



Research Article

Investigating the effect of elevated temperatures on the utilization of demolished paving block powders as supplementary cementitious materials

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Abstract: The study investigated the effects of construction waste powders exposed to elevated temperatures on the properties of cement mortar. The waste powders were obtained from demolished granite and clay blocks after more than 15 years of their service life. The exposure temperatures were 200 °C, 400 °C, 600 °C, and 800 °C. The heat-treated and untreated waste powders replaced cement in mortars at 10% and 20% by weight. The use of untreated recycled granite and clay powders adversely affected the mechanical strength and transport properties of the cementitious mixtures. On the other hand, the effects of thermal exposure varied for the two powder materials. As the exposure temperature increased, the performance of mixtures containing granite powder gradually deteriorated, while mixtures containing clay powder improved. The efficiency was both worst and best at 800 °C. For instance, a mixture containing 10% clay powder treated at 800 °C exhibited compressive strength equivalent to the reference mortar with no waste material. In contrast, under the same conditions, the compressive strength of the mixture containing granite powder was 33% lower. These results indicate that identifying the type and characteristics of recycled materials is essential for their utilization and application of enhancement methods.

Keywords: waste cementitious powder, thermal treatment, waste paving block, waste calcination, cement-based materials.

1. Introduction

Due to increasing global concern for the environment, various countries and institutions have proclaimed forward-looking goals such as a circular economy, zero waste, and net-zero emissions. One strategy for achieving these goals involves reducing waste, but there is still no clear sign of a decrease in the amount of waste generated. In Korea, the replacement of paving blocks, particularly carried out around the year-end for the execution of unused budgets, has been pointed out as a long-standing social bad practice. In 2023, a policy implemented by a city to 'abolish unnecessary year-end paving block replacement works' has been selected as one of the innovative cases by the Ministry of the Interior and Safety. The replacement of

paving blocks raises issues such as inconvenience in traffic and budget waste. From an environmental perspective, one critical consideration is the disposal of demolished paving blocks. Currently, well-preserved demolished blocks are provided to those in need, while severely damaged blocks are being disposed of in landfills. Therefore, effective strategies are needed to utilize blocks in poor condition.

Construction waste, such as demolished paving blocks, is often utilized as supplementary cementitious materials (SCMs) to reduce the environmental burden caused by the cement industry. In previous studies that utilized recycled powders obtained from concrete waste, brick waste, glass, clay, and stone dust as a partial cement replacement, it was reported that the pore structure of cementitious mixtures could be improved at low replacement rates (5%, 15%) due to the filling effects and pozzolanic reactions of recycled powders (Gleize et al., 2007; Likes et al., 2022; Silva et al., 2020; Silva & Delvasto, 2021). However, in general, the use of such recycled powders led to a deterioration in the performance of the final products (de Siqueira & Cordeiro, 2022). For instance, the 28-day compressive strength of concrete containing 30% recycled concrete/mortar powder was 30.5% and 28.8% lower than that of the control group (Yang et al., 2022). This is attributed to the dilution effect of cement when the replacement rate of alternative cementitious materials is high (Silva et al., 2019). Naturally, the dilution effect is dependent on the content of recycled powder, so recycled powders are recommended to be used at low replacement rates (Aquino Rocha & Toledo Filho, 2023; Xiao et al., 2018). This dilution effect is particularly pronounced in mixed construction waste, which can be possibly attributed to variables such as the presence of organic materials and averaging effects due to the integration of low- and high-quality recycled materials (Angulo et al., 2010; Montero et al., 2010; Tam & Tam, 2007). Some researchers have pointed out the difficulty in quality control of recycled materials as a barrier to their practical use (Kim, 2021; Silva et al., 2017; Tam et al., 2018). Therefore, for practical utilization, recycled materials of consistent and predictable quality are needed. The removal of paving blocks is often carried out by manually stacking the blocks on pallets or vehicles for transportation. In this operation process, there is the potential to obtain recycled materials with relatively homogeneous characteristics as the removed blocks remain uncontaminated by other materials.

Heat treatment has been reported as one method to improve the characteristics of recycled powders obtained from construction waste. For examples, concrete incorporating recycled cement powder calcined at 800 °C, with a replacement rate of 25%, exhibited a 35.3% increase in compressive strength compared to concrete containing non-calcined recycled cement powder (Kim & Kim, 2023). This is related to dehydration and decarbonation that occur at high temperatures. Wu et al. (2021) noted that the CaO formed due to this chemical reaction participates in rehydration in cementitious materials. Real et al. (2020) also observed that a significant amount of C₂S was formed in recycled powder treated over 600 °C and that it had high reactivity. At temperatures above 1000 °C, gypsum is formed in the recycled powder, reducing mechanical properties and durability. The optimal temperature range has been suggested to be between 600-800 °C.

Despite the contribution of calcination to the improvement of some recycled powder properties, powders obtained from construction waste other than concrete have been scarcely addressed. Therefore, this study aims to investigate the effect of calcination on granite and clay brick powders obtained from demolished paving block waste. Each powder was heated in the range of 200-800 °C and used to produce mortar samples by replacing 10% and 20% of Portland cement. The properties of the mortar samples, including workability, density, compressive strength, and capillary absorption, were tested, and the analysis was conducted based on replacement rates and temperatures. Additionally, the efficiency of calcination was discussed. This study can provide an initiative for the efficient utilization of construction waste.

2. Materials and methods

2.1. Materials

As materials for the preparation of mortar samples, ordinary Portland cement (OPC), complying with KS L5201 (KS L5201, 2016), was purchased from the market. For fine aggregate, river sand with specific gravity, water absorption, and fineness modulus of 2.59, 1.48%, and 2.62, respectively, was used.

The recycled powders used as SCMs were obtained from waste granite and clay blocks. The blocks were used for over 15 years in an area where freeze-thaw action occurs, and were demolished for renovation work (Kim & Kim, 2022). The demolished blocks were washed with tap water to remove debris and ground in a Los Angeles drum milling machine. By sieving,

granite powder (GP) and clay brick powder (CP) smaller than 0.15 mm were collected, respectively. The chemical composition and physical characteristics of GP and CP were investigated in the authors' previous work, and these are shown in Table 1, Figure 1 and Figure 2 (Kim et al., 2023). The CP, with a total content of SiO₂, Al₂O₃, and Fe₂O₃ exceeding 70%, satisfied the chemical requirements specified by ASTM C618 (ASTM C618, 2022) for pozzolan materials.

Table 1. Chemical composition and physical characteristics of recycled brick and recycled granite by percentage.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	CaO	TiO ₂	MgO	SO ₃	Specific gravity	Water absorption, %
OPC	13.11	2.84	4.62	1.48	73.39	0.28	1.05	2.82	3.14	-
GP	52.48	10.62	3.03	5.54	26.17	0.31	0.37	1.12	2.62	0.58
CP	64.22	18.98	7.57	5.10	1.75	1.21	0.77	0.11	2.26	7.70

Figure 1 shows the particle size distribution curves of the materials used in this study (river sand, OPC, GP, and CP) (Kim et al., 2023). The D₅₀ values for GP and CP were 47.7 μm and 48.5 μm, respectively. The particle sizes of both materials were quite similar, allowing for the elimination of the particle size difference as a variable in the interpretation of experimental results.

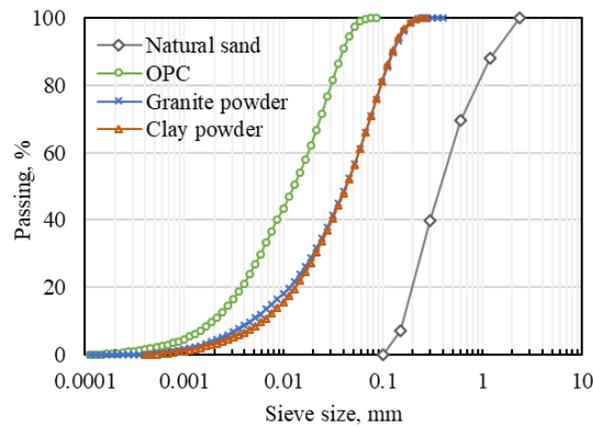


Figure 1. Particle size distributions of materials used in the study.

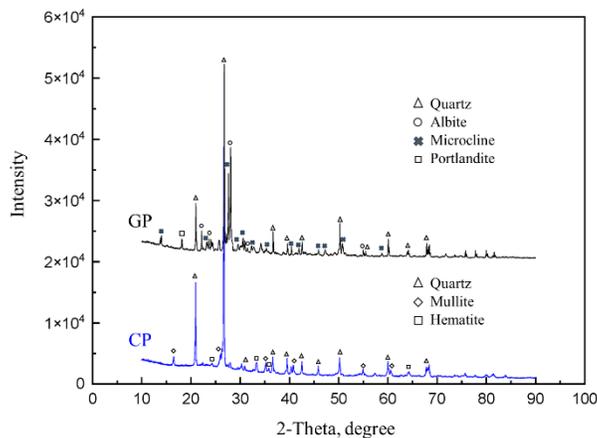


Figure 2. X-ray diffraction of granite and clay powders.

Figure 3 shows the results of thermogravimetric analysis (TGA) of GP and CP conducted under N₂ atmosphere and at a heating rate of 10 °C/min. At 900 °C, GP and CP exhibited total weight losses of 5.35% and 0.57%, respectively.

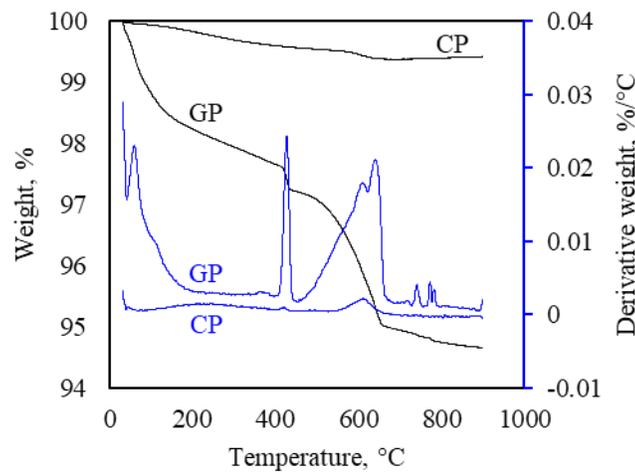


Figure 3. Thermogravimetric analysis of granite and clay powders.

Typically, clay blocks are manufactured by firing at high temperatures to achieve the desired strength. The thermal stability of the CP confirmed by TGA and the presence of mullite identified by X-ray diffraction suggest that the CP had previously undergone calcination (Akindahunsi et al., 2020; Tabana et al., 2020). The GP used in this study significantly differs in chemical characteristics from granite dust typically obtained from quarries reported in the literature (Singh et al., 2016). For instance, the high CaO content observed by X-ray fluorescence, the presence of portlandite identified by X-ray diffraction, and the weight loss due to the dehydroxylation of portlandite at 400-450 °C in TGA indicate the presence of cement components in GP. This is presumed to be because the cement mortar used for leveling and adhesion during the installation of granite blocks was likely mixed during demolition, and these characteristics demonstrate the quality variables that can actually occur in demolition waste.

2.2. Heat treatment of granite and clay powders

To investigate the influence of elevated temperature, GP and CP were contained in alumina crucibles and heated in a furnace at 200 °C, 400 °C, 600 °C, and 800 °C, respectively. The temperature increased at a constant rate of 10 °C per minute, and the exposure duration was 2 hours from the point of reaching each target temperature. After the completion of the heating process, the powders were cooled down to room temperature inside the furnace (Figure 4).



Figure 4. Granite and clay powders at elevated temperatures.

2.3. Mortar compositions

In this study, a total of 21 different mortar samples were prepared, including a reference mortar with only OPC as the cementitious binder. Table 2 shows the compositions of various mortar samples containing GP and CP with different exposure temperatures and replacement rates. Each mixture had proportions of 0.5:1:3 for water, cementitious binder, and sand, respectively. The heat treated and untreated GP and CP replaced OPC by 10 wt.% and 20 wt.%. Mortar samples were labeled following the format: 'mixture type - replacement ratio - exposure temperature'. For example, GM10 denotes the GP-based mortar where untreated GP replaces 10% of OPC. CM20-800 stands for the CP-based mortar where 20% of OPC is replaced with CP treated at 800 °C.

Table 2. Mix proportion of mortar with waste cementitious powders in grams.

No.	Mix	Cement	GP	CP	Water	Sand
1	Reference	450	0	0	225	1350
2	GM10	405	45	0	225	1350
3	GM10-200	405	45	0	225	1350
4	GM10-400	405	45	0	225	1350
5	GM10-600	405	45	0	225	1350
6	GM10-800	405	45	0	225	1350
7	GM20	360	90	0	225	1350
8	GM20-200	360	90	0	225	1350
9	GM20-400	360	90	0	225	1350
10	GM20-600	360	90	0	225	1350
11	GM20-800	360	90	0	225	1350
12	CM10	405	0	45	225	1350
13	CM10-200	405	0	45	225	1350
14	CM10-400	405	0	45	225	1350
15	CM10-600	405	0	45	225	1350
16	CM10-800	405	0	45	225	1350
17	CM20	360	0	90	225	1350
18	CM20-200	360	0	90	225	1350
19	CM20-400	360	0	90	225	1350
20	CM20-600	360	0	90	225	1350
21	CM20-800	360	0	90	225	1350

2.4. Specimen preparation and test methods

All mortar samples were prepared using an automatic mixer programmed with the procedures of ASTM C305 (ASTM C305, 2020). After mixing each mortar, the flowability was tested using a flow table according to ASTM C1437 (ASTM C1437, 2020). For the density and compressive strength, 50 mm cube specimens were prepared. Both tests were conducted on three specimens after 28 days of water curing. Density was determined by dividing the saturated surface dry weight of the cube specimen by its volume. Compressive strength was determined by dividing the load at the failure point by the cross-sectional area. Capillary absorption was measured for nine days on cylindrical disks with a diameter of 100 mm and a height of 50 mm according to ASTM C1585 (ASTM C1585, 2020).

3. Results

3.1. Flow

The flow table test results for mortar samples are shown in Figure 5. The flow of the reference mortar was 196 mm, and the flows of GM and CM with untreated powders at a replacement rate of 10% were 190 mm and 201 mm, respectively, which were not significantly different from that of the reference mortar. However, as the replacement rate increased to 20%, the flows of GM20 and CM20 slightly decreased to 180 mm and 193 mm. This reduction can be attributed to the rough particle shape of GP and the high-water absorption of CP.

Regarding the effect of heat exposure of the powder, GP treated at elevated temperatures up to 800 °C had no noticeable effect on the flow of the mortar. The flow of GM10 series ranged from 185-190 mm, and that of GM20 series ranged from 178-184 mm. Conversely, as the exposure temperature of CP increased, the corresponding mortar flow showed a tendency to decrease. The flows of CM10-800 and CM20-800 were approximately 10% lower than those of CM10 and CM20. According to previous studies (Carriço et al., 2020), calcination of CP increases the specific surface area and activity, which increases the water demand for CP-based mortars. Nevertheless, all mixtures had adequate workability required for sample preparation.

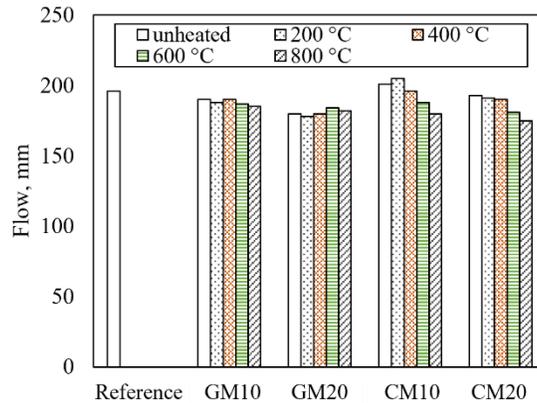


Figure 5. Flow of mortar with granite and clay powders treated at elevated temperatures.

3.2. Density

The density of the prepared mortars is presented in Figure 6. In comparison to the reference mortar, the densities of GM10 and GM20 decreased by 0.3% and 1.8%, respectively. For the CM series, the density decreased by 1.9% to 4.1%, indicating that the influence of CP on density reduction is greater than that of GP. These results can be explained by the difference in specific gravity between OPC and the recycled powder replacing OPC. As indicated in Table 1, the specific gravity decreases in the order of OPC, GP, CP (3.14, 2.62, 2.26, respectively), and the density of the mixtures follows a similar trend: reference mortar (2184 kg/m³), GM (2176-2145 kg/m³ at 10% and 20% replacement), CM (2143-2094 kg/m³). These results are in good agreement with previous studies (Arif et al., 2021; Duan et al., 2020).

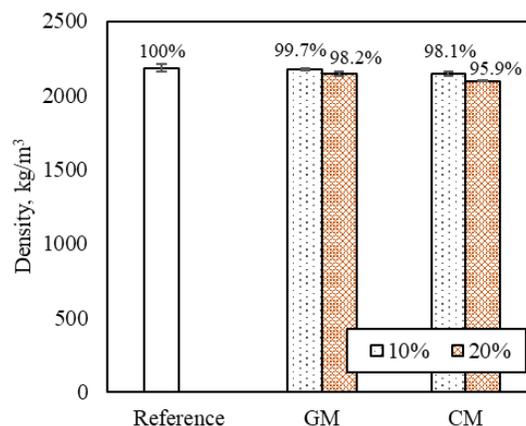


Figure 6. Density of mortar with untreated granite and clay powders.

The densities of mortars containing GP and CP treated at elevated temperatures are shown in Figure 7, the graph clearly shows that the elevated temperature exposure of GP and CP had opposite effects on the density of the cementitious mixture.

The GP treated at elevated temperatures reduced the density. When cement was replaced by 10% and 20% with GP treated at 800 °C, the density decreased by 1.1% and 1.7%, respectively. In contrast, thermal exposure of CP increased the density of the cementitious mixture. Under the same conditions as GP, the density of CP-based mortar increased by up to 1.6%. The interpretation of these divergent behaviors is elaborated in the Discussion section.

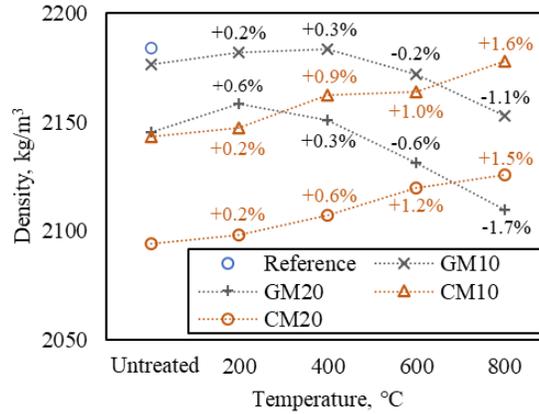


Figure 7. Density of mortar with granite and clay powders treated at elevated temperatures.

3.3. Compressive strength

The 28-day compressive strength is presented in Figure 8. The use of GP and CP decreased the compressive strength of the mixture. At replacement rates of 10% and 20%, the compressive strength of GM was reduced by 15.4% and 23.1% compared to that of standard mortar. Similarly, at given replacement rates, the compressive strength of CM was also reduced by 11.4% and 28.4%, respectively. The loss of compressive strength with increasing replacement rates is commonly reported results in studies involving the use of GP and CP as cement replacement (Kim et al., 2023; Li et al., 2019; Wu et al., 2021). This is primarily associated with the reduced cement content due to the use of SCMs (GP and CP in this study), resulting in the formation of fewer hydration products necessary for strength development. Furthermore, as shown in Figure 1, the larger particle size of GP and CP used in this study compared to OPC may also be one of the reasons for the reduction in strength caused by the insufficient filling effect. However, mixtures containing GP and CP as SCMs have been reported to exhibit superior compressive strength compared to the control group at later ages (e.g., 90-360 days) (Ramadji et al., 2020; Silva et al., 2021, 2024). Therefore, there is a need for additional investigation of their long-term strength.

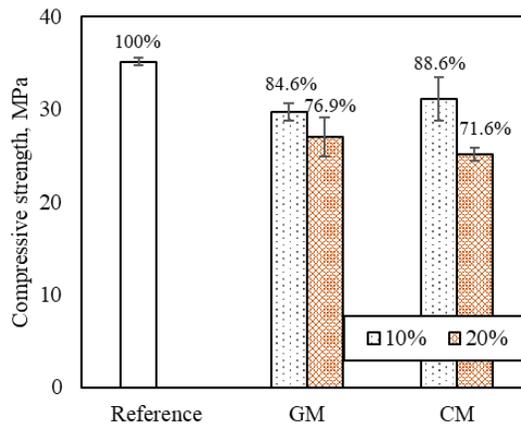


Figure 8. 28-day compressive strength of mortar with granite and clay powders.

The compressive strength of mixtures containing GP and CP treated at different temperatures is presented in Figure 9, showing that treated GP and CP have varying effects on the compressive strength. With the rise in exposure temperature, GP decreased while CP increased compressive strength. Up to an exposure temperature of 800 °C, GP decreased the compressive strength by 21.4%. In contrast, under the same conditions, CP increased the compressive strength by 13% and 17% at replacement rates of 10% and 20%, respectively. The effect of calcined CP on enhancing compressive strength was also reported by Zhang et al. (2023). In particular, CM10-800 achieved compressive strength equivalent to the reference mortar, indicating that utilizing calcined CP at a low replacement rate has the potential to produce cementitious mixtures without strength loss while reducing cement content.

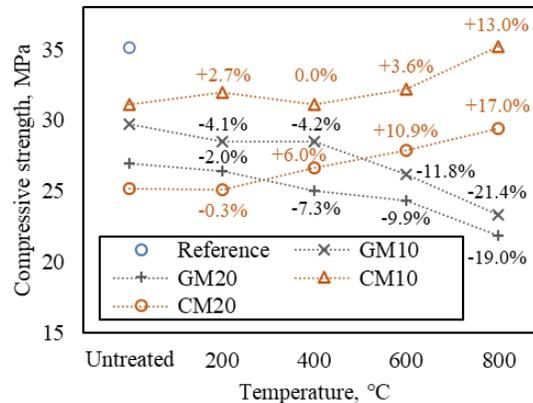


Figure 9. Compressive strength of mortar with granite and clay powders treated at elevated temperatures.

3.4. Capillary absorption

Investigating the moisture transport properties of cementitious mixtures is crucial from a durability perspective, as substances such as chlorides and sulfates, which diminish the durability of cementitious mixtures, move through water as a medium. The absorption rates by capillary action of the prepared mortars are shown in Figure 10. Interestingly, the sorptivity of mortars containing GP was lower than that of standard mortar at replacement rates of 10% and 20%, measuring 1.34 mm and 1.57 mm, respectively. This could be attributed to the combined effect of the low water absorption of GP and the filling effect. Indeed, lower water absorption of GP-based mortars compared to standard mortar has been reported in some studies (Lu et al., 2023; Mashaly et al., 2018). For CM series, a remarkably high capillary absorption (2.29 mm) was observed at a replacement rate of 10%, which is an expected result considering the high absorption capacity of CP. As the replacement rate increased from 10% to 20%, the absorption capacity of both GM and CM increased, indicating that the pore structure of the mixture was loosened due to the reduced cement content by the recycled powders.

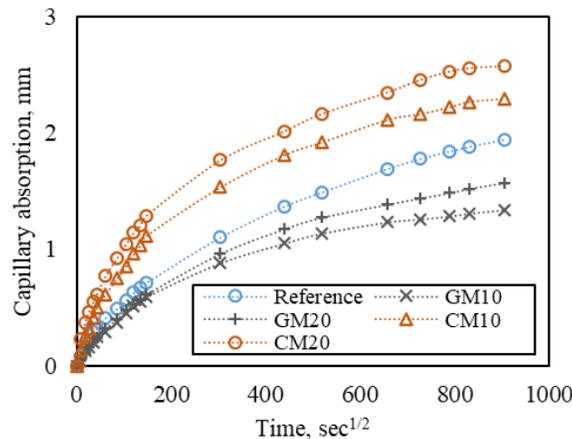


Figure 10. Capillary absorption of mortar with untreated granite and clay powders.

The test results of capillary absorption for mortars containing GP and CP treated at different temperatures are shown in Figure 11. Incorporating GP heated to 800 °C increased the water uptake by capillary action by 22.1% and 8.3% over the mixture with untreated GP at replacement rates of 10% and 20%, respectively. In contrast, as the exposure temperature of CP increased, the capillary absorption of the mixture decreased. The absorption rates of CM10-800 and CM20-800 were 33.9% and 23.0% lower than CM10 and CM20, respectively. This suggests that elevated temperatures adversely affect the pore structure in the case of GP, while CP enhances it.

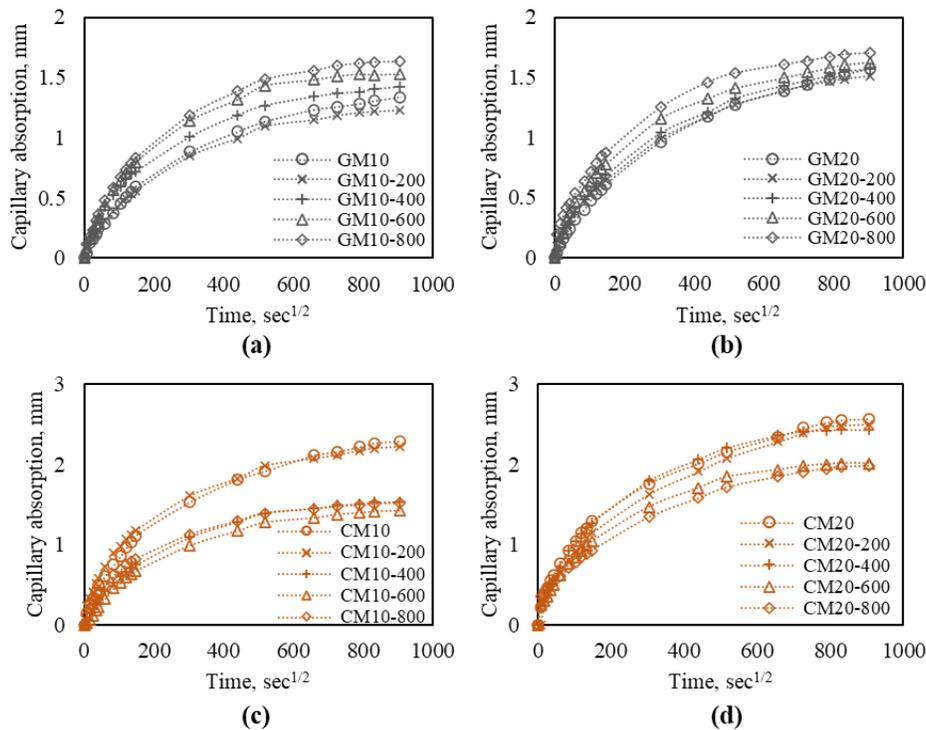


Figure 11. Capillary absorption of mortar with granite and clay powders treated at elevated temperatures: (a) GM10 , (b) GM20, (c) CM10, and CM20.

4. Discussion

4.1. Effect of replacement rate

The experimental results of this study showed that the use of both untreated GP and CP as cement replacements had an unfavorable effect on the properties of the mortar, and the performance of the mortar decreased as the replacement rate increased. Generally, when using recycled materials without improvement techniques, the performance of mortars with recycled materials does not surpass that of standard mortar without recycled materials. This is attributed to characteristics such as the porosity and high-water absorption of recycled materials, as well as damage, such as micro cracks, that occurs during waste processing into recycled materials. Moreover, when recycled materials are used as SCMs, the inferior performance is noticeable due to the dilution effect. For these reasons, GP and CP are recommended for use at low replacement rates (i.e., 5-15%) (He et al., 2021).

4.2. *Effect of elevated temperature*

Depending on the type of recycled powder, the influence of exposure to elevated temperature was different. According to the experiments in this study, the performance of GP-based mixtures gradually decreased with increasing temperature, whereas CP improved the strength and transport properties of the mixtures. This is attributed to the fact that GP and CP undergo different physicochemical reactions at high temperatures.

The quartz component in granite, known as alpha quartz at room temperature, undergoes an inversion to beta quartz at 573 °C, causing thermal expansion (Van der Molen, 1981). Furthermore, exposure to temperatures between 500 °C and 900 °C induces uneven thermal stresses, leading to the breakdown of connections between mineral particles, and a significant increase in porosity (Huang et al., 2021; T. Ma et al., 2021). This heat-induced damage in GP plays a role in reducing the performance of the mixture and well supports the observed performance loss in GM-600 and GM-800.

At 550-800 °C calcination, CP forms pozzolanic amorphous materials such as metakaolin depending on the type of mineral (C. He et al., 1995). The amorphous phase reacts with CH, generating new CSH and contributing to the improvement of the mixture's performance (Zhang et al., 2023). The effectiveness of calcined CP as a SCM has been reported in several studies. However, it is essential to emphasize that the CP used in this study has been calcined once in the past to produce sidewalk blocks. Hence, the calcination of CP performed in this study should be considered as re-calcination, which is particularly important in terms of waste utilization. Kovářík et al. (2015) reported that re-calcination at temperatures between 650 °C and 800 °C increased the specific surface area and reactivity of CP. This finding supports the performance improvement observed in CM-600 and CM-800 in this study.

Interestingly, elevated temperature exposure has been regarded by some researchers as an enhancement method, referred to as thermal activation (Kim & Kim, 2023; Wu et al., 2021), while others perceive it as a result of fire damage (Z. Ma et al., 2021). In other words, investigations into elevated temperature exposure provide not only information as a strengthening method but also insights into the potential utilization of fire-damaged materials. That is, regardless of whether it is thermal activation or fire damage, CP exposed to heat can be considered a promising and feasible option for use as a SCM, whereas this is not the case for GP. The different effects of thermally treated powders on the properties of cementitious mixtures can complicate the practical utilization of recycled materials, especially those mixed with various types of waste. To make more valuable use of materials generated from construction and demolition activities, the European Commission has established guidelines for waste audits before demolition and renovation (European Commission, 2018). This involves identifying the types of waste, as well as estimating the quantity of waste to be reused and recycled in advance of work. Moreover, selective dismantling by material type is recommended if possible. Unlike buildings composed of various types of materials, pavements typically consist of a single type of paving block. Thus, paving blocks are usually not mixed with other materials during demolition works, and can be considered to have a high potential for recycling.

4.3. *Efficiency of heat treatment of clay powder*

The variation of 28-day compressive strength and power consumption at elevated temperatures are shown in Table 3. For GM mixtures, these calculations are of no practical significance and were excluded as their strength deteriorates at elevated temperatures. Data on power consumed for calcination were referenced from the literature (Zhang et al., 2023). Calcination at 400 °C, 600 °C, and 800 °C consumes power of 13.86 kW.h/t, 16.63 kW.h/t, and 18.71 kW.h/t. At given temperatures, the strength of the CM10 series increased by 0.0%, 3.6%, and 13.0%, and that of the CM20 series increased by 6.0%, 10.9%, and 17.0%. To determine the efficiency of elevated temperature exposure, the power consumption was divided by the strength variation, which means the power consumed to increase compressive strength by 1%. As shown in Table 3, CP treated at 800 °C required the lowest power consumption to increase 1% of compressive strength at both 10% and 20% replacement rates, measuring 1.43 kW.h/t and 1.10 kW.h/t, respectively. While the use of thermal energy for enhancing the reactivity of recycled powder may not seem desirable from a sustainability perspective, it is essential to note that the calcination temperature required to achieve optimal performance for recycled powder is significantly lower than that of cement clinker production.

Table 3. Power consumption and strength change by calcination.

Mix	Power consumption (kW.h/t)	Strength variation (%)	Power for increasing 1% of strength
Reference	23.1	-	-
CM10	0	-	-
CM10-400	13.86	0.0	0.00
CM10-600	16.63	3.6	4.60
CM10-800	18.71	13.0	1.43
CM20	0	-	-
CM20-400	13.86	6.0	2.30
CM20-600	16.63	10.9	1.53
CM20-800	18.71	17.0	1.10

5. Conclusions

In this study, recycled powders obtained from demolished paving blocks were treated at elevated temperatures and their effect on the properties of cementitious mixtures was investigated. The following conclusions were drawn:

1. Incorporation of untreated GP and CP into cementitious mixtures resulted in a deterioration in the properties of the mixture, and this loss became more pronounced as the replacement rate increased.
2. The effects of heat treatment on the properties of GP and CP mixtures varied. As the heat treatment temperature increased, GP-based mortars exhibited a gradual decline in properties (reduced density and compressive strength, increased capillary absorption), whereas CP-based mortars showed an increase in those properties.
3. The calcination of CP at 800 °C showed the most favorable 'power consumption/strength increase' efficiency compared to that at other temperatures investigated in this study.
4. The different effects of elevated temperatures on GP and CP indicate that identifying the characteristics of materials to be recycled may be required before use to maximize recycling efficiency. Methods such as waste audits could be considered for this purpose.
5. This study provides information on the recycling of demolished paving block waste at the end of its service life. Given the focus on the engineering properties of cementitious mixtures in this study, microstructure analysis is recommended for further research to elucidate the mechanisms behind these effects.

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Conflicts of interest: The authors have no relevant financial or non-financial interests to disclose.

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