017); (Tennis et al., 2024), beyond which the risk of obtaining diminished short and long-term performance will rise with a lack of enough hydration products (Lothenbach et al. 2008).

Nevertheless, the chemical effect of LFs addition is also validated through thermodynamic modelling and experimental measurements (Lothenbach et al. 2008); (Briki et al. 2021), meaning that LFs are also involved in cement hydration and modifying the properties of concrete. With the sole addition of LFs, C3A and portlandite will react to form Moncarbonate (Mc) instead of monosulphate (Ms), indirectly stabilizing ettringite and leading to a corresponding decrease of the total volume of the hydrate phase (Lothenbach et al. 2008), and thus be detrimental to the mechanical properties of concrete. However, these chemical adverse effects can be reversed when combining the LFs with alumina-rich SCMs, where the pozzolanic reactivity of SCMs will be enhanced by the consumption of alumina to form Mc and simultaneously more C-(A)-S-H and crystalline hydrated phases such as C2ASH8 (Tang et al. 2019). Such SCMs (e.g., fly ash, slag) have been used successfully worldwide to partially replace OPC and produce durable and sustainable concrete. Besides the most applied SCMs, metakaolin (MK) bears the highest alumina content (Figure. 1) and, therefore, is promising when used with elevated amounts of LFs (i.e., more than 15%) to produce sustainable, strong, and durable concrete.

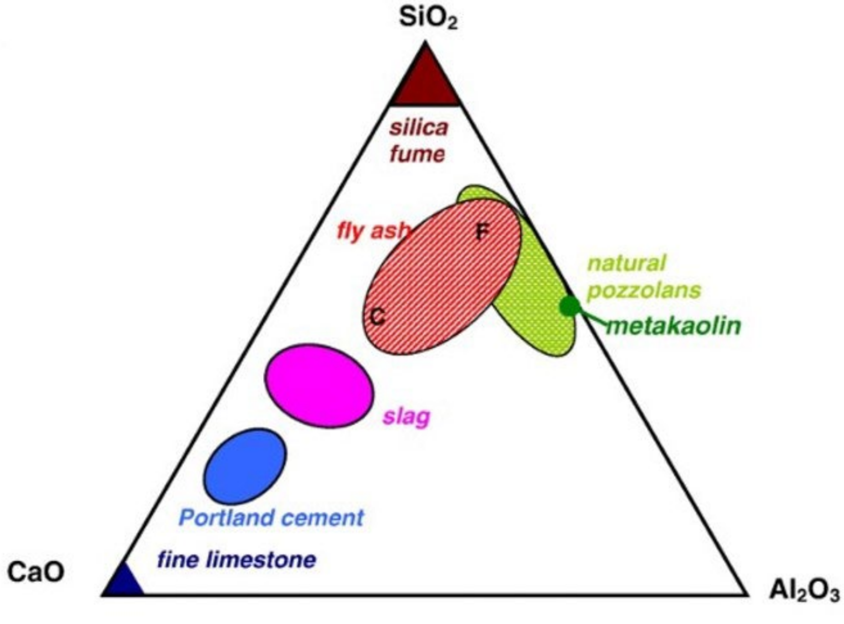

Figure 1. CaO–Al2O3–SiO2 ternary diagram of cementitious materials (Lothenbach et al. 2011).

The reported synergistic effect of MK and LFs in concrete refers to the enhanced mechanical strength achieved when both materials are used together in concrete. When LFs and MK are combined in concrete mixtures, they complement each other's effects. MK provides reactive alumina silica phases, contributing to the formation of additional C-(A)-S-H gel. At the same time, LFs react with the alumina and portlandite in the pore solution to form Hemi-carboaluminate (Hc), which promotes the reaction of LFs. LFs also help optimize the particle packing, leading to denser and more durable concrete. Previous research has focused on exploring the optimal replacement ratio and MK to LFs ratio regarding compressive

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strength. It is reported that at 45% cement replacement, the optimal synergistic impact is achieved when the MK to LFs ratio is 2:1, where the synergy results in concrete improvements with enhanced compressive strength compared to solely incorporating LFs and even pure OPC (Antoni et al. 2012).

Nevertheless, although prescriptive parameters like compressive strength, water-to-cement (w/c) ratio and minimum cement contents are globally applied in standards to reflect concrete quality, it has been challenged by many researchers that they cannot reflect the durability of eco-efficient concrete with the implementation of chemical admixtures, SCMs, mineral fillers etc. (Celik et al., 2015); (Beushausen et al. 2019); (Douglas Hooton 2019).

In this context, this research investigates the electrical resistivity of blends with Portland limestone cement (PLC) and MK. The correlation between different tests on compressive strength is also examined.

2. Scope of the Work

As aforementioned, there is still a lack of assessment of the performance of PLC when used in combination with MK on electrical resistivity. Therefore, the project is divided into two phases.

In the first phase, the target is to evaluate the electrical resistance made of pure PLC with increasing amounts of LFs (from 15% to 25%). Six mixtures were fabricated with the w/c = 0.45. The influence of the fineness of the PLC will also be revealed by comparing the results from sources A and B.

The second phase aims to explore the potential of MK to improve the electrical resistivity of PLC concrete, which displays lower total cement content but with the same w/c ratio (i.e., 0.45). The MK replacement ratios were selected as 8% for all six groups. A mid-range water reducer and superplasticizer (SP) are used to achieve the same slump (100 ± 10 mm) as the mixtures in the first phase.

Bulk and surface resistivity measurements were then recorded over time (i.e., until 91 days), and correlations with the 28-day compressive strength results of the materials were evaluated.

3. Experimental Program

3.1 Raw Materials

A total of 12 mixtures based on PLC with different replacement ratios (i.e., 15%, 20% and 25%) from 2 companies (i.e., A and B) were fabricated. The physical properties and chemical composition of the PLCs and MK are shown in (Table 1). (Figure 2) shows the particle size distribution for the binders. Nature sand and limestone coarse aggregate of 4.75mm to 19 mm were used for the casting.

Table 1. Chemical composition, mineralogical phases and limestone contents of the Portland-limestone Cements and Metakaolin.

Components	A-15%	A-20%	A-25%	B-15%	B-20%	B-25%	MK
CaO	60.47	59.25	59.26	62.19	60.83	60.87	0.39
SiO_2	19.64	19.24	18.49	18.71	17.71	17.20	62.5
Al_2O_3	4.82	4.85	4.55	4.51	4.27	4.09	29.8
Fe_2O_3	2.20	2.15	2.06	2.78	2.60	2.55	1.23
MgO	2.29	2.25	2.20	2.19	2.11	2.05	0.42
K_2O	1.09	1.04	0.98	1.04	0.96	1.00	1.75
Na_2O	0.27	0.24	0.29	0.29	0.26	0.23	0.18
TiO_2	0.26	0.24	0.26	0.19	0.19	0.17	0.69
P_2O_5	0.14	0.13	0.13	0.13	0.13	0.12	0.03
MnO	0.05	0.05	0.05	0.06	0.05	0.06	0.01
Na_2Oeq	0.98	0.92	0.93	0.97	0.90	0.89	1.33

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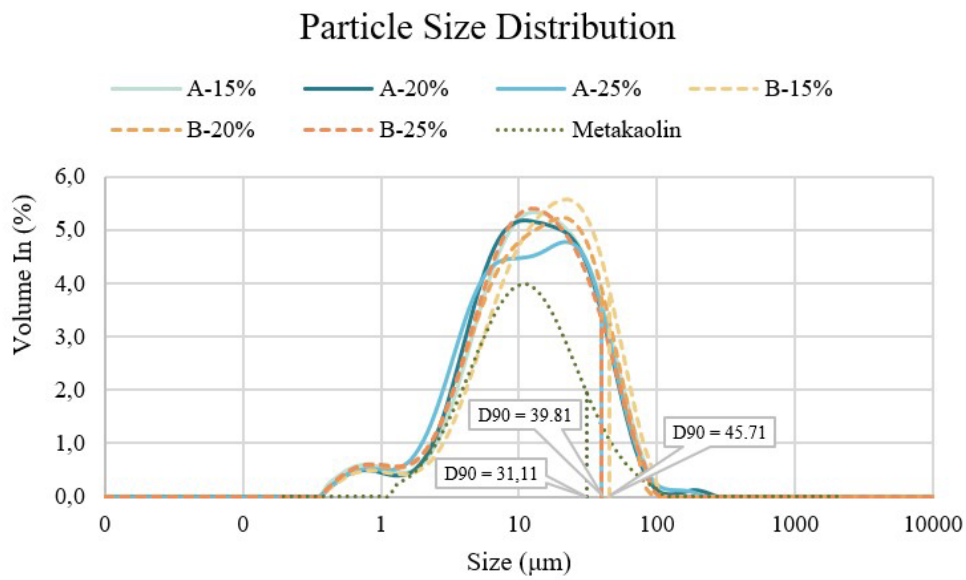

Figure 2. Particle Size Distribution of the Binders.

3.2 Mix Proportion and Curing

The mix design was prepared as shown in (Table 2). All the concrete mixtures were designed to have a constant water-to-cement ratio (i.e., 0.45), with or without MK. For all the MK-bearing families, the PLC content is reduced to further enhance the eco-efficiency of the mix. A water reducer and superplasticizer (i.e. 1.5% by weight of the PLC) were added to adjust the fresh-state performance.

After the production of each mix, the cylindrical concrete specimens (100 mm in diameter and 200 mm in length) were molded and covered with a wet cloth and a plastic sheet for one day. All the specimens were then demolded and kept in moisture curing boxes (RH = 100%, temperature = 25℃) until the designated testing age.

Table 2. Concrete mixture proportions.

Mixture	Water	PLC	MK	Coarse Aggregate	Fine Aggregate	WR	SP
PLC15- A/B	204.2	455.6	0	1071.7	713.6	0	0
PLC20 - A/B	204.2	455.6	0	1071.7	713.6	0	0
PLC25 - A/B	204.2	455.6	0	1071.7	713.6	0	0
PLC15M8 - A/B	135.9	299.9	26.1	1179.8	866.3	4.5	4.5
PLC20M8 - A/B	135.9	299.9	26.1	1179.8	866.3	4.5	4.5
PLC25M8 - A/B	135.9	299.9	26.1	1179.8	866.3	4.5	4.5

WR: Mid-range water reducer.
SP: Superplasticizer.

3.3 Test Methods

3.3.1 Bulk Electrical Resistivity

The bulk resistivity (BR) test provides a rapid indication of the concrete's resistance to the penetration of chloride ions by diffusion. The apparatus comprises a resistivity test device (Giatec RCON), electrical cables, sponges, and stainless steel electrically conductive plate electrodes, as shown in (Figure 3).

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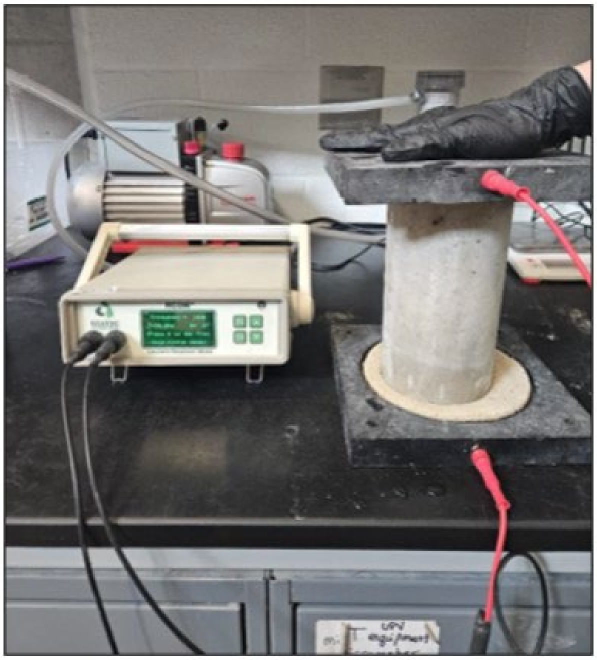

Figure 3. Bulk Electrical Resistivity Set-up.

As per ASTM C1876 (C09 Committee, 2019), the electrical resistivity was determined by calculating the current passing through all phases of the specimen. Cylindrical specimens with 100 mm in diameter and 200 mm in height were taken from each family for measurements until 91 days. Before conducting the test, the specimens are removed from the curing container. Excess water is then blotted off, and the specimens are transferred to a test device designed to hold specimens vertically. The equipment calculates and provides electrical resistance (R), and then the BR of the specimen is calculated from (Equation 1).

$$\rho = \frac{RA}{L} \quad \textbf{(1)}$$

where
ρ = bulk resistivity (Ω·m)
A = Specimens cross-sectional area (m2)
L = Average length of the specimen (m)

CSA A23.2-26C provides the correlation between BR and chloride ions penetrability, as shown in (Table 3).

Table 3. Correlation between BR and Chloride Ion Penetrability (CSA, 2024).

Chloride Ion Penetrability	Bulk Electrical Resistivity (Ω·m)
High	<50
Moderate	50-100
Low	100-200
Very low	200-2000
Negligible	>2000

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3.3.2 Surface Electrical Resistivity

The surface resistivity (SR) is assessed by using a four-point Wenner array probe resistivity meter (Proceq Resipod). The longitudinal array probe is placed and centred against the specimen's surface, as shown in (Figure 4). Subsequently, the device is pressed for 3 to 5 seconds until a stable reading is achieved, which is expressed in KΩ·cm and indicates the surface resistivity of the specimens.

Figure 4. Surface Electrical Resistivity Set-up.

The electrical resistivity results correlate well with chloride exposure tests, such as ASTM C1556. AASHTO T 358-15 (AASHTO, 2015) provides the correlation between BR, SR, and chloride ions penetrability, as shown in (Table 4).

Table 4. Correlation between SR and Chloride Ion Penetrability (AASHTO, 2015).

Chloride Ion Penetration	Surface Resistivity Test	
	100-by-200-mm (4-by-8-in.) Cylinder (kΩ·cm) a = 1.5	150-by-300-mm (6-by-12-in.) Cylinder (kΩ·cm) a = 1.5
High	<12	<9.5
Moderate	12–21	9.5–16.5
Low	21–37	16.5–29
Very low	37–254	29–199
Negligible	>254	>199

a = Wenner probe tip spacing

3.3.3 Compressive Strength

The uniaxial compressive strength was performed on cylinders (100mm x 200mm) according to ASTM C-39 (C09 Committee, 2024), with a 2000 N/s loading rate. After 28 days of curing in saturated lime water and room temperature (i.e., 25°C), both ends of the cylinders are ground to obtain flat surfaces. For each tested mixture, two specimens were tested, and the mean value of each was recorded.

4. Results

4.1 Bulk Electrical Resistivity

The evolution of bulk resistivity results demonstrates an increasing trend for all mixtures over the curing period from 28 to 91 days, as demonstrated in (Figure 5).

Without the addition of metakaolin, the pure PLC families consistently exhibit lower BR values, remaining below 75 Ω·m at 91 days. It is also noticeable that the PLC-A families generally obtain higher resistivity compared to PLC-B families (except PLC25-A). A similar trend is also observed in concrete with 8% MK, where a distinguished divide can be seen between PLC15/20//25M8-A (from 174.11 Ω.m to 191.58 Ω.m) and PLC15/20/25M8-B (from 133.24 Ω.m to 139.75 Ω.m) families. Additionally, regardless of the source, the rate of resistivity development for these

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pure PLC mixtures from 28 to 91 days is relatively marginal (around a 15% increase), indicating a limited enhancement in electrical resistivity without adding metakaolin.

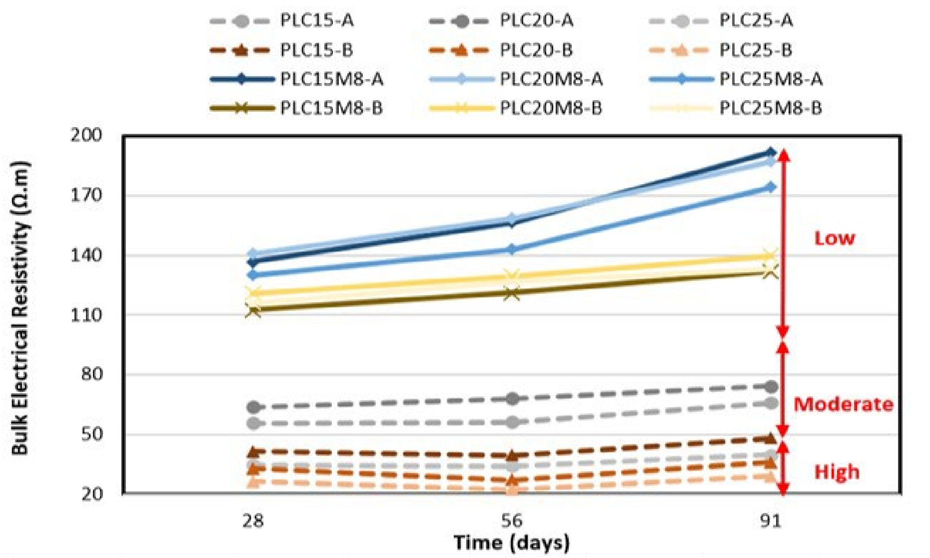

Figure 5. Results of Bulk Electrical Resistivity.

The variation in LFs content (15%, 20%, and 25%) also influences the BR development. Nevertheless, the impact is not the same for PLC from both sources. In pure PLC-A concrete, the resistivity keeps increasing until the LFs content is 20%, whereas PLC-B concrete illustrates a lower threshold and LFs content (15%) exhibits slightly higher resistivity than those with higher LF (20% and 25%). Nevertheless, most pure PLC families are still characterized as high chloride penetrability according to the standard (Table 3). Only the PLC15-A and PLC20-A mixtures achieved a BR value exceeding 50 Ω·m, indicating moderate chloride penetrability.

In contrast, mixtures incorporating 8% metakaolin exhibit significantly higher BR values. All PLC groups with 8% of metakaolin addition exceed 110 Ω·m, with PLC15M8-A achieving the highest BR value of 191.58 Ω·m at 91 days. All groups achieved low chloride penetrability after 28 days of curing.

4.2 Surface Electrical Resistivity

The surface resistivity (SR) results, depicted in (Figure 6), demonstrate the progression of SR values for various concrete mixtures over curing periods of 28, 56, and 91 days.

A clear trend of increasing SR values over time is observed across all mixtures, with notable differences between the pure PLC mixtures and those incorporating MK. The MK-bearing mixtures exhibit consistently higher SR values at all curing ages, indicating their enhanced resistance to chloride penetration contributed by refined pore structure (Tang et al. 2019). Among these families, PLC20M8-A achieves the highest SR value at 91 days (33.8 kΩ·cm), followed closely by PLC25M8-A (32.8 kΩ·cm) and PLC15M8-A (32.7 kΩ·cm). In contrast, mixtures containing 25% limestone (PLC25-A and PLC25-B) show the lowest SR values, measuring 6.05 kΩ·cm and 4.30 kΩ·cm, which is around five times lower than the value of the corresponding concrete with MK. The PLC20-A mixture is the only mix that achieves moderate chloride penetrability, with an SR value of 12.4 kΩ·cm at 56 days.

Additionally, a slight variation is observed between sources A and B, with mixtures from resource B generally showing marginally lower SR values than those from resource A. This trend, which is also reflected in the bulk resistivity (BR) results, suggests the potential impact induced by the fineness.

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In summary, all MK-containing mixtures consistently exhibit low chloride penetrability at all test ages, further highlighting the effectiveness of MK in enhancing the concrete's resistance to chloride ingress.

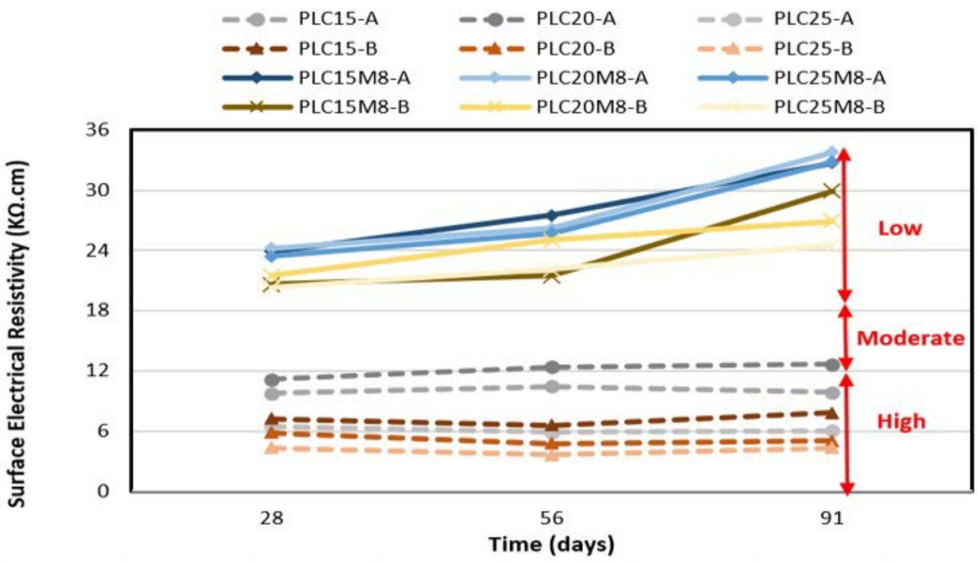

Figure 6. Results of Surface Electrical Resistivity.

4.3 Compressive Strength

The 28-day uniaxial compressive strength results are shown in (Figure 79. Without the addition of MK, for PLC-A groups, the compressive strength increases when the LFs content is increased from 15% to 20% but decreases with a further increase from 20% to 25%, with the highest value observed in PLC20-A at 42.43 MPa. Nevertheless, PLC-B families indicate a consistent declining trend, decreasing from 35.78 MPa in PLC15-B to 24.61 MPa in PLC25-B.

For the mixtures containing MK, the PLC-A group exhibits a continuous decrease, reaching a value of 42.92 MPa in PLC25M8-A. Differently, PLC20M8-B shows a significant reduction from 34.77 MPa to 23.33MPa with 5% more LFs, followed by an increase to 34.1 after a further 5% LFs.

Based on the results, there is no direct correlation between the amount of LFs and compressive strength. However, the PLC-A families generally obtained a higher compressive strength than their corresponding specimens made of PLCs from source B, regardless of the MK addition.

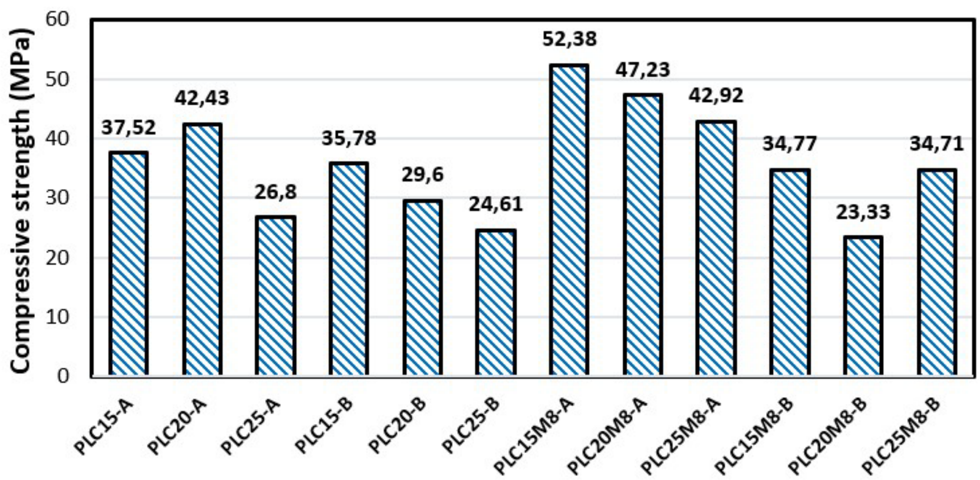

Figure 7. Results of Compressive Strength.

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5. Discussion

5.1 Surface and Bulk Electrical Resistivity

The results of BR and SR suggest that increasing the LFs content can reduce resistivity without MK addition, potentially due to a dilution effect and chemical effect that altered the overall hydration and microstructural development (Lothenbach et al. 2008); (Wang et al. 2018). Nevertheless, the boundary beyond which the adverse effects will overwhelm the advantages of the filler effect also depends on other factors, such as fineness, as evidenced by the difference between PLCs from sources A and B.

The improved resistivity after MK addition emphasizes the benefit of the synergistic effect where more secondary products (C-A-S-H) are generated, leading to refined microstructure and reduced porosity measured by mercury intrusion (Tang et al. 2019). Additionally, the advantages of adding MK will become more evident with extended curing time, which aligns with the longer hydration period for pozzolans and subsequent pore structure refinement. As curing progresses, without the addition of MK, the growth in BR and SR occurs relatively slower, indicating a marginal and limited gain in electrical resistivity. In contrast, mixtures containing MK exhibit significantly faster and more resistivity development, highlighting the beneficial influence of MK.

(Figure 8) illustrates the correlation between BR and SR. Consistent with the literature (T. De Grazia et al. 2021), a linear correlation between BR and SR is observed in the Portland cement system. This study focuses on PLC concrete with the addition of metakaolin to achieve a low carbon footprint. A strong linear correlation between BR and SR is observed, with a high coefficient of agreement (R^2 = 0.9809) at a level of confidence of 0.95. The results support the use of surface resistivity as an indicator of bulk resistivity, which in turn enhances the feasibility of using non-destructive tests (NDTs) techniques for quality control in field construction.

5.2 Compressive Strength

Traditionally, the baselines for designing concrete mixtures are set primarily on engineering properties such as minimum compressive strength, which are largely governed by prescriptive parameters such as water-to-cement ratio (w/c) and minimum cement contents. Durability design received only secondary consideration; thus, the provision of deemed-to-satisfy requirements for structural design also serves as an indirect method of specifying durability in a particular aggressive exposure (Douglas Hooton 2019).

Although those prescriptive requirements were able to provide satisfactory quality in the past when the compositions of cement and concrete were basic and straightforward (Alexander, 2017); (Douglas Hooton, 2019), it turned out that such deemed-to-satisfy requirements cannot reflect the durability of the modern concrete with the evolution of manufacturing process and implementation of chemical admixtures, SCMs, mineral fillers etc. (Alexander and Beushausen 2019); (Douglas Hooton 2019), which is also revealed by this research.

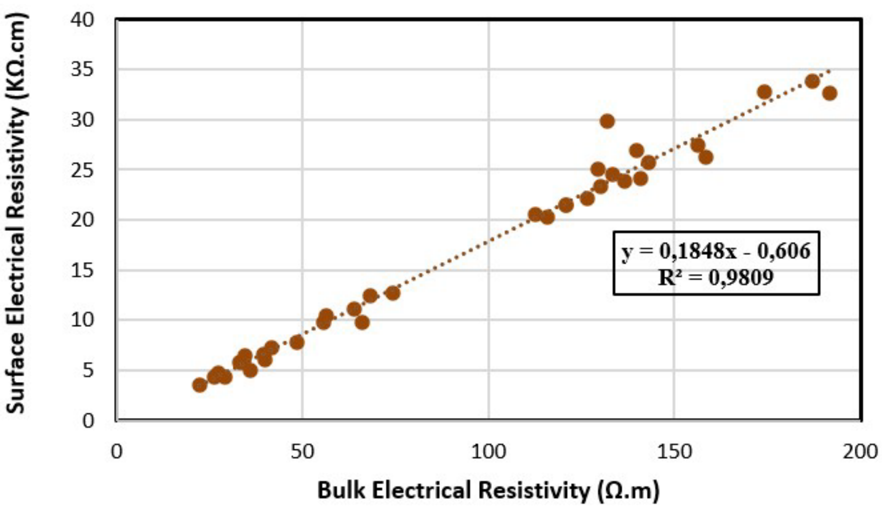

Figure 8. The correlation between BR and SR.

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(Figure 9a) and (Figure 9b) illustrate the correlation between bulk resistivity (BR), surface resistivity (SR), and compressive strength. Both bulk and surface electrical resistivity exhibit a positive correlation with compressive strength, though the correlation is moderate in both cases. Compressive strength, as a critical mechanical property of concrete, has been shown in previous studies to have a potential linear relationship with surface resistivity (SR) in Portland cement systems (T. De Grazia et al. 2021). However, the correlation coefficients of 0.317 for BR and 0.300 for SR indicate considerable variability, suggesting that electrical resistivity alone does not have a reliable correlation with compressive strength in the PLC system containing metakaolin.

These findings, which align with the rising concern about adopting a performance-based design approach in the standard, reveal the discrepancy between performance tests and prescriptive requirements, particularly for eco-efficient concrete, which incorporates PLC and SCMs.

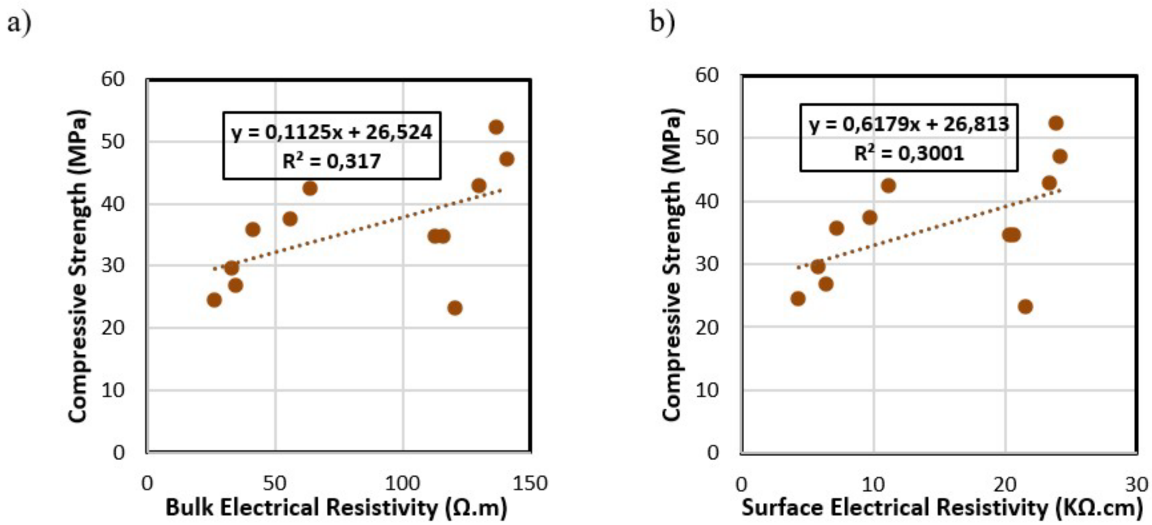

Figure 9. The correlation between a) BR and compressive Strength and b) SR and compressive Strength.

6. Conclusion

The previous life cycle assessment (LCA) on concrete has revealed the eco-efficiency of LFs and SCMs while also raising concern about the substantial cost of repair (Celik et al. 2015); (Renne et al. 2022). It is apparent that enhancing the durability (and hence prolonged service life, less cost on maintenance and repair) of concrete structures is of the same significance as sustainability. Therefore, a transition from prescriptive to performance-based guidelines is urgently needed to provide more precise durability assessment and service life prediction of the concrete structures while enabling innovations in sustainable concrete technologies.

In this situation, concrete producers can adopt more imaginative and innovative choices of materials, such as supplementary cementing materials, chemical admixtures, blended cement, polymers, fibers, and mineral fillers, which will facilitate the decarbonization of the cement and concrete industry.

This research analyzes the use of Portland-limestone cement with the incorporation of metakaolin in terms of electrical resistivity. A summary of the key observations is shown below:

• At the same water-to-cement ratio, the combination of PLC and 8% metakaolin in concrete mixtures has demonstrated promising improvements in electrical resistivity, even with lower total cement content. This indicates the potential of achieving high eco-efficiency while simultaneously obtaining a significant refinement of concrete's pore structure by the beneficial synergistic effect between PLC and MK.

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• No clear correlation between the LFs content and the electrical resistivity of the PLC concrete can be established in this research. The incorporation of a mixture with other replacement percentages close to those obtained is needed for future study. However, PLC of higher fineness generally demonstrated elevated performance regarding chloride penetrability.

• Regardless of the fineness, pure PLC families with a w/c = 0.45 can only achieve a maximum moderate chloride penetrability according to the standard even after 91 days of curing in this research. Nevertheless, with the addition of 8% MK, all the families can reach low chloride penetrability after curing for 28 days.

• The linear correlation between bulk resistivity (BR) and surface resistivity (SR), with a high coefficient of determination (R^2 = 0.98), persists in PLC systems, both with and without metakaolin. This finding highlights the potential of using SR as a non-destructive testing (NDT) method to facilitate the quality control of concrete structures made of Portland-limestone cement without coring.

• Compressive strength is not a suitable indicator for the electrical resistivity of the PLC concrete, which is revealed by the scattered data points with R^2 values of around 0.3. This signifies the necessity of transition from prescriptive design to a performance-based approach for the chloride resistance of eco-efficient concrete.

7. Notes on Contributors

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Special Issue 2024
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