Evaluation of the Enhancement of the Mechanical Properties of Cement Mortar Incorporated with Porcelain and Marble Powder

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ABSTRACT

It is widely accepted that cement and concrete are substantial sources of CO_2 emissions. Researchers have been looking to replace cement with industrial waste to reduce the environmental impact of concrete production. The current research focuses on successfully using marble powder (M) and porcelain powder (P) instead of cement to produce cement mortar. Both mechanical and microstructural characteristics of several single and binary mixtures consisting of M and P were examined, with 13 mixes with M and P having been replaced by 2.5, 5, 7.5, and 10% of cement weight. For all mixtures in this work, compressive strength, flexural strength, SEM analysis, and thermogravimetric analysis (TG/DTA) were applied. The mixtures containing 10% M, 10% P, and 5% M+5% P demonstrated the best results. The maximum values recorded in compressive strength were 52, 55, and 50 MPa, whereas the control mix had 30 MPa. The maximum values for flexural strength were 9.24, 10.64, and 8.4 MPa in comparison with the 7 MPa of the control mix. Moreover, SEM analysis demonstrated the existence of a thick and compacted microstructure, which is predominantly the result of the formation of both Calcium Hydroxide (C-H) and

high-density Calcium Silicate Hydrate (C-S-H) phases in all blends. SEM images demonstrated how hydration compounds formed and how the presence of M and P improved bonding. The difference in the quantities and types of phases distinguished by the different types of substitute material was also noted. The decrease in C-H, which is most prominent after a hydration period of 28 days, is primarily attributed to the interaction between C-H and M and P. Thus, this study demonstrates that M and P, by themselves or in combination, can be utilized as alternative resources in the production of high-performance and sustainable concretes.

Keywords-marble powder; porcelain powder; sustainable concrete

I. INTRODUCTION

The swift expansion of industrialization and infrastructure will result in the depletion of natural materials for construction. Furthermore, industries generate a substantial amount of waste that has detrimental effects on the environment. To address this concern, a potential solution is to explore alternative approaches to reducing the excessive consumption of natural resources. An example of such an approach could involve the rational utilization of industrial waste [1]. It has been estimated that more than 30% of the daily production within the ceramic sector lasts in some form of waste. Presently, ceramic waste, which possesses long-lasting qualities, hardness, and a high level of resistance against biological, chemical, and physical deterioration, is not being systematically recycled. The ceramics industry faces significant pressure due to the rapid escalation of waste production, urging them to identify an appropriate disposal solution. Meanwhile, conventional crushed stone aggregate stocks are being depleted, particularly in certain arid regions [2]. The utilization of inorganic industrial byproducts in the production of concrete will give rise to the development of sustainable concrete design, thereby fostering a more environmentally friendly production system [3, 4]. Marble, a metamorphic rock, is significant as a decorative construction material. It is worth noting that approximately 20-30% of a marble block is transformed into marble powder during the cutting procedure. Thus, it must be acknowledged that the presence of marble powder poses risks to both human health and the environment [5]. Therefore, it is essential to provide workable strategies for recycling these resources, especially those with the highest level of integrated energy. One of the best ways to recycle these waste materials is to incorporate them into mortar and concrete production. Such re-use would not only take advantage of the incorporated energy, but also the number of refuse tips would be reduced [6]

Several researchers have employed marble (M) and porcelain (P) powder as alternatives to cement. Authors in [7] observed an increase in compressive strength at 28 days by up to 92.22% when incorporating 50% of P residue as an additive. Additionally, they found that this approach reduced equivalent CO_2 emissions by up to 38.18%. Therefore, using P waste in concrete development offers the potential to create more resistant and durable structures than those generated with the employment of traditional concrete. Authors in [8] explored the impact of replacing cement with waste ceramic powder and found it had pozzolanic properties, impeding strength development and flowability to the mortar. Authors in [9] investigated the properties of concrete blended with M and P powder within the range of 0-10% by weight. The findings indicated that the compressive and splitting tensile strengths of the 2.5% M + 2.5% P sample exhibited an increase of 8.90% and 8.30%, respectively [9]. Authors in [10] conducted a study investigating the impact of substituting cement with waste from marble and granite (MGW) at various water-to-cement ratios (w/c). The findings indicated that the MGW functioned as a filler, improving the particle packing, preserving compressive strength, and refining the porous structure. Authors in [11] determined that the utilization of marble dust at a substitution rate of 8% led to a decline in the efficacy of the cement mixture. Conversely, the integration of both marble dust and crushed brick in the cement yielded a noteworthy enhancement in the potency of the cement mixture compared to that of the marble dust-infused cement mixture. Authors in [12] replaced cement with ceramic waste powder by 10%, 15%, and 20% by weight and found that the compressive strength of concrete increased when the addition of dosage was less than 15%. Authors in [13] determined that the replacement of traditional fine aggregate and cement with P results in a notable augmentation in compressive strength, rendering it appropriate for cement mortar production. Authors in [14] asserted that the incorporation of M residue as a partial replacement did not impact the process of cement hydration. However, it did offer enhancements in terms of packaging and served as a filler material. In [16], M dust powder was utilized as a substitute for cement by 0% to 30%. The outcomes of this substitution demonstrated a commendable enhancement in both the strength and quality aspects, surpassing those achieved by conventional concrete. Notably, it has proven to be particularly productive in ensuring high cohesiveness within mortar and concrete. Authors in [17] reported that including 10% M powder as a substitute for cement led to enhanced concrete microstructure and increased strength. Authors in [18] documented that the incorporation of M powder with cement at a 10% substitution level is a feasible option due to the absence of any detrimental impact on the technical properties of the mixture. Furthermore, their findings indicated that substituting beyond the 10% threshold results in a retardation of the hydration process, leading to increased porosity and decreased compressive strength. Authors in [19] replaced cement with marble powder by 0%, 5%, 10%, 15%, and 20% by volume and concluded that marble powder could be replaced by up to 10%, reducing the need for cement and enabling the efficient use of waste products [19].

Even though there are a few studies regarding the use of P and M waste in concrete or cement mortar, only a few studies have used M and P powders simultaneously as potential alternatives to cement. Furthermore, because these wastes are produced in huge numbers locally, this research is becoming increasingly important to solve issues associated with them at the local level. In the current study, the primary inquiry was the

employment of Supplementary Cementitious Materials (SCMs) procured from industrial sources to diminish the construction sector's carbon footprint. The present study aims to elucidate the impacts of P and M dust, either separately or in conjunction, on structural mortar's fundamental characteristics and determine the appropriate replacement ratio.

II. EXPERIMENTAL WORK

A. Materials

Sand supplied from Arena, complying with the requirements of EN 196-1:1994 and ISO 679: 2009 [20], was used in the mortar mixes (sieve analysis can be seen in Table I). In this study, Ordinary Portland Cement (OPC) of type I, which conforms to the Iraqi Standard Specifications No. 5, was employed [21]. The chemical composition of this cement is detailed in Table II. Additionally, a Glenium 54 superplasticizer with a density of 1.08, classified as type F, was utilizeed in the mixtures. M dust powder M and P waste powder (P), serving as alternatives to cement, were incorporated into the mixes.

TABLE I. SAND SIEVE ANALYSIS

Size (mm)	Passing %
2	100
1.6	95
1	65
0.8	33
0.16	8
0.08	0.5

TABLE II. CEMENT CHEMICAL ANALYSIS

Oxide	Content%
CaO	63
SiO_2	24
Al_2O_3	4
Fe_2O_3	5
MgO	1.1
SO_3	1.64
L.O.I	3.38
Chemical compound	**Content%**
C_3S	55
C_2S	16
C_3A	3
C_4AF	15

The chemical composition of M and P are presented in Table III ascertained with the XRF method. The M powder originated from crushing marble sludge, a byproduct of the local marble-cutting industry. The characterization of the M powder was performed deploying established international experimental tests. The P waste was initially processed in a jaw crusher and then sieved with a 1.18 mm sieve to remove larger fragments. The ceramic material was further refined in a modified Los Angeles abrasion machine, which is equipped with six stainless steel bars, each 18 mm in diameter and 800 mm in length. Only the fine ceramic particles that successfully passed through a 45 µm sieve at an 80% rate were collected and used in the blending process [22, 23]. The color of the ceramic powder following its treatment exhibited a shade of dark grey, which closely resembled that of conventional OPC. Subsequently, the ceramic powder was employed as a substitute material for cement upon being subjected to grinding. Both replacements have particle sizes less than 75 µm. Figure 1 represents the graph of M and P powders.

TABLE III. CEMENT PHYSICAL PROPERTIES

Property	Value
Initial setting time (hr:min)	1:20
Final setting time (hr:min)	3:05
Fineness (m^2/kg)	305
Compressive strength (MPa)	22
Compressive strength (MPa)	26

TABLE IV. CHEMICAL ANALYSIS OF M AND P POWDERS

Oxide%	M	P
SiO_2	22.5	71.8
Al_2O_3	1.8	13.4
CaO	42	3.06
Na_2O	1.5	1.45
K_2O	1.2	5.7
MgO	0.75	0.71
SO_3	3.2	0.04
Fe_2O_3	0.4	3.45
$MgCO_3$	27	-
Cr_2O_3	1.6	0.311

(a)

(b)

Fig. 1. (a) Porcelain powder, (b) marble powder.

B. Mixture Design

For this study, 13 mixes were designed and cast. Twelve combinations were created by substituting cement with M, P, or both, while one control mixture (without any substitution) was also made. The water-to-binder ratio was set to 0.4, and the cement-to-sand ratio to 1 to 2.75. Table V shows the proportions of the mortar mix. A pan-style mixer was utilized to combine the components for the mortar. The mixing was the following: Initially, the mixer was filled with all the dry ingredients, including sand, cement, M, and P powders (if applicable), and it was mixed for 2 min. The superplasticizer and water were introduced after they had been previously combined. After that, the mix was mixed for another 2 min. The wet materials were mixed for one additional min after

being stopped for 30 s. After mixing, the freshly created mortar was poured into cube and prism molds. The molds were removed after around 24 h, and the samples were placed in a water basin to cure until testing.

TABLE V. DETAILS OF MIX PROPORTIONS

Mix symbol	Cement (g)	Sand (g)	Marble (g)	Porcelain (g)
C	800	2200	0	0
M2.5	780	2200	20	
M5	760	2200	40	
M7.5	740	2200	60	
M10	720	2200	80	
P2.5	780	2200		20
P5	760	2200		40
P7.5	740	2200		60
P10	720	2200		80
MP2.5	780	2200	10	10
MP5	760	2200	20	20
MP7.5	740	2200	30	30
MP10	720	2200	40	40

III. TESTING

A. Compressive Strength

Compressive strength testing was conducted on cubic samples with dimensions of 50×50×50 mm. The compressive strength, expressed in MPa, was determined by dividing the load that caused the sample to fail (N), by the cross-sectional area of the specimen (mm^2). Three samples were tested in accordance with ASTM C109 [24], and the average value was considered. A test specimen is depicted in Figure 2. Cement ages of 14 and 28 days were selected for evaluation.

Fig. 2. Compressive strength test.

B. Flexural Strength

The flexural strength test on the mortar samples was carried out following the guidelines of BS EN 196-1 [25], as illustrated in Figure 3. To determine the flexural strength, prisms with dimensions of 40 × 40 × 160 mm were utilized. The calculation of the flexural strength involved taking the average of the results of three such prisms. This test was systematically performed when the mortar samples reached 28 days of curing.

C. SEM Characterization

Broken sample pieces were used for this purpose after the mechanical testing (28-days) for Scanning Electron Microscopy (SEM). This test was done implementing the Philips XL30 model. Prior to the SEM analysis these pieces underwent processing with a coater device. This step is essential because the intrinsic nature of concrete does not allow it to conduct electron rays. Therefore, the samples are coated with conductive materials, such as gold, to facilitate the electron microscopy examination.

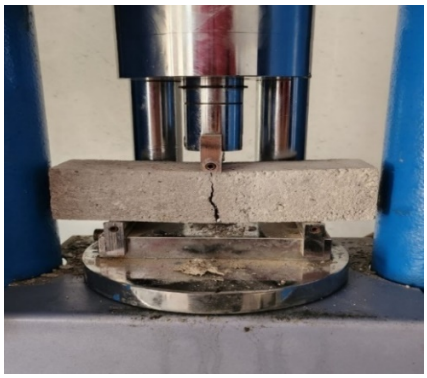


Fig. 3. Flexural strength test.

D. Thermal Analysis

Eleven samples underwent thermogravimetric analysis (TG/DTA) using the TG 209 thermo-analyzer (NETZSCH). The powder samples were placed in a ceramic crucible and heated under static conditions. Nitrogen was deployed as the medium, and the temperature was increased at a rate of 10 °C/min, reaching up to 1000 °C. Powdered alumina was utilized as the reference material. The DTA and TG processes were run concurrently. Prior to TG/DTA analysis, all samples were refrigerated at room temperature and dried in an oven set to 105°C.

IV. RESULTS

A. Mechanical Properties

The results of the mortar compressive strength tests at 14 and 28 days are displayed in Figures 5-7. Fourteen days later, all mixes containing waste in lieu of cement demonstrated an enhancement in compressive strength. It was also observed that the compressive strength increased with the replacement rate. In each replacement type, the mix with the greatest replacement (10%) demonstrated greater compressive strength than that of the other mixes. In the same context, the compressive strength outcomes at 28 days exhibited a similar trend to those observed at 14 days, but with an increased rate of growth. This enhancement is attributed to the continuous hydration of the cement and the pozzolanic reactions of the waste materials over time. This increase in compressive strength can be linked to the pozzolanic activities of both M and P, which contain a significant amount of calcium oxide, as evidenced in Table IV.

Corresponding outcomes with M powder have been also reported in earlier studies [17, 26]. Additionally, marble comprises silica, calcium oxide, and alumina, which interact with calcium hydroxide during the hydration of cement to produce additional C-S-H gel [27-29].

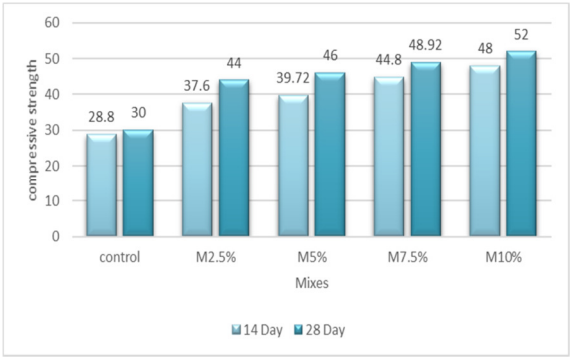


Fig. 4. Results of compressive strength test of M mixtures at 14 and 28 days.

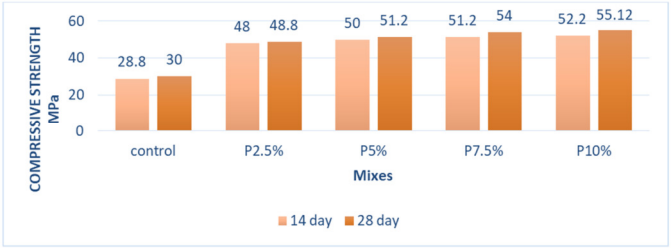


Fig. 5. Results of compressive strength test of P mixtures at 14 and 28 days.

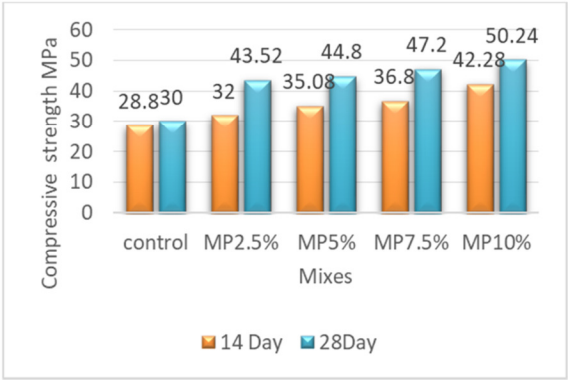


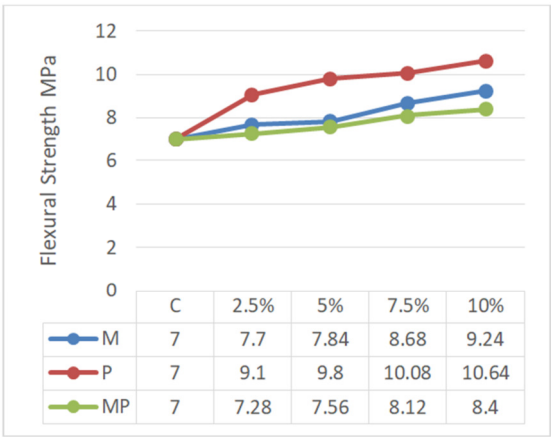
Fig. 6. Results of compressive strength test of M+ P mixtures at 14 and 28 days.

These results agree with [30-32]. M residue is frequently utilized following the grinding process to act as a filler in cement matrices, where the finely divided particles assume this function. The application of M residues in this manner is believed to be attributable to their packaging effects, which are contingent upon the size of the particles. Furthermore, the smaller particles are postulated to facilitate the nucleation mechanism by becoming lodged within the interstices of the clinker crystals, thereby promoting the filler effect [15]. Furthermore, the compressive strength of mixes with P was increased faster than the one of the mixes with M. This can be attributed to the higher silica content of P, which helps strengthen the matrix through the pozzolanic effect and densification, thus increasing its compressive strength. Moreover, the results demonstrated that combining M and P resulted in lower compressive strength than when utilizing each material individually, yet still surpassing that of the reference mix. This newly formed C-S-H gel improves the microstructure's density and fortifies it, enhancing its compressive strength.

B. Flexural Strength

Concrete's flexural strength significantly impacts several critical properties, including brittleness, shear strength, and deflection [33]. Flexural strength is, therefore, a crucial aspect of the design. Figure 7 exhibits the effect of partially replacing cement with M and P on the flexural strength of the mortar after 28 days of curing. The results demonstrated that adding waste material in place of cement greatly boosted the mix's flexural strength, with the improvements growing as an amount of garbage was added. This improvement followed the same pattern as the 28-day compressive strength data. In particular, at a 10% replacement ratio, the best values for flexural strength were recorded at 9.24, 10.64, and 8.4 MPa for the mixes 10M, 10P, and 10M+P, respectively, whereas the reference mix recorded 7 MPa. The densification of the microstructure and the hydraulic and pozzolanic impacts of the fine marble and porcelain particles are responsible for this improvement in flexural strength [19, 34, 35].

Furthermore, the filler effect of waste particles compared to cement contributes to higher flexural strength. These findings align with previous research [33, 36], which indicated that using M and ceramic waste produced a denser mix at higher replacement ratios, therefore improving long-term properties. The new mixtures containing this waste are technically viable and have higher reactivity than that of a conventional mix.



	C	2.5%	5%	7.5%	10%
M	7	7.7	7.84	8.68	9.24
P	7	9.1	9.8	10.08	10.64
MP	7	7.28	7.56	8.12	8.4

Fig. 7. Flexural strength test results.

C. Results of Microstructural SEM Analysis

Figures 8-11 manifest the morphology of samples containing M, P, and M+P along with the morphology of the control mix. The main hydration products in the plain mortar

(Figure 8) were found to be portlandite (in the form of a plate) and C-S-H (with a sheet-like structure) in all samples. Additionally, a needle-like structure was seen, suggesting the existence of ettringite [37]. In the samples with M powder (Figure 9), ettringite was absent due to the presence of calcium carbonate in the sample, significantly reducing ettringite's stability. More gel was detected due to the presence of more silicate compared with conventional mortar. The utilization of M powder has the potential to occupy the voids within the concrete, thereby enhancing the compactness of the microscopic composition of concrete mixtures and subsequently augmenting its mechanical prowess [17]. Figure 10 shows the presence of large quantities of gel with small amounts of ettringite with a dense structural composition. This agrees with the findings of [38, 39], where it was explained that using porcelain reduced porosity. It should be mentioned that incorporating CWP as a cement replacement improved several microstructure characteristics [38-39].

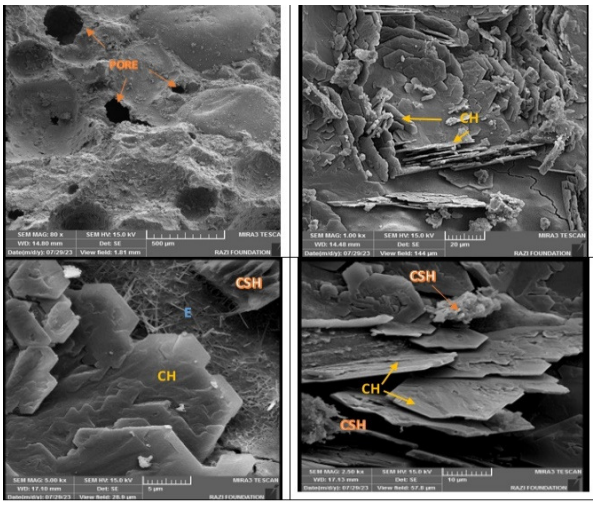


Fig. 8. SEM micrograph of the control mix.

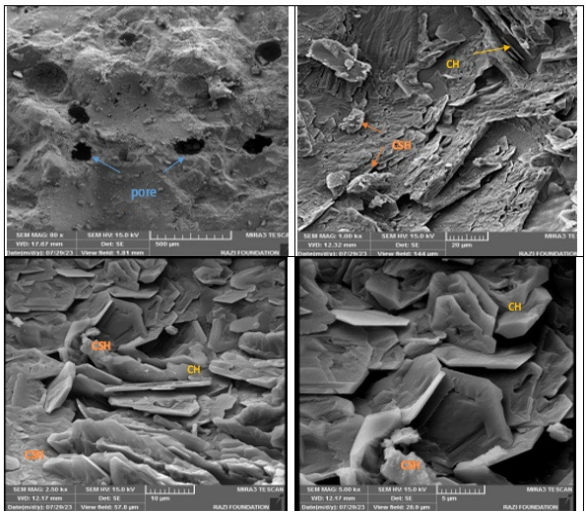


Fig. 9. SEM Micrograph of the mortar mix with marble powder.

All mortar specimens displayed a rich presence of hydration products, featuring both clustered or stacked calcium hydroxide particles and flocculent C-S-H gels. The SEM micrographs in Figure 11 demonstrated that replacing cement with M powder caused a wider spread of C-S-H compared to that of the standard mortar mix. In these formulations, the extensive formation of CSH arose from the interaction between CH and micro CaO from M and P, promoting the creation of more CSH and ettringite. This process accounts for the increased strength of the mortar when MP is used as a partial cement substitute. The formation of hydration products in the microstructure of different mortar mixes can be analyzed to comprehend this enhancement. Other hydration products, including C-S-H and ettringite, are identifiable by comparing all figures, although their development differs depending on the type of replacement used.

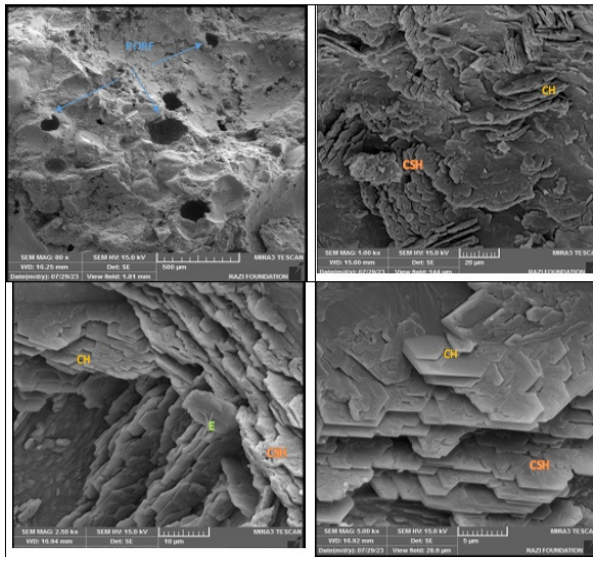


Fig. 10. SEM micrograph of mortar mix with porcelain powder.

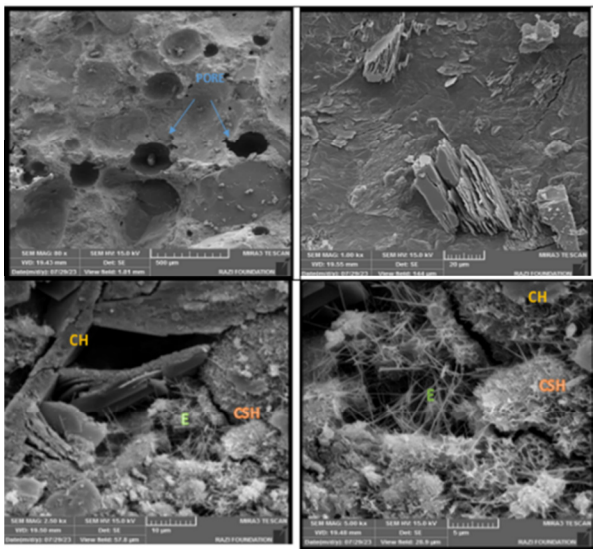


Fig. 11. SEM micrograph for mortar mix with porcelain+marble powder.

D. Results of Differential Thermal Analysis (DTA)

DTA has been extensively deployed in clay mineralogy and has recently been employed in other fields. The method consists of tracking the changes in the heat that occur as a substance is heated gradually, which is caused by physical or chemical transformations. Thermal alterations suitable for DTA analysis include dehydration, crystalline transition, lattice breakdown, oxidation, and decomposition. These changes are typically marked by a substantial increase or decrease in temperature.

The thermal analysis results for the examined samples are presented in Figures 12-15. The DTA charts for all samples reveal four distinct regions. The initial effect is related to the water evaporation adsorbed on the surface at temperatures between 25 and 100 °C. This occurs because the samples absorb moisture from the air after cooling at room temperature and again during drying at 104 °C. The second endothermic event, occurring from 100 to 400 °C, is caused by the dehydration of C-S-H and ettringite. The temperature at which each component in the mix starts to lose water is influenced by the ratio of CaO to SiO_2 in the hydrated cement mixture. The peak temperatures of the third phase range between 430 and 460 °C, suggesting the decomposition of $Ca(OH)_2$ formed during the hydration process, as indicated by the following reaction:

$$Ca(OH)_2\,(s) \rightarrow CaO\,(s) + H_2O\,(g)$$

At about 790 °C, an endothermic process occurs when calcium carbonate decarbonizes the hydrated molecule. The $CaCO_3$ was determined using the weight loss by the assumption of the decarbonation process shown below [40]:

$$CaCO_3\,(s) \rightarrow CaO\,(s) + CO_2\,(g)$$

As illustrated in Figure 13, in the M samples, the decomposition of $CaCO_3$ begins at a temperature of 740 °C. The peaks reveal an endothermic reaction, in contrast to the control mix depicted in Figure 12. The M mix showed greater intensity due to the elevated levels of calcium carbonate that existed in the mix. Figures 14 and 15 display more pronounced peaks at temperatures from 420 to 450 °C, indicating another reaction involving the consumption of CH. This phenomenon is linked to the added silica and alumina from the P, which intensifies the pozzolanic reaction in these mixtures to a higher degree [41-42].

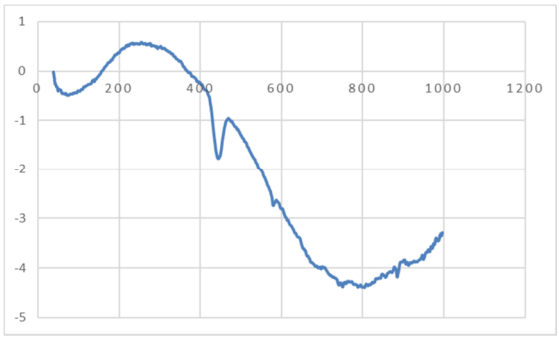


Fig. 12. DTA curve of the control mix.

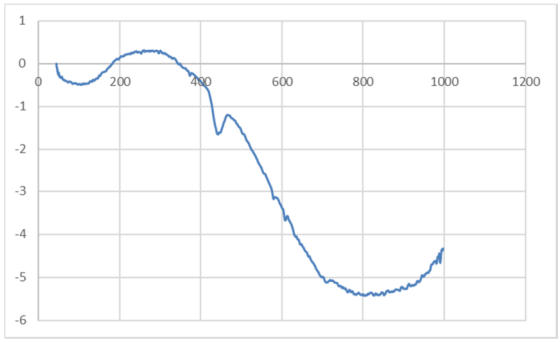


Fig. 13. DTA curve of the M mix.

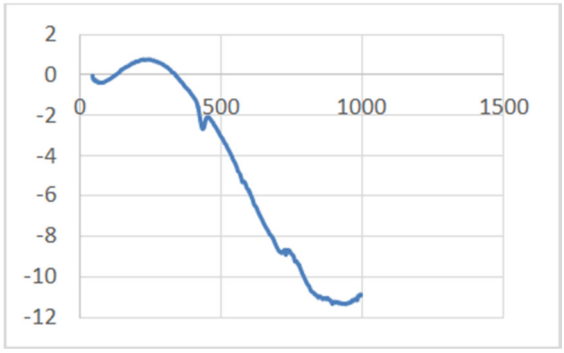


Fig. 14. DTA curve for the P mix.

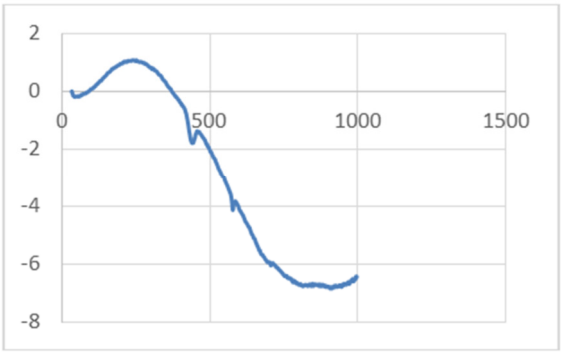


Fig. 15. DTA curve for the M + P mix.

V. CONCLUSIONS

This research aims to develop an environmentally friendly mortar that can be made by replacing cement with marble and porcelain at replacement ratios of 2.5, 5, 7.5, and 10%. The specimens were cured for 14 or 28 days before mechanical property tests, microstructure analysis, and thermal analysis were conducted. The most important conclusions drawn from the results of this study are:

- The findings revealed increased mortar strength with all M and P replacement percentages of cement. Also, a significant increase in strength was observed in binary mixes (M+P). The maximum compressive strength was 52, 55, and 50 MPa with 10% of M, P, and M+P mixes of cement, compared to the 30 MPa of the control mix. The maximum flexural strength was 9.24, 10.64, and 8.4 MPa

for 10% of M, P, and M+P mixes compared to the 7 MPa of the control mix.

- Microstructure SEM analysis data illustrate the cooperation of M and P as cement replacements, which results in the densification of the paste matrix by reducing the Ca/Si ratio in both the C-H and C-S-H phases. Replacement ratio of 10% M or P suggests improved microstructure refinement for these mixes, eventually leading to greater engineering performance.
- The DTA analysis revealed that substituting cement with M or P led to a decrease in the C-H content, which has a favorable impact on the mortar's properties.

In conclusion, both M and P wastes can be recycled and utilized as cement substitutes to produce an eco-friendly binder. This substitution markedly enhances both the mechanical and microstructural properties of the mortar. Additionally, the integration of these materials offers a promising approach to create a sustainable ternary blend with enhanced properties.

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