Received: 8 July 2024 | Revised: 25 November 2024 | Accepted: 2 December 2024

DOI: 10.1002/suco.202400889

ARTICLE

fib CEB·FIP WILEY

Enhancing concrete sustainability with spent diatomaceous earth from the wine industry: Long-term experimental and statistical analysis

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Funding information
European Regional Development Fund, Grant/Award Number: POCI-01-0247-FEDER-069583

Abstract

Spent diatomaceous earth, a by-product of wine filtration, holds significant promise for use in concrete mixtures due to its pozzolanic properties, which enhance concrete performance. This research explores the application of spent calcined diatomaceous earth (SCDE), heat-treated at 700°C to remove organic content, as a partial substitute for cement or sand in concrete. The high silica content of SCDE contributes to the formation of additional calcium silicate hydrates during the hydration process, leading to improvements in mechanical strength over time. The study includes a preliminary analysis of cement replacements ranging from 5% to 10% and sand replacements from 2.5% to 15%, assessing their effects on workability, density, water absorption, and compressive strength at 7 and 28 days. The testing program focuses on three key compositions: the reference concrete and optimal mixtures with 10% cement and 5% sand replacements. Compressive strength tests are conducted at 7, 28, 90, 180, and 360 days. The results, validated through ANOVA, demonstrate the influence of SCDE on concrete strength over time, with distinctive behavior patterns identified for different mixtures. The strength of concrete with replacements correlates with the reference strength, with a multiplicative factor that varies from 7 to 90 days and then. When SCDE replaces cement, the factor is less than one before 28 days due to the slow pozzolanic reaction, whereas for sand replacement, it always exceeds one because of the initial filler effect. After 90 days, the strength multiplicative factors are 1.13 for cement replacement and 1.30 for sand replacement, demonstrating the potential of SCDE for sustainable concrete manufacturing and its positive long-term impact on mechanical performance.

KEYWORDS

cement replacement, compressive strength, concrete mixtures, sand replacement, spent diatomaceous earth

1 | INTRODUCTION

Since the onset of the industrial era,[1,2] human activities have increasingly impacted the environment, with greenhouse gas emissions rising over time.[3,4] This trend has led to the current climate crisis, which calls for urgent measures across all sectors.[5] In the construction industry, cement production is a significant contributor, currently responsible for nearly 8% of global CO_2 emissions.[6,7] Additionally, this process requires substantial utilization of natural resources and thermal energy, with an approximate requirement of 3460 MJ per ton of clinker in 2018,[8] primarily due to the high temperatures (1400–1500°C) necessary for clinker manufacturing.[9] In this context, transitioning towards sustainable construction practices is critically important to address the environmental challenges faced. As a result, there has been a growing interest in the development of alternative materials with a reduced environmental footprint.[10]

The search for sustainable alternatives in cement production has led to the exploration of various pozzolanic materials, such as metakaolin, fly ash, and blast furnace slag.[11–14] These materials allow for the adjustment of cement content in concrete mixtures, without compromising their strength,[13,15] and when used as cement replacement in appropriate amounts (10%–30% of the cement mass), they contribute to the development of low-carbon cementitious materials.[16] This is achieved through chemical reactions between the cement and the calcium (Ca), silicon (Si), and aluminium (Al) components of the additives, forming hydration products like calcium–silicate–hydrate (C–S–H) gel and ettringite. However, reducing cement content by just 10%–30% does not sufficiently lower CO_2 emissions. As a result, researchers are turning to novel materials with compositions similar to cement components, such as diatomaceous earth (DE), which is rich in silica and aluminium.[16]

In addition, it is also important to study the replacement of sand in concrete and mortar mixtures, as it is a scarce natural resource that needs to be preserved. Sand is the second mostly exploited resource worldwide after water[17] and its excessive extraction affects inland and coastal ecosystems.[18,19]

The DE used in this research is a by-product of wine filtrations, generated during the winemaking process at Caves Campelo S.A. After filtration, the DE becomes saturated and loses its effectiveness, at which point it is referred to as spent diatomaceous earth (SDE). Once it has reached this state, SDE can no longer be reused in filtration and becomes industrial waste, typically sent to waste treatment plants. This research stems from the "BacchusTech—Integrated Approach for the Valorisation of Winemaking Residues" Project, POCI-01-0247-FEDER-069583,[20] which aims to valorize SDE generated through wine filtration processes, along with solid and liquid waste from distillation processes. Waste will be valorized by incorporation into food and cosmetic formulations, while the SDE will be repurposed for the construction industry.[20,21]

DE is a natural, biogenic and sedimentary material, rich in aluminosilicates[22,23] with pozzolanic properties very similar to those of metakaolin and fly ash.[14,24,25] These properties present an opportunity to develop a unique cement by harnessing the distinct properties of this material. DE has physical properties such as low bulk density, high porosity and large surface area which make this material valuable in the filtration industry.[22] For this use, DE undergoes a calcination process at temperatures above 1000°C, resulting calcined diatomaceous earth (CDE). This treatment enhances the hardening of the exoskeletons of diatoms, improving its filtering effectiveness.[26] However, once used in filtration, CDE loses its filtering capabilities, becoming a waste product that requires proper disposal.[22] In accordance with the principles of economic-sustainable development, companies that generate this by-product aim to reuse this SDE for alternative applications, helping reduce its ecological footprint.

Previous studies have researched the introduction of raw DE,[26–30] CDE,[28,29,31–36] SDE from wine and beer filtrations,[26,37,38] and spent calcined diatomaceous earth (SCDE)[26,37–39] in cement mixtures, obtained through a calcination process at temperatures ranging from 400 to 900°C in order to eliminate all organic matter.[26,37–39] Research has shown that incorporating CDE and SCDE into concrete and mortar mixtures can improve compressive strength over time,[26,28,29,32,34,35,39] with better results than those from mixtures containing raw DE[26–29] and SDE.[26] Several studies also revealed that concrete and mortar mixtures present better compressive strength results introducing quantities ranging from 5% to 15% as a replacement of cement.[28,29,31–39] However, the high porosity and the water absorption characteristics of CDE and SCDE can reduce workability in the concrete mixture.[26,36,39] To maintain workability, superplasticizers or the increase of water in the composition may be required.[31,33] Moreover, the low particle density of CDE and SCDE leads to a lower-density concrete when used to replace cement and sand.[40,41] At the same time, the large quantity of fine and porous particles in SCDE leads to more impermeable concrete, thus reducing the water absorption content of the concrete.[26] These properties make SCDE a promising alternative binder, offering an opportunity to reduce the ecological footprint of concrete production.

The aim of this study is to investigate the potential utilization of SDE derived from the wine industry in concrete mixtures, following a calcination process at 700°C, as a substitute for cement and, as a key novelty, for sand. The optimal replacement levels were established in a preliminary study, included in the paper, which analyses the effect of cement replacements ranging from 5% to 10%, and sand replacements ranging from 2.5% to 15% on workability, density, water absorption, and compressive strength. This study uniquely focuses on the long-term effects of SCDE on compressive strength, conducting in-depth analyses at the optimal replacement levels for both cement and sand. Compressive strength evolution is monitored over time, with data collected at 7, 28, 90, 180, and even 360 days, to evaluate the potential for long-term strength improvements and address concerns about long-term strength declines, as observed in other research.[42,43] An analysis of variance (ANOVA) is also performed to evaluate statistical differences between the three mixtures and to determine a confidence interval of 5%–95%.

2 | MATERIALS AND METHODS

2.1 | Diatomaceous earth

To valorize this by-product in the production of concrete, it is necessary to subject the SDE to a thermal treatment to remove any potential organic material. In this study, the SDE was air-dried for 30 days to minimize the emission of odors and smoke during calcination. Previous research has reported a wide range of calcination conditions, with temperatures set at 400°C for 3 h,[37] 650°C for 1 h,[38] and 900°C for 2 h.[26] Calcination in the range of 600–800°C not only eliminates all organic matter but also increases the amount of silica present in SDE.[26,38,39] In this study, calcination at 600 and 700°C were tested, confirming that complete organic matter removal was achieved at 3 h for 600°C and at 2 h for 700°C. Given the similar energy consumption, calcination at 700°C was chosen. Figure 1a shows the SDE rejected by the wine industry, while Figure 1b presents the SCDE after undergoing the mentioned treatment.

The effectiveness of the treatment was verified through thermogravimetric analysis (TGA) conducted with a TGA/DSC 3+ Mettler Toledo SAE, using an alumina crucible. As shown in Figure 2, the thermogravimetry (TG) curve indicates a residual mass loss of less than 1.86%, with an inflection point at 183.67°C.

The importance of aluminosilicates found in SCDE, such as silica and alumina, has been highlighted in previous studies.[37–39] Silica, in particular, plays a crucial role in its reaction with cement.[32,35,44] Consequently, to characterize the residue used in this study, it was essential to determine the chemical composition of the SCDE using inductively coupled plasma-optical emission spectrometry (ICP-OES), according to ISO 11885.[45] The chemical composition of SCDE is presented in Table 1. The comprehensive chemical analysis does not provide information regarding the reactivity of the tested diatomaceous earth in an alkaline environment. Therefore, it is crucial to determine the content of reactive amorphous phases.[29,46] To address this, X-ray diffraction (XRD) analysis was conducted on the diatomite samples, and the results are shown in Figure 3a, indicating the absence of crystalline peaks, suggesting an amorphous structure for the residue. In Figure 3b, a scan of the SCDE obtained through scanning electron microscopy (SEM) is presented. Fossilized cells from microscopic organisms, known as diatoms, are visible.

(a) (b)

FIGURE 1 (a) Spent diatomaceous earth and (b) spent calcined diatomaceous earth.

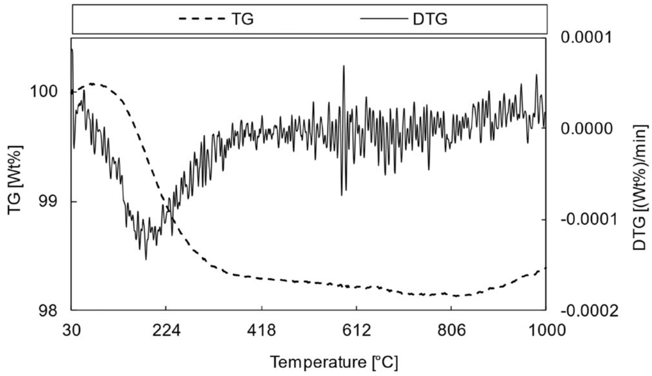


FIGURE 2 TG and DTG curves of SCDE.

TABLE 1 Chemical composition of SCDE.

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MnO
% in mass	84.29	13.86	0.81	0.80	0.20	0.04

The Fourier transform infrared spectroscopy (FTIR) spectrum of diatomaceous earth, shown in Figure 4, was obtained using a Tensor 27 Bruker FTIR spectrometer with a SPECAC "Golden Gate" accessory. The Si—O—H stretching vibrations, typically observed at 3250–3450 and 960–975 cm^{-1} in silicate materials,[47–49] were not present, as the spent diatomaceous earth used in this study originated from a commercial material that had undergone calcination during manufacturing.[50] Different characteristic bands were observed, which are mainly due to vibrations of the silicon–oxygen bond. The strong absorption band at 1000.42 cm^{-1} corresponds to the asymmetric stretching vibration modes of the Si—O—Si bond.[47,50,51] Also, one of the other two characteristic bands of Si—O—Si, typically at 792 and 468 cm^{-1},[50–53] was observed at 785.22 cm^{-1}.

The density of diatomaceous earth was analyzed in accordance with ISO 17892-3,[54] revealing values of 2340 kg/m^3 for commercial DE and 2460 kg/m^3 for SCDE. The calcination process raised the density of SCDE by 5.13% compared to commercial DE. This observation aligns with findings from other researchers.[25,55,56] The granulometric analysis of the SCDE was conducted through wet sieving, using the specifications outlined in ISO 17892-4.[57] The results of this assay are shown in Table 2. Another interesting parameter tested to describe the fineness of the SCDE is the specific surface area, obtained according to ISO 9277,[58] and the porous structure, in accordance with ISO 15901-2,[59] showing values of specific surface of 15.60 m^2/g, pore volume of 0.0156 cm^3/g, and average pore radius of 1.815 nm.

2.2 | Sand and gravel

The particle size characterization of the limestone aggregates was conducted in accordance with EN 933-1,[60] whose result is shown in Table 3. The density of these aggregates was assessed in accordance with EN 1097-6,[61] with sand and for gravel registering values of 2610 and 2660 kg/m^3, respectively.

2.3 | Preliminary analysis of replacement

A preliminary experimental analysis was developed to establish the SCDE replacements considered in the research. For this purpose, nine concrete mixtures were manufactured, one reference concrete (REF) composition and eight compositions incorporating SCDE as a replacement for cement (RC) or sand (RS). REF was manufactured using Portland cement class II B-L 32.5N, sand, gravel, and water. For cement replacement, SCDE proportions ranged from 5% (RC5) to 10% (RC10), while for sand replacement, SCDE proportions varied from 2.5% (RS2.5) to 15% (RS15), as shown in Table 4. The water content remained constant across all mixtures, with the water/cementitious material ratio[34,62,63] also provided in Table 4. The concrete components were mixed in a mixer for 15 minutes. The resulting fresh concrete was poured into 100 × 100 × 100 mm plastic molds and compacted using a vibrating table. The curing was conducted, in a humid chamber, at 25°C with 100% relative humidity. The workability of the fresh concrete was assessed in accordance with EN 12390-2,[64] with results presented in Table 4.

As expected, it was noted that the workability of concrete mixtures decreases with the increase of SCDE replacement,[26,39] due to the increase in the surface area of the mix. The reference composition presents a slump of 50 mm, which places it in consistency class S2 (50–90 mm).[65] The quantities of SCDE added to the composition were gradually increased, resulting in a loss of workability. As a consequence, all of these compositions achieved class S1 consistencies (10–40 mm)[65] (Table 4). As shown in Figure 5, the loss of workability depends not only on the amount of SCDE introduced into the concrete mixture but also on the material that was replaced in the composition.

Concrete cubic specimens were tested for compressive strength at 7 and 28 days, and density at 28 days, according to EN 12390-3[66] and EN 12390-7,[67] respectively. The average results of compressive strength (MPa) and density (kg/m^3) are listed in Table 5, where "CC" is concrete composition, "N" represents the number of specimens

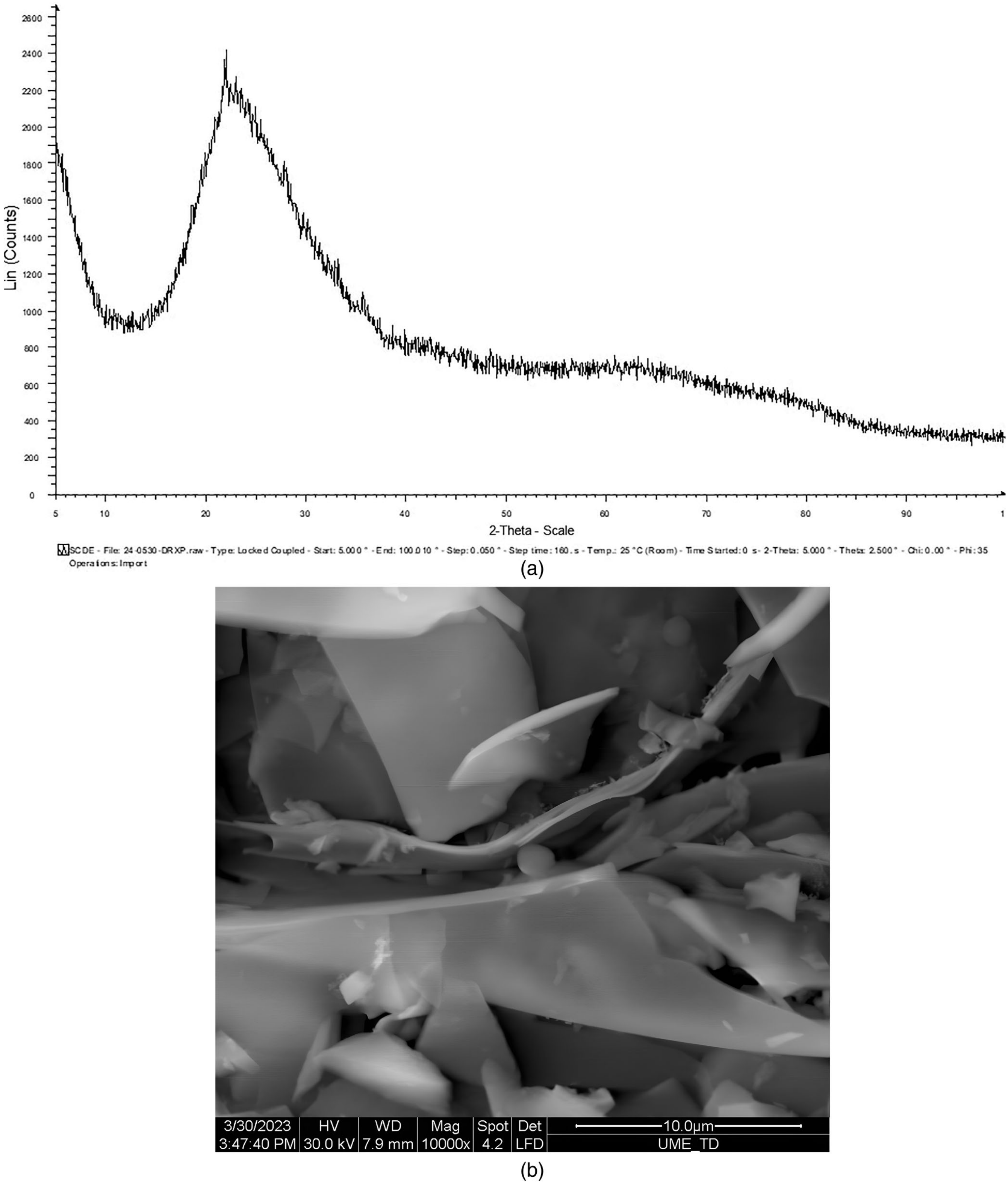


FIGURE 3 (a) XRD diagram and (b) SEM image of SCDE.

tested, and “CoV” is the coefficient of variation. These results are also plotted in Figures 6 and 7.

At 28 curing days, all concrete compositions with the incorporation of SCDE showed a lower density of hardened compared to REF. This reduction is attributed to the low particle density of SCDE, which is significantly lower than that of cement and sand. Similar trends have been reported in previous studies involving CDE.[40,41]

Three key factors have been identified that influence the contribution of diatomaceous earth to the mechanical strength of cement mixtures. These include the filler effect,[24,46] acceleration of ordinary Portland cement hydration, and the pozzolanic reaction between diatomite and Portland cement hydrates.[29]

The primary pozzolanic reaction can be summarized as:

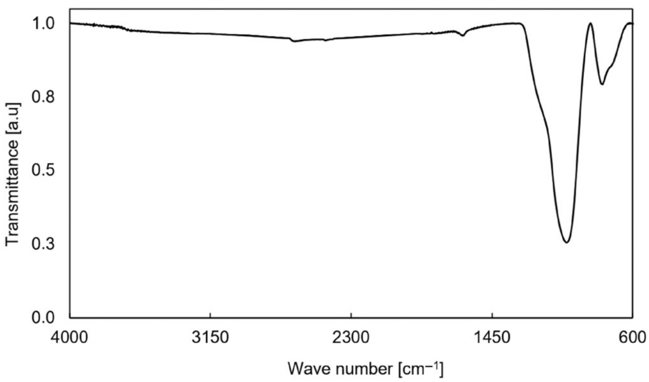


FIGURE 4 FTIR spectrum of SCDE.

$$CH + S + H \rightarrow C\text{-}S\text{-}H \quad (1)$$

The kinetics of this reaction are similar to the slower hydration rate of C_2S. Consequently, the incorporation of pozzolans, such as diatomaceous earth, has an effect comparable to increasing the C_2S content in cement. As a result, their addition reduces early heat release and initial strength development without affecting long-term strength.[29] For this reason, RC specimens show an initial loss of strength at 7 days. However, a slight increase in strength at 28 days is observed, as shown in Figure 6. This trend confirmed by findings from other previous studies.[26,28,29,32,34,35,38,39] This increase is primarily attributed to the filler effect[46] and the acceleration of cement hydration. The fine particles of SCDE enhance the compactness of the mix and act as nucleation sites, promoting the early formation of hydration products and accelerating the hydration process.[68,69] This results in reduced concrete porosity and improved properties such as strength. A minor contribution from a pozzolanic second generation of C–S–H[31,32,38] could play a role in this observed strength increase, as it starts to be perceived around 28 days, and extends up to 90 days, exceeding the

TABLE 2 Particle size distribution of spent calcined diatomaceous earth.

Diameter of particles (μm)	63	59.3	42.1	26.7	15.8	11.5	7.4	3.8	1.6	1.1
Accumulated passing (%)	100	88.7	83.1	77.5	53.9	9.0	3.4	2.2	2.2	2.2

TABLE 3 Particle size distribution of sand and gravel.

Sieve (mm)	12.5	10	8	6.3	4	2	1	0.5	0.25	0.125	0.063	<0.063
Accumulated passing (%)												
Sand	100	100	100	100	98.3	91.9	84.1	61.3	19.0	5.3	0.6	0.0
Gravel	100	97.5	69.1	33.4	4.4	1.5	1.2	1.1	1.0	0.8	0.5	0.0

TABLE 4 Mixture proportions and slump values of specimens in the replacement study.

Concrete composition	Cement (kg/m^3)	Water (kg/m^3)	SCDE (kg/m^3)	Sand (kg/m^3)	Gravel (kg/m^3)	W/(C + SCDE) ratio	Slump (mm)
REF	350	218.50	-	804.35	1010.06	0.62	50
RC5	332.50		17.50	804.35		0.62	40
RC7.5	323.75		26.25	804.35		0.62	30
RC10	315		35.00	804.35		0.62	20
RS2.5	350		20.11	784.24		0.59	45
RS3.5	350		28.15	776.20		0.58	40
RS5	350		40.22	764.13		0.56	35
RS10	350		80.44	723.92		0.51	20
RS15	350		120.65	683.70		0.46	15

timeframe covered in this preliminary analysis of the replacement.

Given the lack of no significant differences in compressive strength between RC7.5 and RC10, the RC10 composition was chosen for further research due to its greater material savings. In this previous study, no decrease in strength was observed at 28 days for cement replacements up to 10%, compared to the REF. However, when the CDE and SCDE replacement level exceeds 10%, significant strength reduction has been reported.[29,34,39]

In RS specimens, an increase in compressive strength at 7 days was expected because of their composition. While the amount of water and cement remained constant, the addition of a pozzolanic filler reduces the water to cementitious material ratio.[70] This reduction further densifies the concrete compared to the RC replacements, leading to improved strength,[36,71] to which is added the filler effect and the increase in the hydration rate of cement. However, this effect was only observed for replacements of up to 5%, as shown in Figure 7. Beyond this point, a decrease in compressive strength is observed compared to the reference concrete. To analyze this occurrence, diffractograms obtained through PerkinElmer Spectrum Two FTIR spectrometer are presented in Figure 8 for REF, RS5, and RS15.

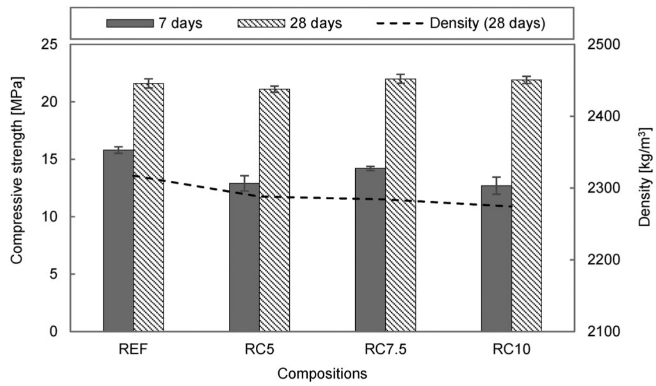


FIGURE 6 Cement replacements: compressive strength and density at 7 and 28 days.

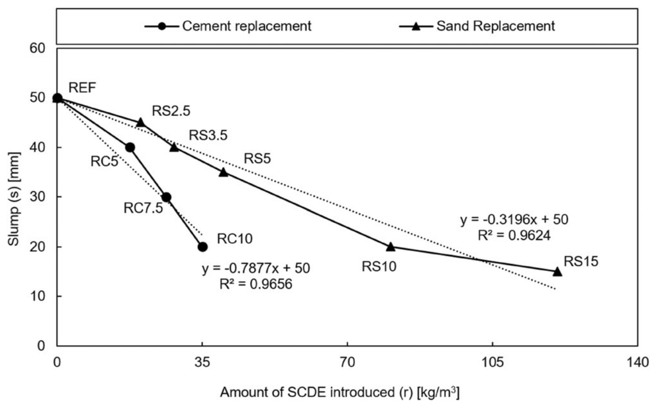


FIGURE 5 Effect of cement and sand replacement on workability.

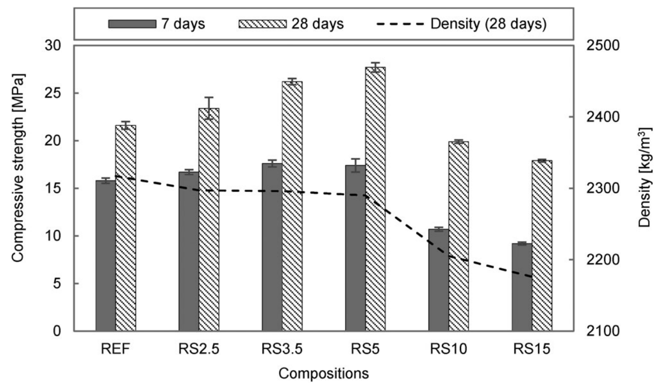


FIGURE 7 Sand replacements: compressive strength and density at 7 and 28 days.

TABLE 5 Concrete results at 7 and 28 days.

		Density (kg/m³)		Compressive strength (MPa)			
CC	*N*	Mean value (28 days)	CoV (%)	Mean value (7 days)	CoV (%)	Mean value (28 days)	CoV (%)
REF	3	2317	0.34	15.8	1.78	21.6	1.83
RC5		2288	0.14	12.9	5.20	21.1	1.32
RC7.5		2284	0.38	14.2	0.49	22.0	0.87
RC10		2274	0.21	12.7	5.91	21.9	1.45
RS2.5		2297	0.19	16.7	1.60	23.4	4.87
RS3.5		2296	0.39	17.6	2.02	26.2	1.25
RS5		2290	0.29	17.4	3.94	27.7	1.78
RS10		2205	0.26	10.7	1.87	19.9	0.35
RS15		2176	0.16	9.2	0.57	17.9	0.76

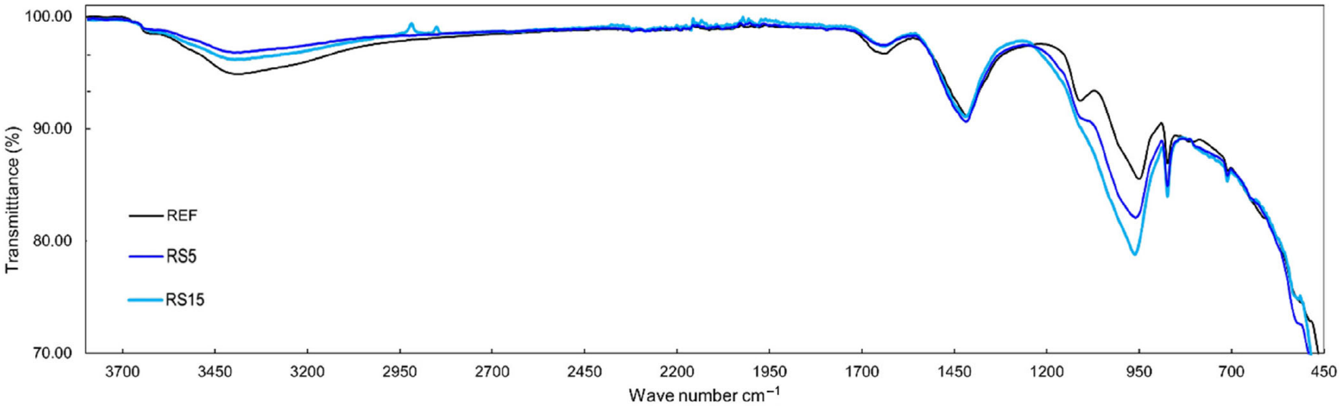
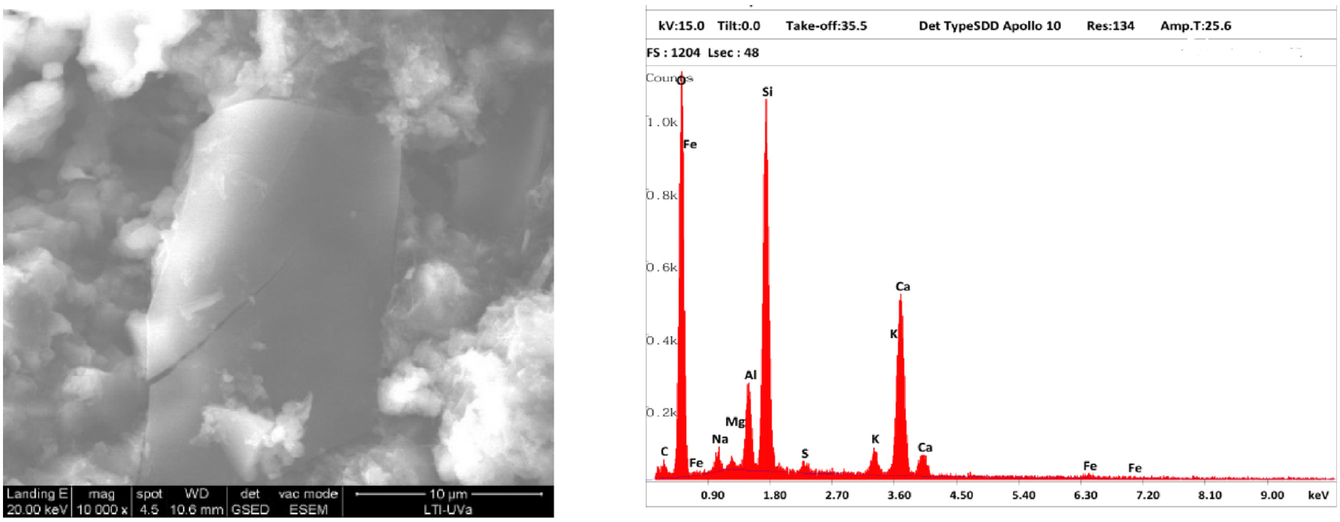


FIGURE 8 FTIR curves of REF, RS5 and RS15 at 28 days.



(a) (b)

FIGURE 9 (a) SEM and (b) EDS images of RS5 mixture.

In the FTIR analysis, a reduction in band intensity is observed in the range of 3420 cm^{-1} and around 1640 cm^{-1}. This decrease is attributed to a reduction in water associated with hydration products,[72,73] linked to a lower water-to-cementitious material ratio. The deformations in the bands around 1000.42 and 785.22 cm^{-1}, corresponding to SCDE, indicate that sand replacement remains as a filler at 28 days. However, when the percentage of SCDE surpasses the optimal filler level, it negatively impacts the strength of the concrete. This is because excess SCDE reduces the proportion of active cementitious materials, leading to a decrease in final strength and the formation of additional voids in the mix, which can act as weak points.[62] This explains the observed strength reduction when the SCDE content exceeds 5% of the sand.

In Figure 9, unreacted DE at 28 days is shown in the SEM (a) and EDS images (b), demonstrating that not all the diatoms have dissolved and that the C–S–H gel structure is still evolving even after 28 days, as in previous works.[24]

For these three chosen compositions: REF, RC10 and RS5, this preliminary study also analyzed the water absorption by immersion at atmospheric pressure, according to the specifications described in LNEC E394.[74] The RC10 and RS5 compositions showed better waterproofing results compared to the REF composition after 28 days of curing, with RC10 showing a water absorption value of 1.78% and RS5 1.93%, lower than the REF composition which showed a value of 3.36%. The introduction of SCDE can improve the impermeability of concrete because SCDE is a finer material than cement and sand, which leads to a greater number of voids to be filled, thus increasing the impermeability of concrete, an issue also verified in other authors' research.[26] This highlights the important durability advantages of concrete manufactured with the introduction of SCDE, due to the reduction in concrete density,[40,41] the increase in compressive strength over time,[26,39] and the reduction in water absorption.[26]

2.4 | Experimental research program

The experimental program analyses three concrete compositions: REF, RC10, and RS5. The density of the hardened concrete was obtained over time using the specifications described in EN 12390-7,[67] and the compressive strength over time was performed according to EN 12390-3.[66] A total of 240 cubic specimens, each measuring 100 × 100 × 100 mm, was produced, Figure 10. For each of the three composition analyzed, 20 specimens were used to evaluate the compressive strength at 7, 28, and 90 days. For the 180-day analysis, 15 specimens per composition were tested, while for the 360-day analysis, only 5 specimens per composition were used. Table 6 summarizes the distribution of specimens. Analyzed compositions in this program are identical to those investigated in the preliminary study, detailed in Table 4.

Understanding the long-term behavior of new materials in concrete is crucial, and can only be achieved after extensive investigations. Previous studies have highlighted potential compressive strength losses at advanced curing ages when incorporating novel materials.[42,43] For example, replacing 7.5% of cement by sugar cane bagasse ash caused compressive strength losses of approximately 8% between 45 and 65 days of curing,[42] and introducing raw DE in lightweight concrete resulted in compressive strength losses of approximately 20% between 28 and 56 days of curing.[43] Since SCDE is a relatively new material in the construction materials sector, one of the novelties of this study is the evaluation of the potential improvements in the compressive strength that SCDE can offer over 1-year period.

After the compression test at 28 days, small fragments of REF, RC10 and RS5 specimens were crushed and sieved using a 0.063 mm sieve. The powdered samples, containing primarily cementitious material and a percentage of aggregate filler, were used for TG and FTIR analysis, performed using a Mettler Toledo SAE TGA/DSC 3+ thermogravimetric analyzer and a Bruker Tensor 27 with a SPECAC 'Golden Gate' accessory. Additionally, SEM investigations used small, crushed fragments of concrete resulting from the compressive strength test, at 28 days.

2.5 | Statistical analysis

This study assessed the impact of cement and sand replacements on the compressive strength of concrete. The experimental results underwent an ANOVA test to determine whether the selected replacements significantly impacted concrete properties. A one-way ANOVA was chosen, as it allows comparison of the mean compressive strength between the three concrete mixtures (REF, RC10, and RS5) across different time points. Since the study focused on comparing different replacement levels within each mixture, with time as a separate variable, a one-way ANOVA was appropriate for evaluating the significance of differences in compressive strength. This analysis was repeated at 7, 28, and 90 days with 20 specimens per composition, at 180 days with 15 specimens, and at 360 days with 5 specimens, due to issues outlined in the experimental program. The statistical analysis was conducted using IBM SPSS Statistics Software.[75]

A null hypothesis, suggesting no significant differences between mean values, was set with a 95% confidence level. If the p-value from the ANOVA was below 0.05, the null hypothesis was rejected. Levene's test ensured homogeneity (p-value >0.05), while Shapiro–

(a) (b)

FIGURE 10 (a) Compositions under study; (b) compressive strength test.

T	CC	N	Density (kg/m^3) Mean value	Density (kg/m^3) CoV (%)	Compressive strength (MPa) Mean value	Compressive strength (MPa) CoV (%)
7	REF	20	2305.8	0.55	16.67	4.17
	RC10		2294.8	0.42	14.55	4.54
	RS5		2303.8	0.47	17.88	2.74
28	REF	20	2297.9	0.43	20.44	1.84
	RC10		2286.5	0.45	20.61	4.18
	RS5		2289.1	0.79	24.15	3.98
90	REF	20	2312.9	0.46	23.29	1.46
	RC10		2303.6	0.48	26.23	3.30
	RS5		2299.6	0.70	30.85	2.76
180	REF	15	2302.9	0.62	26.33	3.14
	RC10		2292.8	0.34	29.67	3.73
	RS5		2302.5	0.37	34.40	2.33
360	REF	5	2311.6	1.16	27.43	3.01
	RC10		2296.8	2.14	31.14	4.52
	RS5		2297.0	2.57	35.34	2.90

TABLE 6 Density and compressive strength values over time.

Wilk's test assessed normality (p-value >0.05). Tukey's HSD test was then used to determine mean differences at a significance level of 5%.[76–79]

In cases where the Shapiro–Wilk test indicated non-normality, or when sample sizes below 15,[76] Welch ANOVA and Dunnett's T3 for multiple comparison test were employed to handle variance heterogeneity. These tests ensure robust results even despite violations of the normality assumption.[80]

Similarly, when the Levene's test indicated non-homogeneous variables,[77] the Kruskal–Wallis test was used. Mean comparisons were conducted using the Bonferroni–Dunn test[81,82] to maintain the reliability of the results, even in presence of variance heterogeneity.

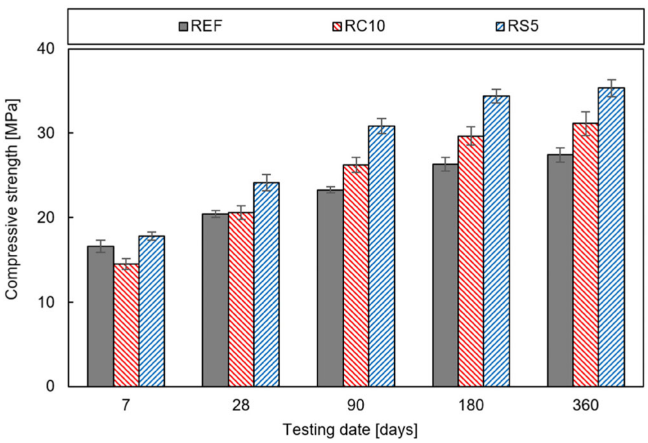


FIGURE 11 Compressive strength of concrete at test date.

3 | RESULTS AND DISCUSSION

3.1 | Compressive strength results

Average results of density and compressive strength obtained during the experimental campaign are listed in Table 6, where "T" is the testing days, "CC" is concrete composition, "N" is the number of specimens tested and "CoV" is the coefficient of variation (%). These results are also plotted in Figures 11 and 16.

As stated in the preliminary analysis of replacement, both RC10 and RS5 exhibited a lower density compared to REF, with average values of 2295 and 2298 kg/m^3, respectively, compared to REF at 2306 kg/m^3. The reduction in density is due to the replacement of cement with more porous, lower-density materials, a trend also confirmed by previous studies.[40,41]

According to the analysis in Section 2.3, the compressive strength of RC10 at 7 days was lower compared to REF. However, due to the acceleration of the cement hydration processes with the addition of SCDE, and the initiation of the pozzolanic reaction, the strength of RC10 and REF at 28 days is nearly identical. From that point onwards, an increase can be observed, with RC10 showing 12.8% higher strength than REF at 90 days due to the reaction between the silica in the SCDE and the calcium hydroxide from the cement,[31,32,35] as shown in Figures 11 and 12. After 90 days, the pozzolanic reaction appears fully developed, and the evolution of the compressive

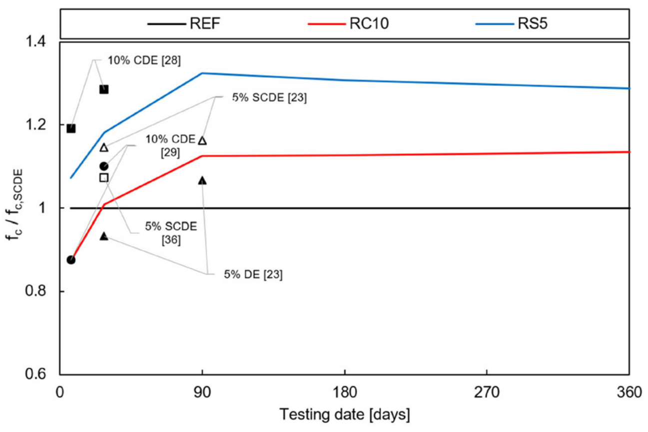


FIGURE 12 Relation between $f_c/f_{c,SCDE}$ over time.

strength of RC10 becomes similar to that of REF. These results are consistent with previous studies involving the incorporation of SCDE[26,39] and CDE[31,32] in concrete.

Concerning the compressive strength results of RS5, it consistently outperformed those of REF. This is related to the fact that SCDE was introduced into the RS5 composition without changing the amount of cement and only reducing the amount of sand, reducing the water to cementitious material ratio. The higher quantity of fine particles in SCDE compared to sand leads to a greater quantity of filled voids, thus densifying the concrete mixture and resulting in an increase of strength.[36,71] As explained in Section 3.2, this densification, combined with the reduced water-to-cement ratio and the higher rate of strength increase between 7 and 28 days due to the acceleration of hydration, contributes to the observed results. RS5 showed compressive strength values 7.13% higher at 7 days compared to REF, 18.15% higher at 28 days and 32.46% higher at 90 days. After this period, as shown in RC10, the strength over the REF stabilizes, this time around 28.84%. These results can be analyzed in Figures 11 and 12.

As shown in Table 6 and Figure 11, none of the three compositions show long-term declines in compressive strength.

Regarding the research of Hasan et al.,[32] which employed a 10% replacement of cement by CDE, it was found that the loss of compressive strength at 7 days was in the same order of magnitude as that found in RC10, associated with the reduction of cement in the mixture. At 28 days, this author shows higher compressive strength values compared to RC10, which is due not only to the higher pozzolanic activity of CDE compared to the SCDE used in this work, with a SiO_2 value of 78.7%, but also to the use of iron ore powder in the concrete composition.

Comparing the results of RC10 with those obtained in the research of Zhang et al.,[31] it was found that the latter showed greater increases in compressive strength at 7 and 28 days, with the same substitution of cement by CDE compared to RC10. The use of CDE with higher SiO_2 content (92.1%) increases pozzolanic activity compared to RC10. Additionally, this study uses superplasticizers in the concrete composition.

About the research of Letelier et al.,[39] this author uses a 5% replacement of cement by SCDE and shows an increase in compressive strength greater than RC10 at 28 days. The SCDE used by this author has much higher SiO_2 values (94.6%) than the SCDE used in this study, which explains the increase in compressive strength recorded at 28 days.

Replacing 5% of the cement by raw DE or SCDE was studied in the research of Rodrigues et al.[26] The composition with raw DE showed worse compressive strength behavior at 28 and 90 days compared to RC10. Although pozzolanic activity was detected in concrete with raw DE, which exceeded the compressive strength of the reference composition at 90 days, the raw DE did not undergo a calcination process and its pozzolanic properties were not improved, presenting a SiO_2 value of 54.8%. This SiO_2 value is much lower than that found in the SCDE used in RC10, resulting in a concrete with worse compressive strength behavior compared to RC10. Moreover, the composition with SCDE showed a greater increase in compressive strength at 28 and 90 days compared to RC10, which is also due to the high amount of SiO_2 present in the SCDE (90.7%) used by this author.

However, it should be noted that at the end of 90 days the increase in compressive strength of RC10 is close to that of the composition with 5% SCDE, showing that although RC10 does not show such rapid increases in compressive strength, it reaches values higher than REF and an increase that is close to the concrete with SCDE obtained in the research of Rodriguez et al.[26]

Despite the SCDE used in this study does not have a very high amount of silica, the compressive strength increases at the end of 90 days are very close to those found in studies using pozzolans with a higher amount of silica.[26,31,32,39] The cement or sand replacements, also shows higher compressive strength results than the reference concrete over time. The comparison of results with those of these authors can be seen in Figure 12.

The TG and DTG curves of the REF, RC10 and RS5 concretes at 28 days are shown in Figure 13. Three inflection points can be identified in TG curves at approximately 95, 440, and 730°C. The first one is associated with the evaporation of physically bound water[83] and the disintegration of cement and SCDE hydration products.[16,63] The second inflection point is associated with the dehydration of $Ca(OH)_2$, and the last one to the decomposition of $CaCO_3$, which primarily comes from the limestone aggregate that was incorporated into the

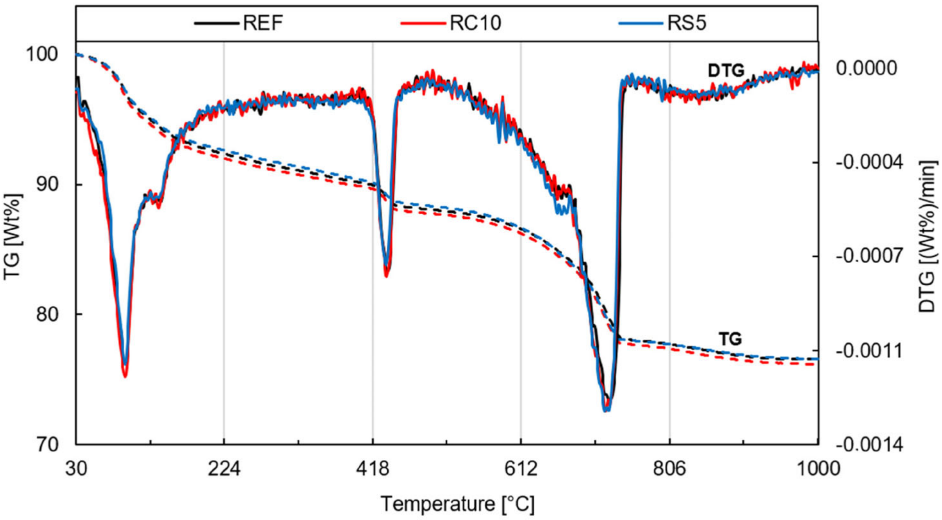


FIGURE 13 TG/DTG curves of REF, RC10 and RS5.

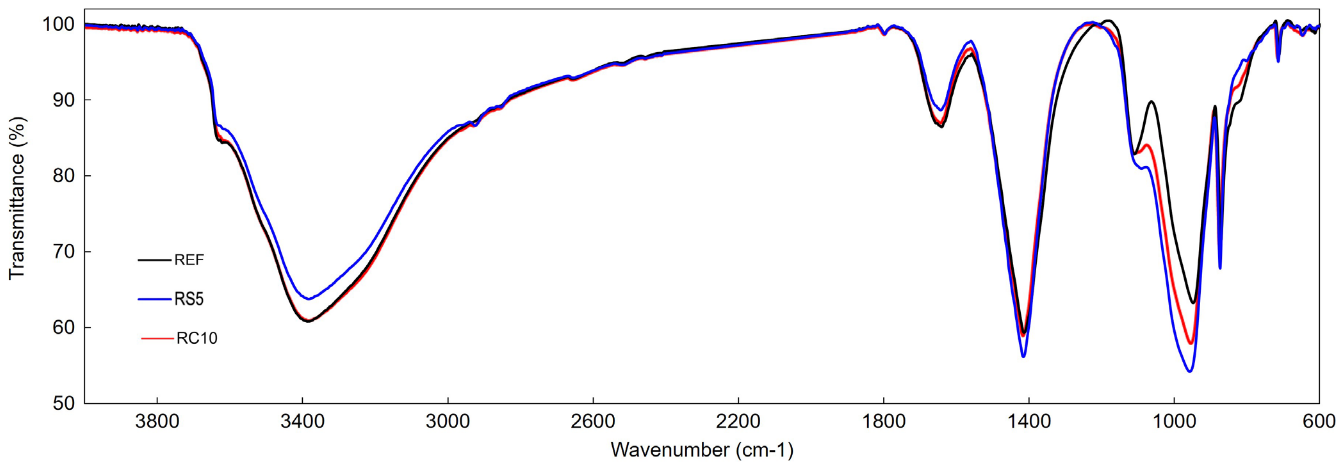


FIGURE 14 FTIR curves of REF, RC10 and RS5.

sample after the concrete compression test.[16,63,83] No significant differences are observed in the TGA results between the three samples. The mass losses associated with the decomposition of $Ca(OH)_2$ show no appreciable variation with the addition of diatomaceous earth, indicating that after 28 days, there is no significant pozzolanic reaction.

Figure 14 shows the FTIR spectrum of REF, RC10 and RS5 at 28 days. In REF and RC10, the peaks at 3400 and 1640 cm^{-1} are identical. The first peak corresponds to the stretching vibration of the —O—H groups in aluminosilicates and/or the moisture present in the materials, while the second peak corresponds to the bending of the H—O—H bonds in water molecules. Due to the lower water-to-cement ratio, the RS5 sample shows lower transmittance at both peaks. At 1400 cm^{-1}, corresponding to the stretching vibration of the C—O group in the carbonate, all samples are similar. The carbonates mainly originate from the aggregate, which, in filler form, is present in the powdery samples. The peak at 1109 cm^{-1}, attributed to the Si—O—Al vibration, is similar in all the samples. The main difference between samples occurs at 947 and 871 cm^{-1}, which seem to correspond to the presence of SiO_2. As indicated in Section 2.3, the SEM images show unreacted SCDE remains, acting as a filler. Another difference between samples can be observed around 850 cm^{-1}, which could be attributed to the interaction of SCDE with the cement matrix, influencing the formation or density of the C—S—H gel, the main hydration product.[16,31,63,72,83–86]

Figure 15 shows SEM and EDS images of RC10 and RS5, revealing a substantial reduction compared to REF. SEM analysis highlight the presence of unreacted SCDE remains and $Ca(OH)_2$ in the RC10 and RS5 mixtures, as illustrated in Figures 9 and 15.

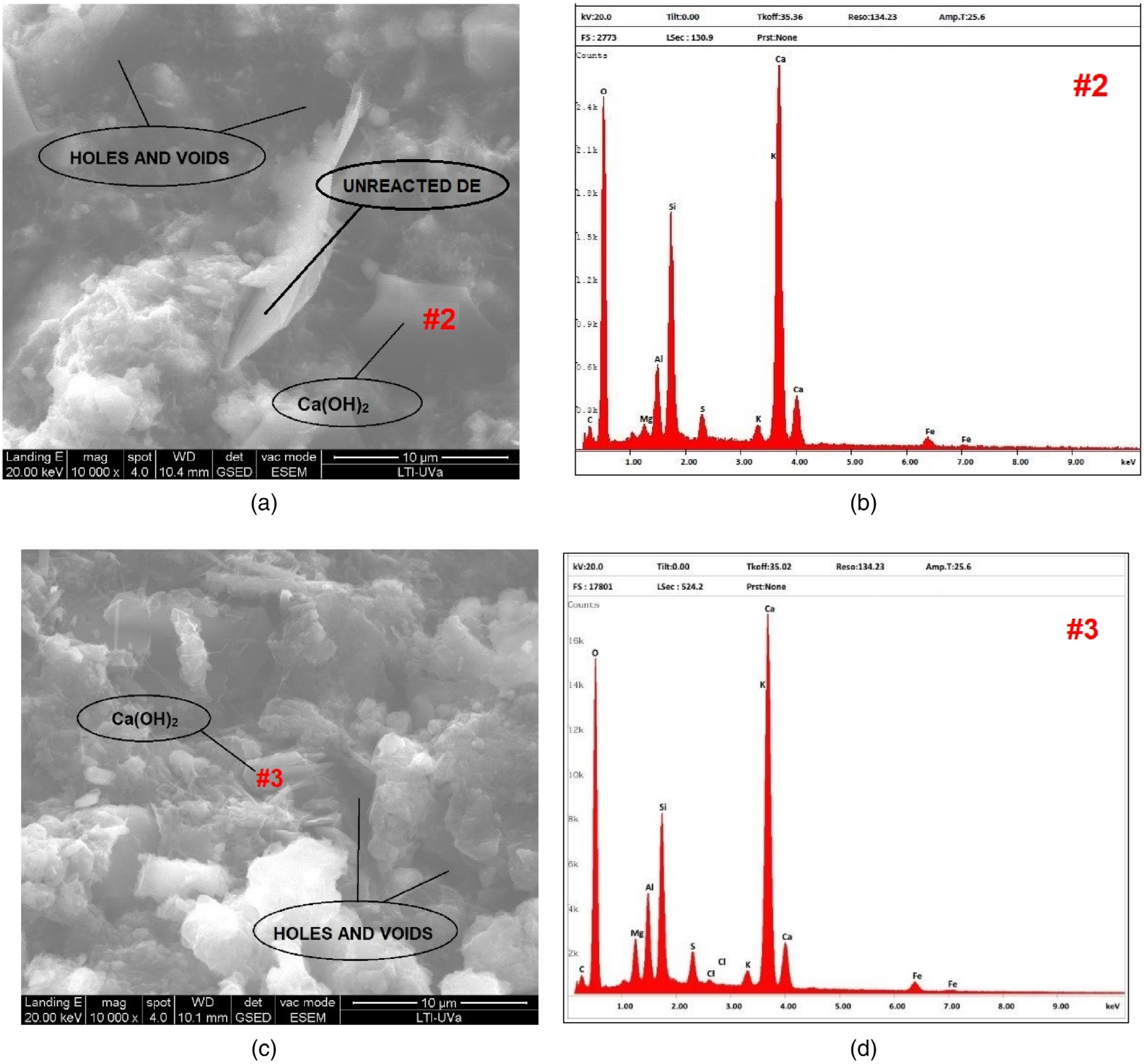


FIGURE 15 (a) SEM and (b) EDS images of RC10; (c) SEM and (d) EDS images of RS5.

3.2 | ANOVA results

The results of the statistical analysis performed on the compressive strength of the specimens over time are presented in Table 7, where "*T*" is the testing days, "CC" is concrete composition, "CI" is the 5%–95% confidence interval (MPa), and "S-W" is the Shapiro–Wilk test.

The statistical analysis over time shows that at 7 days the normality of the obtained values for the different compositions was confirmed using S-W test, with *p*-values greater than 0.05. The homogeneity of the variances of the compositions was confirmed through Levene's test, which yielded a *p*-value of 0.104, greater than 0.05. With the assurance of normality and homogeneity assumptions, a "one-way" ANOVA was applied, which showed a *p*-value lower than 0.05. Consequently, the "null hypothesis" was rejected. This indicates that the average compressive strength values of the different compositions are completely independent at 7 days of age, and the introduction of SCDE into the composition directly influences the concrete's compressive strength results. The comparison between means was performed using the Tuckey HSD test. In the comparisons performed, *p*-values lower than 0.05 were found. This implies that there is no similarity between the means of REF, RC10 and RS5 at this age.

TABLE 7 Statistical analysis over time.

		CI					Multiple comparisons	
T	CC	Lower bound	Upper bound	S-W *p*-value	Levene *p*-value	ANOVA *p*-value	Compared compositions	*p*-value
7	REF	16.33	17.00	0.146			REF–RC10	<0.001^{T}
	RC10	14.23	14.86	0.069	0.104	<0.001^{C}	REF–RS5	<0.001^{T}
	RS5	17.65	18.11	0.682			RC10–RS5	<0.001^{T}
28	REF	20.26	20.62	0.931			REF–RC10	0.833^{D}
	RC10	20.19	21.02	0.192	0.003	<0.001^{W}	REF–RS5	<0.001^{D}
	RS5	23.68	24.61	0.967			RC10–RS5	<0.001^{D}
90	REF	23.13	23.46	0.166			REF–RC10	<0.001^{D}
	RC10	25.81	26.64	0.142	<0.001	<0.001^{W}	REF–RS5	<0.001^{D}
	RS5	30.44	31.26	0.319			RC10–RS5	<0.001^{D}
180	REF	25.87	26.78	0.871			REF–RC10	0.005^{B}
	RC10	29.06	30.28	0.030	0.931	<0.001^{K}	REF–RS5	<0.001^{B}
	RS5	33.96	34.85	0.211			RC10–RS5	0.005^{B}
360	REF	26.41	28.46	0.166			REF–RC10	0.001^{B}
	RC10	29.39	32.89	0.054	0.051	0.002^{K}	REF–RS5	<0.001^{B}
	RS5	34.07	36.61	0.060			RC10–RS5	<0.001^{B}

Note: C, one-way ANOVA; D, Dunnett T3; E, Bonferroni Dunn; K, Kruskal–Wallis; T, Tuckey HSD; W, Welch ANOVA.

At 28 days, the strength values obtained were verified as normally distributed, as all compositions had p-values greater than 0.05 in S-W test. However, there was no homogeneity of variances, as evidenced by Levene's test with a *p*-value of 0.03, less than 0.05. Failing the assumption of homogeneity of variances, Welch's ANOVA was applied, showing a *p*-value lower than 0.05. This rejection of the "null hypothesis" indicates that the means of the compression values at 28 days of age are entirely independent. In this case, the Dunnett's T3 test was used for mean comparisons. For both REF–RS5 and RC10–RS5 comparisons, the *p*-value was below 0.05, which proves that there is no similarity between these means. Regarding the REF–RC10 comparison, the *p*-value was 0.833, surpassing 0.05, which meant that, at 28 days, significant similarities in the compressive strength results between these two compositions.

At 90 days of testing, the normal distribution of the compressive strength values was confirmed with *p*-values greater than 0.05 for all compositions. Similar as occurred at 28, at 90 days, the compressive strength results did not exhibit homogeneity of variances, showing a p-value lower than 0.05. As the assumption of homogeneity of variances failed, a Welch ANOVA was applied revealing a *p*-value lower than 0.05. Consequently the "null hypothesis" was rejected. The multiple comparison between means was performed using Dunnett T3, and the test results indicate that for all the compositions compared, the *p*-value was less than 0.05, which means a lack of similarity between the means of REF, RC10 and RS5 and significant differences between the three compositions at this age.

At 180 days, homogeneity of variances was assumed, with a *p*-value greater than 0.05. However, normal distribution was not verified in the compressive strength values of the RC10 composition. The S-W test shows normality in the REF and RS5 compressive strength values, with *p*-value of 0.871 and 0.211 respectively, but it does not show normality in the RC10 specimen data, with a *p*-value of 0.03, less than 0.05. As the assumption of normality failed, the non-parametric Kruskal–Wallis test was applied to analyze the means obtained from the three compositions. The test showed a *p*-value of less than 0.05, meaning that at 180 days the "null hypothesis" is still rejected. The comparison between means was performed using the Bonferroni Dunn test, where it can be seen that for all comparisons between compositions the *p*-value is less than 0.05, so there is no similarity between the REF, RC10 and RS5 means at this age.

Lastly, at 360 days, the results obtained show normal distribution with *p*-values greater than 0.05 in all compositions. The homogeneity of variances was also verified with a *p*-value greater than 0.05. Despite the assumptions of normality and homogeneity being guaranteed, there are uncertainties about the normal distribution of the compressive strength values due to the sample data size being less than 15 samples. Therefore, the non-parametric Kruskal Wallis test was applied, revealing a *p*-

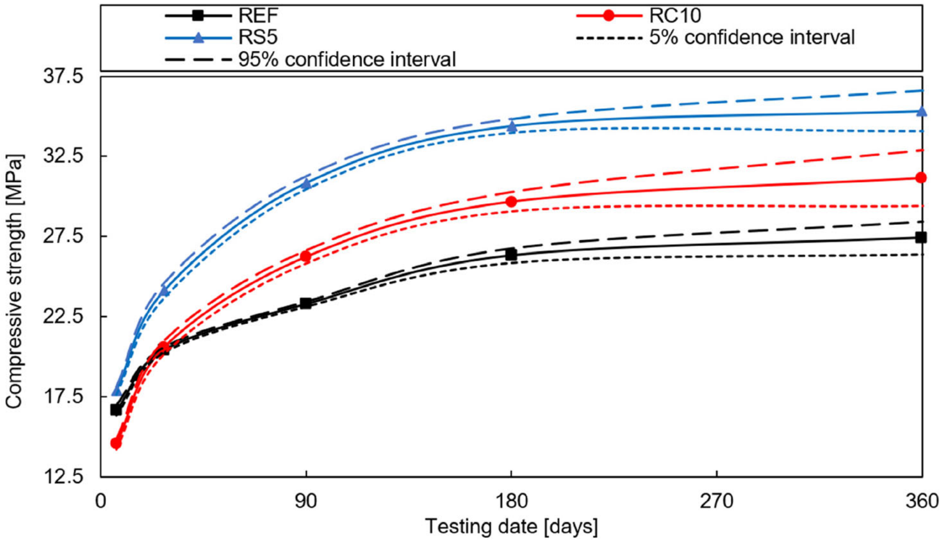


FIGURE 16 Five to 95% confidence interval of compressive strength over time.

value lower than 0.05, and thus rejecting the "null hypothesis". The comparison between means was performed using the Bonferroni Dunn test, with this test showing a *p*-value lower than 0.05 in all comparison cases, which means that there is no similarity between all means at the end of 360 days.

For the three compositions analyzed, the statistical analysis also provided a 5%–95% confidence interval of compressive strength over time. These results can also be visualized in Figure 16. For this confidence interval, with a 95% confidence level, the statistical analysis over time verified that there are significant differences between the means of the three compositions so they are therefore completely different materials.

The shape of the REF curve follows a normal pattern, with an early-age rapid strength gain followed by a slower development of strength over the long term, until it approaches an asymptote that represents the final compressive strength of the concrete.[65,87] RS5 and RC10 show the same trend, with a higher slope with respect to REF at early age, more pronounced in the case of the RS5.

4 | CONCLUSIONS

The possibility of introducing SCDE into concrete compositions by replacing cement or sand was investigated in this study. It focuses on replacing 10% of SCDE by cement (RC10) and 5% of SCDE by sand (RS5), comparing the results of the workability of fresh concrete, density of hardened concrete and compressive strength over 7, 28, 90, 180 and 360 days with a reference composition (REF), and performing a statistical analysis on the compressive strength results over time. According to the obtained results and the discussion presented in this work, the following conclusions can be drawn:

- The replacement of 10% of cement by SCDE in RC10 results in a decrease in initial compressive strength after 7 days of 12.8%. However, it exhibits a faster increase in strength over time compared to REF, which stabilizes after 90 days, with compressive strength values approximately 13% higher than those of the REF.
- Comparing with other authors, it is concluded that the effect of cement replacement by CDE or SCDE depends not only on the replacement percentage but also on the pozzolanic activity, primarily concerning the percentage of SiO_2 and the previous calcination treatment, rather than whether or not it is based on a by-product.
- The replacement of 5% of sand by SCDE in RS5 enhances compressive strength compared to the REF. At 7 days, values are 7.13% higher than the reference, and after 90 days, they tend to stabilize around 30% higher than the reference values.
- The inclusion of SCDE in concrete mixtures leads to a reduction in workability, as evidenced by a 30 mm decrease in slump for the RC10 composition and a 15 mm decrease for RS5, compared to REF, due to the high porosity and water absorption properties of SCDE. For effective implementation, the proper use of superplasticizers may be required.
- The density of RC10 and RS5 concretes was lower when compared to REF.
- The study shows the evolution of average and characteristic compressive strength values over time for REF, RC10 and RS5 up to 360 days, which does not show a long-term decrease.

- Replacements with CDE and SCDE yield similar improvements in concrete compressive strength, which highlights the possibility of valorization of SDE, a by-product from the wine industry, in concrete production, reducing landfill waste and contributing to the development of sustainable concretes.
- The statistical analysis confirms the influence that SCDE has on the concrete compressive strength up to 360 days. Materials with statistically different behavior were found over time, for which the 5%–95% confidence interval for compressive strength is displayed.
- This study highlights the potential of SCDE not only as a cement substitute but also as a partial sand replacement, opening the door to more extensive applications in eco-friendly concrete production. Future research could explore both cement and sand replacements in mixed proportions, to maximize compressive strength around the 90-day mark while reducing cement content. This approach could help optimize the balance between sustainability and performance, contributing to the development of more durable and environmentally friendly concrete.

ACKNOWLEDGMENTS

This article is a result of the project "BacchusTech—Integrated Approach for the Valorisation of Winemaking Residues" (POCI-01-0247-FEDER-069583), supported by the Competitiveness and Internationalization Operational Programme (COMPETE2020), under the PORTUGAL 2020 Partnership Agreement, through the European Regional Development Fund (ERDF).

The support of the Department of Mechanical Engineering at the University of Salamanca is also acknowledged, as it was essential for conducting the complementary tests that complete this publication.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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How to cite this article: Magalhães LJR, Ferreira DM, Ramos-Gavilán AB. Enhancing concrete sustainability with spent diatomaceous earth from the wine industry: Long-term experimental and statistical analysis. Structural Concrete. 2024. https://doi.org/10.1002/suco.202400889