DURABILITY OF NEW RECYCLED GRANITE QUARRY DUST-BEARING CEMENTS

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Abstract

Imperative to the design of new cements that bear different types of waste as additions is a parallel study of the mechanical strength and durability of the new materials to ensure their performance will be satisfactory throughout their service life. This study explored the effect of adding 10 % or 20 % granite quarry dust to cement on properties such as transport (total and capillary water absorption and electrical resistivity), dimensional stability (drying shrinkage and expansion), the alkali-silica reaction, heat of hydration and colour. No alkali-silica reaction was observed in the new materials and expansion and contraction were less intense than in conventional cement. The water absorption and capillary absorption coefficients rose less in the additioned cements than the replacement ratio, whilst their higher resistivity values afforded greater corrosion protection than found in the reference. The inclusion of this waste also prompted a rise in lightness and a decline in peak heat of hydration. The multivariate analysis of variance (MANOVA) conducted showed that the factors time and replacement ratio affected the properties significantly, whereas the combined effect of the two was statistically significant or otherwise depending on the property analysed. The findings showed that the partial replacement of cement with quarry dust is not detrimental to product durability and the recycled material qualifies as a strength class 42.5, type II/A. Additionally, the binders bearing 20 % granite quarry dust meet the requirements to qualify as low heat cements (CEM II/A LH).

Keywords: *transport, dimensional stability, heat of hydration, granite quarry dust, recycled cements*

1. INTRODUCTION

Of the 4.6 billion tonnes of cement produced by the cement industry in 2016, 47 % was used in residential construction, 32 % in commercial construction and 21 % in civil works [1]. Those figures are indicative of the vast amounts of natural resources consumed by the industry and of its enormous potential to reduce that consumption by reusing waste, either generated in construction per se or in other industries.

In recent years the cement industry has made a huge effort to develop alternative inputs, known as supplementary cementitious materials (SCMs), in the design of new cements. Their use would lower energy consumption and the GHG emissions intrinsic to cement manufacture. Much research has been conducted the world over to analyse the viability of valorising waste, primarily from the fired clay, agroforestry, steelmaking and ornamental stone industries.

Granite quarrying is estimated to account for over 30 % of the total volume of ornamental stone waste. The vast amounts (125 Mt in 2012) [2] involved are presently deposited in land fills, with the concomitant damage to the environment and risk to human health, both causes for concern in modern societies.

With a view to solving these problems, in recent years researchers have tested the viability of valorising (coarse, fine and dust) quarry waste as a raw material in new eco-efficient materials. The literature identifies three major applications for valorising industrial waste: i) fine or coarse aggregates for sustainable concretes [2-5] or mortars [6]; ii) partial cement replacements in the design of new concretes (up to 30 %) [7, 8] or cements (up to 10 %) [9]; and iii) other uses, such as geopolymers [10] or roof tiles [11]. Much of the scant research conducted worldwide to date on the valorisation of such waste in cement has focused primarily on mechanical strength or chloride penetration-related durability.

That has left a large scientific-technical gap in the understanding of other properties associated with material durability, such as: i) indirect indicators of transport intensity (water absorption, capillarity and electrical resistivity) [12]; ii) volume change resulting from drying shrinkage or early-age swelling that may induce micro- or macrocracking, compromising service life [13]; iii) the alkali-silica reaction due to the simultaneous presence of moisture, reactive silica and cement-borne alkalis, forming silica gel and subsequently map-like cracking [14]; and iv) the heat released during hydration, possibly generating steep thermal gradients that may prompt cracking, a major concern in structures requiring large volumes of concrete such as dams [15].

This study consequently sought to further knowledge of the effect of partially (10 % or 20 %) replacing cement with granite quarry dust on the physical and mechanical characteristics, durability and colour of type II/A cements. The variables studied included 28 d mortar mechanical performance and pore size distribution, along with transport (water absorption, capillarity and resistivity), drying shrinkage and swelling, the alkali-silica reactivity of the new pozzolan and the calorimetric properties and colour of these new binary cements. A multivariate analysis of variance (MANOVA) was conducted to determine the effect of age and replacement ratio on mortar performance and bivariate correlations between the variables studied were established.

2. MATERIALS AND EXPERIMENTAL

2.1. Materials

The granite quarry dust (AF) analysed was furnished by an ornamental stone quarry at Quintana de la Serena in the Spanish province of Badajoz (region of Extremadura). The waste generated by the quarry is presently carried directly to uncontrolled spoil banks, a practice with obvious environmental implications (Figure 1). Representative samples collected by random selection from three heights on the on-site stockpile were subsequently homogenised in the laboratory. After selection, the waste was dried and characterised chemically, physically and mineralogically.

Figure 1. Stockpiled granite quarry dust

The X-ray fluorescence analysis of this dust performed in an earlier study [16] showed that it was acidic ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 85.0$ wt%) with 1.5 wt% to 4.0 wt% of Na_2O and K_2O , as well as other minority oxides. Tectosilicates, phyllosilicates and hematites prevailed in its mineralogical composition [16].

CEN-standardised, European standard EN 196-1 [17]-compliant sand with a particle size ranging from 2.0 mm to 0.075 mm was used.

A non-reactive limestone (≈ 97 % CaCO_3) sand with a particle size of 4.0 mm to 0.063 mm was also used to analyse the alkali-silica reactivity of the granite quarry dust included as a pozzolan in the design of type II/A cements.

The EN 197-1 [18]-compliant CEM I 42.5 R ordinary portland cement (OPC) used was furnished by Lafarge, a Spanish cement manufacturer sited in the province of Toledo.

2.2. Blends

The blended cements were prepared in a high-speed powder mixer to guarantee homogeneity. Blend proportions were computed by weight, with OPC/AF ratios of 100/0 (OPC), 90/10 (OPC+10%AF) and 80/20 (OPC+20%AF). These replacement ratios were adopted on the grounds of the percentages allowed for type II/A (6 % to 20 %) cements in European standard EN 197-1 [18].

The pore size distribution of the cements shown in Figure 2 was obtained by measuring N₂ absorption isotherms on a Micrometrics ASAP 2000 analyser. All the cements were observed to have pore diameters ranging from 0.7 nm to 190 nm. The OPC had a small shoulder at 2.0 nm and cements OPC+10%AF and OPC+20%AF had two each, at 1.6 nm and 2.0 nm. The new shoulder at 1.6 nm would be attributed to the addition, as observed by Medina et al. [19].

Figure 2. Pore size distribution for cements OPC, OPC+10%AF and OPC+20%AF

Including the addition induced a minor decline in the BET specific surface of the cement, from the 1.37 m²/g in OPC to 1.34 m²/g in OPC+10%AF and 1.32 m²/g in OPC+20%AF. This property would not, then, affect the performance of the new binary cements designed.

2.3. Standard methods and mortar manufacture

Table 1 lists the physical and mechanical properties and durability variables assessed for the new cements, as well as the methodology and specimen sizes used.

Table 1. Physical, mechanical and durability variables studied

With the exception of the mortars used to analyse the possible alkali-silica reactivity of the new addition, all the mortars were standardised and prepared with a sand/cement ratio of 3. The mortars used to test for the alkali-silica reaction, in contrast, had a sand/cement ratio of 2.26.

The mortars were prepared as specified in standard EN 196-1 [17].

Mortar colour was measured on powdered blends of cement and waste pressed into pellets to a density of 1.8 g/cm³.

2.4. Characterisation

The instrumental techniques used to characterise the microstructure of the new cements and assess the heat released, heat of hydration and colour are described below. The pore size distribution of the 28 d mortars was determined on a Micromeritics Autopore IV 9500 mercury

porosimeter able to operate at pressures of up to 33 000 psi (227.5 MPa) and measure pore diameters of 0.006–175 μm . This trial was conducted to ASTM standard D 4404 [20].

The microstructural damage induced by immersing the mortars in an NaOH solution for the alkali-silicate reactivity test was assessed with backscattering electron microscopy (BSE). Samples removed from the centre of the specimens were embedded in epoxy resin, epoxy-coated, precision-sawed and carefully polished for study under a Hitachi S4800 scanning electron microscope coupled to a Bruker Nano XFlash 5030 silicon drift EDX detector.

The colour parameters were quantified on a Minolta CM-2500d portable spectrophotometer for positioning on the $L^* a^* b^*$ colour space (where L^* is lightness and a^* and b^* the chromaticity coordinates) defined by the International Commission on Illumination (CIE) (Commission internationale de l'éclairage) in 1976 [21].

2.5. Statistical analysis

A MANOVA was conducted to assess the findings recorded for the mortars studied (OPC, OPC+10%AF and OPC+20%AF), testing for the significance of the effect of age and replacement ratio, as well as the interaction between them, on macroscopic properties.

As the significance level defined in the statistical study was $\alpha=0.05$, factors with a lower p-value were determined to have a significant effect on the results obtained.

Pearson's coefficient was calculated to determine the strength of the bivariate linear relationships at 5 % significance.

IBM SPSS Statistics 2.0 software, version 22, was used for the statistical analyses.

3. RESULTS AND DISCUSSION

3.1. Bulk density

The bulk density for the 28 d mortars is given in Table 2. The data showed that including granite quarry dust lowered density relative to the reference by 0.08 % with a 10 %, and by 0.16 % with a 20 % replacement ratio.

Table 2. Bulk density and mechanical performance in 28 d mortars

That finding was directly related to the effect of including the new addition on total porosity (see Table 3), which was linearly related to density further to the equation $y=-3.96x+2334$ ($R^2=0.997$), where y is density and x total porosity. The trend observed was consistent with

earlier observations by researchers working with other types of waste, such as marble [22] and fired-clay blocks [23], as cement additions.

3.2. Mechanical strength

According to the data in Table 2, all the mortars studied exhibited 28 d compressive strength ≥ 42.5 MPa (42.5N/ 42.5R). Strength was lower in the materials bearing granite quarry dust, due primarily to two factors: i) increase in total porosity (see Table 3), with a larger volume of macropores, the fraction with the heaviest impact on mechanical strength [24]; and ii) the quarry dust's slow pozzolanic reaction rate [16, 19]. That behaviour was also observed in previous studies when additions of a similar nature, namely ornamental stone such as marble [25] or slate [26] or others characterised by low reactivity such as copper slag [27], fly ash [28, 29] or silico-manganese slag [30], were added to cement. The strength values nonetheless showed that the new cements met the strength requirements laid down in the standards in place for strength class 42.5 MPa [18].

The flexural strength data followed a similar pattern, with strength declining with rising granite quarry dust content.

3.3. Total porosity and pore size distribution

The total porosity and pore size distribution in the 28 d mortars and the linear equations relating these properties to the replacement ratio are listed in Table 3. Total porosity was observed to be 3.35 % higher in mortar OPC+10%AF and 7.28 % higher in mortar OPC+20%AF than in the reference OPC.

Table 3. Total porosity and pore size distribution in 28 d mortars

Both added mortars were observed to have slightly higher macropore ($\phi > 0.05 \mu\text{m}$) and capillary pore ($0.007 < \phi < 0.05 \mu\text{m}$) fractions than the reference. In the latter fraction, the smallest capillary pores were the ones most heavily impacted, due to waste pozzolanicity.

These findings were consistent with earlier reports on the behaviour of industrial waste (fly ash [31], bagasse ash [32], bottom ash [33] and waste water sludge [34]) included as pozzolanic additions in new cement design.

3.4. Electrical resistivity

The electrical conductivity of the mortars analysed is plotted versus time in Figure 3. Three regions can be distinguished on the curves: i) a first, up to 4 d and particularly on the first day

when the mortars bearing granite quarry dust had a higher conductivity than the OPC; ii) a second, up to 16 d when conductivity was similar in the three mortars, and iii) a third, where the conductivity pattern was $\text{OPC}+20\%\text{AF}<\text{OPC}+10\%\text{AF}<\text{OPC}$. Those findings were consistent with earlier studies on mortars containing fly ash [35] or sugar cane bagasse ash [36], which like granite quarry dust are characterised by low early age pozzolanicity.

Figure 3. Electrical conductivity versus time

Moreover, resistivity, defined as the inverse of conductivity and which rose with age and replacement ratio, was observed to be 9.1 % higher in $\text{OPC}+10\%\text{AF}$ and 17.2 % higher in $\text{OPC}+20\%\text{AF}$ than in OPC. Similar patterns were observed by other authors studying this property in cements with additions such as andesite [37], fluid catalytic cracking catalyst residue (FCC) [38], rice husk [39], silica fume [40] and blast furnace slag [40].

As noted by Baroghel et al. [41], this improved behaviour is associated with the addition-prompted decline in alkalis and OH^- ions in the pore system, which reduces ion mobility in the pore solution. The outcome, higher resistivity as observed here, would counter the decline in that property induced by a more intensely connected pore system [41, 42].

Figure 3 also revealed a potential correlation between conductivity and time. That relationship was defined in an earlier study by Andrade et al. [43] as $y = y_o \cdot (t)^q$, where y is the value of conductivity at a given age; y_o is conductivity at the first given age; t is time; and q is the ageing factor that represents hydration progress. The fourth parameter, q , is essential to establishing the durability models proposed by Andrade and D'Andrea [44] and laid down in Spain's Code on Structural Concrete, EHE-08 [45] and Model Code 2010 [46]. The q values were observed to be higher for the new type II/A cements than for type I conventional cement, as observed by Andrade and D'Andrea [43] for cement types II/V and II/P. Moreover, these values for ageing factor are within the 0.2-0.8 range showed for this parameter in Model Code 2010 [47].

The inference drawn from the values for 28 d electrical resistivity was that the inclusion of granite quarry dust had no adverse effect on that durability indicator, for all the materials exhibited values ranging from $100 \Omega \cdot \text{m}$ to $200 \Omega \cdot \text{m}$, i.e., defined by a number of authors [41, 48] as moderate steel corrosion risk (M).

3.5. Drying shrinkage

The drying shrinkage measured in the mortars dried for 40 d in air at 20 °C and 60 % RH is shown in Figure 4. Whilst contraction was similar in all three mortars in the first 48 h, the presence of granite quarry dust subsequently mitigated shrinkage, which was 13.5 % lower in OPC+10%AF and 19.1 % lower in OPC+20%AF than in the reference OPC.

Those findings were also consistent with the results of earlier studies on additions such as blast furnace slag [49], fly ash [50] and rock wool waste [51].

Figure 4. Drying shrinkage in mortars dried in air for 40 d at 20 °C and 60 % RH

The better drying shrinkage behaviour observed in the additioned mortars was attributed to a number of factors: i) the linear relationship between heat of hydration and drying shrinkage (see 3.11), observed by Hu et al. [52] and Zhan et al. [49]; ii) the effect of fineness on shrinkage [49], with greater fineness ($\text{OPC} > \text{OPC}+10\%\text{AF} > \text{OPC}+20\%\text{AF}$) translating into greater shrinkage; iii) the higher free water content [34] in OPC than in the additioned mortars, which have a higher water demand [19]; and iv) the pore structure, which conditions the capillary pressure generated in the pore system as per the Kelvin-Laplace equation. With respect to iv), a critical pore size of 0.10 μm was found for this property in the present study (see Table 3), which also identified a linear relationship between total shrinkage and volume of pores under 0.10 μm . The equation fitted to that curve was $y=0.22x-0.49$ ($R^2=0.966$), where y is drying shrinkage and x the volume of pores under 0.10 μm . The same effect was observed by Kwon et al. [53], who noted that mortars with lower total porosity, a smaller volume of pores $>0.10 \mu\text{m}$ and a greater volume of pores $<0.10 \mu\text{m}$ exhibited greater drying shrinkage.

Figure 4 also shows the log regressions for the relationship between drying shrinkage and time, along with the correlation coefficients (all over 0.95).

3.6. Expansion

The expansion observed in the mortars after immersion in water for 40 d at a temperature of 20 °C is shown in Figure 5. The increase in volume found for all the mortars tested was attributed to water absorption by C-S-H gels, which tend to sponge when in contact with fluids [54, 55]. The lower C-S-H content in the additioned than in the OPC mortar [19] would explain why OPC+10%AF exhibited 20 % less expansion and OPC+20%AF 40 % less than OPC.

Figure 5. Expansion in mortars immersed in water at 20 °C

The figure shows that 25 % to 33 % of the expansion took place within the first 24 h, which is consistent with reports by Martauz et al. [56]. Figure 5 also shows the log regressions for the relationship between expansion and time and the respective correlation coefficients, all over 0.93.

3.7. Alkali-silica reaction

Mortar expansion after immersion in a 1N NaOH solution for 28 d at 80 °C is shown in Figure 6. Less expansion was observed in mortars OPC+10%AF and OPC+20%AF than in OPC and the 14 d and 28 d values were lower than the 0.10 % ceiling stipulated in the national legislation [57] and the 0.20 % recommended in international standards [58]. These data confirmed that the granite quarry dust added to the cements exhibited no alkali-silica reactivity.

Figure 6. Expansion in mortars immersed in a 1N NaOH solution at 80 °C

The decline in expansion with higher proportions of the addition was directly associated with the lower alkali content in the pore solution and lower Ca(OH)_2 content in OPC+20%AF, an effect generally accepted by the research community, as noted by Kawabata and Yamada [59].

The present findings were also consistent with observations reported by Al-Fadala et al. [60] and Saha and Sarker [61], according to which the inclusion of volcanic or fly ash failed to induce the alkali-silica reaction. Those authors also recorded lesser expansion in the added mortars.

The BSE micrographs recorded at the end of the test for the alkali-silica reaction (Figure 7) showed that all the cements exhibited a similar appearance, with no indication of the formation of expansive silica gels or therefore of cracking.

Figure 7. BSE micrographs (500x) of 28 d mortars tested for the alkali-silica reaction: a) OPC; b) OPC+10%AF; c) OPC+20%AF

3.8. Total and capillary absorption

The water absorption and capillary absorption coefficient values for 28 d mortars are given in Table 4. Mortar OPC+10%AF absorbed 7.1 % more water, and mortar OPC+20%AF 14.8 % more water than the reference OPC.

Those findings were consistent with earlier reports for additions with pozzolanicity similar to that of the granite dust studied here, such as copper slag [27], fly ash [28], ground granulated blast furnace slag [28] and concrete waste ash [62].

Table 4. Total water absorption, capillary absorption coefficient and peak temperature in the mortars studied

The data recorded were directly related to the linear relationship between this property and both density (see 3.1, the higher the density the lower the absorption values [63]) and porosity and pore size distribution (Figure 8) [64]. As Figure 8 shows, total porosity and the size of the macropore fraction were more closely correlated to total absorption than capillary pore volume.

Figure 8. Water absorption and capillary absorption coefficient versus porosity: a) OPC; b) OPC+10%AF; c) OPC+20%AF

Further to the data in Table 4, the capillary absorption coefficient was slightly higher in the mortars bearing granite waste cement than in the reference OPC, by 10.3 % in OPC+10%AF and 13.7 % in OPC+20%AF.

Those observations were the direct result of the increase in both total porosity and the volume of capillary pores in the mortars bearing granite quarry dust (see Figure 9) ($\Phi < 0.05 \mu\text{m}$). The effect is visible in Figure 8, which shows the linear relationship between the increase in porosity (total porosity and macro- and capillary pore volume) and the capillary absorption coefficient in the mortars studied. The capillary fraction was observed to have a greater impact than total porosity on capillary absorption, a finding reported previously by Martys and Ferraris [65].

Rises in capillary absorption as a direct effect of the increase in capillary pore volume were also recorded by researchers studying the impact of additions such as biomass bottom ash [66], calcined marl [67], fly ash [68] and granulated blast furnace slag [69] on that property.

The capillary absorption curves plotted in Figure 9 proved to be similar for all three mortars studied (OPC, OPC+10%AF and OPC+20%AF), while also showing that the inclusion of the addition induced a rise in water absorbed and lengthened the time taken to reach saturation due to the higher volume of capillary pores. The curves were characterised by rapid early age (up to 24 h) absorption, which subsequently slowed due to rehydration and hydration of the paste components, developments that modified their microstructure and with it pore connectivity [70, 71]. In the final, saturated stage sample weight remained constant, during which period the pores filled with air as a result of diffusion [72].

Figure 9. Capillary absorption curves in mortars

3.9. Heat of hydration

The peak temperature of the heat released by the mortars analysed is given in Table 4. The data showed lower maximum heat in the mortars containing granite quarry dust than in the reference: 5.5 % lower in OPC+10%AF and 19.3 % lower in OPC+20%AF. Similar behaviour has been reported for other pozzolanic materials included in new cements at similar replacement ratios, such as paper mill sludge [73], fly ash [74], fired clay-based sanitary ware [15] and milled ash [29].

Heat of hydration in the mortars analysed is plotted against time (up to 120 h = 5 d) in Figure 10.

Figure 10. Total heat of hydration in mortars versus time

Heat of hydration rose rapidly in the first 15 h, and then more slowly, peaking at 30 h and remaining essentially flat through the end of the test.

Heat of hydration was also observed to decline as the replacement ratio rose. The mortar containing 10 % granite quarry dust released 6.4 % less heat and the 20 % additioned mortar 20 % less than the control OPC. That behaviour can be explained by the linear relationship between heat of hydration and the replacement ratio observed by Medina et al. [15]. The relationship observed here fit the equation $y = -2.79x + 317$ ($R^2 = 0.974$), where y is heat of hydration and x the replacement ratio.

The decline observed was consistent with a trend reported by authors [74, 75] studying the early age effect of low pozzolanicity mineral additions (fly ash), irrespective of the replacement ratio. Sánchez de Rojas et al. [76, 77] and Rodríguez et al. [73], analysing additions with higher pozzolanic activity such as spent FCC and paper sludge, observed that for replacement ratios of 20 % or higher, heat of hydration declined due to the slower pozzolanic reactivity resulting from the shortage of portlandite in the mortars.

In light of the foregoing, OPC+20%AF can be regarded as a low heat cement and classified as CEM type II/A 42.5R LH, inasmuch as its 41 h heat of hydration value was lower than 270 J/g.

3.10. Colour

The colour coordinates for the anhydrous cements and the granite quarry dust are shown in Figure 11. Although all three cements lay in a range (0-0.03 (a^*), 8.4-9 (b^*)) indicative of grey tones [78], the addition of granite quarry dust induced a slight shift to the right in the chromatic

quadrant defined by the $+a^*/+b^*$ axes. A similar shift was recorded by other authors assessing the effect of adding paper sludge [79] or coal mining waste [80] to cement raw mixes.

Figure 11. Colour coordinates (a^* , b^*) for the anhydrous cements and granite quarry dust
Adding granite dust, with a lightness (L^*) value of 77.1 %, to the cement raised L^* from the 65.4 % recorded for OPC to 68.3 % in OPC+10%AF and 69.5 % in OPC+20%AF.

The increase in lightness toward 100 % (white) was linearly correlated to the replacement ratio further to the equation $y=0.20x+65.73$ ($R^2=0.941$), where y is lightness and x the percentage of the addition in the cements.

3.11. Statistical analysis

The results of the bivariate analyses of the variables studied are listed in Table 5. The data given for each variable include Pearson's correlation coefficient (R^2), a measure of the strength of the linear relationship between two variables (which is strongest when closest to one of the two extreme values, +1 and -1); and the p-value, an indication of the statistical significance of the correlation between variables (assuming significance at $p<0.05$) at a confidence level of 95 %.

Table 5. Bivariate analysis data

The variables (properties) water absorption, capillary absorption, electrical resistivity, shrinkage, expansion and heat of hydration were all observed to be inter-related, and related as well to the mechanical properties studied. The correlations obtained for the variables water absorption, density and compressive strength were consistent with findings reported by Baeza et al. [63] for mortars prepared with binary and ternary cements.

The multivariate analysis of variance (MANOVA) findings on the effect of the factors time and replacement ratio on the variables electrical resistivity, water absorption, capillary water absorption, expansion, shrinkage, alkali-silica reaction and heat of hydration are given in Table 6. The statistical model applied explained from 65 % to 99 % of the variability observed, depending on the variable analysed.

Table 6. Multivariate analysis of variance (MANOVA) data

The data showed that replacement ratio and age had significant effects ($p<0.05$) on all the variables analysed, as observed in the findings discussed above. The interaction between replacement ratio and age also had a significant effect ($p<0.05$) on the variables electrical

resistivity, drying shrinkage and heat of hydration. Interaction between the two factors was not significant for any of the other variables studied, however ($p = 1 > 0.05$).

4. CONCLUSIONS

The conclusions that may be drawn from this study are set out below.

- New cements bearing 10 % or 20 % granite quarry dust meet the strength requirements laid down in the standards in place for cement type II/A, strength class 42.5 N or 42.5 R.
- When included in cement, the waste induces a small rise (≈ 17 %) in electrical resistivity and raises the q (ageing factor) value, affording the resulting mortars greater protection against corrosion than available to conventional materials.
- The new cements exhibit slightly higher total (15 %) and capillary (14 %) absorption than the reference OPC.
- The partial replacement of cement with granite quarry dust reduces drying shrinkage and expansion in the new materials, lowering the risk of early age cracking that could compromise their durability and shorten their service life.
- As granite quarry dust exhibits no alkali-silica reactivity, it is apt for use in cement manufacture.
- Heat of hydration is 19 % lower in OPC+20%AF cement than in OPC, narrowing the likelihood of early age cracking.
- Binders bearing 20 % granite quarry dust meet the requirements to qualify as low heat cements (CEM II/A LH).
- The new cements have higher L^* values than OPC, rendering them apt for aesthetically sensitive applications.

In short, granite quarry dust induces no substantial change in the durability parameters assessed and is apt for use in the design of new type II/A conventional as well as type II/A special low heat cements. The present findings to that effect further the institution of the circular economy in the cement industry.

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Table 1. Physical, mechanical and durability variables studied

Property	Standard	Specimen size (cm)	Water/ cement ratio
Bulk density	-	4 x 4 x 16	0.50
Compressive and flexural strength	EN 196-1 [17]		
Electrical resistivity	UNE 83988 [20]		
Total water absorption	UNE 83980 [21]		
Capillary absorption	UNE 83982 [22]		
Drying shrinkage	UNE 80112 [23]	2.5 x 2.5 x 28.5	0.47
Water-induced swelling			
Alkali-silica reaction	UNE 146508 [24]		
Calorimetry	EN 196-9 [25]	-	0.40
Colour	UNE 80117	4 x 4 x 16	0.50

Table 2. Bulk density and mechanical performance in 28 d mortars

Property	Mortar		
	OPC	OPC+10%AF	OPC%+20%AF
Bulk density (kg/m ³)	2284.51 ± 2.71	2282.68 ± 0.76	2280.86 ± 2.20
Compressive strength (MPa)	65.67 ± 1.27	58.08 ± 1.07	51.12 ± 0.94
Flexural strength (MPa)	9.65 ± 0.08	8.68 ± 0.29	7.97 ± 0.63

Table 3. Total porosity and pore size distribution in 28 d mortars

Property (%vol.)		Mortar		
		OPC	OPC+10%AF	OPC%+20%AF
Total porosity		12.52	12.94	13.44
		$y = 0.05x + 12.51 [R^2=0.99]$		
Macropores (>0.05 μm)		9.85	9.94	10.14
		$z = 0.02x + 9.83 [R^2=0.96]$		
Capillary pores (0.007 < ϕ < 0.05μm)		2.27	2.73	2.81
		$j = 0.03x + 2.33 [R^2=0.86]$		
Pores	ϕ > 0.10 μm	5.50	6.73	7.10
	ϕ < 0.10 μm	6.62	5.94	5.86
Note. – y: total porosity; z: macropores; j: capillary pores; x: granite quarry dust replacement ratio				

Table 4. Total water absorption, capillary absorption coefficient and peak temperature in the mortars studied

Property	Mortar		
	OPC	OPC+10%AF	OPC+20%AF
Water absorption (%)	4.99	5.35	5.73
Capillary absorption coefficient ($10^{-4}\text{kg/min}^{0.5}\text{m}^2$)	2.90	3.21	3.32
Peak temperature ($^{\circ}\text{C}$)	32.70	30.90	26.40

Table 5. Bivariate analysis data

		WA	SC	DE	EX	RT	AS	CS	FS	RE	HH
WA	R^2	1	0.903	-0.698	-0.913	0.721	-0.699	-0.954	-0.926	0.768	0.882
	p		0.001	0.037	0.001	0.028	0.036	0.000	0.000	0.016	0.002
SC	R^2	0.903	1	-0.667	-0.777	0.634	-0.689	-0.815	-0.958	0.640	-0.683
	p	0.001		0.050	0.014	0.067	0.040	0.007	0.000	0.048	0.042
DE	R^2	-0.698	-0.667	1	0.465	-0.198	0.220	0.700	0.635	-0.290	0.328
	p	0.037	0.050		0.208	0.610	0.570	0.036	0.066	0.450	0.388
EX	R^2	-0.913	-0.777	0.465	1	-0.832	0.701	0.984	0.855	-0.912	0.942
	p	0.001	0.014	0.208		0.001	0.011	0.000	0.003	0.001	0.000
RT	R^2	0.721	0.634	-0.198	-0.832	1	-0.459	-0.828	-0.654	0.886	-0.779
	p	0.028	0.067	0.610	0.001		0.134	0.016	0.056	0.001	0.013
AS	R^2	-0.699	-0.689	0.220	0.701	-0.459	1	0.772	0.692	-0.685	0.772
	p	0.036	0.040	0.570	0.011	0.134		0.055	0.069	0.062	0.055
CS	R^2	-0.954	-0.815	0.700	0.984	-0.828	0.772	1	0.874	-0.902	0.965
	p	0.000	0.007	0.036	0.000	0.016	0.055		0.002	0.001	0.000
FS	R^2	-0.926	-0.958	0.635	0.855	-0.654	0.692	0.874	1	-0.693	0.764
	p	0.000	0.000	0.066	0.003	0.056	0.069	0.002		0.039	0.017
RE	R^2	0.768	0.640	-0.290	-0.912	0.886	-0.685	-0.902	-0.693	1	-0.864
	p	0.016	0.048	0.450	0.001	0.001	0.062	0.001	0.039		0.003
HH	R^2	-0.882	-0.683	0.328	0.942	-0.779	0.772	0.965	0.764	-0.864	1
	p	0.002	0.042	0.388	0.000	0.013	0.055	0.000	0.017	0.003	

Note. - **WA**: water absorption; **SC**: capillary absorption coefficient; **DE**: density; **EX**: expansion; **RT**: drying shrinkage; **AS**: alkali-silica reaction; **CS**: compressive strength; **FS**: flexural strength; **RE**: electrical resistivity; **HH**: heat of hydration; **p**: p-value; **R^2** : Pearson's correlation coefficient. **Shading** denotes statistical significance.

Table 6. Multivariate analysis of variance (MANOVA) data

Source of variation	Dependent variable	Type III sum of squares	Degrees of freedom	Mean square	F	p-value
Corrected model	Electrical resistivity ^a	142850.46	41	3484.16	646.01	0.00
	Water absorption ^b	9191.24	26	353.51	19.33	0.00
	Capillary absorption ^c	848560	56	15.15	1.68	0.00
	Expansion ^d	0.34	89	0.004	3.49	0.00
	Drying shrinkage ^e	13.58	89	0.15	160.68	0.00
	Alkali – silica ^f	0.001	35	2.43*10 ⁻⁵	2.39	0.00
	Heat of hydration ^g	3406769.47	137	24866.93	176.35	0.00
Replacement ratio	Electrical resistivity	242.39	2	121.19	22.47	0.00
	Water absorption	2069.16	2	1034.58	56.58	0.00
	Capillary absorption	72.56	2	36.28	4.02	0.02
	Expansion	0.28	2	0.14	126.12	0.00
	Drying shrinkage	1.12	2	0.56	590.32	0.00
	Alkali - silica	0.000	2	0.000	22.55	0.00
	Heat of hydration	139126.88	2	69563.44	493.33	0.00
Age	Electrical resistivity	139937.78	17	8231.63	1526.26	0.00
	Water absorption	7105.52	8	888.19	48.57	0.00
	Capillary absorption	756.83	18	42.05	4.66	0.00
	Expansion	0.06	29	0.002	1.85	0.01
	Drying shrinkage	12.36	29	0.43	448.73	0.00
	Alkali - silica	0.000	11	3.43*10 ⁻⁵	3.38	0.00
	Heat of hydration	3230399.00	45	71786.64	509.10	0.00
Interaction between replacement ratio and type of waste	Electrical resistivity	2596.14	22	118.01	21.88	0.00
	Water absorption	16.56	16	1.03	0.057	1.00
	Capillary absorption	19.17	36	0.53	0.06	1.00
	Expansion	0.005	58	9.34*10 ⁻⁵	0.09	1.00
	Drying shrinkage	0.102	58	0.002	1.85	0.01
	Alkali - silica	1.48*10 ⁻⁵	22	6.74*10 ⁻⁷	0.07	1.00
	Heat of hydration	37243.60	90	413.82	2.94	0.00
a. $R^2 = 0.997$ (Adjusted $R^2 = 0.995$)			e. $R^2 = 0.981$ (Adjusted $R^2 = 0.975$)			
b. $R^2 = 0.903$ (Adjusted $R^2 = 0.856$)			f. $R^2 = 0.637$ (Adjusted $R^2 = 0.625$)			
c. $R^2 = 0.652$ (Adjusted $R^2 = 0.602$)			g. $R^2 = 0.989$ (Adjusted $R^2 = 0.983$)			
d. $R^2 = 0.735$ (Adjusted $R^2 = 0.682$)

Figure 1. Stockpiled granite quarry dust

Figure 2. Pore size distribution for cements OPC, OPC+10%AF and OPC+20%AF

Figure 3. Electrical conductivity versus time

Figure 4. Drying shrinkage in mortars dried in air for 40 d at 20 °C and 60 % RH

Figure 5. Expansion in mortars immersed in water at 20 °C

Figure 6. Expansion in mortars immersed in a 1N NaOH solution at 80 °C

Figure 7. BSE micrographs (500x) of mortars tested for 28 d for the alkali-silica reaction: a) OPC; b) OPC+10%AF; c) OPC+20%AF

Figure 8. Water absorption and capillary absorption coefficient versus porosity: a) OPC; b) OPC+10%AF; c) OPC+20%AF

Figure 9. Capillary absorption curves in mortars

Figure 10. Total heat of hydration in mortars versus time

Figure 11. Colour coordinates (a^* , b^*) for the anhydrous cements and granite quarry dust