REFINING NATURAL PERLITE FOR ENHANCED CEMENTITIOUS PERFORMANCE IN CONCRETE

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Abstract

Replacing a portion of ordinary Portland cement (OPC) with natural pozzolans may be an effective solution to reduce the carbon footprint of the concrete industry and construction costs, and to improve concrete durability in general terms. However, some natural pozzolans, such as perlite, need to be pretreated to be reactive enough and satisfy the minimum standard qualifications of the concrete industry. This research investigated the effectiveness of milling of natural perlite as an alternative to calcination, which is the more common reactivation method. For this purpose, the effects of replacing OPC with natural perlite powder (NPP) were studied at three different levels of fineness and replacement ratios. A variety of tests for the mechanical and transport properties of the concrete were carried out, including a compressive strength test, a surface electrical resistivity test, a water penetration test, a rapid chloride permeability test, and a rapid chloride migration test. These tests conducted on 14 different types of concrete mixtures with various ratios of water-to-cementitious materials (W/Cm), cement content, and replacement ratios. The results demonstrated that increasing the fineness of natural perlite could be an effective method to reactivate NPP, leading to almost the same compressive strength while improving the transport properties significantly.

Keywords: Natural Pozzolan reactivation, Natural Perlite powder, concrete corrosion, supplementary cementitious materials, Chloride ion ingress and concrete mass transport properties.

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1. Introduction

Cement plants are the major industrial contributor to CO2 emissions, second after electric power generation. It comprises 5% of the total CO2 emissions that represent the environmental carbon footprint of the concrete industry [1]. A possible solution to help mitigate the substantial environmental effects of the concrete industry is to replace a portion of the OPC used in the concrete mixture design with supplementary cementitious materials (SCM). Reducing the consumption of OPC helps to develop a sustainable concrete that also may cost less or have better mechanical or durability properties [2-5].

SCMs can be categorized as industrial waste materials, such as silica fume or fly ash, and natural pozzolans, either raw pozzolans, such as pumice, or calcined pozzolans, such as metakaolin. Industrial waste materials like fly ash has been the predominant pozzolan for use in concrete construction since the 1930's. However, the availability has become a problem in recent years due to reduction in the use of coal for electric power generation. This is why there has been an increased focus on natural pozzolans such as Perlite powder lately.

Pozzolans are aluminosilicate materials that can react with hydroxide calcium, and they generate a calcium-silicate-hydrate (C-S-H) gel. Generating a C-S-H gel may help improve the mechanical and durability properties of cementitious materials by making the pore structure denser, cutting the connectivity of pores, and affecting the chemical properties of the pore solution [2,7]. This may lead to suppressing both internal and external attacks to the concrete [3, 6]. Natural pozzolans are formed by amorphous or vitreous volcanic-eruption materials that have quickly cooled under various environmental conditions [8]. However, their inherent properties are not sufficiently active to meet the minimum standard requirements for concrete mixtures. Therefore, pretreatment is required by means of either pre-calcination, alkaline activation, or further milling [9-12]. Several studies [13-15] have delved into assessing the efficacy of additional milling as a preliminary treatment technique for Supplementary Cementitious Materials (SCMs). Increasing the specific surface area of SCMS leads to making them more efficient by increasing both the filler effect and the heterogeneous nucleation effect. On the other hand, it results in more secondary pozzolanic reactions due to the higher contact surface between the pozzolan and calcium hydroxide ($Ca(OH)_2$) [16]. The secondary pozzolanic effect becomes more substantial at greater ages than the other two effects.

Supplementary Cementitious Materials (SCMs) offer advantages that can aid concrete materials in better resisting both internal and external attacks more effectively. Internal attacks may be prevented by choosing the right raw materials or construction method. Bektas et al. [17] showcased the ability of replacing Ordinary Portland Cement (OPC) with either Expanded Perlite Powder (EPP) or Natural Perlite Powder (NPP) to mitigate alkali-silica reactions (ASR). These substitutions act as effective measures against internal attacks, countering the reactivity of aggregates, recognized as a critical source of deterioration.

The first line of defense against external attacks are the mass transport properties of concrete, which can be evaluated by a variety of test methods [18, 19]. Ramezanianpour et al. [20] conducted a study examining the impacts of substituting a fraction of Ordinary Portland Cement (OPC) with Expanded Perlite Powder (EPP) on diverse transport properties of concrete mixtures. They found almost constant mechanical properties while transport properties were remarkably improved mainly because of both the consumption of hydroxide ions (-OH) in the pore solution and the reduction in the connectivity and volume of pores [6, 21]. Kotwica et al. [23] showed that replacing OPC with expanded perlite powder, which is an industrial waste material, results in the increasing 1 and 28-day strength of mortar and concrete mixtures. They studied replacement ratios up to 35%.

Investigation of the microstructure of mixtures revealed that the main reason of increasing the strength of cement-based materials is pozzolanic reaction of waste expanded perlite powder. On the other hand, another study on NPP indicated that early-age strength loss is possible as a result of replacing a portion of OPC with perlite powder in the mixture design; however, the results getting closer over time, maybe because of the pozzolanic reactions with a lower rate [22]. Few investigations have been done on studying microstructure and degree of hydration of such mixtures to prove this hypothesis.

The focus of this paper is to explore the efficacy of additional milling as a pretreatment method aimed at reactivating Natural Perlite Powder (NPP). The objective is to replace a portion of Ordinary Portland Cement (OPC) within a designed ratio for concrete mixtures. Substituting OPC with NPP might offer benefits in terms of concrete properties, yet it could potentially impact these properties negatively due to a dilution effect. Hence, it became imperative to scrutinize the effectiveness of various milling techniques and the ideal replacement ratio. To accomplish this, 14 concrete mixtures underwent evaluation for concrete compressive strength, surface electrical resistivity (SER), water penetration (WP) resistance, rapid chloride permeability (RCP), and rapid chloride migration (RCM) at three different ages (7, 28, and 91 days). It was anticipated that although the substitution of OPC might result in decreased early-age mechanical strength, the durability of the concrete would substantially improve in later stages, while retaining nearly identical mechanical strength to that of fine NPP.

2. Experimental program

2.1Materials and mixture proportions

The chemical composition and physical characteristics of OPC Type I and NPP utilized in this study are presented in Table 3.1. These specifications adhere to ASTM C150 for OPC and ASTM C618 for NPP. Additionally, a high-range water-reducing agent (HRWRA) formulated with neutral polycarboxylate ether (PCE) met the standards delineated in ASTM C494.

Natural river sand was used as a fine aggregate, having a fineness modulus of 2.95, a specific gravity of 2.56, and water absorption of 1.82%, which addressing a specification from ASTM C33. Crushed calcareous aggregates were used as the coarse aggregate, with a nominal maximum size of 19 mm, a specific gravity of 2.58, and 1.61% water absorption, according to ASTM C33. The concretes were mixed and cured using potable treated tap water sourced from the municipal pipeline.

Table 3.1. Chemical and physical properties of cementitious materials

Chemical Composition[a] (%)	**OPC**	**NPP**
CaO	65.3	1.12
SiO_2	20.8	70.28
Al_2O_3	4.3	12.97
Fe_2O_3	2.2	1.46
MgO	2.17	0.41
K_2O	0.63	4.51
Na_2O	0.36	2.96
Loss in ignition (%)	0.91	3.02
Physical properties		
Specific gravity	3.15	2.87
Fineness (cm^2/gr)	2800	-

[a] Chemical composition determined based on ASTM C114

In the laboratory, a simple rotational ball mill that was 450 mm in length and 420 mm in diameter was used for the grinding operation, as it is a commonly used method in the cement industry. The rotation speed was selected as 30 rpm. The charge of the mill contained 96-kgspherical balls and cylpebs in total. Before milling, the natural perlite underwent a drying process in a 105°C oven for a day. Seven kilograms of the dried material were ground in each cycle to ensure the uniform grinding of the material by the procedure's conclusion. Periodically, a 100-gr sample was taken throughout the milling procedure to measure the specific gravity and Blaine fineness, and the procedure continued until the materials reached the desired Blaine fineness (3100, 3500, and 3900 cm^2/gm) [16]. Particle size distributions of the grounded NPPs are presented in Figure 3.1

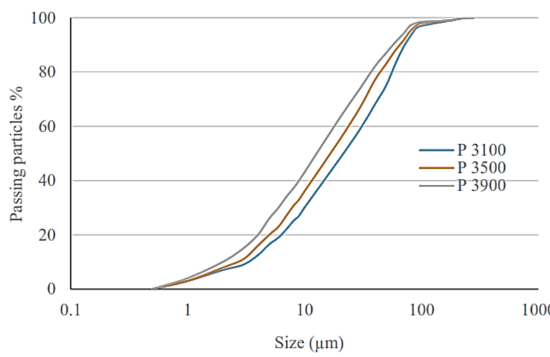

Figure 3.1. Particle-size distribution of grounded natural perlite powder.

Concrete mixture proportions were designed based on the unit volume method. No air-entraining agent used, and it was assumed that the air content for fresh concrete would be around 3%. Table 3.2 presents the mixtures in detail. Two different ratios for water per cementitious materials (W/Cm), equal to 0.32 and 0.4; the cementitious materials content, and OPC replacement ratios were considered in the design of concrete mixtures as well as three different levels of fineness for NPP. Various dosages of HRWRA were used to achieve the design slump (7.5±2.5 cm) based on the fresh properties of each mixture.

Table 3.2. Concrete mixture designs of various proportions

	Code	W/b	Cementitious Material (kg/m^3)	Perlite		Portland Cement (kg/m^3)	Aggregate	
				(%)	Blaine (cm^2/gr)		Coarse (kg/m^3)	Fine (kg/m^3)
1	G1CRL	0.32	430	0	-	430	843	946
2	G1B31P10		430	10	3100	387	841	944
3	G1B31P15		430	15	3100	365.5	840	943
4	G1B35P10		430	10	3500	387	841	944
5	G1B35P15		430	15	3500	365.5	840	943
6	G1B39P10		430	10	3900	387	841	944
7	G1B39P15		430	15	3900	365.5	840	943
8	G2CRL	0.4	380	0	-	380	844	947
9	G2B31P10		380	10	3100	342	843	946
10	G2B31P15		380	15	3100	323	842	945
11	G2B35P10		380	10	3500	342	843	946
12	G2B35P15		380	15	3500	323	842	945
13	G2B39P10		380	10	3900	342	843	946
14	G2B39P15		380	15	3900	323	842	945

[a] Super plasticizer percentage is presented by mass for the cementitious material

2.2Specimen preparation and test methods

All concrete mixtures were prepared using a laboratory pan mixer. Specimens were taken, and cured in lime-saturated water at 23±2°C by the testing age, following ASTM C192. To assess the initial workability of the mixtures, slump tests were carried out immediately after the mixing process, following the guidelines outlined in ASTM C143. For the evaluation of compressive strength, three 100-by-200-mm cylindrical specimens were arranged in two layers and utilized to measure the concrete mixtures' compressive strength at ages 7, 28, and 91 days, in accordance with ASTM C39. The reported results were the averages obtained from these tests. Surface Electrical Resistivity (SER) tests, as per AASHTO T358 [24], were performed on two 100-by-200-mm cylinders at ages 7, 28, and 91 days, and the average of these tests was reported.

A four-probe Wenner-array resistivity meter was used for this purpose, having 1.5-inch probe spacing, as illustrated in Figure 3.2 [25]. Water penetration tests were carried out at ages of 28 and 91 days, following BS EN 12390-8 [26], as there is no ASTM or AASHTO standard for this type of test. The equipment and the broken specimen after 72-hour test can be seen in Figure 3.3. The rapid chloride permeability test, the most common test used to evaluate transport properties over the past few decades, was conducted at ages of 28 and 91 days, complying with ASTM C1202 [27]. The rapid chloride migration test also was conducted at ages of 28 and 91 days, complying with AASHTO T357 [28]. The device can be seen in Figure 3.4.

Figure 3.2. Measuring SER using 4-probe Wenner-array device

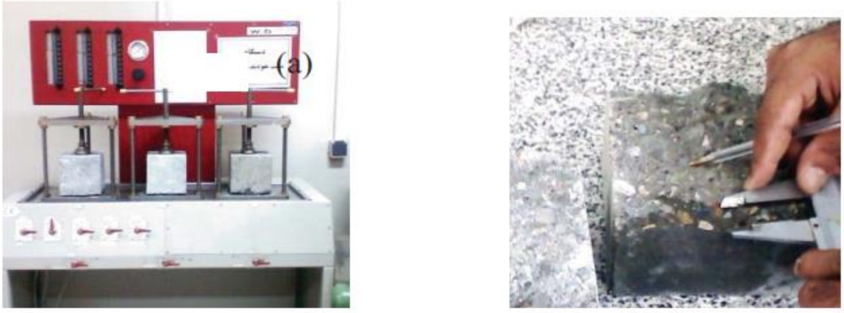

Figure 3.3. Measuring water penetration resistance: a) equipment b) test specimen

Figure 3.4. The equipment for measuring rapid chloride migration

3. Results and discussion

3.1Fresh and mechanical properties

The required dosage of HRWRA to obtain the slump of each mixture in the desired range is presented in Table 3.3. Replacing a portion of OPC with some SCMs may adversely affect the workability of concrete mixtures due to the change in the particle surface texture or specific surface area [29].

Table 3.3. Required dosage of HRWRA* to achieve designed slump

	Code	Slump (cm)	HRWRA (%)
1	G1CRL	7	0.41
2	G1B31P10	8.5	0.58
3	G1B31P15	8	0.54
4	G1B35P10	7.5	0.69
5	G1B35P15	8	0.7
6	G1B39P10	8	0.82
7	G1B39P15	7	1.04
8	G2CRL	8.5	0.24
9	G2B31P10	9	0.26
10	G2B31P15	8.5	0.31
11	G2B35P10	8	0.29
12	G2B35P15	8	0.37
13	G2B39P10	8	0.35
14	G2B39P15	7	0.44

* To the weight of the cementitious material

The results show that while the W/Cm ratio was constant, a higher dosage of HRWRA was required to compensate for the effect of increasing the fineness or replacement ratio of NPP in order to obtain almost the same workability. On the other hand, it was demonstrated that with up to a 15% replacement ratio, an increase in the fineness of NPP from 3100 to 3900 cm^2/gm did not extensively increase the HRWRA demand.

The compressive strength of concrete mixtures at the ages of 7 days, 28days, and 91 days was measured, and the results are presented in Figure 3.5. The strength decreased by increasing the W/Cm ratio and by decreasing the cementitious material content. This led to increasing the volume and size of the pores in both the cement paste and the interfacial transition zone (ITZ), which caused a decrease in the strength of the mixture [22,16,30]. Moreover, elevating the substitution rate of NPP from 10% to 15% resulted in a slight decrease in the compressive strength of the mixtures. The maximum reduction values recorded were 9%, 11%, and 13.8% at the ages of 7, 28, and 91 days, respectively. This suggests that while replacing NPP may offer certain advantages due to a nucleation effect and improved particle packing by acting as a filler in smaller voids, the predominant influence observed was the dilution effect, especially with the escalated replacement ratio.

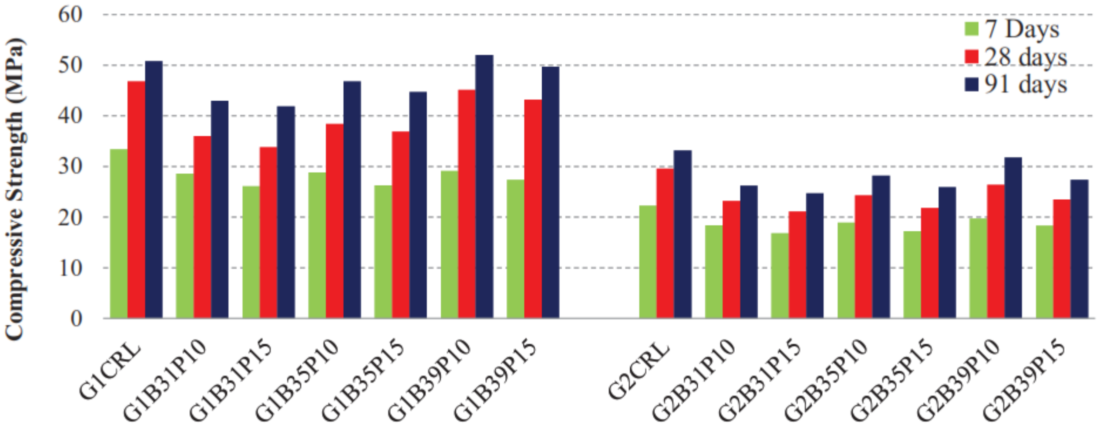

Figure 3.5. Compressive strength of Group 1 concrete mixtures

With the standard curing process persisting until the testing age, it was observed that an increase in the concrete's age correlated with higher levels of compressive strength. However, the rate of gaining strength between ages from 7 to 28 days was much higher than for ages from 28 to 91 days. This was due to a reduction in the rate of OPC hydration reactions over time. In addition, secondary pozzolanic hydration reactions after ages greater than 28 days were not that significant in generating the C-S-H gel, which makes the pore structure stronger.

On the other hand, increasing the specific surface area of NPP from 3100 to 3900 cm^2/gm increased the compressive strength at an age of 28 days by 25% for 10 % replacement and 28% for 15% replacement ratios, respectively. It illustrates that making NPP finer by further milling is an effective method to enhance the beneficial effects of NPP by increasing the specific surface area.

4.2Transport properties

Water penetration depth of designed mixtures was measured at both ages of 28 and 91 days, as shown in Figure 3.6. The depth of water penetration over a period of 72 hours under a pressure of 500 kPa serves as an indicator of both the volume of penetrable pores within the pore structure and their interconnectivity.

The results indicate that resistance against water penetration improved by decreasing the W/Cm ratio and increasing the cementitious material content. It is also demonstrated that although increasing the age is a remarkable term in declining water penetration depth, it is more significant in lower W/Cm ratios. The results showed a decrease by 56% and 28%, on average, for mixtures with 0.32 and 0.4 W/Cm ratios, respectively. Raising the replacement ratio from 10% to 15% stood out as another advantageous factor, resulting in an average reduction of water penetration depths by 11.8% and 13.5% at the ages of 28 and 91 days, respectively.

This suggests that augmenting NPP content not only enhances particle packing and fosters greater C-S-H gel formation but also disrupts pore connectivity. This disruption stands as the primary advantage of substituting cement with NPP, given that it does not enhance the mechanical properties of the concrete mixtures, as previously discussed. Grinding NPP can be an efficient method of reactivating it. The results, as shown in Figure 3.6., indicate that concrete mixtures that incorporated NPP with the highest fineness also reduced the water penetration depth up to 40%, compared to mixtures with coarser NPP. This occurred because the SCM had a higher specific surface area as well as better performance in cutting the connectivity of pores in the paste over time.

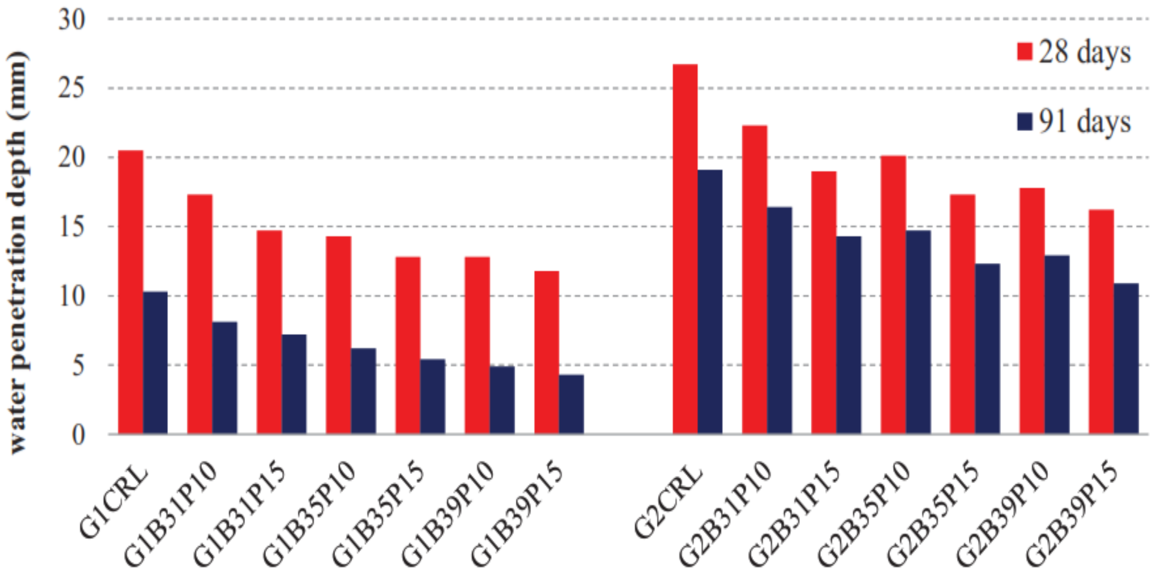

Figure 3.6. Water penetration depth of concrete mixtures

Electrical resistivity is an intrinsically important parameter in the corrosion of steel rebars embedded in concrete. It also affects the rate of corrosion [31]. The electrical resistivity of concrete demonstrates how difficult it is to move ions in the body of concrete; in other words, it can be an indication of the time required for external chloride ions to ingress in concrete and to reach the critical threshold value to significantly increase the rate of corrosion [32-34]. Results for SER of the mixtures measured at ages of 7, 28, and 91 days are shown in Figure 3.7.

These findings suggest that Surface Electrical Resistivity (SER) is influenced by both the Water-to-Cementitious Materials (W/Cm) ratio and the content of cementitious materials. Additionally, the results were impacted by the NPP replacement ratio. SER demonstrated an increase of up to 21% when the NPP replacement ratio was elevated from 10% to 15%. Essentially, the dilution effect might play a decisive role in augmenting the SER of concrete mixtures by reducing the concentration of ions released from dissolving OPC particles within the pore solution. Consequently, SER rises when a larger portion of OPC is substituted with NPP in lower values. However, if the dilution effect leads to an increase in pore size and connectivity within the cement paste, SER could decrease significantly.

The rate of cementitious hydration reactions of Ordinary Portland Cement (OPC) experienced an exponential decrease over time. However, secondary pozzolanic hydration reactions primarily took place during later stages or longer ages. Pozzolanic reactions can notably enhance the Surface Electrical Resistivity (SER) of concrete mixtures due to various contributing factors.It can make the pore structure denser and decrease the total volume of pores because it generates more C-S-H gel. It also can make the pore network more tortuous by discontinuing the connectivity of pores due to generating extra C-S-H gel. Conversely, hydroxide ions, known for their high conductivity in the pore solution [35,36], are utilized during pozzolanic reactions. It significantly decreases the conductivity of the pore solution, and increases the SER of the mixture.

As shown in Figure 3.7., the SER of control mixtures was improved by 37% due to increasing the testing age from 28 to 91 days. Nevertheless, the percentage increased up to 178% and 159% for mixtures with 0.32 and 0.4 W/Cm ratios, respectively. Moreover, the improvement was more significant in mixtures with lower W/Cm ratios, caused by the lower volume of pores and lower connectivity. Consequently, the filling of certain capillary pores led to heightened tortuosity in pore connectivity [37].

The results also indicated that increasing the specific surface area of NPP by further milling was an effective method to improve the SER of concrete mixtures. SER results for mixtures at an age of 91 days improved by 40% and 63% for concrete mixtures with 0.32 and 0.4 W/Cm ratios, respectively. A finer grade of NPP may yield superior effects in terms of filling and nucleation during the earlier stages. Meanwhile, augmenting the specific surface area could potentially facilitate enhanced secondary pozzolanic reactions, particularly at later ages, such as 91 days.

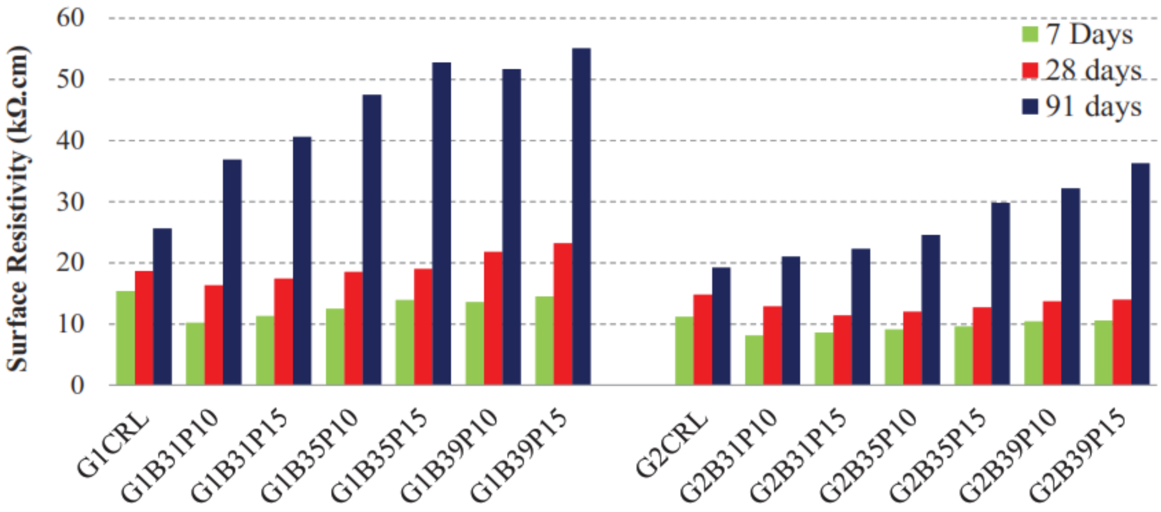

Figure 3.7. Surface electrical resistivity of Group 2 concrete mixtures

Over the past few decades, the RCP test has been the test most commonly used to evaluate the transport properties of concrete mixtures. Plenty of structures have been built using concrete mixtures that were designed based on the results of this test. Even though it is relatively expensive and time-consuming test, it still is required to conduct this test and compare the results with other mixtures. However, there were some problems and limitations in the test procedure. During the RCP test, passing a current through the specimen indicates the concrete's ability to resist against chloride ion transport under a 60V potential. The results at the ages of 28 days and 91 days are shown in Figure 3.8.

Results indicated that increasing the W/Cm ratio and decreasing the cementitious material content adversely affected the RCP of the mixtures. This is the same trend similar to the SER test, and is remarkable increasing with the age being tested; it indicates that significant additional pozzolanic reactions occurred between the ages of 28 days and 91 days. In addition, a slight increase in the NPP replacement ratio from 10% to 15% had a positive effect on RCP test results due to the dilution effect on the ions present in the pore solution of the mixtures.

As anticipated, concrete mixtures integrating finer Natural Perlite Powder (NPP) demonstrated enhanced performance during the Rapid Chloride Permeability (RCP) test. Increasing the specific surface area made the SCM more reactive as well as improving both the filler and nucleation effect. The findings demonstrated that there was a decrease of 54% and 48% in Rapid Chloride Permeability (RCP) when the fineness was increased for concretes with W/Cm ratios of 0.3 and 0.4, respectively.

The RCM test measures the chloride penetration depth under a 0 to 60 DC voltage potential. It has some benefits compared to RCP test, such as being able to conduct the test under room temperature and measuring chloride ion penetration only, and not the conductivity of all the ions in the pore solution. Therefore, results from the RCM test results are more reliable that other methods, especially for concrete mixtures incorporating SCMs.

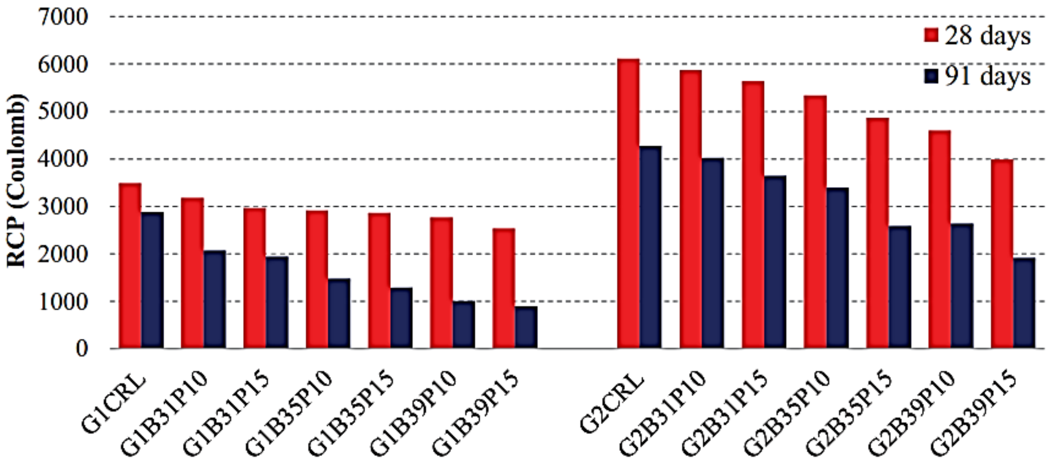

Figure 3.8. RCP test results for concrete mixtures

As shown in Figure 3.9., chloride migration coefficients increased for concrete mixtures with higher W/Cm ratios and lower contents of cementitious material. Elevating the NPP replacement ratio resulted in a decrease in the chloride migration coefficient, primarily attributed to the beneficial effects of dilution. In addition, conducting the test at longer ages led to measuring substantially less chloride migration coefficients for the concrete mixtures because the further pozzolanic hydration reactions made the pore network smaller and more tortuous.

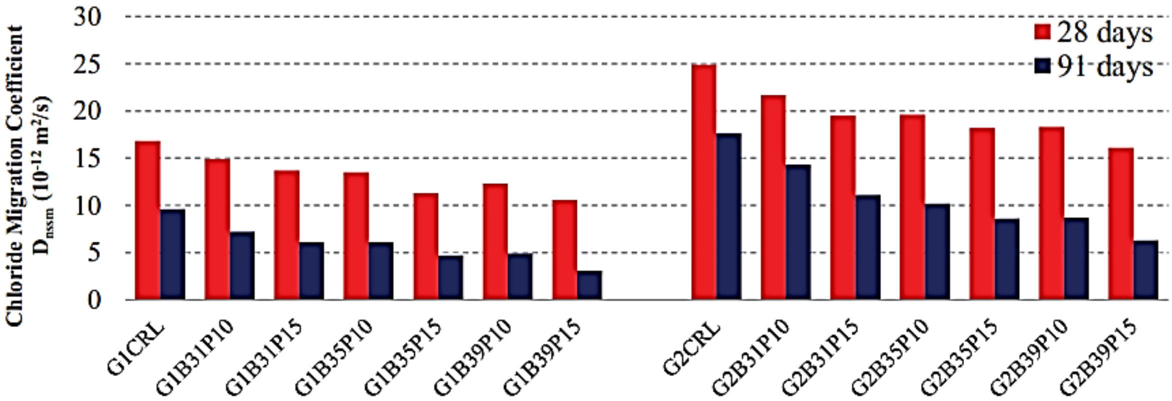

Figure 3.9. RCM test results of concrete mixtures

Although using NPP as an SCM, with even the lowest specific surface area, can improve the chloride migration coefficient, the results demonstrated that further Milling proved to be an effective pretreatment method, contributing to the improvement of the chloride migration coefficient within the concrete mixtures.

The results revealed that using the finest NPP instead of coarse NPP may decrease the chloride migration coefficient to the half the value. These outcomes parallel those observed in the Surface Electrical Resistivity (SER) and Rapid Chloride Permeability (RCP) tests, affirming the advantages of employing NPP possessing higher specific surface areas. This supports the notion of encouraging hydration reactions and augmenting the impermeability properties within concrete mixtures [38].

5. Conclusion

The main conclusions based on the investigations presented in this paper are as follows.

- Partially replacing OPC with NPP slightly increases the water demand in fresh concrete, a concern that can be addressed by increasing the HRWRA dosage. Moreover, the increased fineness of NPP requires a higher HRWRA dosage to achieve equivalent results.

- Further milling proves to be an effective technique to enhance the performance of NPP in concrete mixtures, particularly regarding compressive strength. Concrete mixes containing 10% NPP with the finest particles display minimal variation in strength even after 91 days.
- Replacing OPC with NPP significantly improves the transport properties of mixtures due to several factors: the filler effect, heterogeneous nucleation effect, pozzolanic effect, and dilution effect.
- Notably, there is a considerable enhancement in the transport properties of the mixtures between the ages of 28 and 91 days, indicating that the pozzolanic effect of NPP becomes more pronounced at longer ages, especially with increased specific surface area.
- Different test methods proposed for evaluating the mass transport properties of concrete corroborate each other, affirming their reliability and consistency in assessing concrete properties.
- The strongest correlations between SER and other test results follow a power function. However, the results of other tests (including WP, RCP, and RCM tests) have linear relationships with each other.

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