





## Article

# Properties of Cement-Based Materials Incorporating Ground-Recycled Diatom

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**Abstract:** This research investigates the use of recycled diatomaceous earth (diatomite) from the wine, beer, and oil industries as supplementary cementitious materials in cement-based mixtures. This study aims to reduce embodied energy and promote circular economy practices by incorporating these industrial by-products. The research evaluates the compressive strength, durability, and pozzolanic activity of the mixtures over 7, 28, and 90 days of hydration. The results demonstrate that uncalcined diatoms from wine and oil showed lower compressive strength than natural diatomite, whereas calcination at 500 °C significantly improved performance. Beer diatoms exhibited the lowest mechanical strength because of the organic matter content in their composition. The incorporation of quicklime failed to induce pozzolanic activity in uncalcined diatoms; however, calcination at 500 °C led to improved long-term performance, highlighting the importance of heat treatment for activating diatoms' pozzolanic properties. This study concludes that recycled diatoms, particularly when calcined, have potential as sustainable cementitious materials.

**Keywords:** diatom; recycled diatomite; wine; beer; oil; compressive strength; water demand; setting time; circular economy



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## 1. Introduction

Diatomite, also known as diatomaceous earth, dyahydro, celite, tripolite, kieselguhr, or moler (impure Danish form), is a siliceous mineral formed by microscopic algae fossils (diatoms) via sedimentation. The country that leads the exploitation of this resource is the USA, which produced 830,000 tons in 2021, representing 36% of the global production that year. Other countries with remarkable figures are Denmark, Turkey, and China, with a global share of diatomite production of 17%, 9% and 6%, respectively [1]. The production of diatomite in Spain over the last years has been almost constant and around 60,000 tons per year [2]. The most common use of diatomite is the filtration of water, beer, wine, liquors, and oils, contributing to the elimination of contaminants [1]. On some occasions, the diatomite is calcined before its use as a filter [3].

Alternatively, the use of diatomite as a supplementary cementitious material is increasing its popularity, becoming the second-ranked application in some countries, such as the USA [1], because of its pozzolanic properties and low embodied energy. It should be noted that the use of supplementary cementitious materials is encouraged all around the world as the production of Portland clinker still generates important amounts of greenhouse gas emissions. For instance, in Spain, the CO<sub>2</sub> emissions intensity of gray Portland clinker was 0.827 tons per ton of clinker in 2017; however, more research is still needed to completely

understand the performance of alternative binders and fillers and, thereafter, decrease the proportions of Portland clinker in new types of cement. In this context, many scientific publications have addressed the feasibility of using diatomite powder as a supplementary cementitious material.

Generally, the methodology followed in these works includes substituting Portland cement by weight and in low proportions, typically up to 20%, without compensating the water demand. Additionally, diatomite is usually pretreated to enhance its reactivity by crushing and grinding [4], drying [5], calcination at different temperatures, or exposure to some chemicals such as silicic acid [6]. Regarding calcination, the most common temperatures used are between 850 and 1000 °C [7–10], although some studies consider lower temperatures, between 600 and 700 °C as more efficient [6,11].

Generally, the substitution of Portland cement for diatomite decreases the strength of cementitious materials at an early age due to the dilution effect; however, some studies have reported similar short-term strength values when using low replacement ratios (around 5%) because of the filler effect, as diatomite powder can be finer than Portland cement [12]. In any case, there is a strong agreement regarding the possibility of attaining similar or even superior compressive strength in the long term because of pozzolanic reactions [7,12–15]. Some studies have supported this hypothesis by thermogravimetric analysis, measuring the decrease in Portlandite content of cement pastes [12,14] and lime pastes [16]. Complementarily, some research works point out the internal curing effect as a contributor to strength development in diatomite blended cements [9,17,18]. The positive effect of diatomite as a supplementary cementitious material is maintained even after heating concrete at temperatures up to 1000 °C; however, only low replacement ratios (< 15%) lead to satisfactory strength results, as the dilution of Portland cement compensates any other positive effects with higher contents [4,15,17,19]. Parallel conclusions have been reached regarding splitting-tensile strength [20].

The replacement of OPC by diatomite increases the water demand (workability decreases) of cement-based mixtures [4,12,15,20,21]. This phenomenon has been pointed out as the main drawback to be overcome regarding the use of this material in concrete production [10]. The setting times are delayed when the water demand of diatomite is compensated [14] but shortened when it is not [10,15]. In any case, this effect can be slight [12]. The use of diatomite as a supplementary cementitious material decreases the heat release of concrete [11] and decreases its weight [5,15]. Furthermore, positive effects over durability properties have been detected, such as higher resistance to sulfate attack and freeze–thaw cycles [5] and a decrease in the chloride diffusion coefficient [19].

Similar conclusions have been drawn when replacing hydraulic lime instead of OPC in concrete mixtures: the presence of diatomite increases the mechanical strength because of pozzolanic reactions, decreases the density, and improves the durability by increasing the resistance to acid and sulfate attack [22]. The same beneficial effects are detected when replacing fly ash in blended hydraulic lime mixtures [23]. Diatomite has also been compared with other pozzolanic powders in OPC-based mixtures. For instance, Ouyang et al. replaced quartz powder with a similar silica content, attaining a faster setting and higher strength [24]. Another example is the comparison with silica fume carried out by Sun et al. [9], where higher strength and durability against freeze–thaw cycles were attained despite the coarser particle size of diatomite.

Other authors have included diatomite as a supplementary cementitious material in ternary blends together with OPC and metakaolin [8], ground granulated blast furnace slag, fly ash, and limestone filler [4,15]. Geopolymers and alkali-activated materials have also been proven to admit diatomite as an alternative silica source to sodium silicate [25–27] and as a replacement for fly ash as a precursor [28]. Furthermore, some research works propose the use of diatomite as an aggregate in concrete [29,30].

Given the promising results of natural diatomite as a supplementary cementitious material, recycled diatomite, coming from the filtration of different liquids, has also been proposed to play the same role. In this case, not only the embodied energy of cementitious mixtures is decreased, but the reuse of a waste sub-product contributes to the circular economy. It must be noted that diatomite has a limited life as a filter, as its pores end up filled with organic matter, which is costly to clean. The main sources of diatoms to be recycled as supplementary cementitious materials are the filtration of wine [31–33] and beer [32–37]. As for the case of natural diatomite, several pretreatments have been proposed to improve its performance, including washing [37], grinding [31], and calcination. The latter has been proven to be more beneficial as it can eliminate a high proportion of the organic matter remaining in the filtering diatomite. Several authors have tested different calcination temperatures: 200 °C [35], 500 °C [31], 650 °C [32], 800 °C [36], and 900 °C [33].

The obtained results show a decrease in the workability of cementitious mixtures when incorporating the recycled diatomite [31]; however, the strength can be increased due to beneficial phenomena also reported when using natural diatomite, such as the filler effect [34,36]. Only if organic matter is not conveniently erased, can the strength development be inhibited [33]. Furthermore, the use of diatomite instead of OPC increases concrete drying shrinkage [35], reduces thermal conductivity [35], increases water absorption due to capillary action [33], and increases the resistance to chloride ingress [33]. Finally, it can be remarked that some studies have compared recycled diatomite with other supplementary cementitious materials, such as rice husk ash, obtaining slightly lower strength [32].

Certainly, most of the scientific literature focuses on the study of fresh and mechanical properties, as they are the basic requirements to be fulfilled; however, there is still some scarcity regarding the study of long-term and durability properties when using both natural and recycled diatomite. The few works dealing with this issue conclude that the use of diatomite can have a beneficial effect on several long-term properties (freeze–thaw resistance [9,19], chloride ingress permeability [19,33], and resistance to acid and sulfate attack [22]). Nevertheless, it must be considered that diatomite is a porous material and, therefore, increases the water absorption of concrete [33]. Then, special attention should be paid to any possible durability issues.

Despite the consistency in certain patterns regarding the influence of natural and recycled diatomites in cement-based materials, it must be considered that the features of this by-product strongly depend on the source; therefore, basic physical and chemical characterization tests must be performed prior to the use of diatoms to anticipate their performance properly. Specifically, the chemical composition and weight loss of this waste material when exposed to different temperatures must be carefully evaluated. This way, the composition of diatomite from different sources can be regulated.

Some research works have also surpassed the cement stage and scaled their investigations to actual construction materials. For instance, Li C. et al. [4], Li J. et al. [15], Lv Z. et al. [17], Zhang H. et al. [19], and Domínguez-Santos D. et al. [34] tested basic properties on concrete with diatoms. Moreover, some more specific mixtures have been tried, such as previous concrete by Liu J. et al. [38]. Gerengi H. et al. [39] carried out their research on reinforced concrete, which means a further step. Finally, some authors have casted concrete elements ready to be placed on the construction site, such as concrete pipes, as it can be seen in Figure 1 [33].

On the one hand, dealing with diatomite as a waste material from the food industry has an important negative impact (landfilling). On the other hand, the literature reviewed shows promising results regarding the substitution of OPC for recycled diatomite. Given this background, this research work proposes the characterization of new blended types of cement incorporating pretreated recycled diatomite from the wine, beer, and oil industries; therefore, this study aims to contribute to the circular economy and reduce embodied energy in the construction industry.



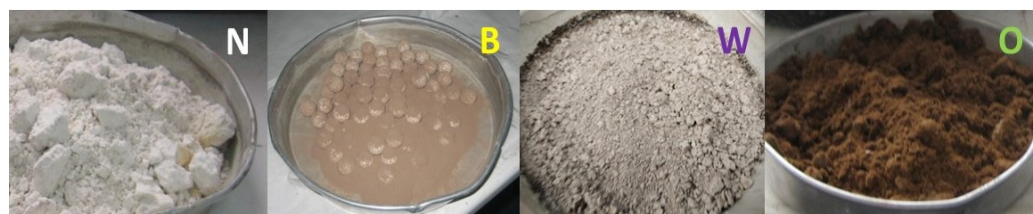
**Figure 1.** Concrete pipes made with diatomite cement [33].

## 2. Materials and Methods

The materials used in this study were Ordinary Portland clinker (OPK) 42.5R, gypsum, quicklime (QL), natural and recycled diatoms, CEN standard sand according to EN 196-1 [40], and tap water. OPK, gypsum, and QL were supplied by Cementos Cruz Company, Murcia, Spain, and natural and recycled diatoms from filtration in different industries were used:

- Natural diatomite (N) was supplied by CEKESA, whose quarries are located in Hellín, Murcia, Spain.
- Recycled diatomite from beer (B) filtration, supplied by Estrella Levante in Murcia, Spain.
- Recycled diatomite from wine (W) filtration, supplied by Bodegas Jumilla in Jumilla, Spain.
- Recycled diatomite from oil (O) filtration, supplied by Almazara Valle Ricote in Murcia, Spain.

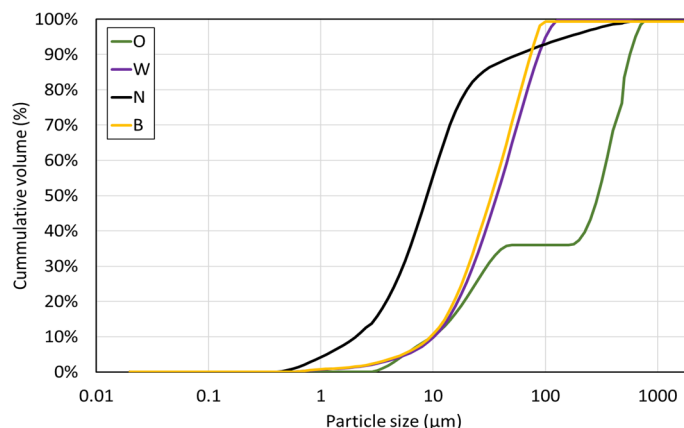
All the sources are from the industry in the area, favoring the circular economy. Natural and recycled diatoms can be seen in Figure 2.



**Figure 2.** Natural diatoms (N) and recycled diatoms from the filtering of beer (B), wine (W), and oil (O). Beer diatoms (B) appear together with some mill balls in the picture.

The apparent density of N, B, W, and O, tested according to UNE-EN 1097-6 [41], are 0.34, 0.41, 0.24, and 0.51 gr/cm<sup>3</sup>, respectively. The particle size distributions were analyzed by laser diffraction and are represented in Figure 3.

Figure 3 shows the particle size of the natural and recycled diatoms determined by laser granulometry after grinding has been performed on each of the diatoms. The particle size groups were obtained by using a Mastersizer 3000 Aero S analyzer, from Malvern Panalytical, Madrid, Spain. N shows an average particle size of 9.07  $\mu\text{m}$ . 10% of the particles of these diatoms (Dv10) pass through the 2.04  $\mu\text{m}$  sieve, and 90% (Dv90) through the 78.49  $\mu\text{m}$ . B shows an average particle size after conditioning of 35.45  $\mu\text{m}$ . A total of 10% of particles (Dv10) pass below 10.05  $\mu\text{m}$ , and 90% (Dv90) pass through 86.95  $\mu\text{m}$ . W shows an average particle size after conditioning of 36.83  $\mu\text{m}$ . A total of 10% of particles (Dv10) pass below 10.15  $\mu\text{m}$ , and 90% (Dv90) pass through 87.07  $\mu\text{m}$ . O shows an average particle size after conditioning of 294.59  $\mu\text{m}$ . A total of 10% of particles (Dv10) pass below 10.90  $\mu\text{m}$ , and 90% (Dv90) pass through 564.46  $\mu\text{m}$ . Furthermore, the pH of all the samples before drying, in fluid appearance (slug), was tested. The obtained results are 6.87, 4.89, 7.72, and 5.75 for N, B, W, and O, respectively.



**Figure 3.** Particle size distribution of natural diatomite (N) and recycled diatoms (B, W, and O).

Chemical analyses were performed using an S8 Tiger Bruker spectrometer (XRF), from Malvern Panalytical, Madrid, Spain. The chemical composition of all the used materials is presented in Table 1. The composition of the diatoms given in Table 1 shows that SiO<sub>2</sub> is the major compound and CaO for natural diatomite. Significant amounts of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> were also identified. Although the N and O diatoms exhibited organic material values higher than 15%, the calcination of each one reduced these values to 0% at 900 °C, producing enrichment in SiO<sub>2</sub> (from 54.82% to 65.49% for N and from 41.85% to 52.75% for O diatoms). For the other recycled diatoms, W and B, growth is minimal and practically uncalcined.

**Table 1.** Chemical composition of clinker, gypsum, quicklime, and diatoms (\* Loss on ignition at 1100 °C).

Oxides	Clinker	Gypsum	QL	N	W	B	O
Na <sub>2</sub> O	0.48	0.03	-	0.20	2.07	1.11	1.28
MgO	1.89	0.18	0.42	0.68	0.15	0.64	0.14
Al <sub>2</sub> O <sub>3</sub>	5.23	0.01	1.2	1.77	2.37	4.18	1.29
SiO <sub>2</sub>	19.89	0.41	3.03	54.82	90.67	85.21	41.85
K <sub>2</sub> O	0.51	0.01	0.74	0.33	0.19	1.01	1.22
CaO	64.10	31.47	92.1	19.98	0.80	0.89	0.55
TiO <sub>2</sub>	0.05	-	-	0.09	0.50	0.34	0.39
Fe <sub>2</sub> O <sub>3</sub>	3.78	0.06	0.34	0.78	2.14	2.40	1.53
P <sub>2</sub> O <sub>5</sub>	0.18	0.01	-	0.14	0.09	0.32	0.27
SO <sub>3</sub>	2.05	48.34	0.39	0.22	0.03	0.14	0.14
LOI *	0.91	19.87	0.15	20.65	0.85	3.54	51.15

The results of TGA, which were analyzed by SDT Q600 TA Instruments, of the samples are shown in Figure 4. It can be seen in Figure 4 how the diatom from the oil, O, loses all weight until it reaches 400 °C; this is reflected because of the percentage of LOI present in its composition, see Table 1. The second compound with more % of LOI is N, which is also reflected in Figure 4; most of the adsorbed organic matter has been removed after heating to greater than 600 °C. On the other hand, the low percentage of LOI of W and B means that the loss of mass after the test is carried out is minimal.

The mineralogical study of the natural and recycled diatoms was carried out using XRD. The mineral structures of the natural and recycled diatoms were examined by means of a D8 Advance from Bruker AXL, from Malvern Panalytical, Madrid, Spain. The diffraction patterns are interpreted using PANalytical's X'Pert HighScore Plus 3.0 software and version 2.1 of Bruker's software DiffraPlus TOPAS, qualitatively reconstructing the mineral profiles of the compounds by comparison with the PDF databases of the International Centre for Diffraction Data (ICDD). The quantification of crystalline and amorphous phases

has been carried out by the Rietveld method, for which a standard or intentional standard (ZnO at 20wt%) was added to the powdered samples. The semi-quantitative XRD mineralogical analysis, Table 2, showed that the main crystalline phases in all the diatoms were quartz and cristobalite, two different forms of silica. Minor amounts of other crystalline phases were also identified, including tridymite ( $\text{SiO}_2$ ) in N, anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ) in B and W diatoms, and calcite ( $\text{CaCO}_3$ ) and anorthite in B and W diatoms. Aragonite was also present in the N sample.

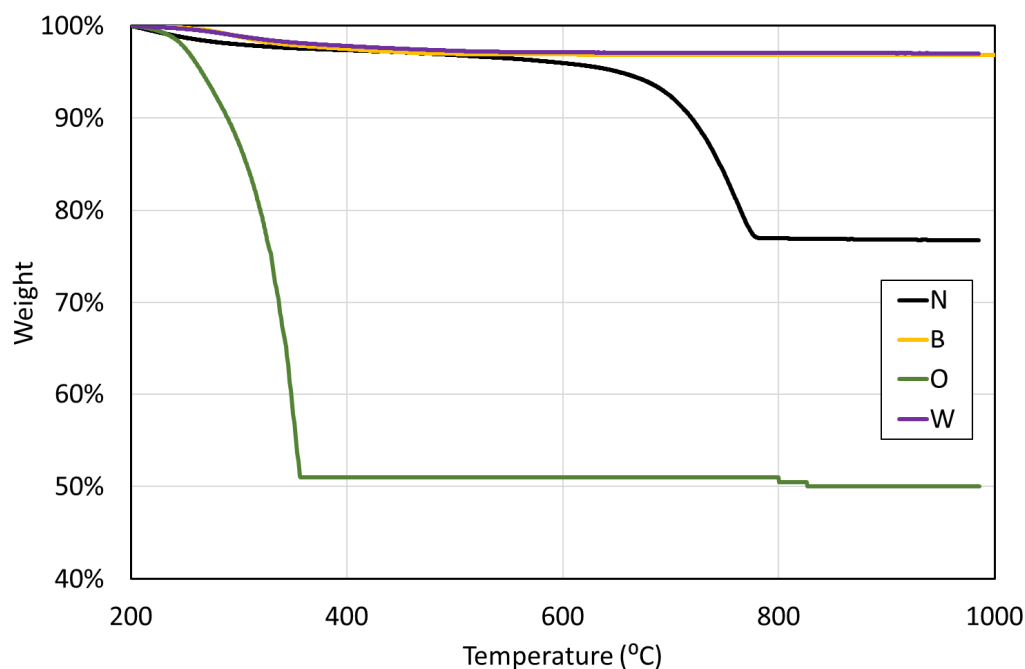


Figure 4. TGA of diatoms (N, B, W, and O).

Table 2. Semi-quantitative XRD mineralogical analysis of N, B, O, and W.

Mineral	N	B	O	W
Quartz	MJ	MJ	MJ	MJ
Calcite	-	TR	-	TR
Aragonite	TR	-	-	-
Silicon Oxide	MD	-	MD	MD
Anorthite	-	TR	-	TR
Cristobalite	MJ	MJ	MJ	MJ

Explanatory notes: MJ = major component, MD = medium component, TR = minor/trace component.

All four samples contained, besides the crystalline constituents, an amorphous silica phase (opal-A), as shown by the presence of a broad peak (bump) between  $20^\circ$  and  $26^\circ$   $2\theta$  in the X-ray diffractogram.

The content in amorphous phases was estimated as 65.4%, 89.1%, and 62.8% for W, B, and O diatoms without any calcination pretreatment. When the samples were heated up to  $500^\circ\text{C}$ , those proportions barely changed, and results of 66.2%, 88.76%, and 62.5% were obtained, respectively; however, calcination at  $900^\circ\text{C}$  increases the ratio of amorphous content to 81.6%, 89.5%, and 72.6% for W, B, and O diatoms.

Therefore, given the high increase in LOI after the filtration process, calcination pretreatments to be carried out before the incorporation in cementitious mixtures were proposed to minorize jeopardizing effects on setting and strength development. Specifically, recycled diatoms were heated up to  $500^\circ\text{C}$  and  $900^\circ\text{C}$  for 2 h. Reference samples without calcination pretreatment were oven-dried at  $60^\circ\text{C}$  for 2 days before its use.

A wide range of mixtures, including a reference mixture without diatomite nor quicklime, mixtures with all the presented recycled diatoms with the corresponding pretreatments and without them, and mixtures including quicklime in their binding composition, were tested.

All the samples were elaborated under laboratory conditions (Temperature ( $T^a$ ):  $20 \pm 2$  °C; Relative Humidity (RH):  $40 \pm 5\%$ ) and de-molded after 24h. Compressive strength was tested at 7, 28, and 90 days of hydration, respectively, according to the standard EN 196-1 [40].

The water demand of the developed mixes was determined according to EN 1015-3 [42]. The initial and final setting times were determined according to the procedures indicated in EN 196-3 [43].

The mortar mix design is shown in Table 3. The nomenclature used refers to the Reference mortar (REF), the type of diatomite (X)—natural (N), beer (B), wine (W), and oil (O)—the percentage of diatoms used, 5% (5), 15% (15), and 20% (20), and finally, the use of the temperature treatment at 500 °C (500) and 900 °C (900).

**Table 3.** Mix proportions (weight%).

	Clinker + Gypsum	Quicklime (QL)	Diatoms (wt%)			Oil Filtering (O)	Temperature Treatment (°C)
			Natural (N)	Beer Filtering (B)	Wine Filtering (W)		
REF	100	0		0			0
REF-QL	85	15		0			0
X-5	95	0		5			0
X-15	85	0		15			0
X-20	80	0		20			0
X-5-QL	80	15		5			0
X-15-QL	70	15		15			0
X-20-QL	65	15		20			0
X-5-500	95	0		5			500
X-15-500	85	0		15			500
X-20-500	80	0		20			500
X-5-900	95	0		5			900
X-15-900	85	0		15			900
X-20-900	80	0		20			900

### 3. Results

#### 3.1. Compressive Strength Development

The compressive strength of the mortar was measured following the standard UNE-EN 196-1 [40] at 7, 28, and 90 days of hydration. Tables 4–7 show the results of the mortar strengths.

**Table 4.** Evolution of Compressive Strength of N, B, W, and O mortars.

Samples	Compressive Strength (MPa)						
	7 d	28 d	90 d	Samples	7 d	28 d	90 d
REF	41.03	50.22	56.72	W5	31.95	41.50	46.55
N5	37.12	45.72	52.72	W15	33.20	42.40	46.55
N15	37.18	48.10	56.88	W20	30.88	39.01	42.36
N20	33.07	45.07	52.40	O5	2.70	16.90	21.77
B5	-	-	-	O15	2.60	15.80	21.77
B15	-	-	-	O20	2.36	14.52	18.10
B20	-	-	-				

**Table 5.** Evolution of Compressive Strength of N, B, W, and O mortars calcinated at 500 °C.

Compressive Strength (MPa)							
Samples	7 d	28 d	90 d	Samples	7 d	28 d	90 d
N5-500	40.63	51.90	59.53	W5-500	39.10	52.20	53.50
N15-500	37.80	50.08	57.90	W15-500	37.50	50.20	53.70
N20-500	34.95	48.65	55.53	W20-500	36.70	51.60	53.50
B5-500	39.20	48.60	59.60	O5-500	39.10	50.10	56.45
B15-500	35.10	47.00	56.10	O15-500	36.80	48.20	53.90
B20-500	30.20	42.70	51.60	O20-500	33.87	47.83	51.69

**Table 6.** Evolution of Compressive Strength of N, B, W, and O mortars calcinated at 900 °C.

Compressive Strength (MPa)							
Samples	7 d	28 d	90 d	Samples	7 d	28 d	90 d
N5-900	39.23	48.53	60.63	W5-900	40.10	50.30	54.50
N15-900	30.83	41.33	50.50	W15-900	37.00	48.00	54.30
N20-900	25.83	42.45	53.08	W20-900	31.70	43.40	51.70
B5-900	35.40	43.80	59.80	O5-900	37.10	47.20	57.52
B15-900	21.70	31.70	40.70	O15-900	29.35	39.85	47.70
B20-900	17.00	38.50	51.50	O20-900	24.90	40.87	51.60

**Table 7.** Evolution of Compressive Strength of N, B, W, and O mortars with Quicklime (QL).

Compressive Strength (MPa)			
Samples	7 d	28 d	90 d
REF-QL	38.85	45.30	45.70
N15-QL	29.85	38.40	43.15
B5-QL	17.26	21.81	23.93
B15-QL	16.20	20.85	23.45
B20-QL	14.97	18.10	20.74
W15-QL	27.25	34.35	39.05
O5-QL	25.95	31.20	35.05
O15-QL	21.75	25.45	26.60
O20-QL	19.74	22.17	24.16

At early hydration ages, such as 7 d, it can be observed how the compressive strengths of mortars with natural diatoms (N), 5%, 10%, and 15% (N5, N15, and N20) are lower than the value of the reference mortar (REF) being the value of this at 7 days of 41.03 MPa, while for N5, N15, and N20 it is 37.12 MPa, 37.18 MPa, and 33.07 MPa, respectively. At 28 days of hydration, N5, N15, and N20 values are still lower than the results obtained for REF, 50.22 MPa. Meanwhile, the values of N5, N15, and N20 are 45.72 MPa, 48.10 MPa, and 45.07 MPa, respectively. At 90 days, it can be observed that diatoms have a pozzolanic character that generates an increase in mechanical properties to 52.72 MPa, 56.88 MPa, and 52.40 MPa for N5, N15, and N20, with the N15 value being identical to that obtained by REF of 50.22 MPa.

For all mixtures with beer diatoms (B), from 5% to 15%, the mechanical properties cannot be measured because the material, once cured, cannot be de-molded.

Mortars with recycled diatoms from wine (W) at 7 days show a compressive strength of 31.95 MPa, 33.20 MPa, and 30.88 MPa for W5, W15, and W20, respectively. This resistance is lower than the value of 41.03 MPa of REF. The mechanical properties continue to grow continuously during the hydration of the material, reaching 28 days, 41.50 MPa, 42.40 MPa, and 39.01 MPa for W5, W15, and W20, respectively. These values increase slightly until 90 days, when they reach 41.50 MPa, 42.40 MPa, and 39.01 MPa for W5, W15 and W20, respectively.

Mortars with diatoms from oil (O) at 7 days show a very low compressive strength, with values of 2.70 MPa, 2.69 MPa, and 2.36 MPa for O5, O15, and O20, respectively. The main reason for these resistances is because of the high percentage of LOI, as can be seen in Table 1, present in the chemical composition of the diatom, see Table 1, where it is 51.15%. A high organic material content interferes with chemical hydration reactions that occur between water and cement components, delaying the development of the phases necessary for concrete to reach initial stiffness. This inhibits setting times, prolonging the time required for concrete to move from a plastic to a solid state and affecting its early strength.

During the hydration of the material, at 28 days, low values are observed compared with natural diatoms and recycled diatoms, since 16.90 MPa, 15.80 MPa, and 14.52 MPa are reached for O5, O15, and O20, respectively. These values increase slightly until 90 days, when they reach 21.77 MPa, 20.35 MPa, and 18.10 MPa for O5, O15, and O20, respectively.

The compressive strengths achieved via calcination at 500 °C and 900 °C are shown in Tables 5 and 6. Mortars with natural diatoms calcined at 500 °C and 900 °C, N500 and N900, show a higher compressive strength at 7 days when the percentage of substitution is 5%, 40.63 MPa, and 39.23 MPa for N5-500 and N5-900, respectively. As the hydration time increases, compressive strengths grow to 59.53 MPa and 60.63 MPa at 90 days of hydration for N5-500 and N5-900, respectively. The increase in the percentage of replacement of clinker by activated natural diatoms means that the mechanical properties are lower throughout the hydration time studied, reaching 90 days, 55.53 MPa, and 53.08 MPa for N20-500 and N20-900.

Tables 5 and 6 show the compressive strength values of recycled diatoms from beer, wine, and oil. It can be observed that by comparing them with the mechanical properties of the uncalcined recycled diatoms, it is possible to measure the mechanical properties of beer diatoms (B-500 and B-900). Obtaining values higher than the reference and similar to N5-500 and N5-900, reaching 59.60 MPa and 59.80 MPa, respectively, at 90 days. Samples with oil diatoms show considerable improvement once calcined, both at 500 °C and 900 °C. It reaches O5-500 and O5-900 after seven days of hydration, 39.10 MPa and 37.10 MPa. As with other recycled diatoms, the values of the resistances decrease with increasing percentage of substitution. Over the hydration period, O5-500 and O5-900 increase their compressive strengths at 90 days to reach 56.45 MPa and 57.52 MPa, respectively.

The samples with wine diatoms showed a higher value when undergoing calcination at both temperatures, reaching values of 54.50 MPa and 57.52 MPa at 90 days of hydration for W5-500 and W5-900, respectively.

Table 7 summarizes the compressive strength values of various cement mixtures with different types of diatoms and the addition of quicklime (QL), evaluated at 7, 28, and 90 days of curing. A complete study of the compressive strength of recycled diatoms from oil (O-QL) and beer (B-QL) has been carried out since they have the worst mechanical strength values. On the other hand, for natural diatoms (N-QL) and those from the wine industry (W-QL), a control dosage (N15-QL and W15-QL) is carried out to compare with the mechanical properties obtained in Table 4.

The reference sample (REF-QL) without diatom substitution showed the highest resistance at all ages, reaching 45.70 MPa at 90 days. On the other hand, the addition of natural diatoms (N15-QL) reduced the initial strength, but a significant improvement was observed over time, achieving a strength of 43.15 MPa at 90 days, which is close to the baseline. Calcined diatoms (W15-QL) also exhibited good resistance development capacity, reaching 39.05 MPa at 90 days. This suggests that the use of natural or calcined diatoms can maintain adequate strength in cementitious mixtures modified with quicklime.

In contrast, samples that incorporated beer residues (B-QL) showed the lowest resistance values, in particular the B20-QL sample, which reached only 20.74 MPa at 90 days. This indicates that the addition of beer residues negatively affects the material's ability to develop strength. In addition, samples with diatoms from other sources (O-QL) showed a progressive decrease in resistance as the percentage of substitution increased, with the

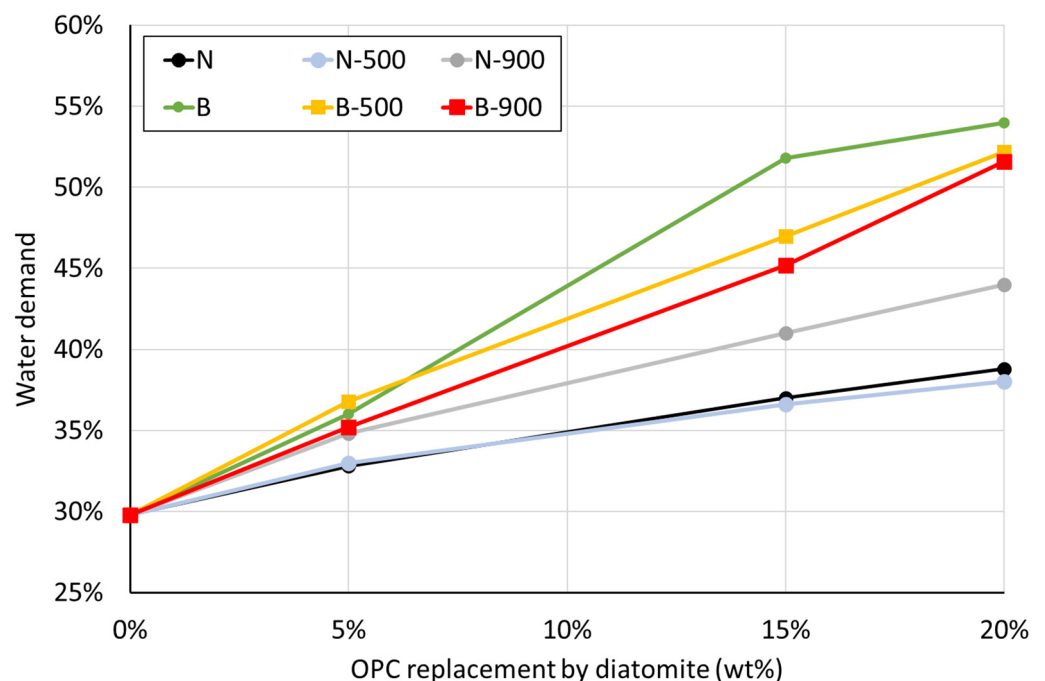
O5-QL sample being the best-performing within this series. In general, the results show that the substitution of cement by diatoms, depending on their origin and treatment, has a significant impact on mechanical properties, with natural and calcined diatoms being the most viable options to maintain an acceptable resistance over time.

Therefore, the addition of quicklime does not manage to add pozzolanic activity in the dosages studied. As shown in Table 7, the recycled diatoms that show the worst uncalcined performance are those from wine and oil; therefore, the results are compared with the B-500 and O-500 samples. It is observed that the 7-day compression values are 20% higher for B5-500, B15-500, and B20-500 than for B5-QL, B15-QL, and B20-QL, respectively, since each of the specimens reaches 17.26 MPa, 16.20 MPa, and 14.97 MPa. While after 90 days of hydration, the values are 16.67% lower for B5-QL, B15-QL, and B20-QL than for B5-500, B15-500, and B20-500.

This trend is reflected in the samples with diatoms from the oil since, comparing the values of compressive strength at 7 days, the results are 20% higher for O5-500, O15-500, and O20-500 than for O5-QL, O15-QL, and O20-QL, respectively, since each of the specimens reaches 17.26 MPa, 16.20 MPa, and 14.97 MPa. While, at 90 days of hydration, the values are 16.67% lower for O5-QL, O15-QL, and O20-QL than for O5-500, O15-500, and O20-500.

### 3.2. Water Demand

Figures 5 and 6 show that, in all dosages, with both diatoms, the demand for water increases.



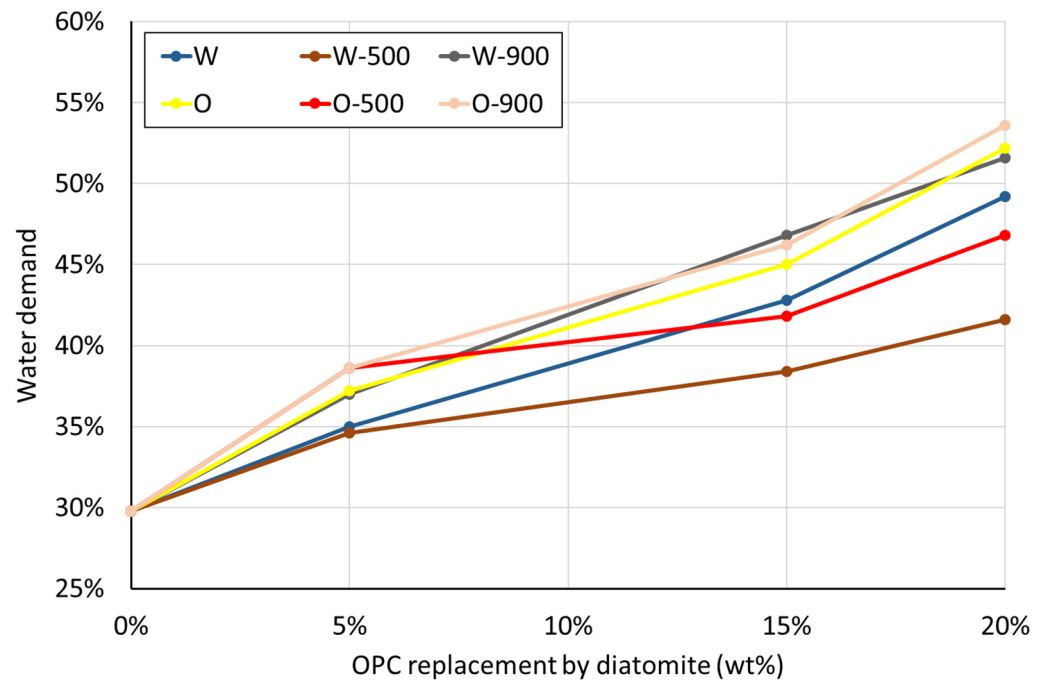
**Figure 5.** Evolution of Water demand replacing OPK by Natural (N) and Beer (B) diatoms (wt%).

In Figure 5, it can be seen as the percentage of natural diatoms and beer increased, an increase in the amount of water required was observed.

Focusing on the results obtained by the natural diatom, N, both uncalcined and calcined at 500 °C and 900 °C, it is observed that there is a greater demand for water in N-900. Whereas, for beer diatom, B, heat treatment does not have the same result.

Comparing the series, the N series (natural diatomite) has the lowest water demand, which indicates that, at the same percentage of substitution, this series is less demanding in terms of water content. In contrast, the B-500 and B-900 series (derived from beer waste)

show increased demand for water at all substitution levels, with the B-900 series revealing a steeper increase.



**Figure 6.** Evolution of Water demand replacing OPK by Wine (W) and Oil (O) diatoms (wt%).

Figure 6 shows the evolution of water demand, replacing OPK with Wine (W) and Oil (O) diatoms. The replacement of OPK by diatoms provokes an increment in the amount of water required.

It is observed that at low replacement levels (5%), mixtures show a similar water demand, over 30%, regardless of heat treatment; however, with greater replacement, heat-treated samples begin to show a higher demand for water compared with untreated samples. The curves corresponding to the samples without heat treatment show the lowest water demand throughout the range, while those treated at 500 °C and 900 °C exhibit progressively increasing behavior.

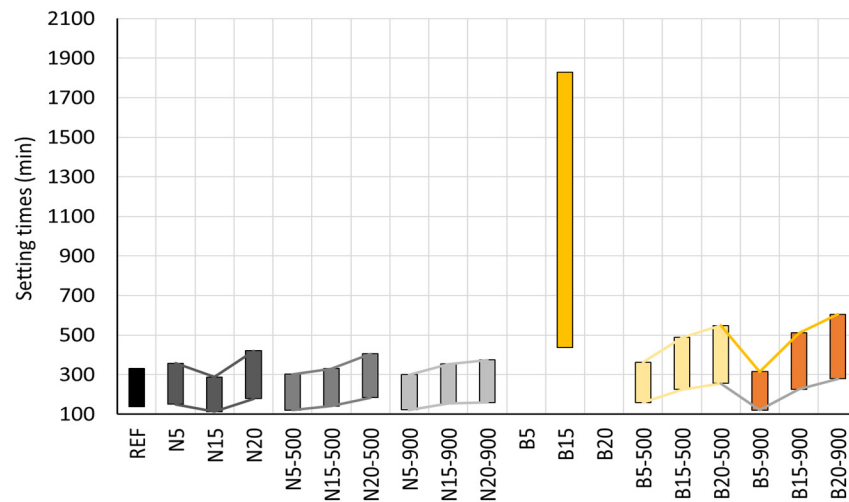
In samples heat-treated at 900 °C, W-900 and O-900 reach the highest water demand, exceeding 55%. This suggests that heat treatments at high temperatures modify the microstructure of the diatom particles, increasing their absorption capacity.

### 3.3. Setting Time

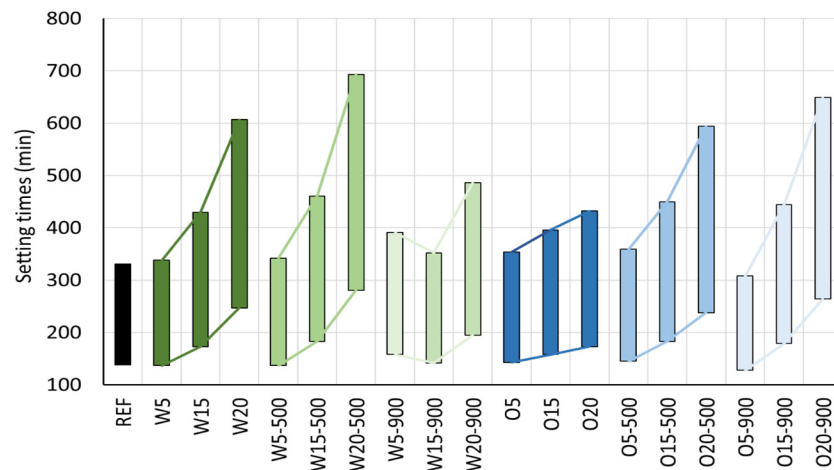
Figures 7 and 8 show the setting times of the different formulations used according to the origin of the diatom, natural, and recycled ones, according to the proportion of the substitution material and the heat treatment.

Figure 7 shows the setting time evolution of different formulations according to the origin of the diatom (Natural, N, and Beer, B). The results show that the reference material (REF) has a shorter setting time, with a value close to 150 min. On the other hand, formulations with diatoms show an increase in setting times as the percentage of substitution increases.

Mixtures with natural diatoms, N (N5, N15, and N20), show a trend in setting times with increasing proportions of diatoms. The variability in the setting times of the N15 sample is remarkable, which presents a wide distribution, which suggests a lower consistency in the setting behavior for this formulation. Heat treatment variations (N5-500, N15-500, N20-500, N5-900, etc.) indicate that by increasing the treatment temperature to 500 °C and 900 °C, setting times are significantly lengthened, with N20-900 recording the longest times.



**Figure 7.** Setting time of different formulations according to the origin of the diatom.



**Figure 8.** Setting time of different formulations according to the origin of the diatom.

On the other hand, formulations with diatoms from beer, B (B5, B15, and B20), which are represented with a yellow color scheme, follow a similar trend to the N series, showing longer setting times as the proportion of diatomite and the heat treatment temperature increases. B20-900 has one of the longest setting times, indicating that the most intense heat treatments prolong the setting process.

However, diatoms from beers without chemical treatment had problems performing the setting time test. Samples B5 and B20 could not be measured because they did not harden, as in the mechanical properties section, while for B15, the start of setting is at 437 min; therefore, it is necessary to carry out heat treatment for samples that use diatoms from the brewing industry.

Figure 8 shows that mixtures with low levels of OPK replacement by diatoms (W5 and O5) do not present important variations, while for a 20% replacement, the setting times increase considerably. Samples treated at 500 °C reduce setting times compared with untreated samples, while samples treated at 900 °C show much longer setting times, some close to 700 min (W20-500).

## 4. Discussion

### 4.1. Compressive Strength Development

The percentage of SiO<sub>2</sub> in diatoms and their fineness are the main factors, so incorporating diatoms does not affect the mechanical properties. These values are in concordance with other research [33], where an improvement in compressive strength is observed when

10% is replaced, indicating that the resistance values depend mainly on the reactivity of diatoms. The reactivity of diatoms may improve the mechanical properties mainly because they participate in pozzolanic reactions that strengthen the cement matrix by forming calcium silicate hydrate (C-S-H).

As shown in Section 2, Table 1 shows how recycled diatoms (B, W, and O) have a high percentage of organic material in their sample, causing problems in the setting and, consequently, in the evolution of the mechanical properties of the specimens; therefore, according to Font et al. and C. Rodríguez et al. [32,33], diatoms, both natural and recycled, are calcined at two temperatures, 500 °C and 900 °C for two.

Therefore, given the exposed values of the compressive strengths of recycled diatoms from wine and oil, the calcination pretreatment at 500 °C improves the behavior with respect to the addition of quicklime, both at early and long ages of hydration, mainly due to the phenomenon of hydration of clinker at early ages produced by the hydration of quicklime.

In these cases, the pozzolanic activity of diatoms must be low; therefore, it can be concluded that treatment with a temperature of 500 °C is necessary to endow diatoms with pozzolanic capacity. In fact, once again, it is mentioned that the test specimens with untreated beer diatoms could not even be manufactured because they did not develop any resistance.

#### 4.2. Water Demand

It appears that heat treatment at 900 °C of beer residues not only increases water demand but also affects the reactivity and microstructure of the mix, which can negatively influence the workability of the concrete.

Therefore, the effect of heat treatment on the values of uncalcined diatoms for N and B is reduced.

The results of Figures 4 and 5 are in accordance with the literature [44–47]. This phenomenon is attributed to the porous and high-adsorption properties of recycled diatoms, which generate a higher water demand [13].

This event occurs due to the physical and chemical properties of diatoms. On the one hand, it is due to the high porosity and water absorption of diatoms since their internal structure has a large surface area, which allows them to absorb a significant amount of water, decreasing the amount of free water available for the hydration of the sample, generating the need to increase the amount of water in the mixture [10,12,13,21,33].

In addition, the particle size of diatoms (see Figure 2) is smaller than that of traditional Portland cement, resulting in a denser material that requires more water to maintain the mixture's workability.

In conclusion, the graph shows that the substitution of OPKs by diatomite increases the water demand, which has practical implications in the formulation of cement mixtures. The choice of diatomite type and heat treatment are determining factors that must be considered for the design of efficient and sustainable cement mixtures

#### 4.3. Setting Time

The behavior suggests that both the percentage of OPK replacement by diatomite and the heat treatment significantly affect the setting times of the mixtures. In particular, samples treated at 900 °C have a higher setting, which could be related to microstructural changes in the diatom due to heat treatment.

Taken together, these results suggest that increasing the proportion of diatomite, together with heat treatment, delays cement setting. This could have important implications for the viability of mixes in concrete applications where setting times must be carefully controlled. This is consistent with what has been researched by different research groups [7,12,13,35].

The observed differences in setting times between the Natural and recycled one series can be attributed to the inherent characteristics of the materials used.

## 5. Conclusions

This study explored the potential of using recycled diatomaceous earth from the wine, beer, and oil industries as supplementary cementitious materials in cement-based mixtures. The research focused on evaluating the mechanical properties, specifically compressive strength, of these mixtures over time, as well as the influence of heat treatments and the addition of quicklime on the performance of the recycled diatoms, water demand, and setting time of the mixture. The findings offer insights into the viability of using industrial by-products to promote sustainability in the construction industry while maintaining adequate mechanical performance.

1. Samples treated with quicklime failed to achieve strengths comparable to those heat-treated with lime, confirming that heat treatment is essential for improving the reactivity of diatoms in mixtures.
2. The incorporation of natural and recycled diatoms in substitution of OPKs has a significant impact on compressive strength, especially depending on the heat treatment applied and the origin of the diatoms. Samples that have not been heat-treated show an increase at 90 days of hydration. Meanwhile, heat treatment at 500 °C and 900 °C significantly improves mechanical properties, especially in diatoms from the brewing industry.
3. The demand for water increases as Portland clinker (OPK) is replaced by diatom. In addition, heat-treated samples, especially at 900 °C, increase significantly. This is mainly due to the physical and chemical properties of diatoms, such as their high porosity and smaller particle size.
4. The results of this study of the setting time in diatom mixtures show that the setting time increases as diatom replacement increases, both in untreated and heat-treated samples.
5. The study of mechanical behavior, water demand, and setting time makes diatoms from wine the most optimal as an addition.

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