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Use of Ceramic Tile Wastes as Raw Substitution Material in the Production of Blended Cement

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Abstract: In this study, the feasibility of using ceramic wastes in the production of blended cement was evaluated by substituting limestone with ceramic waste at the percentages of 5, 10, 15, 20, and 28% before the milling stage. The chemical, physical, and mechanical properties of the cements were determined according to relevant standards, and the results were compared. The chemical analysis showed that the SiO_2 content of the cements increased with higher ceramic waste substitution percentages, while the $CaCO_3$ content decreased. The grindability of cements decreased with increased ceramic waste ratios, slightly reducing the Blaine specific surface area values. The water consistency for the cements was set at 28%, and all the cements met the standard limitations. The spread diameters for all types of cements were similar and practically usable in terms of workability. The cements containing ceramic waste either maintained or extended the setting time. All cements with ceramic waste exhibited higher flexural and compressive strength compared to the reference cement. The highest flexural strengths were achieved with a 28% ceramic waste substitution ratio across all curing ages. Regarding compressive strengths, all cements exhibited higher compressive strength than 10 MPa at 2 days and 32.5 MPa at 28 days, classifying them as 32.5 R-type blended cements. When the medium- (56–90 days) and long-term (365 days) compressive strengths were compared, the highest strength values were obtained from the cement with a 28% ceramic waste substitution. Although limestone-blended cement is emerging as a promising alternative to traditional Portland cement, these types of cement still contribute to environmental degradation due to the extraction of natural limestone resources through quarrying. This study showed that blended cements can be produced using ceramic waste, providing a more sustainable and environmentally friendly solution for the construction industry.

Keywords: ceramic tile waste; limestone; blended cement; chemical; physical; mechanical

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1. Introduction

Clinker is a key component of many conventional cements. The production process for clinker is energy-intensive, consuming approximately 850 kcal/kg, and contributes to greenhouse gas emissions, releasing about 0.85 tons of CO_2 per ton of clinker [1,2]. Various studies have been conducted over the years to address these issues and develop greener cements. One of the possible solutions is using waste materials as a partial replacement for clinker in cement production, as mentioned earlier by several studies [1,3,4]. This approach not only reduces the demand for clinker but also promotes an environmentally friendly strategy through the utilization of waste materials in the cement industry.

Portland-limestone cement is a blended cement produced by replacing up to 15% of the clinker with limestone, resulting in an average reduction in the carbon footprint of 10% while maintaining the intended performance in concrete production. The use of Portland-limestone cement began in the 1960s and rapidly became widespread with its adoption into the EN 197-1 [5] standard in the 2000s. According to the statistical reports of the Turkish Cement and Cement Products Assembly, the market share of Portland-limestone cements in Turkey has increased from 0.07% to 12.20% of the total cement market between 2000 and 2023 (Figure 1). However, the use of limestone in the production of blended cement continues to have environmental impacts due to the quarrying of natural limestone resources, transportation, machinery, milling, etc., which contribute to additional greenhouse gas emissions.

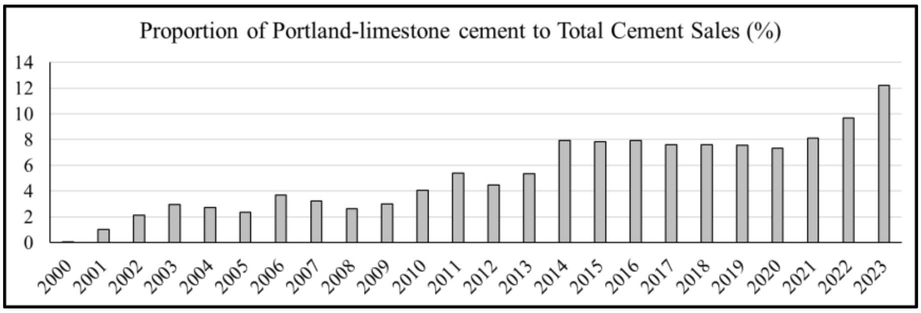

Figure 1. The sale ratios of Portland-limestone cement to the total cement market in Turkey.

The ceramic industry plays a significant role in construction, with ceramic tiles being among the most commonly produced materials for use in flooring and wall coverings. The global production volume of ceramic tiles was reported as 18.2 million m^2 in 2021 [6]. Turkey is among the top 10 countries in the world in ceramic coating material production. In 2022, Turkey ranked ninth in the world with a production share of 2.3%. Turkey's ceramic tile production is expected to be 461 million m^2 by 2025, according to the World Production and Consumption of Ceramic Tiles Report [7]. It has been reported that approximately 30% of the ceramic products produced are released as waste. The accumulation of these wastes is expected to lead to environmental and health problems in the long term.

Many studies in the past have attempted using ceramic tile wastes in concrete as an alternative to cement. Alsaif [4] reviewed studies that investigated the use of ceramic waste as a replacement material for cement in mortar and concrete production. The findings indicated that substituting up to 20% of cement with ceramic waste can have beneficial effects on rapid chloride permeability, electrical resistivity, and sulfate resistance. Additionally, improvements in compressive strength were observed, particularly in the medium term (56 and 90 days). El-Dieb and Kanaan [8] investigated the use of ceramic waste powder as an alternative to cement at 10, 20, 30, and 40% substitutions by mass in three different concrete grades (25, 50, and 75 MPa). The rate of compressive strength development decreased at 7 and 28 days; however, the target compressive strengths were achieved for all substitution rates by 90 days. Raval et al. [9] replaced cement with ceramic waste powder at substitution percentages of 10, 20, 30, 40, and 50% in concrete production. Optimum results were reported at a 30% substitution, with a compressive strength of 22.98 MPa and a 12.67% reduction in cost. Similarly, in a recent study by Faldessai et al. [10], cement was substituted with ceramic waste powder in concrete production at percentages of 10, 20, 25, and 30% by mass and achieved a compressive strength of 41.22 MPa and a 14.89% cost reduction. Samadi et al. [11] prepared mortars by replacing cement with ground ceramic waste at percentages of 20, 40, and 60%. The results showed that at up to a 40% replacement ratio, compressive strengths were enhanced at 28 and 90 days, and energy consumption was expected to decrease from 3.02 GJ/m^3 to 2.13 GJ/m^3, with significant cost savings. Hoppe Filho et al. [12] used ceramic waste as a supplementary cementitious material. In this study, a reference cement and a cement incorporating ceramic waste (30% of cement in mass replaced by ceramic waste) were prepared, and their physical and chemical properties were analyzed. The mechanical performance of the prepared mortars was evaluated at three different water-to-cement ratios over curing periods between 7 and 182 days. They reported that the ettringite phases were intensified and mono-carbo-aluminates were formed when using ceramic wastes, although compressive strengths generally decreased. Mohit et al. [13] replaced ceramic waste powder and limestone powder at various ratios by weight of cement in the production of ternary cement. They concluded that the ternary cement mortar with 10% ceramic waste powder and 15% limestone powder had the highest compressive strength. Samples containing 30% ceramic waste powder by mass of cement showed approximately 51% less alkali–silica reaction compared to the reference. The initial and final setting times of the samples increased compared to the reference sample as the ceramic waste powder content was increased up to 30%.

Puertas et al. [14] indicated that some ceramic wastes could be utilized in cement manufacturing due to their suitable chemical and mineralogical properties. However, there are relatively few studies in the literature on the use of ceramic wastes in cement production. Puertas et al. [1] published an article on the use of ceramic waste in the production of cement clinker. They prepared raw cement mixes using limestone, clay, Fe_2O_3, and ground ceramic wastes. The mixes were burned at temperatures up to 1500 °C, and their chemical, mineralogical, and morphological properties were investigated. They reported that the mineral composition and phase distribution in the resulting clinker were comparable to those of conventional reference clinker. Mas et al. [2] conducted a study on the use of ceramic wastes in the production of pozzolanic cement. They found that the workability of mortars incorporating ceramic waste was similar to the control mortar, and the addition of ceramic waste to between 15 and 35% of cement met the strength activity index requirements established by fly ash regulations. Abdul-Wahab et al. [15] notably highlighted that industrial wastes, such as ceramic, concrete, marble, etc., have the potential to serve as sources of calcium, silica, and alumina, which also contribute to the sustainability of raw materials and waste management.

In this study, blended cements were prepared by replacing limestone with ceramic wastes at percentages of 5, 10, 15, 20, and 28% before the milling stage. The chemical, physical, and mechanical properties of cements were determined in accordance with the relevant standards and results were compared. It is noteworthy that many studies in the literature generally investigate mechanical properties up to 28 days. In this study, medium and long-term mechanical performances were also investigated.

2. Materials and Methods

Clinker, limestone, pozzolana, and gypsum were used to produce a reference limestone blended cement. Then five different ceramic waste-blended cements were formulated by replacing limestone with ceramic waste at percentages of 5, 10, 15, 20 and 28%. According to the recent EN 197-1 [5] standard, cement types containing limestone are classified as "CEM II/A-L", "CEM II B-L", "CEM II A-LL", and "CEM II B-LL". The proportion of limestone used in the grinding process ranges between 21% and 35% by weight for the "CEM II A-LL" and "CEM II B-LL" cements. The 28% ratio was selected as it represents the average value between 21% and 35%.

The design and mix ratios of the materials are presented in Table 1. The materials used in the preparation of the cements are shown in Figure 2. Clinker, limestone, pozzolana, and gypsum were obtained from BAŞTAŞ (Baştaş Başkent Cement Industry and Trade Inc., Ankara, Turkey), while the waste ceramics were obtained from local ceramic manufacturer (Ece Bathroom Equipment Industry and Trade Inc., Çorum, Turkey). Note that all materials were initially crushed into clinker size using a laboratory-type mill (Figure 3). In the production stage of all types of cement, the ingredients were ground together in the mill for 30 min.

Table 1. Mixing ratios of materials for the reference and ceramic waste-incorporating cements.

Mix. Materials (%)	Reference	Ce5	Ce10	Ce15	Ce20	Ce28
Clinker	63					
Limestone	28	23	18	13	8	0
Ceramic Waste	0	5	10	15	20	28
Pozzolana	5					
Gypsum	4					

(**a**) (**b**) (**c**) (**d**) (**e**)

Figure 2. (**a**) Clinker, (**b**) limestone, (**c**) ceramic waste, (**d**) pozzolana, and (**e**) gypsum.

Figure 3. Laboratory-type mill used in the grinding process of cement.

After the preparation of each cement, its chemical, physical, and mechanical properties were determined. For the chemical analysis, a FLUXANA X-ray fluorescence (XRF) (FLUXANA® GmbH & Co., Bedburg-Hau, Germany) analyzer conforming to ISO 29581-2 [16] was used (Figure 4a). Loss on ignition values was determined using a ProTherm (Alser Technical Ceramic Co. Inc., Ankara, Turkey) high temperature furnace conforming to ASTM C114 [17] (Figure 4b). Blaine specific surface areas of cements were calculated via an Atom Technic Blaine test device conforming to ASTM C 204 [18], automatically (Figure 4c). The chemical structure of each cement was analyzed through X-ray diffraction analysis using a PANALYTICAL Empyrean (Malvern Panalytical Ltd., Malvern, UK) brand test device.

(**a**) (**b**) (**c**)

Figure 4. Determination of (**a**) chemical properties by XRF, (**b**) loss on ignition, and (**c**) Blaine specific surface area.

After the determination of basic cement characteristics, water consistency demand and setting properties of cements were assessed using a Vicat apparatus in accordance with ASTM C 187 [19] and ASTM C 191 [20], respectively (Figure 5). Cement mortars were prepared with a cement–sand–water ratio of 1:3:0.50, and spread flow diameters were determined conforming to ASTM C230 [21]. A total of 18 pieces of 40 × 40 × 160 mm^3 prismatic specimens were cast from each mortar made with produced cements. Mechanical tests were conducted using a ToniTechnic convertible test device. The flexural strengths of mortars were determined at 2, 7, 28, 56, 90, and 365 days following the ASTM C348 [22] (Figure 6a). All specimens were water-cured until the test day. The compressive strength test was performed on both sections separated during the flexural strength tests, conforming to the ASTM C349 [23] standard (Figure 6b).

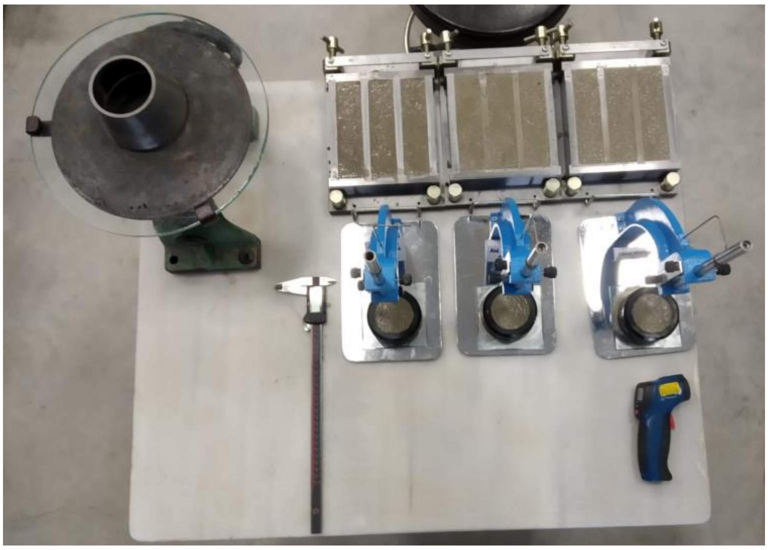

Figure 5. Fresh state cement paste and cement mortar tests.

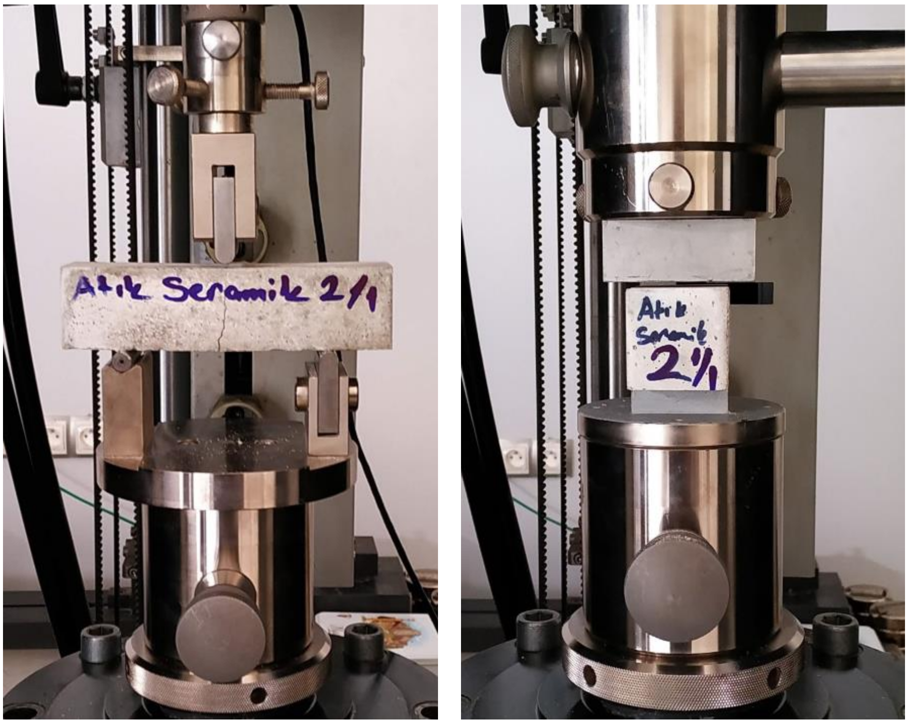

Figure 6. Mechanical tests. (**a**) Three-point flexural test and (**b**) compressive test. ("Atık seramik" means "waste ceramic"; "2/1" means "Ce10/specimen no:1").

3. Results

3.1. Raw Cement Properties

3.1.1. Chemical Properties of Cements

The chemical analysis results of cements obtained from XRF tests are given in Figure 7. A significant increase in the SiO_2 and Al_2O_3 contents of the cements was observed with an increasing percentage of ceramic waste. On the other hand, the CaO ratio by mass gradually decreased as the ceramic waste percentage increased. This result is expected due to the replacement of limestone (CaO-based) with ceramic waste, which is primarily composed of SiO_2 and Al_2O_3. These findings are consistent with the study by Mas et al. (2016), which reported that ceramic tile waste contains high amounts of SiO_2 and Al_2O_3 when used as a replacement material in Portland cement [2].

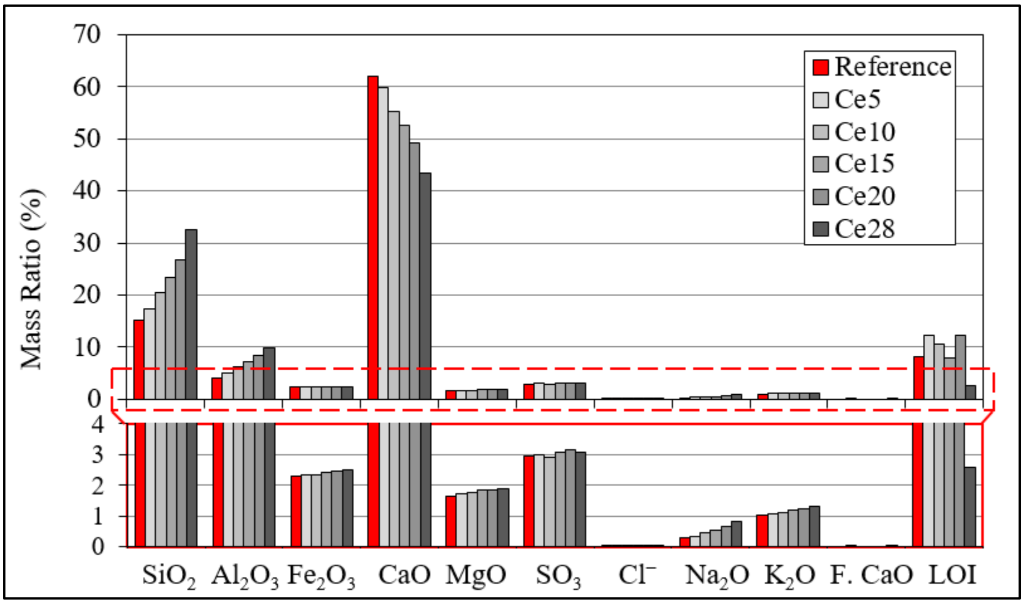

Figure 7. Chemical analysis results of the cements.

There were also slight increases in the Fe_2O_3, MgO, Na_2O, K_2O, and SO_3 contents of the cement as the ceramic waste percentage increased. There was an unsystematic change in the case of the loss on ignition (LOI) value. The LOI values were high in all waste ceramic-incorporating cements. A study by Deniz (2012) indicated that grindability has a strong correlation with LOI content, where grindability decreases as LOI increases [24]. This change can be correlated to the grindability of the studied cements. As the ceramic wastes have a higher hardness value than the limestone, the grindability of cements may have decreased, leading to the formation of air voids (related to LOI) that can be sensitive to humidity during storage.

The XRD results of the cements are presented in Figure 8. No significant changes were observed in the intensities of the basic oxides and the amorphous phases of the cements. However, the $CaCO_3$ intensities of the cements were decreased while the SiO_2 intensity increased as the replacement ratio of limestone with waste ceramic increased. This situation also supports the results obtained by the chemical analysis.

3.1.2. Comparison of the Blaine Specific Surface Areas

The Blaine specific surface areas of cements were calculated and are given in Figure 9. When the substitution of waste ceramic was 5%, the Blaine value of the cement slightly increased. However, the Blaine values generally tended to decrease as the substitution rate increased. This can be explained by two different scenarios depending on the use of waste ceramics: (i) since the waste ceramic particles used in relatively small amounts are harder than limestone, they can facilitate grindability by creating an additional abrasive effect in the grinding stage, and (ii) with the continued increase of the ceramic dosage, the raw cement mix becomes harder due to the higher hardness of the waste ceramic particles and the reduced amount of softer limestone particles. This may generate resistance during the grinding, leading to a decrease in Blaine values.

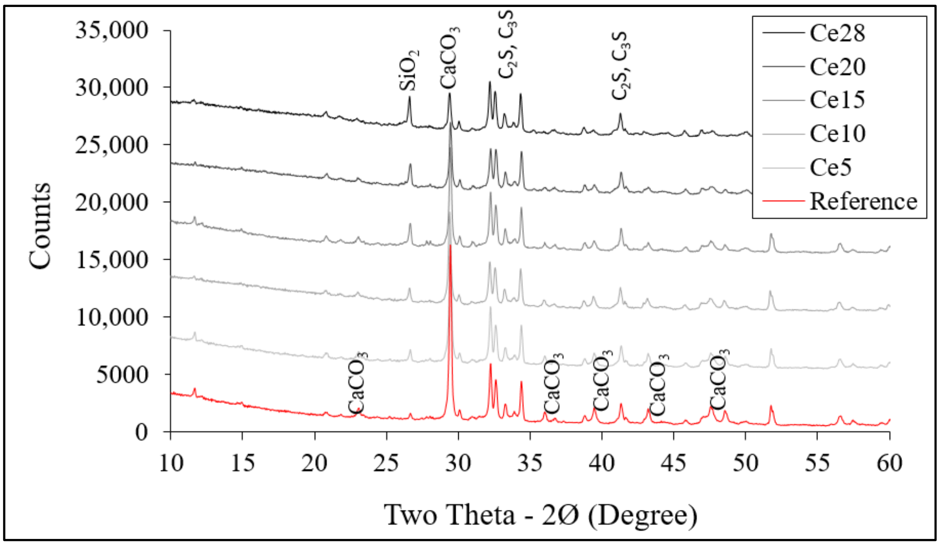

Figure 8. XRD analysis results of the cements.

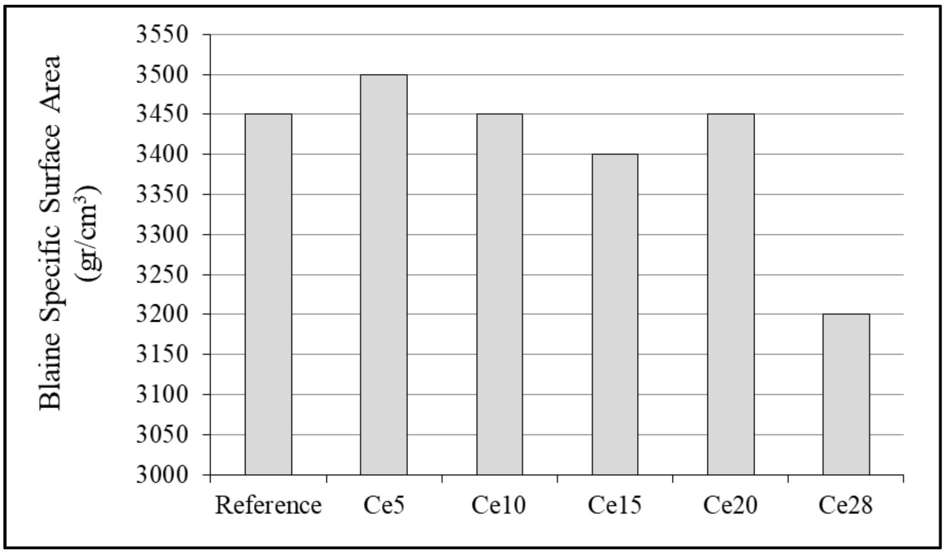

Figure 9. Blaine specific surface areas of the cements (g/cm^3).

Similar findings can be found in the literature. Kaissar et al. (1977) reported that when two materials with different grindability properties are ground together, the softer material will be preferentially ground. They also demonstrated a wide range of Blaine specific surface areas for various additives (sand, clinker, gypsum, fly ash, basalt, pumice stone, slag, etc.) between 3000 and 1000 cm^2/g after 64 min of grinding [25]. Pokorný et al. (2014) showed that the Blaine specific surface area of ceramic powder (2750 cm^2/g) was relatively lower than that of ordinary Portland cement (2920 cm^2/g), indicating that ceramic powder had lower grindability [26]. Another study conducted by Ahmed et al. [27] (2021) showed that micro-ceramic powders obtained from waste could be used as a supplementary cementitious material up to 40% in blended cement production. They investigated the effects of mill speed and air classifier speed on the performance of an industrial ball mill and demonstrated that by optimizing these parameters, the Blaine fineness could be refined from 2800 to 3200 cm^2/g. They also observed that higher SO_3 content leads to lower grindability. In the XRF examinations conducted in this study, SO_3 values slightly increased from 2.96% to 3.17% with the addition of waste ceramic powders, which may have also reduced grindability.

The grinding time within this study was kept constant at 30 min for accurate comparison. Since all Blaine values obtained in this study range were between 3200 and 3500 g/cm^3 and are acceptable in terms of practical usability, no additional grinding investigations were made.

3.1.3. Water Consistency and Setting Properties of Cements

Vicat probe heights obtained from the water consistency examinations are given in Figure 10. All water consistency tests were carried out using 28% water. This ratio was obtained from the preliminary tests of the reference cement which started at 25% water. As the ceramic waste ratio increased, the Vicat probe heights ranged between 5 and 6 mm. All probe heights were found lower than the reference; however, all probe heights provided the desired range of the standard. Additionally, the probe heights were gradually decreased with increased ceramic waste content, which could be counted as the need for constituent water decreased. This situation can be related to the fineness of cement. As previously mentioned, the fineness of the cement became coarser with increased ceramic waste content, which decreased the water demand to cover the particles of cement. This caused the cement paste to become watery and the probe to sink deeper, depending on the same amount of water used in the tests.

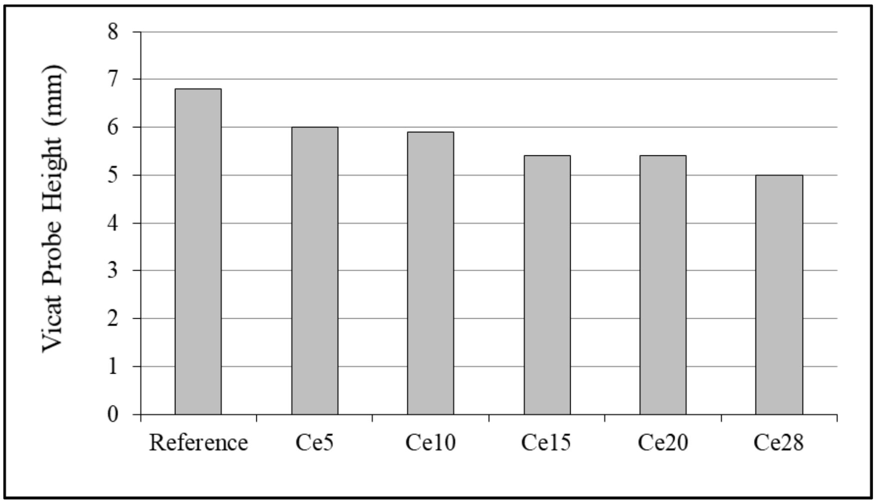

Figure 10. The Vicat probe heights at 28% water consistency.

The Vicat needle heights obtained during the setting tests are given in Figure 11a. It is clear from the figure that as the ceramic waste content increased, the slope of the Vicat curve decreased. Based on this finding, it can be said that the setting duration of cements was prolonged with the addition of ceramic waste instead of limestone. For a detailed understanding, the initiation and final times of setting for each cement are presented in Figure 11b. The initiation time of setting increased with an increased ceramic waste percentage (except Ce20). For ceramic waste replacement up to 10%, the total setting durations were calculated as 240 min. At 15% or more ceramic waste, both the initial setting time and final setting time of the cements were increased, and the total setting duration was prolonged between 50% and 88% compared to reference cement. According to the EN 197-1 [5] standard, the initial time of setting should be higher than 75 min, 60 min, and 45 min for the classification of cement grades of 32.5, 42.5, and 52.5, respectively. Considering these classifications, it is noteworthy that all cements prepared in this study ensure the initial times of setting for all grades.

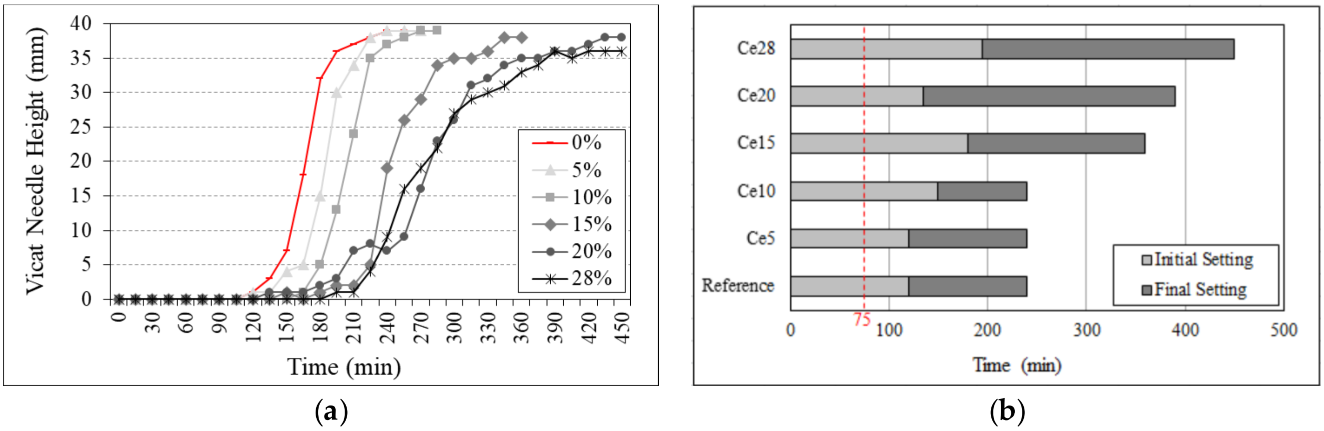

Figure 11. (**a**) The curve of Vicat needle heights, and (**b**) the initial and final times of setting.

3.2. The Fresh and Mechanical Properties of Cement Mortars

3.2.1. The Workability of Cement Mortars

The workability of the cement mortars was investigated by performing flow table tests. The spread flow diameters of mortars obtained from the tests are given in Figure 12. The use of ceramic wastes instead of limestone slightly decreased the spread flow diameters, between 1.89% and 3.96%. All mortars prepared with each cement type exhibited similar flow performance and their spread flow diameters ranged between 106.39 and 108.68 mm.

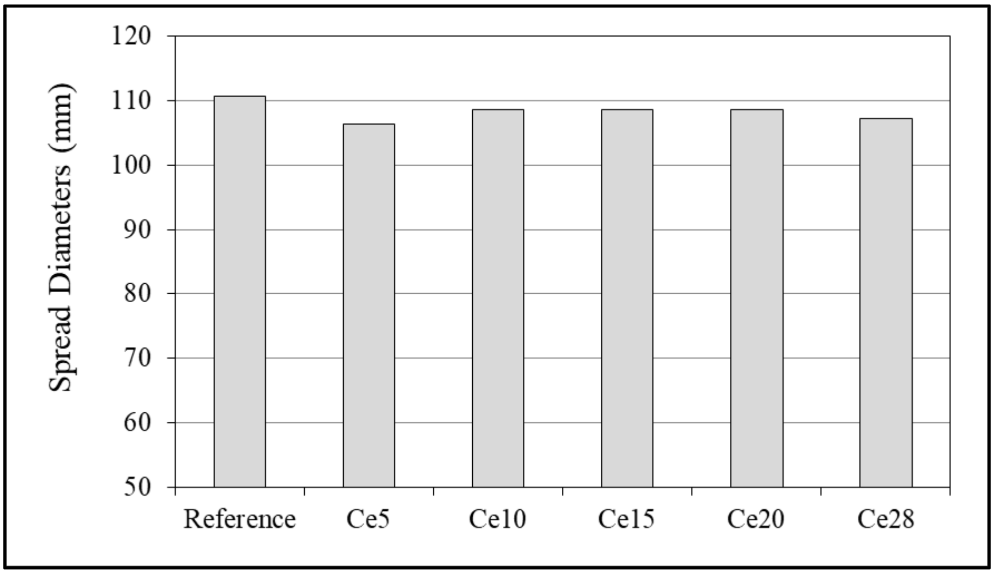

Figure 12. Spread flow diameters of cement mortars.

3.2.2. The Mechanical Properties of Cement Mortars

Flexural Strength of Cement Mortars

The flexural strengths of the cement mortars are presented in Figure 13, taking both cement type and curing age into consideration. The flexural strengths of mortars gradually increased with increased ceramic waste replacement, except for 2 days of curing. At 2 days of curing, there was a slight fluctuation in the flexural strengths of up to 15% ceramic waste substitution. However, all the flexural strengths were found to be higher than the reference mortar for all curing ages. Additionally, the highest flexural strengths were obtained from the ceramic waste substitution ratio of 28% for all curing ages. Unless the flexural strengths are not used in the strength classification of cement, the curing ages of 7 and 28 days are critical for the building industry. Therefore, it takes attention to examine the strength carefully for these ages. At the curing day of 7, flexural strengths were calculated to be higher than the reference mortar, between 3.24 and 22.03%, while at the curing day of 28, this increment ranged between 2.10 and 11.75%. Moreover, the flexural strengths at 365 days of curing were investigated and 13.46, 17.51, 33.93, 35.40, and 61.20% higher flexural strengths were achieved for the ceramic waste replacement ratios of 5, 10, 15, 20, and 28%, respectively.

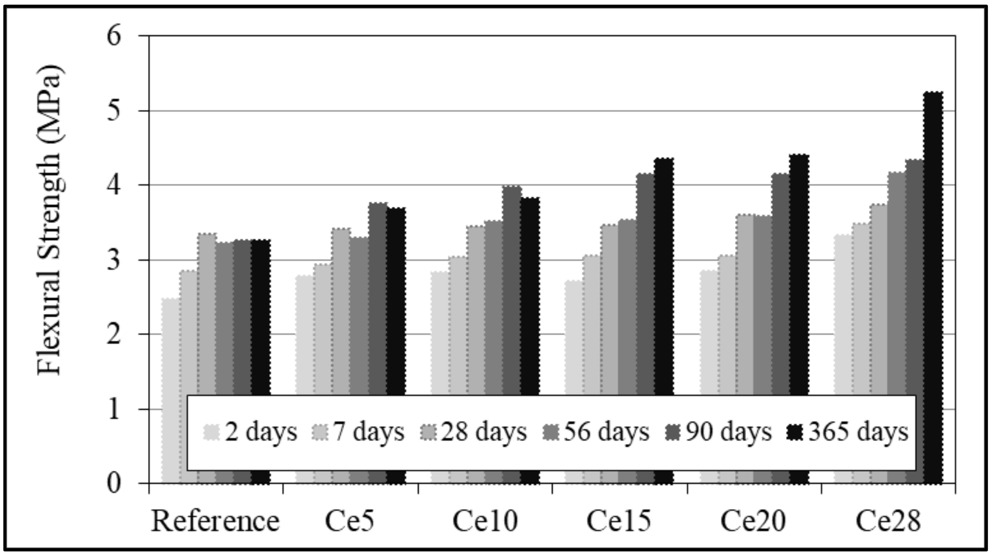

Figure 13. The flexural strengths of cement mortars considering all curing ages.

Compressive Strength of Cement Mortars

The compressive strengths of the cement mortars are presented in Figure 14, taking both cement type and curing age into consideration. Regardless of the cement type, the compressive strengths gradually increased with the increased curing age, and all the cements produced with ceramic waste either maintained the compressive strength or demonstrated higher compressive strengths than the reference. The compressive strengths tended to increase with an increased ceramic waste replacement of cement, generally. When the compressive strengths at 2 days and 28 days of curing were examined, all the cements exhibited higher compressive strengths than 10 MPa and 32.5 MPa, respectively. These curing ages are crucial for the determination of the strength grade of cement. Therefore, all the cements ensured the requirements of the EN 197-1 [5] standard and were classified as 32.5 R type cement. Additionally, all the cements exhibited a compressive strength of over 30 MPa at 7 days. To evaluate the moderate strength gain rates of the cements, the compressive strengths at 56 and 90 days were investigated. At 56 days of curing, 6.31, 13.66, 9.79, 13.66, and 25.52% higher compressive strengths than the reference cement were obtained for the ceramic waste ratios of 5, 10, 15, 20, and 28%, respectively. When the curing age was 90 days, these strength gains were calculated as 0.36, 8.49, 14.86, 11.79, and 19.58%. For the long-term strength comparisons, the compressive strengths at 365 days of curing were investigated. With the increased ceramic waste ratio from 5 to 28%, the compressive strengths were calculated as 6.53, 2.69, 7.49, 12.38, and 23.32% higher compared to the reference cement. Note that all the cements demonstrated compressive strength over 50 MPa in the long term. The ongoing strength gain with increased curing ages can be related to the probable pozzolanic reactivity of ceramic waste. During the production of cement, an increase in mechanical properties may have resulted from the addition of ceramic waste, which likely gained high pozzolanic activity due to heat treatment. This effect is more pronounced in the long-term mechanical results.

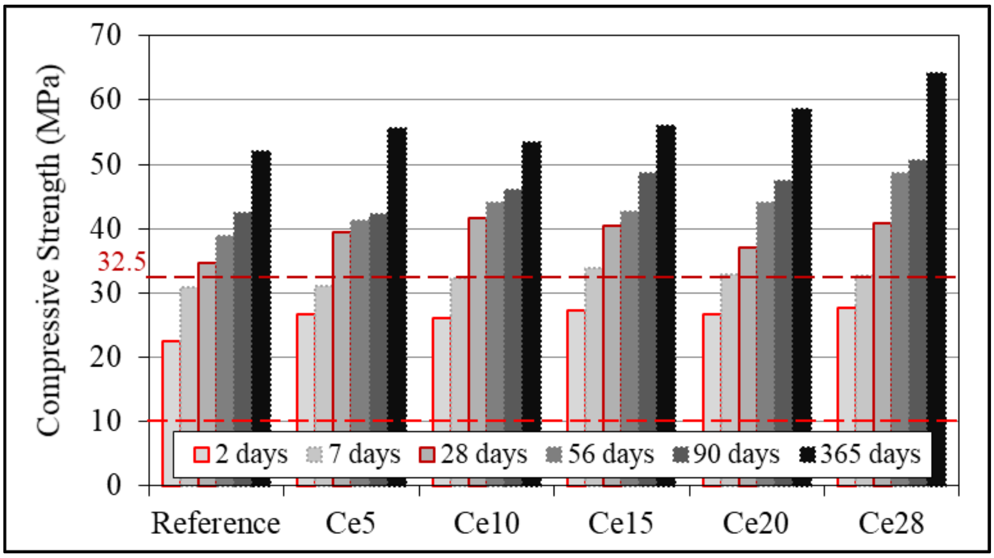

Figure 14. The compressive strengths of cement mortars, considering all curing ages.

4. Effect of Using Waste Ceramic on the Estimated CO_2 Emission

Although limestone-blended cement is emerging as a promising alternative to traditional Portland cement, the limestone used in cement production is still routinely extracted from nature through the quarrying of natural resources. This quarrying process not only harms the environment but also contributes to greenhouse gas emissions due to the use of heavy machinery for processes such as crushing, shredding, and grinding.

Approximately 0.85 tons of CO_2 are released for every 1 ton of clinker produced. Therefore, the CO_2 emissions of the reference cement, due to the clinker, were calculated to be approximately 0.54 tons of CO_2 per ton of cement. According to Shan et al. (2016) [28], the emission factor for lime was reported as 0.683 tons of CO_2 per ton of lime. By factoring in the emissions from the added limestone, the estimated total CO_2 emissions of the reference cement were calculated to be 0.72 tons of CO_2 per ton of cement. During the preparation of blended cements in this study, only the amount of limestone additive was reduced. Therefore, the estimated CO_2 reduction due to the decreased limestone additive ranged between 4.70% and 26.32% (Figure 15). It should be noted that this calculation does not include the CO_2 emissions from waste ceramics due to a lack of available data; however, it may provide insight for future studies, particularly with regard to the conservation of resources.

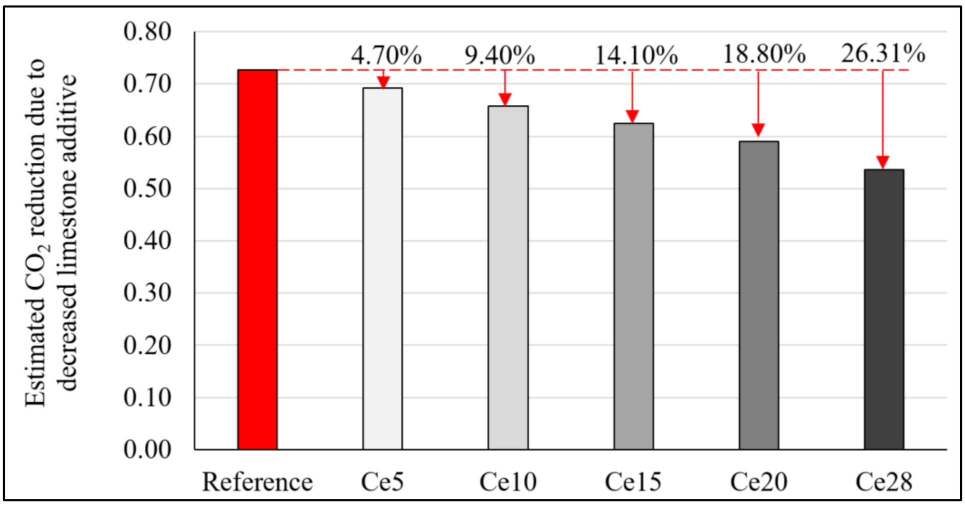

Figure 15. Estimated CO_2 reduction due to decreased limestone additive.

5. Conclusions

In this study, the possibility of using ceramic wastes in the production of blended cement was evaluated by substitution of limestone with ceramic waste at the percentages of 5, 10, 15, 20, and 28%. The following results were obtained:

- The chemical analysis showed that the SiO_2 content of cements was increased with an increased ceramic waste substitution ratio, while the $CaCO_3$ contents were decreased. Similarly, the peak intensity of SiO_2 was increased and the peak intensity of $CaCO_3$ was decreased in the XRD analysis of the cements.
- Since the hardness of ceramic wastes was higher than of limestone, the grindability of cements was decreased with the increased ceramic waste percentage, which decreased the Blaine specific surface area values.
- The consistency of water for cements was accepted as 28%, where all the cements achieved the standard limitations. However, the Vicat probe penetrated the cement pastes more easily at the same water consistency level, related to the decreased Blaine values.
- The spread diameters obtained for all types of cements were similar and practically usable in terms of workability.
- The cements including ceramic waste up to 10% maintained the setting time; however, when the ceramic waste substitution percentage increased to 15% and above, the setting time was prolonged.
- When the mechanical performances were taken into account, all the ceramic waste-included cements demonstrated higher flexural and compressive strength compared to the reference cement, regardless of the substitution percentage.
- The highest flexural strengths were obtained when the ceramic waste substitution was 28%, for all the curing ages.
- In the case of compressive strengths, all the cements exhibited higher compressive strength than 10 MPa at 2 days and 32.5 MPa at 28 days, which classified them as 32.5 R-type blended cements. The 10% ceramic waste-substituted cement exhibited the highest compressive strength; however, it is remarkable that over this replacement percentage, the compressive strengths were found to be similar.
- When the medium- and long-term compressive strengths were compared, the highest strength values were obtained from the cement with a 28% ceramic waste substitution.

When all results are taken into account, it is thought that by using waste ceramics in cement production, the manufacture of sustainable cements that exhibit better mechanical performance compared to traditional blended cements can be achieved, not only reserving natural limestone resources, but also recycling ceramic wastes.

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Conflicts of Interest: The authors declare no conflicts of interest.

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