INVESTIGATING THE EFFECTS OF MICRO MONTMORILLONITE ON CEMENT MORTAR PHYSICAL PROPERTIES

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Resume

The effects of micro-sized montmorillonite clay (MMT) on Portland cement concrete were examined in this study, focusing on regular and calcined MMT as partial cement replacements. Key properties assessed include consistency, setting time, flexural strength, and compressive strength at 7 and 28 days. Regular MMT initially acts as an inert filler, potentially reducing compressive strength, while calcined MMT improves compatibility and strength. Both types extend the setting time, with regular MMT causing more delay. The study highlights that the regular MMT increases water demand and setting time, whereas calcined MMT enhances strength and mitigates these issues. The results emphasize the importance of carefully choosing MMT type and dosage.

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1 Introduction

The incorporation of micro clay additives in cementitious materials has garnered significant attention due to their potential to enhance various properties of cement-based composites [1-3]. Micro clay, consisting of finely divided particles typically in the micrometer range, offers a sustainable and cost-effective means to improve the performance of cement mortar and concrete [4-6]. This introduction focuses on the effects of micro clay additives on the setting time of cement-based materials. Initial and final setting times are crucial parameters within the context of cement hydration, as they determine the workability and early strength development of concrete mixtures. Manipulating the setting times through the addition of micro clay can influence construction schedules, improve placement practices, and ultimately impact the mechanical properties and durability of hardened concrete structures. The present study investigates the influence of both virgin and calcined micro clay additives on the setting time of cement mortar. Virgin micro clay refers to untreated clay particles, while calcined micro clay undergoes a thermal treatment process to modify its properties. By comparing the effects of these two forms of micro clay at various dosages, insights into their respective contributions to setting time alterations can be gained. A study conducted by [7] explores alternative materials for replacing fine aggregate in building construction. Marine clay, combined with the microorganism Bacillus subtilis MTCC441, was investigated to enhance the mechanical properties of the mixture. In the study is found that the microbial marine clay mortar mix exhibited significant improvements in compressive strength and a reduction in water absorption compared to the control mix. However, the microbial excavated soil mixes did not demonstrate crack healing properties through bioprecipitation of calcium carbonate crystals, even after observing an induced crack of 0.5 mm width for 60 days at one-week intervals [8-10]. Alani (2020) [11] investigated the effects of incorporating nano-calcined montmorillonite clay (NCMC) into cement mortars on their mechanical strength and microstructure when exposed to high temperatures. NCMC, obtained through thermal activation of nano-clay, was added to mortars in varying proportions. The mortars were then tested at temperatures up to 900 °C. Analytical techniques including thermogravimetric analysis, X-ray diffraction, and scanning electron microscopy were used to assess the fire-resistant properties and microstructural changes. Results indicate that incorporating thermally treated NCMC reduced density and microcrack widths, enhanced mass-loss behavior, decreased calcium hydroxide crystal content, and ultimately strengthened the matrix, leading to higher residual mechanical strengths in the mortars [11-12]. Khand and Nomana (2019) [13] evaluated Pakistani montmorillonite (Mmt) clay as a partial cement replacement in mortar, testing different temperatures and replacement levels. Calcined Mmt clay at 800 °C showed optimal strength performance, with a 25% replacement yielding strong compression results. Montmorillonite-modified mortars also exhibited better durability, particularly against mixed aggressive environments (SCS) compared to sulfate environments (SS) [13-15]. Chi and Huang (2012) [16] explored the effects of montmorillonite additives on cement paste and mortar properties. Different percentages of montmorillonite were added to mixes with varying water/cement ratios. Generally, montmorillonite enhances compressive strength, though not at 15% content after 28 days. Water absorption decreases in cement pastes but increases in mortars with montmorillonite. Both show higher adsorption-desorption values compared to plain mixes, with denser cement paste observed under scanning electron microscopy. Hydration products include portlandite, hatrurite, and calcite in cement paste, and quartz and portlandite in cement mortar according to X-ray diffraction analysis [16-17]. The objective of this research is to investigate the effect of montmorillonite as a partial replacement for cement on the properties of cement paste and mortar. Mechanical tests, including compressive strength and flexural strength, have been used to determine the properties of cement paste with varying percentages of montmorillonite. Additionally, setting time tests and consistency investigations were performed.

2 Materials

2.1 Sand

In this investigation, the standard sand, obtained from the Iraqi Geological Survey (Ministry of Industry and Mining), is used. The gradation of the sand is presented in Table 1. Based on the grading results, it satisfies the required grading specifications as stated by the ASTM C778 [18] standard.

2.2 Cement

Ordinary Portland Cement Type I was used in all mixes throughout this research. It is manufactured in Iraq by Al-Mass Company type CEM I. The chemical composition and physical properties of the utilized cement are shown in Table 2 and 3, respectively. Test results indicate that the chosen cement complies with the Iraqi specifications IQS 5/2019 [19] and ASTM-C150-21 [20].

2.3 Clay (Montmorillonite)

Clay type Montmorillonite (MM) was used in this investigation, its source is the natural stone available

Table 1 *Standard sand gradation*

Sieve size	Percent passing (%)	Specification limits (%)
1.18 mm	100	100
600 µm	96.8	96-100
425 µm	71.1	65-75
300 µm	22.8	20-30
150	2.9	0-4

Table 2 *Chemical composition of cement*

Component	Results (%)	Specification limit (%)
MgO	1.25	max. 5
Al_2O_3	5.46	--
SiO_2	20.9	--
CaO	62.3	--
C_3A	8.96	min 3.5
SO_3	2.48	max. 2.6
L.O.I.	3.56	max. 4
Fe_2O_3	3.24	--
I.R	0.9	max. 1.5

Table 3 Physical properties of cement

Property	Results	Specification limit
Fineness (m^2/kg)	291.4	230
Compressive strength at 3-days age, (MPa)	17.2	min. 15
Compressive strength at 7- days age, (MPa)	26.1	min. 23
Initial setting time (min)	144	min. 45
Final sitting time (min)	397	max. 600

Regular MM

Figure 1 *Photographs for MM*

Calcined MM

Figure 2 *Photographs for calcined CMM*

Table 4 *Physical properties of Montmorillonite clay*

Physical properties	Result	ASTM C618 class N specification limit
Retained No. 325 mesh (%)	11.2	max. 34%
Specific gravity	2.40	…..
Average particle size (µm)	4 to 5	…..

Table 5 *Chemical properties of Montmorillonite clay*

Chemical composition	Result (%)	ASTM C618 class N specification limit (%)
Sodium oxide (Na_2O)	1.36	max. 5
Magnesium oxide (MgO)	2.68	…..
Aluminum oxide (Al_2O_3)	18.24	…..
Silicon dioxide (SiO_2)	56.64	…..
Potassium oxide (K_2O)	0.66	…..
Calcium oxide (CaO)	3.10	…..
Titanium oxide (TiO_2)	0.97	…..
Ferric oxide (Fe_2O_3)	6.1	…..
Sulphur tri oxide (SO_3)	0.18	max. 4
(SiO_2) +(Al_2O_3) + (Fe_2O_3)	80.98	min. 70
Loss on ignition (LOI)	7.3	max. 10

in the western part of Iraq. The stone was broken into small size pieces that is allowed to be ground by the pug mil to finer size passing sieve No. 200 (0.075 mm) to convert them to micro scale. Two types of MM were used; the first one, which is labeled as regular is the normal type without any processing. The second one is the calcined Montmorillonite (CMM). The calcination process consists of heating the Montmorillonite to 800 ºC for two hours. Figure 1 and 2 shows both types of Montmorillonite used throughout this study. Tables 4 and 5 show the physical and chemical properties of both types of montmorillonite. For ease of reference, the following specimens' legend, as presented in Table 6, are adopted within the course of the experimental work. Besides, Table 7 lists the specimens legend and mix gradient. A comparison was made between the

Table 6 *Chemical composition of tap water*

Component	Results	Limits
Sulfites (mg/l)	20	max. 1000
Chlorides (mg/l)	10	max. 500
Carbonates and bicarbonates (mg/l)	65	max. 1000
Inorganic materials (mg/l)	420	total ions, max. 3000

Table 7 *Specimens legend and mix gradient*

Legend	Replacement rate (%)	Sand (g)	Cement (g)	Clay (g)	Water (ml)	No. of specimens (compressive)	No. of specimens (flexural)
RM	0	1375	500	-	242	6	3
MM1	0.25	1375	498.75	1.25	242	6	3
MM2	0.75	1375	496.25	3.75	242	6	3
MM3	1.25	1375	493.75	6.25	242	6	3
MM4	1.75	1375	491.25	8.75	242	6	3
CMM1	0.25	1375	498.75	1.25	242	6	3
CMM2	0.75	1375	496.25	3.75	242	6	3
CMM3	1.25	1375	493.75	6.25	242	6	3
CMM4	1.75	1375	491.25	8.75	242	6	3

Note: The amount of water added to the mortar mixtures was adjusted according to the findings of the consistency test.

Figure 3 *Mortar mixing process*

montmorillonite assay and the specified limit for ASTM C618 Class N, as stated in reference [21].

2.4 Water

The tap water is used during the preparation of cement mortar. The chemical composition for the tap water is presented in Table 6.

3 Preparation of mortar specimens

According to ASTM C305 [22] in the mortar mixing process, the dry paddle and bowl are initially positioned in the electrical mixer. The materials for a batch are then introduced into the bowl following a specific sequence. First, all the mixing water is placed in the bowl. Next, the cement is added to the water, and the mixer is started at a slow speed (140 ± 5 r/min) for 30 seconds to ensure uniform mixing. Subsequently, the entire quantity of sand is added slowly over a 30 second period while continuing to mix at the slow speed. Afterward, the mixer is stopped, and the speed is changed to medium (285 ± 10 r/min) for another 30 seconds of mixing. Finally, the mixer is stopped again, and the mortar is allowed to stand for 90 seconds. During the first 15 seconds of this interval, any mortar that may have collected on the side of the bowl is quickly scraped down into the batch to ensure uniformity. The mixing process is shown in Figure 3.

4 Specimen casting and curing

The specimen molding procedure, following ASTM C 109 [23] standards, requires initiating the molding within 2 minutes and 30 seconds after the mortar batch

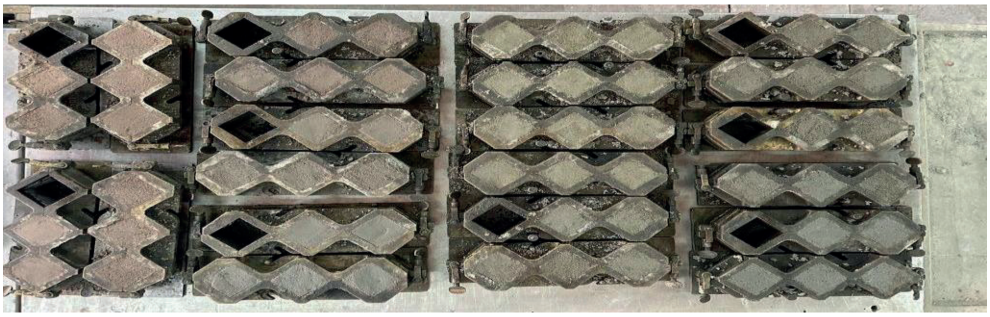

Figure 4 *The casted specimens*

Figure 5 *Vicat apparatus*

mixing is completed. Initially, a 25 mm layer of mortar is placed in the cube molds (50 x 50 x 50 mm). Each cube compartment is then tamped 32 times over four rounds, with each round perpendicular to the other, ensuring uniform filling. Once the first layer in all the compartments is tamped, the remaining mortar is added and tamped similarly. After the tamping, the cube tops slightly extend above the molds. Any excess mortar on top is smoothed with a trowel, and the cubes are leveled. Figure 4 illustrates this process. After 24 hours, samples are removed from the molds, marked, and immersed in tap water for 7 and 28-day curing. For flexural strength specimens, the same process is repeated with prisms measuring 40 x 40 x 160 mm as per the ASTM C 109 standards requirement. A 20 mm layer of mortar is evenly distributed in each mold, compacted with twelve tamper strokes in three rounds, and smoothed with a trowel. Excess mortar is removed, and the surfaces are made plane. Prisms are let to harden for one day in the molds and then removed for curing for 28 days.

5 Physical properties tests

5.1 Consistency

This test's significance lies in determining the water quantity required to achieve a consistent paste with standard consistency. A Vicat device, as per ASTM 187 specification [24], is employed for this purpose. This device comprises a circular piston with a 10 mm diameter, as illustrated in Figure 5. Initially, 650 grams of cement are mixed with a calculated amount of water to form the paste. Subsequently, the mixture is agitated at various speeds in a mechanical mixer, following ASTM C305 specification, as previously mentioned. The paste, confined within a mold and resting on a plate, is positioned beneath the rod with the plunger's tip in contact with its surface, and the screw is tightened. The moving pointer is then aligned with the upper zero mark on the scale, and the initial reading is taken before promptly releasing the rod. This action must occur within 30 seconds of completing the mixing process. After 30 seconds, the screw is tightened once again to halt the piston's descent, and the distance it has descended below the original surface is recorded. The paste achieves standard consistency when the piston descends a distance of (10 ± 1 mm) below the original surface within 30 seconds of its release.

5.2 Setting time of blended cement

According to ASTM C191 [25], the initial and final setting times of the cement and MM pastes were assessed using Vicat's apparatus. The Vicat test, outlined in ASTM C191, is a widely employed method for determining the setting time of cement paste. In this examination, a water-to-cement ratio of 28% was utilized. Figure 6 shows the setting time testing device.

Figure 6 *Vicat testing*

Figure 7 *Flexural strength testing*

Figure 8 *Compressive strength testing*

5.3 Flexural strength

The flexural strength of cement mortar is determined using a flexural test conducted in accordance with ASTM C348 [26] standards. Specimens were tested immediately after removal from storage water at the age of 28 days. The testing setup involves carefully positioning the specimen on the testing device to ensure accurate alignment and load application. This setup includes centering the pedestal beneath the spherical head, placing the specimen on its side on the supports, and ensuring it aligns with the center of both supports. The load is then applied at a controlled rate of 2640 ± 110 N, and the flexural strength is calculated using the formula:

$$\sigma_f = 3PL/2bd\,, \tag{1}$$

where:
σ_f = Flexure strength (MPa),
P = Maximum measured applied load (N),
L = Span's length (mm),
b = Width of the specimen (mm),
d = depth of the specimen (mm).

Photograph for specimen while testing is shown in Figure 7.

5.4 Compressive strength test

Compressive strength testing was performed on cubic specimens, following the guidelines of ASTM C109/C109M-21. The tests were conducted immediately after removing the specimens from the storage water at the predetermined ages of 7 and 28 days. During the testing process, the load was applied to the specimen faces that were in contact with the true plane surfaces of the mold. The load was applied at a controlled rate of movement between the upper and lower platens, ranging from 900 to 1800 N/s. The maximum load, indicated by the testing machine, was recorded, and the compressive strength was calculated using the following formula:

$$\sigma = P/A\,, \tag{2}$$

where:
σ = compressive strength in (MPa),
P = total maximum load in (N),
A = area of loaded surface in (mm^2).

Photograph for specimen while testing is shown in Figure 8.

6 Results and discussion

6.1 Consistency

The data presented in Table 8, as well as in Figures 9 and 10, illustrate the variations in consistency of the Portland cement paste resulting from the incorporation of montmorillonite in its regular and calcined forms. With regular montmorillonite, the proportion of water required to achieve the desired consistency increases as the replacement percentage rises, ranging from 0.25 % to a maximum of 1.75 % replacement. Similarly, with calcined montmorillonite, there is a consistent upward trend in water utilization with increasing replacement percentages, reaching a peak at the maximum replacement percentage. Comparing both types of montmorillonite to the reference mixture at 0 % replacement, it becomes evident that calcined montmorillonite requires a greater amount of water to achieve the desired consistency compared to the regular type. Specifically, there is an approximate 10.71 % increase in water requirement at its peak (1.75 % replacement) compared to the reference mixture. This suggests that the calcination process results in water loss, necessitating a higher quantity of water than typically needed for montmorillonite.

6.2 Setting time

The data presented in Tables 9 and 10 and shown graphically in Figures 11 to 14 indicate that incorporating regular montmorillonite (MMT) into cement leads to a significant increase in both initial and final setting times. This escalation is directly proportional to the montmorillonite dosage, ranging from a 20 % to a 93.33 % increase compared to the control mix. The relationship between the dosage and its effect is pronounced, with the highest dose of 1.75 % causing the most marked delay in the setting process. The likely cause of this delay is the increased water demand and the subsequent disruption of the hydration reactions. In contrast, cement containing calcined montmorillonite also exhibits longer initial and final setting times compared to the control mix; however, the increase is less marked than with regular montmorillonite, ranging from 13.33 % to 60 %. The activation of calcined montmorillonite is thought to elongate the setting time because it releases more heat during the reaction, facilitating the solidification process. On the other hand, regular montmorillonite does not produce such thermal energy, thus hindering the reaction and resulting in more extended setting times when compared to the calcined variant.

Table 8 *Results of consistency test for micro clay samples*

	Control	MM1	MM2	MM3	MM4	CMM1	CMM2	CMM3	CMM4
Consistency (%)	28	29	29	30	30	29	30	30	31
Percentage increase (%)	-	3.57	3.57	7.14	7.14	3.57	7.14	7.14	10.71

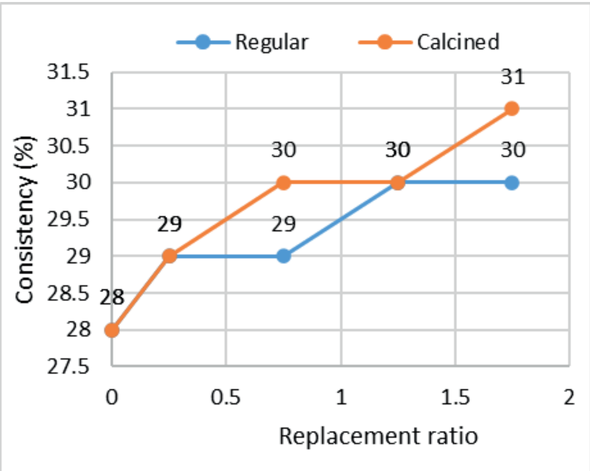

Figure 9 *Consistency test for micro clay*

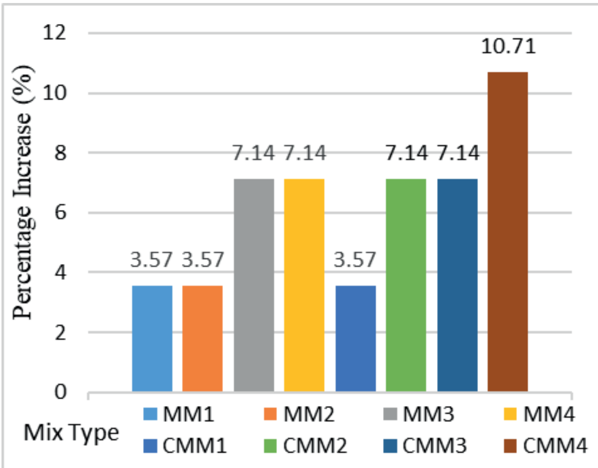

Figure 10 *The percentage increase in the consistency*

Table 9 *Results of setting time (initial) test for micro clay samples*

	Control	MM 1	MM 2	MM 3	MM 4	CMM 1	CMM 2	CMM 3	CMM 4
Initial (min)	75	90	110	125	145	85	95	115	120
Percentage increase (%)	-	20.0	46.66	66.66	93.33	13.33	26.66	53.33	60.0

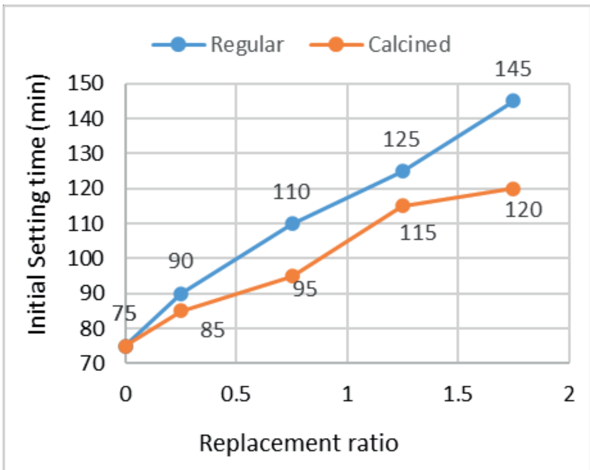

Figure 11 *Initial setting time test for micro clay*

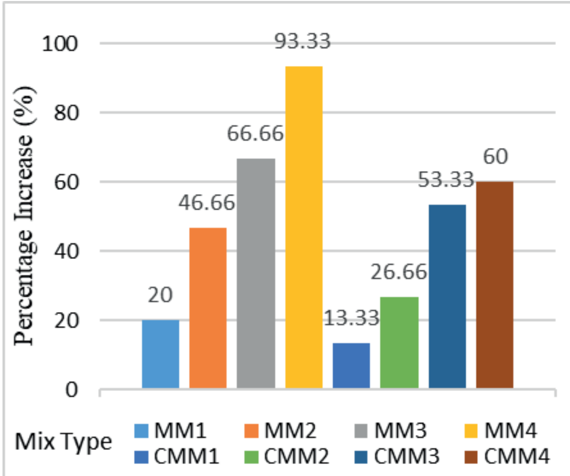

Figure 12 *The percentage increase in initial sitting time*

Table 10 *Results of setting time (final) test for micro clay samples*

	Control	MM 1	MM 2	MM 3	MM 4	CMM 1	CMM 2	CMM 3	CMM 4
Final (min)	285	315	350	395	425	315	325	355	380
Percentage increase (%)	-	10.52	22.81	38.59	49.12	10.52	14.03	24.56	33.33

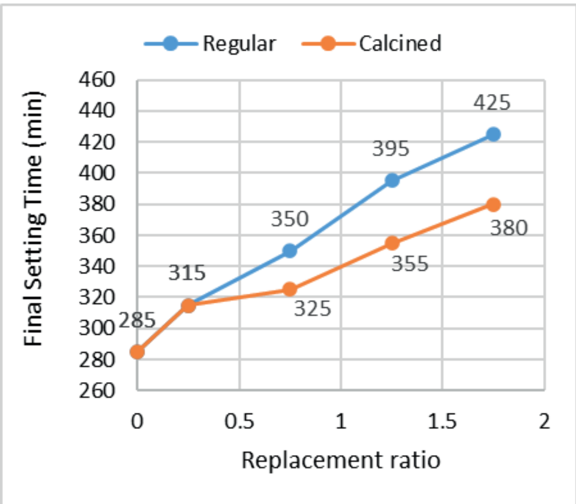

Figure 13 *Final setting time test for micro clay*

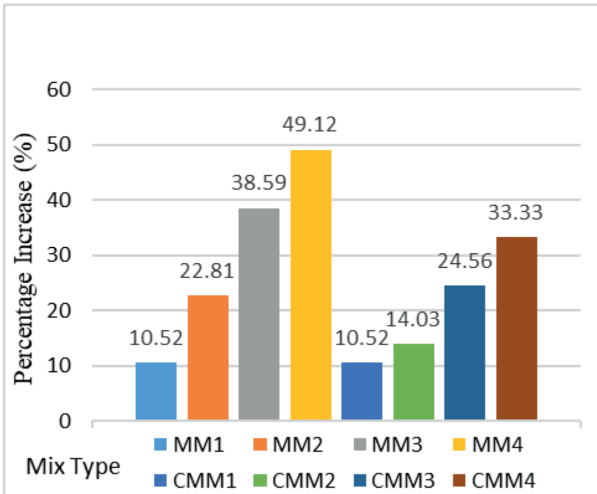

Figure 14 *The percentage increase in final setting time*

6.3 Flexural strength

The results of MMT and calcined MMT are presented in Table 11 and Figures 15 and 16. Initially, replacing montmorillonite in its regular form with a replacement ratio of 0.25% led to a 3% increase in flexural strength compared to the reference sample. However, replacing montmorillonite at higher ratios resulted in a decrease in resistance directly proportional to the increased replacement rate. The initial increase at a low replacement ratio can be attributed to improved particle arrangement, void filling, and strengthening of the mortar structure. However, increasing the replacement ratio led to excessive particle accumulation, which caused the appearance of weak areas. On the other hand, flexural strength increased with calcined MMT up to a concentration of 1.25%. Calcination improved the dispersion and bonding of fine clay particles in the mortar, resulting in a more substantial strengthening effect. However, when the concentration increased to 1.75%, flexural strength decreased. This decrease can be attributed to excessive particle loading, which may cause particle agglomeration or restrict the movement of cement particles during hydration, thereby reducing the ability of calcined clay to improve flexural strength.

6.4 Compression strength

Tables 12 and 13 and Figures 17-20 present the results of the compressive strength test for mortar cubes aged 7 and 28 days. It was observed that adding MMT at all the replacement ratios resulted in a reduction in compressive strength. This decrease is likely attributed to the introduction of defects and brittle voids in

Table 11 *Results of flexure strength test for micro clay samples*

	Control	MM 1	MM 2	MM 3	MM 4	CMM 1	CMM 2	CMM 3	CMM 4
Flexural strength (MPa)	2.85	2.93	2.77	2.68	2.58	2.99	3.11	3.19	3.09
Percentage increase (%)	-	3.0	2.50-	5.73-	9.14-	5.18	9.13	12.08	8.50

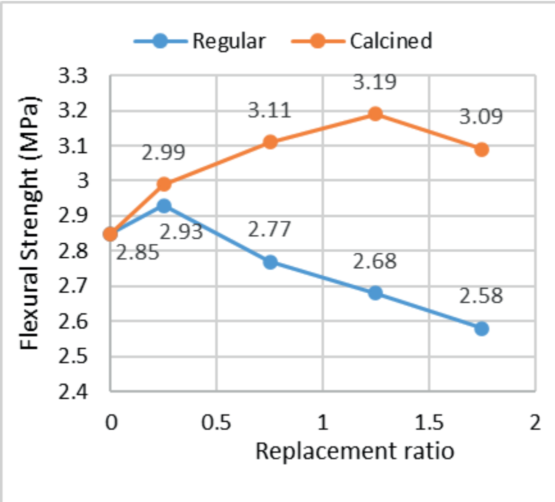

Figure 15 *Flexural strength for micro clay*

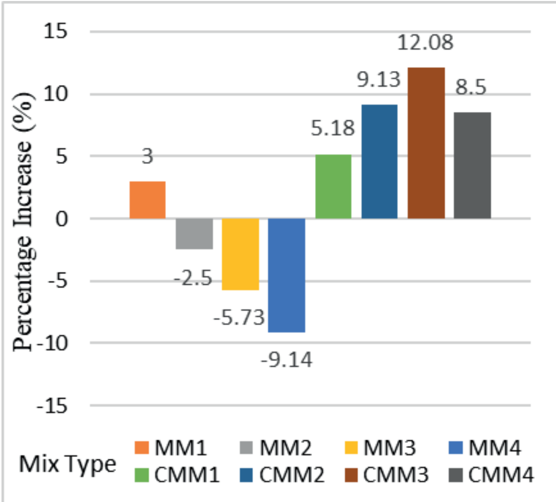

Figure 16 *The percentage increase and decrease in flexural strength*

Table 12 *Results of compressive strength test for micro clay sample at 7 days*

	Control	MM 1	MM 2	MM 3	MM 4	CMM 1	CMM 2	CMM 3	CMM 4
Compressive (MPa)	20.7	19.25	18.83	18.42	17.59	21.36	22.25	21.52	21.19
Percentage increase (%)	-	7.0-	9.03-	11.01-	15.03-	4.49	7.48	3.96	2.36

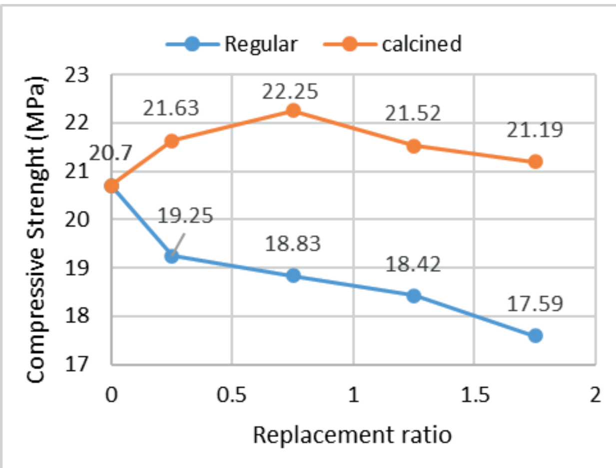

Figure 17 *Compressive strength test at 7 days*

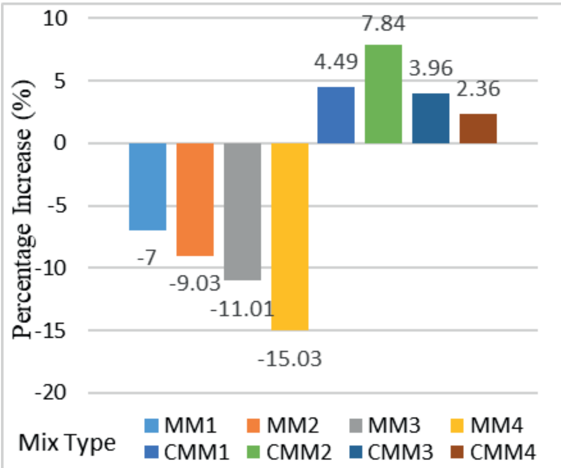

Figure 18 *The percentage increase and decrease in compressive strength*

Table 13 *Results of compressive strength test for micro clay sample at 28 days*

	Control	MM 1	MM 2	MM 3	MM 4	CMM 1	CMM 2	CMM 3	CMM 4
Compressive (MPa)	25.4	23.75	23.26	22.80	21.23	26.67	27.16	26.28	25.94
Percentage increase (%)	-	6.5-	8.40-	10.22-	16.40-	5.00	6.95	3.50	2.14

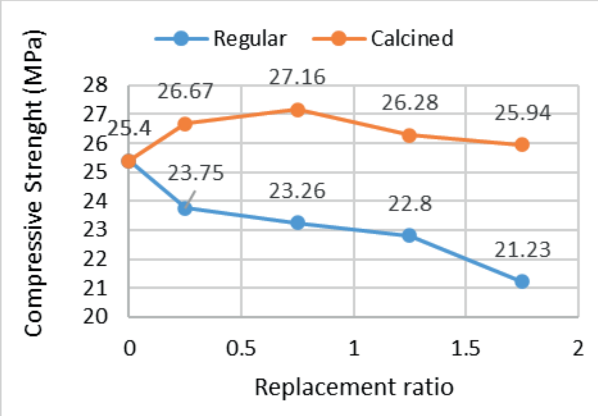

Figure 19 *Compressive strength test at 28 days*

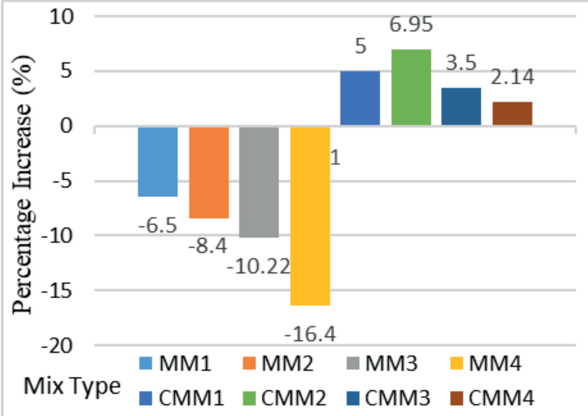

Figure 20 *The Percentage increase and decrease in compressive strength*

the arrangement of cement particles. In contrast, the addition of calcined MMT led to an increase in compressive strength, reaching its peak at a replacement ratio of 0.75 %. At this ratio, the increase reached 7.84 %, and then gradually decreased with each subsequent replacement ratio until reaching a compressive strength of 21.19 MPa. However, this value still exceeded the compressive strength of the reference sample by 2.36 %. The increase in compressive strength, observed when adding calcined clay, is generally attributed to the heat treatment of the clay, which altered its properties such as surface area, shape, and interaction.

7 Conclusions

In the ambit of this study, the multifaceted impacts of micro montmorillonite (MM) on the physical properties of cement mortar have been investigated. Employing two types of MM; regular and calcined at varying percentages (0 %, 0.25 %, 0.75 %, 1.25 %, and 1.75 % by weight of cement), the research delved into the core physical aspects through four fundamental tests: consistency, setting time, flexural strength, and compressive strength. The outcomes of these rigorous examinations are pivotal, providing nuanced insights into the interactions between the MMT and cement mortar. Based on the results of experimental work, the following conclusions can be drawn:

1. Calcined MM, when added to cement, increased the water requirement for desired consistency, with the maximum replacement ratio showing a 10.71 % hike in water demand.
2. Both initial and final setting times of cement were extended by MM incorporation, with regular MM inducing up to a 93.33 % increase in setting time at the highest replacement level, reflecting a direct dose-response relationship.
3. Introduction of regular MM at a 0.25 % replacement ratio enhanced the flexural strength, which is attributable to optimized particle packing. Contrarily, excessive MM resulted in a decline in flexural strength due to the formation of weak zones.
4. The addition of calcined MM up to a 0.75 % replacement ratio significantly improved compressive strength by 7.84 %. However, any further increase in replacement ratio led to a decrease in benefits, although strengths remained higher than the control sample by 2.36 % at 1.75 % replacement.
5. The research delineates the disparate effects of regular and calcined MM on altering mortar properties within the constraints of the experimental conditions and material properties specific to this study. It suggests that while the findings are promising, they are preliminary and should be validated through the extensive field studies to confirm their applicability in real-world scenarios. Future research should continue to refine the application of MM, with a particular focus on the calcination process and precision in replacement ratios to fully realize the material's potential.
6. Swelling of montmorillonite refers to its ability to expand when hydrated or exposed to water. This clay mineral has a layered structure with a high surface area and negative charge, allowing it to absorb water molecules between its layers. This swelling property is significant in various applications, such as in

drilling fluids, soil stabilization, and as an additive in materials like concrete to improve properties such as workability and strength.

7. Washing particles in concrete can negatively affect its strength and durability. These debris, usually clay or silt, can disrupt the cement matrix when excess water is applied during mixing or if segregation occurs. This can lead to decreased compressive strength, increased permeability, and durability issues along with cracking and decreased resistance to environmental elements.

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Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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