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Study of the effects of partial cement replacement with spent diatomaceous earth from the brewing industry in mortar

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Abstract. The present study evaluates the effect of partial replacement of cement in mortars with spent diatomaceous earth (SDE) from beer filtration, aiming to explore sustainable alternatives for civil construction. Three compositions were tested: one with 15% cement replacement by SDE, another with 5% sand replacement by SDE, and a reference composition without SDE addition. The diatomaceous earth (DE), after being used in beer filtration until saturation, was calcined in a furnace to eliminate organic matter and subsequently incorporated into the mortar composition. This research, as an extension of a previous study using residues from wine filtration, evaluates the impact of this replacement on the mechanical properties of the mortar, including compressive strength, tensile strength, and consistency. A detailed characterization of the spent diatomaceous earth was performed through X-ray diffraction and laser granulometry, enabling a better understanding of its structural properties.

1 Introduction

The uncontrolled extraction of natural resources and excessive carbon dioxide emissions, are critical concerns for modern society, driving the search for alternative materials with the potential to replace cement and sand in construction applications. In this context, spent diatomaceous earth (SDE), a byproduct of beverage filtration, presents significant potential for sustainable reuse.

Diatomaceous earth is a sedimentary mineral, derived from fossilized diatom algae [1], composed of approximately 80% amorphous silica [2]. It exhibits unique physical and chemical properties, including high permeability and porosity, a porous structure, small particle size, exceptional adsorption capacity, elevated silica content and a large surface area [3]. To enhance its utility, SDE must undergo calcination, to eliminate all organic matter and impurities, with optimal conditions for this thermal decomposition process having been tested by several authors [4], [5].

SDE, in its various forms, serves a wide range of industrial applications, from construction to environmental remediation [6], showcasing its efficiency and prompting extensive research into its additional uses. SDE has been applicated as a biosorption agent [7], as an herbicide [8], for mineral carbon adsorbents [9], and in vermicomposting [10]. Another prominent use is in the filtration of fermented beverages like wine and beer, due to its low operation cost. However, once saturated with organic material, it loses its filtration capacity necessitating disposal and incurring significant costs.

After use, SDE retains water and organic matter, tripling its initial weight and making disposal the most expensive stage of the process [11]. Large breweries can generate up to 30,000 kg of SDE per month [12]. To address these challenges, the industry is exploring more sustainable waste management solutions, with the potential of using SDE as a substitute for cement and sand in construction materials offering a promising alternative.

This material serves as complementary source of silica in the cement industry, contributing to various aspects of the manufacturing process. Its chemical composition, fine grain size, and low fusion temperature offer advantages, including reduced feed and clinker grinding, facilitated and homogenized reactions, energy savings and enhanced final cement properties. When incorporated into concrete and mortars, diatomaceous earth improves plasticity and hardening characteristics, promotes cohesion and homogeneity, enhances functionality, and reduces efflorescence [13].

2 Experimental Program

2.1 Materials

The spent diatomaceous earth (SDE) used in this study was obtained as a byproduct of the beer filtration process from Reden Brewery (Paraná, Brazil), provided in a saturated state with organic residues. To adapt its physicochemical properties and eliminate organic matter, the SDE underwent a thermal treatment in two stages:

1. Drying in an oven: Conducted at 50°C for 24 hours under static conditions until the sample reached constant mass, eliminating residual moisture associated with the filtration process.

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2. Calcination in an industrial furnace: Carried out at 700°C for 3 hours to thermally degrade remaining organic compounds. The completeness of the process was confirmed by mass stabilization after an additional hour at constant temperature, resulting in a 7% mass loss relative to the dried SDE. This loss was directly attributed to volatile organic matter decomposition, confirming the efficiency of the thermal treatment.

The particle size distribution of the spent diatomaceous earth (SDE) was determined by laser analysis using a Bettersizer S3 Plus instrument with a detection range of 0.01 µm to 3500 µm. The sample, previously dried and calcined, was dispersed in ethanol under mechanical agitation (1600 rpm) and ultrasonic treatment for 2 minutes to disaggregate particles. The results indicated a median diameter (D_{50}) of 35.95 µm, with 10% of particles below 12.42 µm (D_{10}) and 90% below 114.1 µm (D_{90}). The specific surface area (SSA) was 85.78 m²/kg, and the span index, calculated as $(D_{90} - D_{10})/D_{50}$, reached 2.83, reflecting a broad particle size distribution, as illustrated in Figure 1. The granulometric distribution of the brewery SDE closely matched that of SDE from the wine industry [14], shown in Figure 2.

Figure 1. Particle size distribution of SDE.

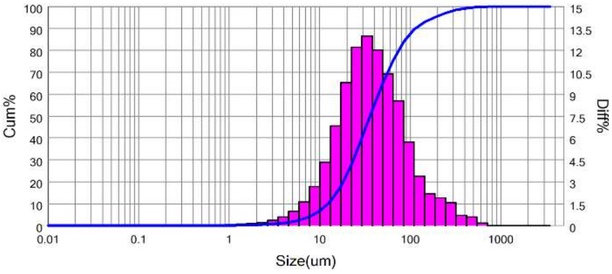
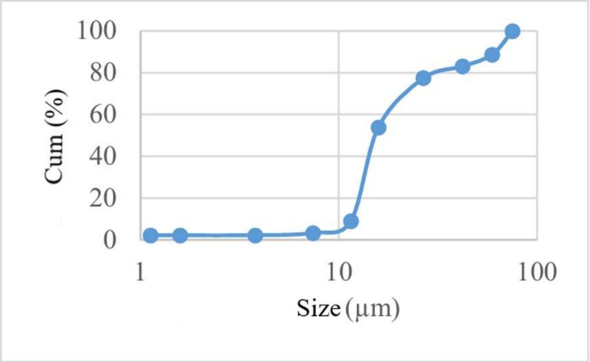


Figure 2. Particle size distribution of SDE from wine filtration [14].



The chemical composition of the SDE was determined by X-ray fluorescence (XRF) using a Bruker S8 Tiger spectrometer equipped with a rhodium (Rh) tube and a wavelength dispersive system (WDS). For analysis, the samples were homogenized by manual quartering, pulverized in an agate planetary mill, and dried at 105°C for 2 hours. The loss on ignition (LOI) was determined after calcination at 1000°C for 5 hours. The results revealed a predominance of SiO_2 (89.62%), followed by Al_2O_3 (3.71%), in addition to a low residual organic matter content (LOI = 0.28%), confirming the effectiveness of the previous calcination, which resulted in a 7% LOI. Table 1 presents the complete chemical composition of the SDE. The high amorphous silica content (89.62%) and fine particle size (D_{50} = 35.95 µm) of the SDE suggest potential pozzolanic reactivity, which could enhance cement replacement efficiency by contributing to the formation of calcium silicate hydrate (C-S-H) gel. While this study did not directly assess pozzolanic activity, future work could include tests to validate this hypothesis.

The cement used was Portland CP V-ARI, selected due to its minimal incorporation of supplementary materials and a maximum limestone filler content limited to 10%, as established by the ABNT NBR 16697:2018 standard [15], aiming to reduce interferences in the cementitious matrix. Table 1 presents the complete chemical composition of the CP V-ARI batch used, as provided by the cement manufacturer.

Table 1. Chemical Composition of the SDE and CP V-ARI.

Chemical Composition (%)	SDE	CP V-ARI
SiO_2	89,62	18,66
Al_2O_3	3,71	4,4
Na_2O	2,73	-
Fe_2O_3	1,50	3,13
CaO	0,72	61,92
K_2O	0,58	-
MgO	0,35	3,92
SO_3	0,30	2,77
P_2O_5	0,27	-
TiO_2	0,19	-
Free CaO	-	1,85
LOI (%)	0,28	3,51

The sand used was standard sand that complies with ABNT NBR 7214:2015 [16]. The additive used to maintain the fluidity of mortars with the addition of SDE, similar to that of the reference mortar, was the TEC-FLOW 8000 superplasticizer, which has a high water-reducing power.

2.2 Methods

The reference mortar was formulated with a mass ratio of 1:3:0.5 (cement:sand:water), as established in preliminary studies by Renan Zolin [14], who investigated mortar with partial replacement of cement and sand by SDE from wine filtration. In that study, the water/cement ratio had to be increased to 0.61 due to the loss of workability caused by SDE addition. However, the current study aimed to correct mortar fluidity by incorporating a superplasticizer admixture, enabling direct comparison of the results.

To evaluate the effect of calcined spent SDE, three compositions were developed:

1. Reference Composition (REF): No addition of diatomaceous earth.
2. C15R: 15% replacement of cement with spent diatomaceous earth.
3. S5R: 5% replacement of sand with spent diatomaceous earth.

The addition of SDE, due to its high fineness (median diameter of 35.95 µm), significantly reduced mortar workability, increasing water demand. To ensure comparability among formulations, consistency was standardized using the flow table test, following the ABNT NBR 13276:2016 standard [17]. All compositions were adjusted to achieve a spread diameter of (160 ± 5) mm by adding a superplasticizer admixture, with experimentally determined dosages: 0.27% by mass of cement for the C15R composition and 0.36% for S5R. The higher superplasticizer demand in S5R is attributed to the need for improved particle dispersion in the altered granular skeleton, where SDE's fine particles filled voids between sand grains, increasing surface area and requiring more admixture to maintain flowability.

The mortars were subjected to compressive and flexural strength tests, as prescribed by the ABNT NBR 13279:2005 standard [18]. For each composition, three prismatic specimens (160 mm × 40 mm × 40 mm) were molded using the material quantities specified in Table 2.

Table 2. Mortar compositions.

Sample	Cement (g)	Sand (g)	SDE (g)	Water (g)	Additive (g)	Ad/Cem Ratio
REF	450	1350	-	225	-	-
C15R	382,5	1350	67,5	225	1,05	0,23%
S5R	450	1282,5	67,5	225	1,62	0,36%

The dry components were mixed in a mechanical mixer following the guidelines of ABNT NBR 16541:2016 [19]. After casting, the specimens were stored in a humidity chamber under controlled conditions: temperature of 23 ± 2 °C and relative humidity of 65 ± 5%, for curing periods of 7 and 28 days.

The mechanical strength tests were conducted according to the NBR 13279:2005 standard [18] using a universal testing machine. Flexural strength was evaluated at 7 days of curing, while compressive strength was tested at 7 and 28 days of curing.

3 Results

The flexural and compressive strength tests revealed distinct behaviours among the formulations with partial replacement of cement (C15R) and sand (SSR) by spent diatomaceous earth (SDE), compared to the reference mortar (REF). The results are presented in Table 3 and discussed in detail below.

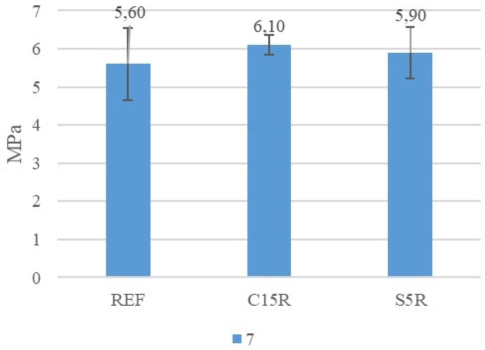
Table 3. Flexural and compressive strength.

Sample	Flexural strength	Compressive strength	
	7 days (MPa)	7 days (MPa)	28 days (MPa)
REF	5,6	38,0	40,9
C15R	6,1	35,3	40,0
S5R	5,9	43,1	46,9

3.1 Flexural Strength

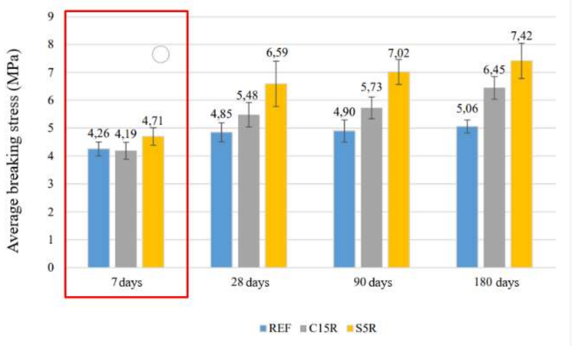
After 7 days of curing, both modified formulations exhibited superior performance compared to the reference mortar, as illustrated in Figure 3.

Figure 3. Flexural strength at 7 days.



This initial gain is consistent with the results obtained in a previous study with SDE from wine filtration [14], where the substitution of sand with SDE also resulted in an increase in flexural strength, as seen in Figure 4:

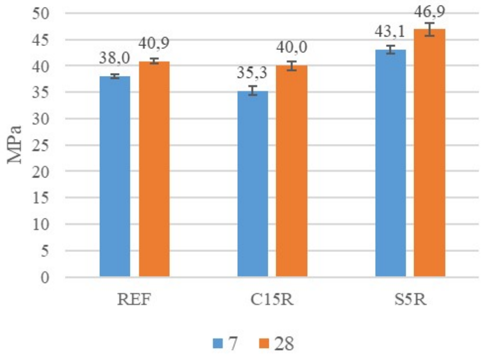
Figure 4. Flexural strength of mortar with incorporation of wine filtration SDE [14].



3.2 Compressive Strength

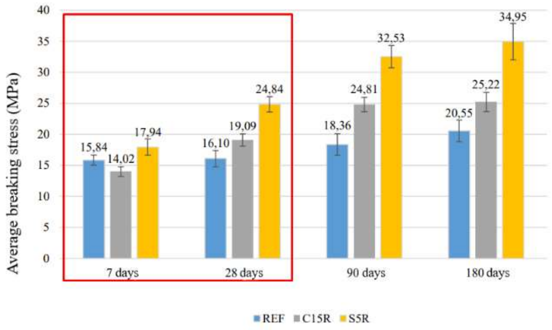
The results of the compressive strength test, as illustrated in Figure 5, show that the S5R composition demonstrated the best results at both 7 and 28 days, with values exceeding those of the reference mortar and the C15R composition, while the C15R was 2.2% lower than the REF at 28 days, though still within an acceptable variation margin.

Figure 5. Compressive strength at 7 and 28 days.



The superiority of the S5R formulation corroborates previous studies with wine-derived SDE [14], in which the substitution of sand with SDE also led to strength gains, as seen in Figure 6. The C15R formulation with wine SDE [14] achieved compressive strength higher than the REF at 28 days, unlike the current study, where it was lower.

Figure 6. Compressive Strength of Mortar with incorporation of wine SDE [14].



4 Conclusion

This study demonstrated that the SDE from beer filtration shows viable potential for partial replacement of cement (15%) and sand (5%) in mortars, aligning with the sustainability principles in civil construction. However, it is crucial to note that the calcination process (700°C for 3 hours) required to eliminate organic matter from the residue is energy-intensive, which may offset some environmental benefits. Comparing the results with the previous study using SDE from wine filtration [14], critical similarities and differences were observed:

In the S5R formulation (5% replacement of sand with SDE), the 28-day compressive strength (46.9 MPa) surpassed that of the reference mortar (40.9 MPa), just as in the study with wine SDE, where the substitution of sand also resulted in significant strength gains. This suggests that SDE, regardless of its origin, acts as a filler material, improving the packing of the granular skeleton due to its fineness (D_{50} = 35.95 µm) and high specific surface area (85.78 m²/kg).

The replacement of 15% of cement with beer filtration SDE (C15R) resulted in a slightly lower compressive strength than the reference at 28 days (40.0 vs. 40.9 MPa), differing from the previous study with wine SDE, where the same replacement showed superior performance. This divergence may be attributed to variations in the materials used.

Concerning flexural strength, both formulations with SDE from the brewing industry (C15R and S5R) outperformed the reference mortar at 7 days, corroborating the results obtained with SDE from the wine industry.

The high fineness of the SDE required the addition of a superplasticizer (0.27–0.36% of the cement mass) to maintain fluidity, a pattern also observed in the previous study. This underscores the need for rheological adjustments in formulations containing SDE, regardless of its origin.

The 5% replacement of sand with SDE not only maintains the mechanical properties but also reduces the demand for natural resources, while cement replacement offers a pathway to mitigate CO_2 emissions. The similarity between the results for SDE from the brewing and wine industries suggests that filtration residues from different industries can be standardized for cementitious applications.

Complementary studies should investigate the porosity and durability of mortars with SDE, evaluate their performance in humid environments to ensure long-term stability, and assess the environmental impact of the SDE calcination.

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