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Mechanical and durability performance of ternary blended calcined clay and pulverized granite mortar composites

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ABSTRACT

Partially replacing cement with pozzolans, apart from the enormous technical benefits, has been reported as an effective approach to reducing greenhouse gases emanating from cement production. Calcined clay and pulverised granite, in recent years, have been experimented individually for their use as filler or pozzolan in concrete to improve workability and compressive strength. This research includes study of the synergistic characteristics of these two mineral admixtures and their influence on the mechanical and durability properties of mortar. Cement was partially substituted with 5–20% by weight of the composite material to form a ternary blended cement composite. The compressive strengths of the ternary blended cements were evaluated at 3, 7, 28 and 90 days. Durability properties such as alkali silica reactivity (ASR), rapid chloride permeability test (RCPT) and performance of the blended cements in aggressive media have been discussed. Test results indicated that, blended cements containing 5% and 10% of the pozzolan recorded similar compressive strengths at 28 days and outperformed it by 2.4% and 0.6% respectively at 90 days. Beyond 10% replacement, compressive strengths declined. The ternary blended cements were found to be highly reactive, potentially causing ASR in concrete than the reference cement.

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KEYWORDS

Pulverized granite; calcined clay; porosity; ASR; electrical resistivity: durability

1. Introduction

One of the most common challenges that has emerged in developing nations over the past few years is the gap between the rapid growth of the urban population and the availability of infrastructure for waste disposal. The difficulties of ineffective waste management strategies having an impact on the degrading ecosystem of fast-growing cities are making this disparity worse every day. Additionally, one of the main trends in the recent expansion of the building and construction sector is the switch to more economical, energy-efficient production methods while retaining the high quality of building materials and buildings. Industrial waste is presently utilised far more frequently in building construction than it ever has been due to the steep rise in energy prices and the need to diversify the supply of raw materials.

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The utilisation of industrial by-products has consistently become an environmentalfriendly approach of disposing large quantities of waste materials that have the potential of causing harm to the environment. The current world production of cement, which is approximately 4 billion tons/year [1], continue to grow each year due to global population growth and the corresponding need for infrastructure. A substantial part of this increase in cement demand will be met using supplementary cementitious materials (SCM) such as calcined clay, metakaolin, ground granulated blast furnace slag (GGBS), fly ash, silica fume, granite dust, etc. [2]. SCMs have been used more frequently by the cement and concrete industries in recent years [3]. This rate of consumption is expected to rise because of the need to change the properties of cements and enhance the quality of cements and cementitious materials [4]. When industrial by-products are employed as a partial replacement for the energy-intensive Portland cement, significant energy and cost savings can be achieved. Mineral admixtures are known to significantly improve the workability and durability of concrete [5].

One of such supplementary materials or fillers is pulverised granite, also known as granite dust. Granite dust, a major by-product in granite stone quarrying, not being used for any application other than filling-up low-lying areas, has been identified as a partial replacement material for cement in concrete [6]. In most regions, there is a significant amount of waste produced in the form of dust due to the high production and consumption of granite. Environmental hazards result from this. In this sector, air and water pollution are the most serious and immediate issues. The action of the wind causes the waste to be deposited on the roads and dispersed everywhere. Additionally, over time, the soil's alkalinity. An alternate use of this waste granite is therefore highly recommended.

As a result, studies [6–11] have been conducted by researchers to find the suitability of granite quarry dust in conventional concrete. According to Venkata et al. [12], 25% of cement can be replaced with granite dust in concrete to achieve desirable results. Ghorbani et al. [13] replaced up to 20% of cement with granite fines and reported an improved strength up to 10% replacement. Beyond 10%, compressive strength declined. Elmoati and Mohamed [14] also replaced up to 15% of cement with granite dust. Results from their experiment showed an improvement in compressive and tensile strengths at most replacement dosages. Concrete containing granite dust exhibited an increase in corrosion cracking time as compared to the reference concrete. Prokopski et al. [9] also studied the effect of granite dust on concrete mixes. The results indicated that, porosity, density and workability were improved by incorporating granite dust.

The incorporation of two or more mineral admixtures in ternary or quaternary blends have been investigated by several researchers [15–24] and reported to affect concrete properties such as porosity, density, strength and durability. Palod et al. [22] studied the synergistic effect of ternary blended steel slag and blast furnace slag and reported a significant improvement in strength, electrical resistivity and ultrasonic pulse velocity. The durability studies conducted by Chandru et al. [25] on ternary blended crushed stone and induction furnace slag in a self-compacting concrete discovered a reduction in water absorption, porosity, permeability and chloride penetration. As replacement increased, these properties decreased to improve overall durability of the concrete.

The granite waste generated by the stone crushing industries (to produce coarse aggregates) has accumulated over the years. Only insignificant quantities have been

utilised and the rest have been dumped resulting in environmental challenges. Valorisation of granite dust is essential and has the potential to boost the economy of the granite quarrying industry, with the added benefit of preserving the environment [7-9,11].

Calcined clay, on the other hand, is a well-known and researched supplementary cementitious material. Several researchers [26–36] have reported different benefits of utilising calcined clay for mortar and concrete applications, including improved workability, compressive strength and durability properties [37]. Calcined clay and pulverised granite have been studied and their individual influence on the properties of mortar and concrete are reported in several scientific publications. However, there is no information on the combined effect of these two pozzolans in cementitious systems. This research has, therefore, studied the possible utilisation of pulverised granite and calcined clay as a composite material in blended cement mortars, focusing on its synergistic influence on the physical, mineralogical, mechanical and durability properties. The effect of this composite material on alkali silica reactivity, porosity, electrical resistivity, chloride permeability and resistance to sulphate environment have been studied. A relation between porosity and compressive strength has been discussed. The results of this study contributes to forming a roadmap for the use of these mineral admixtures in mortar and concrete, especially in regions where conventional SCM's are absent.

2. Materials and methods

2.1. Materials

Ordinary Portland cement (OPC) of Class 42.5N, produced by the Heidelberg Cement Group, and sourced from local distributors, was used as the main binder for this work. The granite and calcined clay were sourced from local suppliers. Pit sand which satisfied BS 4550: Part 6 requirements was used to prepare the mortar preparation. Pyrex glass, which is commonly used for the manufacture of laboratory glassware and known to be a reactive aggregate [34,38] was crushed and used for the ASR determination. Particle size of the pyrex glass ranged between 0.16 and 0.70 mm. The X-ray diffraction (XRD) of the calcined clay and the pulverised granite is shown in Figures 2 and 3. Figure 1 shows samples of the raw materials.



Figure 1. (a) calcined clay, (b) pulverized granite and (c) crushed pyrex glass.



Figure 2. XRD of calcined clay.



Figure 3. XRD of pulverized granite.

2.2. Methods

2.2.1. Materials preparation

The granite was crushed in a motorised jaw crusher and further pulverised by using a laboratory type ball mill to obtain the granite dust (also referred to as pulverised granite in this paper) fineness of $70 \,\mu\text{m}$. It was then uniformly mixed with the calcined clay (1:1) in a motorised mixer to form a composite material (labelled CGD). The composite powder was used to replace portions of the ordinary Portland cement in percentages of 5%, 10%,15% and 20% by weight to obtain a ternary blended cement comprising ordinary Portland, calcined clay and granite dust. Particle size distribution of the calcined clay, pulverised granite and sand are shown in Figure 4.

2.2.2. Testing methods

The X-ray fluorescence (XRF) technique (Spectro X-lab 2000 equipment), due to its accuracy, was employed to study the elemental composition of the calcined clay, granite powder and blended cement samples. Determination of relevant phases in the raw samples and hydrated pastes was carried out by a 3^{rd} generation Malvern Panalytical Empyrean equipped with multicore optics. Readings were taken from 10 to 80 2Θ degrees. The Vicat method, specified by EN 196–3 was used to determine the normal consistency and setting times of all the cement samples. Soundness was determined using the Le-Chatelier's apparatus as prescribed by EN 196–3. Mortar cubes were prepared according to the methods specified in EN 196–1 compressive strength determined after 3, 7, 28 and 90 days.

A thermometric TAM (thermal activity monitor) air conduction calorimeter was used to track the evolution of the hydration heat rate. Samples for this test were prepared by mixing distilled water with 5 g of each blended cement sample and placed in the calorimeter at a working temperature of 20°C. The attached computer automatically generated heat of hydration data continuously for up to 3 days.



Figure 4. Particle size distribution of pulverized granite, calcined clay and sand.

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Porosity was determined using Eq. (1) [39,40], where n is the number of voids, V_T is the total volume, V_C is the change in volume of the test sample because of the existence of voids.

$$n = \frac{V_T - V_C}{V_T} \tag{1}$$

ASR was evaluated according to ASTM C 1260. In this test, $25 \text{ mm} \times 25 \text{ mm} \times 25$ mm mortar bars were prepared using the blended cement composites with a water to binder ratio of 0.47 and binder to aggregate ratio of 1:3. To know the effect of the added pozzolan, the samples were cured for 27 days in water at 20°C, after the initial length data were recorded on the first day. The blended cement mortar bars were subsequently placed in a water bath, having a temperature of 80°C for 24 h after which readings were taken with a length comparator as specified by ASTM C 490. The samples were then soaked in 1N sodium hydroxide solution and readings were taken at specific intervals between 7 and 35 days.

For the aggressive media test, samples were cured for 28 days in water and then immersed in seawater and 5%-Na₂SO₄ solutions. It was allowed to stand for 90 days, and the effect of the two media on their respective compressive strength determined.

With reference to the ASTM C 1202 RCPT procedure, electrical current was made to flow through sliced blended cement mortar samples for a period of 6 h. Two slices of the same sample were placed in sodium chloride and sodium hydroxide solutions, respectively. At the ends of the samples, a potential difference of 60 V was preserved.

The bulk electrical resistivity test method prescribed by ASTM C1760–12 (two-point uniaxial method) was used to determine the extent to which the added composite pozzolan can resist electrical current in the mortar. The set-up for electrical resistivity and a flow diagram of the methodology are shown in Figures 5 and 6.



Figure 5. Arrangement for electrical resistivity test.



Figure 6. Flow diagram of methodology.

3. Results and discussion

3.1. Chemical composition

The chemical compositions of the cement, pulverised granite and the blended cements, as determined by XRF are presented in Table 1. The chemical compositions of the samples were generally found within acceptable limits [41,42]. The pulverised granite and calcined clay contain 71.22% and 62.77% SiO₂ respectively, which exceed the 25% minimum requirement for pozzolans according to ASTM C618. The values of SiO₂ + Al₂O₃ + Fe₂O₃ for both the pulverised granite and calcined clay are also more than the minimum 70%, as required by ASTM C618. The Na₂O eq (Na₂Oeq = N₂O +0.682 K₂O) for all samples were higher than the maximum permissible limit of 0.6% per ASTM C150 and suggest that the blended cement samples could lead to alkali silica reactivity with reactive aggregates.

3.2. Normal consistencies, setting times and soundness

Setting time is the process by which cement loses its plasticity and becomes dense at the onset of hydration. It takes place in two stages, thus, initial and final setting time. Normal consistency is the amount of water needed to form a workable paste [43]. The setting times and normal consistencies are presented in Table 2. OPC recorded the least water demand of 29.1%. This, however, increased by 6.5% when it was replaced with 5% of the

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Composition, %	SiO ₂	AI_2O_3	Fe_2O_3	CaO	Na ₂ O	K ₂ O	N ₂ O eq	SO_3
OPC	26.06	5.8	2.1	57.1	0.6	1.99	1.96	6.27
Pulverized granite	71.22	19.84	2.75	3.56	5.45	3.5		0.18
Calcined clay	62.77	18.71	11.68	0.65	0.03	2.12		0.19
5%CGD	30.32	6.69	2.07	52.69	0.4	2.14	1.87	6.39
10%CGD	31.36	6.98	2.06	51.3	0.97	2.14	2.44	6.37
15%CGD	31.78	6.97	1.96	50.58	1.01	2.48	2.71	5.1
20%CGD	38.28	8.84	2.09	47.08	1.58	2.39	3.22	6.3

Table 1. Chemical composition of OPC, granite dust and calcined clay.

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Sample	OPC	5%CGD	10%CGD	15%CGD	20%CGD
Normal consistency, %	29.1	31	31.6	32.4	33.0
Initial set, min	145	154	163	170	174
Final set, min	242	246	256	260	268
Soundness, mm	1.02	0.85	1.01	0.92	0.64

Table 2. Water demand, setting times and soundness.

composite material. The normal consistency continuously increased as percentage replacement increased from 5% to 20%. This is due to the presence of calcined clay in the composite cement, which requires appreciable amount of water to form a workable paste [44]. It is well known that too much water reduces the mechanical strength of mortar and increases the likelihood of failure due to shrinkage. As a result, the mortar may become too fluid to deal with and the mortar-masonry interface may become less adherent, reducing bond strength [35].

Similarly, both initial and final setting times increased as the composite material content increased. This increase is attributed to the calcined clay content which requires more water to form a workable paste. However, setting times and water demand were all within acceptable limits per EN 197–1. Similar results were reported by Li et al. [45] when Portland cement was replaced with metakaolin up to 20%. Normal consistency and setting time increased with increasing pozzolan content in the mix. This is consistent with results obtained in binary and ternary blended cement systems containing other pozzolans [45–48].

Cement is said to be sound if it maintains its volume after hardening. Excessive expansion of cement is mostly due to the amount of free lime, MgO and SO_3 present in the cement [49]. The test for soundness recorded for all test samples was below the maximum limit of 10 mm specified by EN 197–1 and in line with other ternary blended pastes [11,46,50]. This could be attributed to the low amounts of MgO and SO_3 in the blended cement [51,52]. Compared to reference cement, the composite cement was generally found to exhibit less expansion.

3.3. Mineralogical analysis

XRD patterns of 28-days hydrated reference cement and the 20% calcined clay-pulverised granite blended cement paste are shown in Figure 7. The XRD patterns, as identified by the ICDD database, reveal the presence of Portlandite (shown as CH) in both the reference and blended cement pastes. The blended cement paste was observed to have relatively shorter peaks after hydrating for 28 days which could be attributed to the reaction of the pozzolan with portlandite to produce further cementitious products such as calcium silicate hydrates. Furthermore, the reduction in portlandite formation could be due to the decrease in cement content owing to the composite pozzolan replacement [21,31,53]. Previous study on the effect of ternary blended rice husk ash and nano silica on mineralogical properties of cement showed a similar reduction in portlandite to the continuously decreased as curing age increased. This was attributed to the continuous pozzolanic reaction and the synergistic effect of the nano-particles of the pozzolans involved [54,55].



Figure 7. XRD of hydrated samples.

3.4. Heat of hydration

Heat generated as a result of the exothermic nature of cement hydration could have deleterious effect on concrete structures such as thermal cracking and eventual strength loss [56]. One of the major roles of SCM's in cementitious systems is use up this heat in a secondary reaction with the constituents of cement [57]. The heat flow of the reference cement and blended cement pastes in terms of the time of hydration are shown in Figure 8. Evidently, the inclusion of the pozzolanic material influenced the calorimetric characteristics of the blended cement pastes. It is observed that, increasing pozzolan content in the mix increased exothermic peak values and reduced their corresponding times. This suggests that the presence of the mineral admixture is likely to enhance the degree of hydration and speed up the hydration reaction [27]. Again, a reaction between the Al_2O_3 and SiO_2 present in the pozzolan could interact with portlandite from the cement to produce heat and consequently speed up the hydration process [58]. As the pozzolan content increased, this effect becomes more noticeable. Again, the reference cement peak is seen to exhibit a peak bulge which usually indicates the conversion of afwillite to monosulphate [58,59]. The presence of the pozzolan in the paste appears to cause a disappearance of this peak bulge. This could be attributed to the reactivity of the pozzolan [60]. This is consistent with earlier research on the hydration of ternary blended cement containing varying pozzolan content [58]. Pastes containing 5%-20% metakaolin and limestone powder recorded greater exothermic peak values with shortened corresponding times of peak, than that of the control. This is most likely to accelerate pozzolanic reaction and degree of hydration [48,56,59].

3.5. Compressive strength

The 3, 7, 28 and 90 days compressive strength test results of the reference and blended cement samples with varying pozzolan contents are seen in Figure 9. The reference



Figure 8. Rates of heat of hydration of ternary blended cement pastes.

cement, after curing for 3 and 7 days, obtained compressive strengths of 19.4 MPa and 25.7 MPa, respectively. The addition of the pozzolan, however, caused a reduction in strength and as the pozzolan content increased, compressive strength also decreased. However, at 28 days, cement containing 5% and 10% recorded strengths comparable to the reference cement. Cement replaced with 15% and 20% of the pozzolan obtained strengths which are about 12.7% and 23.9% lower than the reference cement, respectively. When the curing period was extended to 90 days, compressive strengths of the blended cement containing 5% and 10% outperformed the reference cement by 2.4% and 0.6%, respectively. This is due to the slow reactive nature of pozzolans, especially at early ages which significantly improves at later ages [35,61]. Several reports [26,27,29,53,62] confirm the reduction of compressive strength of blended cement mortars at early ages but greatly improves at 28 days and beyond. However, 28 days results obtained from this study could not attain the expected strength gain. This could be due to the relatively larger particle size of the pulverised granite, leading to slow reactivity, even at 28 days [61].

3.6. Porosity

Scatter plots shown in Figures 10 and 11 are plotted to show the relationship between porosity and the pozzolan content and porosity and compressive strength, respectively. It is seen from Figure 10 that increase in pozzolan content caused a reduction in porosity. The goodness-of-fit between pozzolan content and porosity, however, was much less. This is an indication that some other characteristics of mortar pore structure are also likely to influence porosity apart from the pozzolan content [19,63]. Similarly, as shown in Figure 11, there was



Figure 9. Compressive strength of ternary blended cement mortars.



Figure 10. Relationship between pozzolan content and porosity.

a reduction in compressive strength as porosity increased. A power law ($R^2 = 0.9035$) is shown in Figure 11 to express this relationship. This well agrees with results reported by Jittin and Bahurudeen [64], Rukzon and Chindaprasirt [19] and Weiting et al. [65]. In all cases, porosity decreased with increasing pozzolan content.



Figure 11. Relationship between porosity and compressive strength.

3.7. ASR

Expansion of the mortar bars due to ASR is shown in Figure 12. Generally, expansion increased as curing age increased for all composite material replacements [38,66]. Expansion of mortar bars consistently increased to a maximum of 0.55% at 35 days with a pozzolan content of 15%. Reference bars also expanded rapidly as curing age increased up to a maximum of 0.35% at 35 days. Expansion of the mortar bar samples was



Figure 12. Expansion of ternary blended cement mortars due to alkali silica reaction.

all higher than the maximum value of 0.1% specified in ASTM C 1260. The high expansions could be linked to the high Na₂O and K₂O contents of the cement sample as shown in Table 1 [34,36]. Contrary to results obtained in the study, the addition of 25% fly ash in concrete effectively mitigated ASR as compared to the reference cement paste [67]. Other researchers [38,66,68] have reported the effectiveness of pozzolans in mitigating ASR in concrete.

3.8. RCPT

Figure 13 presents the findings for chloride permeability of the blended cement mortars at ages 3, 7, 28 and 90 days. It is evaluated in the form of electric charge (in coulombs) passing through the mortar samples. The charge passing through all samples is observed to decrease with increasing curing age and pozzolan replacement. The results demonstrate that, compared to the reference cement, incorporation of the pozzolan greatly improves chloride penetration resistance. At 90 days, the amount of electric charge passing through the reference cement decreased by 9.1% and 26.8% when 20% of the OPC was replaced with the pozzolan. This improvement in chloride permeability could be attributed to the reaction between the pozzolan and calcium hydroxide from the cement and the low electrical conductivity of blended cement mortars and concrete [69]. This is in agreement with earlier results reported by Garg et al. [70] in studying the effect of ternary blended nano metakaolin and fly ash. The most impressive resistance to chloride permeability was observed in pastes containing 10% metakaolin and 15% fly ash, which greatly influenced strength development.

3.9. Electrical resistivity test

One technique that has been used to assess the hydration process of cementitious systems over the years is electrical resistivity measurements [71]. Generally, the electrical resistivity of blended cements depends on volume fraction and the resistivity of the phases present [72]. In this work, electrical resistivity



Figure 13. Rapid chloride ions permeability of ternary blended mortars.

measurements were taken for all ternary blended cement batches after curing for 3, 7, 28 and 90 days. Figure 14 presents the effect of varying replacement levels on electrical resistivity. Electrical resistivity was seen to increase with increasing curing age. This is due to the formation of calcium silicate hydrates which influences the cementitious matrix, as curing period increased [73]. Samples containing 5%, 10%, 15% and 20% obtained higher electrical resistance values as compared to the control sample. It is observed that, all blended cement samples, irrespective of replacement level, obtained similar electrical resistivity. During the hydration process, SiO₂ from the pozzolan reacts with Ca(OH)₂ to form extra C-S-H, leading to a reduction in pore sizes and consequently decreasing the ionic strength and improved electrical resistance [70]. Some scientific reports [54,71,74] have demonstrated similar trends in the electrical resistivity properties of cements containing binary, ternary and quarternary blended composites.

3.10. Aggressive media test

Many concrete structures are at risk from sulphate attack, a serious durability issue for cement-based materials. Ternary blended mortar samples were stored in ordinary water, seawater and 5%-Na₂SO₄ solution and the results shown in Figure 15. Ninety-day compressive strengths of samples cured in 5%-Na₂SO₄ and seawater are compared to strengths of mortar cubes stored in water. The reference cement obtained 22.5% and 24.6% reductions in strength when stored in seawater and Na₂SO₄ solution respectively whereas the minimum reduction in strength for the blended cements was 9.8% at 5% replacement. Results of



Blended cement mortar

Figure 14. Electrical resistivity of ternary blended mortar.



Figure 15. Performance of ternary blended cement samples in normal and aggressive media for 90 days.

durability studies in aggressive media suggest that the ternary blended cement samples were more stable in aggressive media as compared to the reference cement. Samples stored in 5%-Na₂SO₄ exhibited reductions in strength for all mixed than those exposed to seawater at all ages. Generally, blended cement mortars were observed to offer a better resistance to sulphate and chloride attack as compared to the reference cement mortars. This is attributed to improved porosity and particle size distribution of the blended cement samples [74–76]. The presence of pozzolans in concrete has been known to improve sulphate and chloride resistance. This is mainly due to reduced porosity, portlandite consumption and pore refinement [76], [77].

4. Conclusion

In this study, the behaviour of ternary blended mortar containing a blend of calcined clay and pulverised granite is investigated. The composite material was blended with ordinary Portland cement in proportions of 5%, 10%, 15% and 20%. The influence of the composite material on the mechanical and durability properties on mortar is investigated and the following conclusions presented:

Blended cements containing 5% and 10% of the pozzolan recorded similar compressive strengths at 28 days and outperformed it by 2.4% and 0.6% respectively at 90 days. Beyond 10% replacement, compressive strengths declined.

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- (2) Results of durability studies in aggressive media suggest that the cement samples were stable in aggressive media up to 90 days with the composite material appearing to stabilise the cement.
- (3) The ternary blended cement was found to be highly reactive and had a high potential of causing ASR in concrete than the reference cement. This could be due to the reactive nature of the granite used for this study. This also contributed to its high Na₂O equivalence.
- (4) Increase in the pozzolan content caused a reduction in porosity. The goodness-offit between pozzolan content and porosity, however, was much less. This is an indication that some other characteristics of mortar pore structure are also likely to influence porosity apart from the pozzolan content. Similarly, a correlation between compressive strength and porosity showed a reduction in compressive strength as porosity increased.
- (5) From the RCPT studies, the charge passing through all blended cement samples was observed to decrease with increasing curing age and pozzolan replacements. Compared to the reference cement, incorporation of the pozzolan greatly improves chloride penetration resistance. At 90 days, the amount of electric charge passing through the reference cement decreased by 9.1% and 26.8% when 20% of the OPC was replaced with the pozzolan.
- (6) Electrical resistivity was seen to increase with increasing curing age. This could be due to the formation of calcium silicate hydrates which influences the cementitious matrix, as curing period increased. All blended cement samples, irrespective of replacement level, obtained similar electrical resistivity.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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