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Physical properties of cementitious composite material with Iraqi bentonite as partial cement replacement under elevated temperatures

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ABSTRACT

The impact of higher temperatures on the qualities of normal-strength mortar containing Iraqi bentonite powder (BP) as a partial substation of cement was examined in the present research. The bentonite concrete was made by replacing cement with bentonite in weight ratio of 5 and 10%. A strength test (compressive strength) was performed, and the durability performance (mass loss) and absorption were performed and discussed. The results showed that samples containing bentonite outperformed the control mix regarding heat endurance. The flowability of the mixture decreased by 15% and 25% for 5% and 10%BP, respectively. Bentonite-containing concrete had a lower compressive strength than the control mix at ambient temperature and at 300 ^{o}C by 22.8% and 38.9, respectively for 5%BP and 10.5% and 24.8%, respectively for 10%BP. But at 600 ^{o}C, the compressive strength of mixes containing BP is higher than that of the control mix by 33% and 49.5% for 5% and 10%BP, and it had good resistance to water absorption at all temperature levels. A somewhat significant color change was noted for mortar compositions with higher BP ratios. A color change was observed from gray to pink for samples containing BP.

1. Introduction

Bentonite may include many species of clay minerals, such as kaolinite, illite, palygorskite, and others, in addition to smectite particles. Non-clay minerals such as feldspar, quartz, gypsum, and others might be present [1], [2]. The ability of smectite minerals to swell as a result of absorbing water makes them special [3]. Most of the time, bentonite has a high concentration of the smectite group mineral montmorillonite. As a result, bentonite is defined as clay that primarily consists of the mineral montmorillonite [4].

Through pozzolanic and physical processes, the characteristics of concrete content bentonite in both its plastic and hardened stages changed. When bentonite is added to cement paste, the amount of water needed to reach the desired paste consistency increases linearly [5], [6]. Additionally, the initial

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Subscripts

C-S-H	Calcium-silicate hydrate
CaO	Calcium Oxide
BP	Bentonite Powder
IQS	Iraqi Standered
ASTM	American Standered of Testing Materials
SP	Superplasticizer

and final setting times are prolonged [7–9], the workability of the concrete is decreased [10], and the amount of superplasticizer needed for self-compacted mortar and self-compacted concrete is increased [11], [12]. Because of bentonite's low pozzolanic reactivity in the hardened state, bentonite slows down the pace of strength increase [13]. Thus, at early ages, 21 days or fewer, bentonite incorporation results in a loss in the compressive strength of concrete [9], [10]. However, at later ages, the reductions in concrete strength tend to diminish. Bentonite's primary impact is to increase the resistance of concrete to the alkalis-silica reaction [11], acid attack [13], and penetration by chlorides [10]. Previous investigation on the impact of high temperatures on the characteristics of mortar using bentonite from Iraq source has not been conducted. Sustainable development aims to improve living standards while simultaneously satisfying the basic needs of coming generations. Among other things, it seeks to meet basic needs, improve living conditions, and encourage the preservation and management of ecosystems [14]. A major hazard that developed infrastructure may confront throughout its lifetime is fire. The concrete's bearing capacity is at risk since high temperatures often cause the material's strength to deteriorate and explode [18]. Calcium-silicate hydrate (C-S-H) gel dehydration and the decomposition of calcium hydroxide and calcium aluminates are caused by the evaporation of water as the temperature rises [15]. Around 100 degrees Celsius is when free water vaporizes, 400 to 500 degrees Celsius is when Ca(OH)2 breaks down into calcium oxide (CaO) and H2O, and 600 degrees Celsius is when aggregates change into quartz [16]. Understanding how concrete behaves at high temperatures is crucial for the widespread use of concrete as a building material [17]. Additionally, the need to create materials for a range of uses that standard concrete cannot satisfy has increased as a result of technological improvements. Furthermore, both costs and demand for cement have increased dramatically in recent years [18].

Iraqi bentonite falls into two categories [19]: low-grade or high-calcium bentonite (CaO > 5%) and high-grade or low-calcium bentonite (CaO < 5%). Examining the behavior of mortar containing bentonite after exposure to high temperatures is the primary goal of this study.

2. Experimental programs

2.1. Materials and mix proportions

Ordinary Portland cement (OPC) complies with IQS 5-CEM I 32.5R [20]standards are used. The cement's chemical composition and physical properties are presented in Tables 1 and 2, respectively.

Figure 1 illustrates the procedure for preparing locally supplied bentonite, which is supplied from Rutba city in the Anbar province of Iraq. As seen in Figure 1a, the bentonite is first dried for 24 hours at 105 °C in an oven and then allowed to cool before being ground. Next, the bentonite is ground in the manner seen in Figure 1b and filtered using sieve No. 200. Tables 3 and 4, respectively, provide illustrations of the chemical and physical characteristics of bentonite, which satisfy ASTM C618-08a standards [21]. As a fine aggregate, natural sand is utilized following Iraqi Specifications No. 45 [22].

Table 1. The chemical properties of the used cement.

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3
19.49	04.40	04.38	02.94	63.06	00.14	00.85	02.63

Table 2. The physical properties of the used cement.

Specific gravity	3.01
Average particle size	19 μ_m
Blain fineness (gm/cm2)	1730
Initial setting time (mins)	0079
Final setting time (mins)	0249
Loss on Ignition	01.08

Tap water was used for mixing and curing and Foree W420-EB was used as superplasticizer (SP), which has a Light yellow-coloured liquid with S.G. of 1.04 @ 25°C. Figure 2 shows the materials that have been used in this work.

Figure 1. The preparation process of bentonite, a) the Drying process, and b) the Grinding process.

Table 3. The chemical properties of bentonite.

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3
59.6	14.9	6.9	3.5	7.5	1.16	0.55	1.4

Table 4. The physical properties of bentonite.

Specific gravity	2.76
Average particle size	4.69 μ_m
Blain fineness (gm/cm2)	4810
Loss on Ignition	6.4

Three sets of mixes were produced, a control mix and other two mixes with different replacing percentages of cement by BP (5 &10% from the cement weight). The mixture proportions are listed in Table 5.

Table 5. The proportion of mixes.

Mixes	Cement (kg)	Sand (kg)	Water (kg)	SP%
BP0	500	1500	200	0.85
BP5	500	1500	200	0.85
BP10	500	1500	200	0.85

Figure 2. The materials that have been used in this work

2.2. Tests

There was a temperature-controlled furnace that could reach up to 1200° C. A thermocouple was installed in the furnace to regulate the temperature. The temperatures that the samples were subjected to were 200, 400, and 600 degrees Celsius. The holding time and heating rate per minute were measured using RILEM test protocols; the heating rate utilized in this test was 2.5° C per minute [23]. To guarantee that the tested samples were heated consistently, the temperature was kept in the furnace for two hours after it reached the necessary level. After the allotted time has passed, the furnace door should remain closed and the little ventilation openings should be left open to allow the temperature to gradually drop. Once the heating process is complete and the furnace's internal temperature reaches approximately 200°C, the door gradually opens to facilitate swift cooling. Following the specimens' temperature reaching room temperature, all tests were conducted.

The specimens were tested under axial compression using a 1000 kN Universal Testing Machine. 0.2 MPa/s of stress was used for the compression test, which was carried out in compliance with ASTM C109/C109M-20b [24]. Density was computed by weighing the samples and dividing the recorded weight by the sample volume. Following a 28-day curing period, ASTM C642 [25] determined the dry density using three cubes, each measuring 50 × 50 × 50 mm. The average of the three specimens for each test was the final result. After reaching the required temperature, the cement mortar samples were maintained there for three hours. Before testing, the cement mortar specimens were allowed to cool naturally. After the samples were dried for 24 hours at 110 C in the oven, their mass was measured. After the specimens had burned to the required extent, they were left to cool naturally in the room's atmosphere. Equation (1) was also utilized to ascertain the mass to calculate the mass loss:

Mass loss% = (dry mass of the cube at 110° C × mass of the burning cube at 300 and 600° C)/dry mass of the cube at 110° C.

Following ASTM C642 [25], the absorption using three cubes, each measuring 50×50×50 mm, was utilized to calculate the absorption capacity at normal and after being exposed to elevated temperature. The average of the three specimens for each test was the final result, as follows:

Figure 3. Experimental tests in this work

Water absorption% = [(wet weight - oven dry weight)/oven dry weight]×100. Also, the effect of bentonite on the fresh state has been considered by measuring the following diameter using a flow table test [**26**]. Figure 3 shows the experimental tests that have been achieved in this work.

3. Results and disscusions

3.1 Fresh state flow

One of the fresh state characteristics considered in this research is the fluidity of fresh mortar. Measurements of the fluidity of various mixtures in their fresh form were performed to determine the extent to which bentonite powder (BP) affects the workability of the mixtures. As the amount of BP for constant water and SP content increased, the flowability of the mixture decreased by 15% and 25% for 5% and 10%BP, respectively. (see **Figure 4**). The main reason for this decrease in flow diameter is the chemical reaction between BP and CaOH, which is a result of cement hydration.

It is evident that flowability decreases as BP increases the quantity of replacement cement. This might be explained by the fact that more water was required to wet the particle surface since CB has a higher specific surface area and finer grain than cement [27-28].

According to Lecomte et al. [29**]** and Hama et al. [30], adding minerals used up a significant portion of the superplasticizer. As a result, introducing minerals will partially mobilize the superplasticizer that was intended for the deflocculation and dispersion of cement grains.

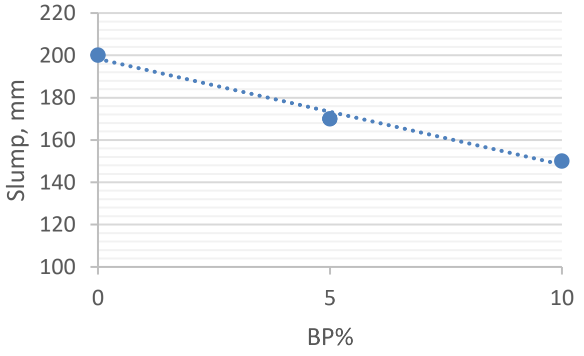

Figure 4. Slump flow vs. BP%

3.2 Hardened properties at ambient and elevated temperatures

3.2.1. Compressive strength

Figure 5 displays the axial compression experimental findings. Bentonite-containing concrete had a lower compressive strength than the control mix at ambient temperature and at 300 °C by 22.8% and 38.9, respectively for 5%BP and 10.5% and 24.8%, respectively for 10%BP. As the cement's substitution level rose, the strength gradually dropped. According to Mehta & Monteiro [31**]** and other researchers [32-35], the strength gain of concrete containing pozzolan is often slow in the early stages. As a result, the compressive strength values could rise with age and be deemed enough for low-cost buildings. The results at ambient temperature (25° C) are close to that obtained by Ahemd et al. [36]. But at 600 °C, the compressive strength of mixes containing BP is higher than that of the control mix by 33% and 49.5% for 5% and 10%BP .

The pozzolanic reaction, which happens more slowly than cement hydration and is coupled with the effect of pozzolanic material filling gaps in concrete materials, is one of the main mechanisms causing this increase in compressive capacity [37]. It has been determined that while BP was able to roughly match the reference strength, it was unable to effectively increase the compressive capacity.

The rise in compressive strength at 300 C, or between 150 and 400°C, has been explained by the removal of moisture from the cement gel interlayer is lower the disjoining pressure and raising the distribution of forces between the hydration product particles, increasing the mortar's compressive strength. The hydration reaction was accelerated by the samples' high temperature [38]. Moreover, a shorter period of exposure to high temperatures may be the cause of the specimen's increased compressive strength after exposure to 150 and 400°C [39].

The reason why the compressive strength of specimens decreases as temperature rises might be attributed to various factors. First off, higher temperatures quickly lose a significant amount of water that would have been used to hydrate the concrete, preventing future hydration and consequent strength growth. Secondly, the generation of steam within the specimens may result in elevated pressures. This pressure has the potential to undermine the concrete's structure and reduce its compressive strength by causing microcracking in the internal structure of the material [**41**].

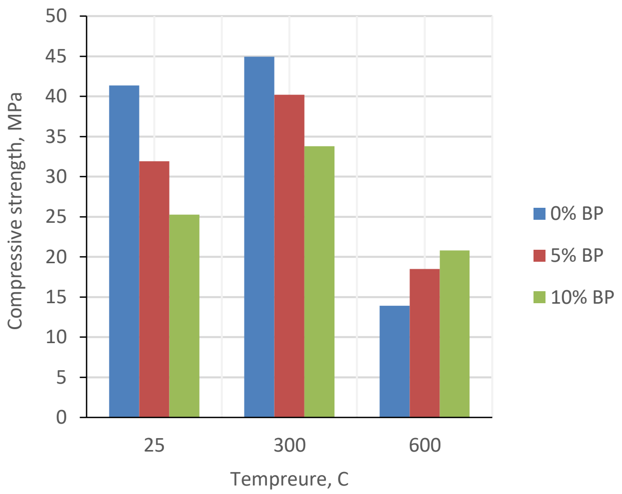

Figure 5. Compressive strength vs. BP% at different temperature levels

3.2.2. Absorption

The impact of BP on the absorption of mixes is illustrated in **Figure 6**. Because the pozzolanic reaction modifies the pore structure and the physical effect of finely ground, unreacted bentonite particles filling the pores, partially replacing cement with BP has a favorable effect and decreases absorption. Because of their comparative equivalency, a small decrease in absorption has been noted at substitution levels of 5% and 10%. On the other hand, Al-Hammood et al. [40] state that at substitution levels of 15% to 25%, the pozzolanic reaction and the physical effect outweigh the negative effects of lowering cement, resulting in a positive outcome. Ur Rehman et al. also found the impact of BP in decreasing absorption [41].

The fact that both free and absorbed water evaporates at temperatures above 200 °C makes it evident in **Figure 6** that water absorption likewise rises as exposure temperature rises. The absorption of water increases significantly as the temperature surpasses 300 °C. When compared to water absorption at 25 °C, water absorption at 600 °C increases dramatically for all combinations. It is clear that, at any target temperature, the amount of water absorbed drops as the bentonite concentration rises. Bentonite is a filler material that has smaller particles than cement; it reduces the porosity of concrete, which in turn reduces the amount of water absorbed through capillary action. Furthermore, because bentonite has a higher thermal capacity than the control sample, it performs better at high temperatures by preventing cracks from spreading [42].

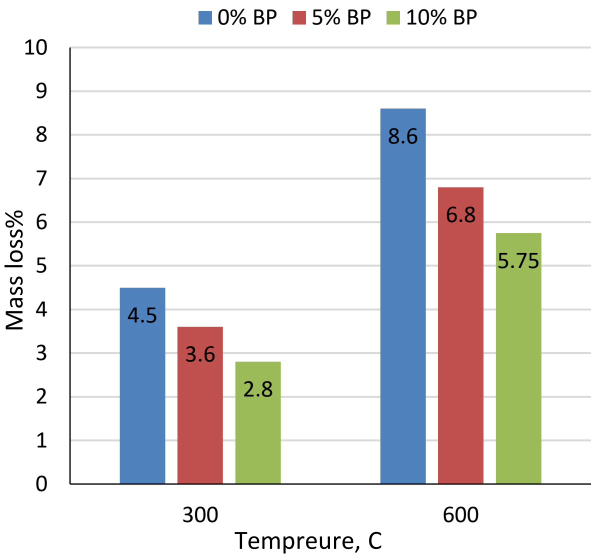

Figure 6. Absorption% vs. BP% at different temperature levels

3.2.3. Density

The impact of BP on the density of mixes is illustrated in **Figure** 7. Mixes' density somewhat decreases as a result of BP replacing cement.

As **Figure 7** illustrates, the density reduction is proportionate to the replacement %. Density decreased with the addition of bentonite substitution; the higher the bentonite percentage, the lower the density. The reason for this is that density depends on specific gravity. The density of the control mix is higher than that of the bentonite-based combination because cement has a higher specific gravity than bentonite [43].

Figure 7. Fresh and dry density vs. BP%

A study additionally stated that when the concrete mix's BP increases, its workability lowers and its compaction affords increase, which in turn causes the concrete mix's fresh and hardened density to fall [44]. Theworkability led to more voids and compaction in the hardened concrete, which decreased the density of the concrete.

3.2.4. Mass loss

Mass loss is an essential variable to consider when assessing the durability of concrete construction. **Figure 8** makes it clear that mass loss increases with temperature and that it decreases with an increase in bentonite amount. Bentonite-containing mixtures outperformed the control mix in terms of behavior, it may be concluded. Bentonite exhibits better performance at high temperatures than cement due to its higher heat capacity and lower thermal conductivity. When compared to the control mix, the bentonite mix with a high heat capacity has a better microstructure and inhibits the spread of cracks [44]. There are four distinct phases to the Changes of mass loss versus exposure temperature. In the range of room temperature to 100°C, there is not much mass fluctuation. The mass loss in this area is correlated with the free water in the capillary pores leaving. For all concretes, there is a noticeable increase in mass loss that amounts to 5.6% of the initial mass when the temperature increases from 100 to 300°C. Both capillary water and gel water are released in this area, which results in mass loss [45]. The rate of mass loss comparatively slows significantly between 300 and 500°C. The mass loss rate rises once more above 500°C. This may result from the loss of the material surface, the release of CO2, and the breakdown of calcareous particles [46].

For mortar compositions with higher BP ratios, a somewhat significant color change was noted. A color change was observed from gray to pink for samples containing BP. The sample color before and after treatment at high temperatures is shown in **Figure 8**. When the reference and 5%BP specimens were heated to 300°C, there was no discernible change in their color. As soon as the temperature reached 600°C, the concrete color of 5%BP began to turn to pink. While the color change At 600°C, the cracks grew more noticeable. This figure additionally displays a color shift. This alteration provides a visible indicator of a substantial change in the characteristics of the material [47].

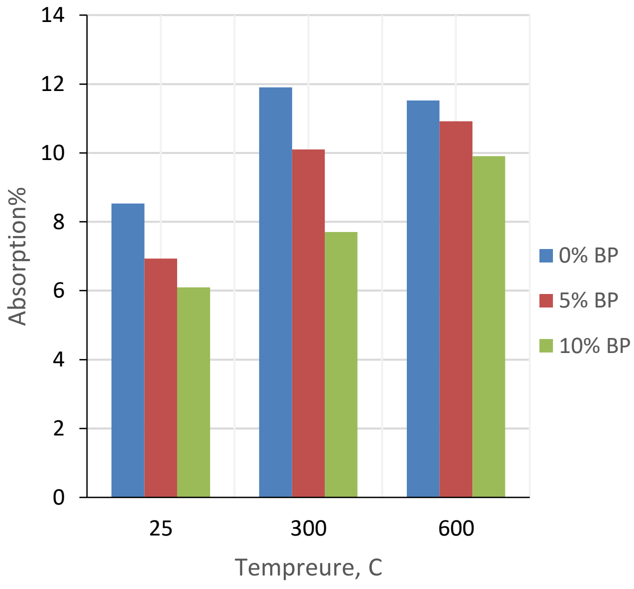

Figure 8. Mass loss vs. BP%

3.2.5. Colour changes of specimens under elevated temperatures

All concrete specimens before exposure were gray, however, at 300 °C, all reference specimens and 5%BP exhibited some minor color variations (light gray) compared with specimens at ambient temperature. However, at 600 C the colors of the 5% specimen changed to (light pink), whereas the colors of the reference remained unchanged (light gray). As shown in **Figure 9**, at 300 C and 600 °C exposure, all 10% of specimens were light pink. This color change was due to the decomposition of CaCO3 into CaO and CO2 under fire exposure [48].

Mineral alterations occurred in mortar following exposure to high temperatures. The breakdown of the C-S-H gel and the conversion of Ca (OH)2 into CaO as a result of the removal of chemically bound water were two of the most significant chemical changes. When 5% and 10% of the CaCO3 composition were used instead of the reference one, the amount of CO2 released during the breakdown of the CaCO3 at 600°C exposure was decreased. Consequently, following exposure to high temperatures, the amount of CaCO3 left behind reduced as the BP replacement level rose. of CaO. CaO and CaSiO2 were produced as a result of the breakdown of Ca(OH)2, CaCO3, and C-S-H. According to Kore Sudarshan and Vyas [48], concrete's mechanical characteristics significantly decreased at 600 °C exposure due to the breakdown of C-S-H.

4. Conclusions

In summary, this study has demonstrated that the use of Iraqi bentonite is effective for resisting high temperatures, and thus it is possible to reduce the use of cement while improving the properties of mortar exposed to high temperatures. The research results can be summarized as follows:

1. BP lowers the mortar workability by 15% and 25% for 5% and 10%BP, respectively. and the degree of the reduction is correlated with the replacement level
2. The delayed reactivity of pozzolan made from Iraqi bentonite retards both the beginning and final setting times, which lowers the rate at which strength is gained.
3. The mortar absorption was found to have decreased with BP increased by 18.75% and 28.5% at 25ºC. All specimens'absorption were increased with increasing the temretures.
4. At room temperature, BP reduces compressive strength according to its concentration. However, when the temperature rose, the compressive strength rose along with the BP increase compared to control specimens and the loss in strength decreased.
5. Compressive strength has been found to be lower than the control mix at ambient temperature and at 300 o C by 22.8% and 38.9, respectively for 5%BP and 10.5% and 24.8%, respectively for 10%BP. But at 600 o C, the compressive strength of mixes containing BP is higher than that of the control mix by 33% and 49.5% for 5% and 10%BP .
6. Mass loss increases with temperature and decreases with an increase in bentonite amount. Bentonite-containing mixtures outperformed the control mix in terms of behavior, it may be concluded. Bentonite exhibits better performance at high temperatures than cement due to its higher heat capacity and lower thermal conductivity.
7. For mortar compositions with higher BP ratios, a slightly color change was noted. A color change was observed from gray to pink for samples containing BP.

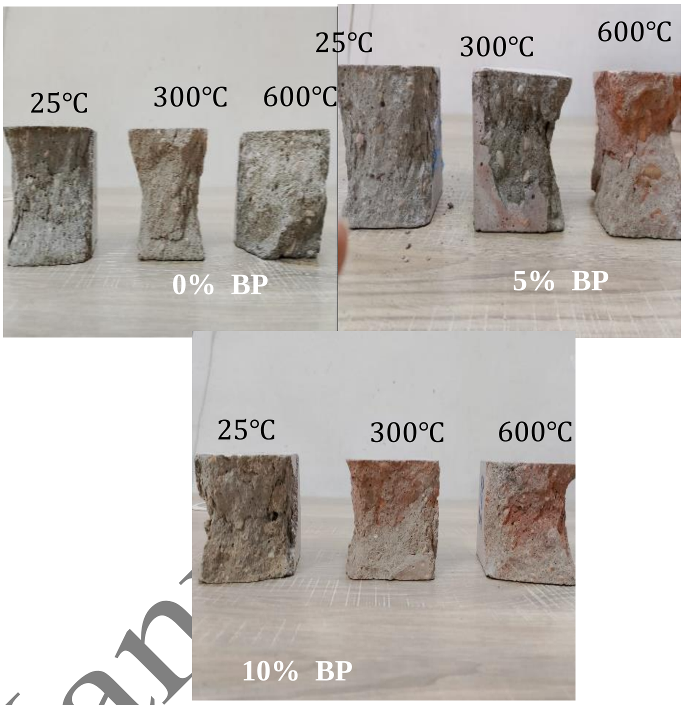

Figure 9. Color shift vs. BP% at different temperature levels

Authors' contribution

All authors contributed equally to the preparation of this article.

Declaration of competing interest

The authors declare no conflicts of interest.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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