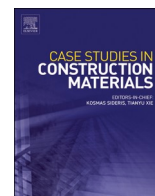




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Effects of raw and different calcined bentonite on durability and mechanical properties of cement composite material

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ABSTRACT

Bentonite is a natural pozzolanic material mostly composed of SiO₂ and employed in cementing materials for the reduction of cement consumption and CO₂ emission besides improving performance of cement composite material by consuming portlandite to form extra C-S-H gel. So, many researchers studied on partial replacement of bentonite to improve concrete performance. However, bentonite mostly exists as a consolidated form that needs treatment for activation of pozzolanic reactivity to improve performance of cement composite materials. However, it is not well known how different replacement of raw and calcined bentonite affects performance of cementing materials in different hazardous environment. Hence, the present study identified the most influential replacement of bentonite, evaluating different calcined and raw bentonite in environments of acids, salt, and elevated temperature in addition to assessing the mechanical and physical properties of cement composite material. So, the results indicate that the employment of raw and calcined bentonite reduces the fresh bulk density, improves compressive strength, and Vicker hardness at 28 days, reduces the mass loss due to 56 days immersion in 5% HCl, 10% NaCl, and 5% HNO₃ compared to the control mixture. Moreover, the employment of calcined bentonite at 800 °C significantly improved the durability and mechanical properties of cement composite materials compared to the control mixture. Specifically, replacing bentonite calcined at 800 °C for 3hs by 20% has the highest compressive strength than all samples after 28 and 56 days. Besides these, the study reveals that the corrosion potential of steel bars embedded in the mortar is reduced as the proportion of raw and different calcined bentonite increases. Generally, the employment of calcined bentonite is very beneficial to get improved performance on mechanical and durability properties of cement composite materials.

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1. Introduction

Ordinary Portland cement production requires a huge amount of energy consumption and pollutes the environment by releasing one tone of CO₂ through every tone of its production [1–7]. Hence, using supplementary cementitious materials commonly known as pozzolana in cement composite materials contributes a lot of significance in improving performance, reducing cement consumption with highly lessening CO₂ emission [8–20]. Pozzolanic materials are mostly used in the production of Portland pozzolana cement which helps to control the heat of hydration, that enables to use in mass concrete projects [21]. Also, it is used in production of cement composite materials to reduce Portland cement consumption and to improve the mechanical and durability properties of cementing materials [22–26]. Besides these, it can provide a significant chemical binding of water, resulting in reduced permeability of concrete matrix [27,28]. Hence, most specimens having cement and pozzolanic materials exhibit smaller deformations and higher compressive strength [9,24,29–31].

Pozzolana has two types that are artificial and natural pozzolana [21,32,33], bentonite is a natural pozzolana rich in SiO₂, Al₂O₃, Fe₂O₃, and its replacement in cement composite materials used for active consumption of free lime to form extra C-S-H gel. The C-S-H gel is the sole that forms strength development in Portland pozzolana cement production and cement-based concrete [34,35]. Employing bentonite in a concrete mixture lessens the amount of portlandite due to the hydration reaction between bentonite and cement [36]. Hence, the use of bentonite in cementing material enhances strength and durability by increasing the resistance to acidic attack of cement matrix [37–44].

Besides these, partial substitution of calcined bentonite in cementitious materials lessens acidic attack by enhancing the resistance of chloride penetration and sulfate resistance compared to reference concrete [42,45–47]. Especially, penetration of acids significantly lessen with rising level of bentonite in concrete, this is because, rising the content of calcined bentonite that more fills pores and improves densification of cementitious materials that can barrier the migrations of aggressive agents [38,45,48]. Also, this is mostly due to the amorphous aluminum silicate matrix of calcined bentonite highly reacting with Portlandite to form a secondary C-S-H gel that can improve the performance of the final hydrated cement matrix, which is primarily dependent on the hydration reaction of



Fig. 1. The colour of raw and calcined bentonite at 400, 600, and 800 °C.

pozzolana and cement phase [49–54]. Hence, Wei & Gencturk [36] reported that adding bentonite in cement composite materials is beneficial by its ability to consume more portlandite compared with some of natural and artificial pozzolana, specifically, more than kaolin replacement in cement composite materials. However, the active pozzolanic reaction of natural pozzolana requires thermal activation [55]. Hence, bentonite is one of the natural pozzolana reflects pozzolanic properties when calcined at the optimum temperature [22]. Especially, bentonite mostly achieves maximum pozzolanic reactivity in between 650 and 900 °C calcination temperature, that can change crystallization to amorphousness, however above 900 °C reactivity rapidly decreases due to re-crystallization of bentonite [56,57]. So, many researchers studied as partial replacement of bentonite improves concrete performance. Reddy & Reddy [58] studied on the replacement of bentonite in concrete by raw and calcined at 700 °C and 800 °C to assess its effect on some of mechanical and physical properties of concrete. Also, Rehman, Yaqub, Noman, et al. [38] investigated on different substitutions of bentonite in concrete by raw and calcined at 800 °C to evaluate its effect on some of mechanical properties and durability such as water absorption and sulfuric acid effects. However, the investigation of various ranges of bentonite calcination temperature with different bentonite substitutions at different adverse environment needs detail studies. So, the present study performed on the employment of raw and different calcined bentonite at 400, 600, and 800 °C having replacement ranges 0, 5%, 10%, 15%, and 20% to assess the effect of different acids, salt, and elevated temperature environments. Besides these, the present study investigated the reactivity of raw and calcined bentonite with detail analysis on physical and mechanical properties of cementing materials. In addition to this, have analyzed the importance of employing different doses of raw and calcined bentonite on preventing the corrosion of steel bars in a salty environment. Hence, from the sampled bentonite replacement the best bentonite dose and calcination temperature that can improve durability and mechanical properties of cement composite materials is recorded.

2. Materials

The mortar specimens used in this study have four components cement, sand, water, and bentonite. The ordinary Portland cement (OPC) of CEM I 32.5 R was used for the preparation of mortar at a plane and different substitutions of raw and calcined bentonite as shown in Fig. 1. The aggregate used is fine aggregate as the ASTM standard presented in Fig. 2. The water used is the distilled water supplied by the Tanga cement PLC., where this study was conducted.

The raw bentonite is extracted from Arusha, Tanzania and the collected raw bentonite is grinded by laboratory ball mill machine sieved by number 325 sieve and the residues is checked, as presented in Table 1. After that, the calcination of bentonite was conducted at 400, 600, and 800 °C for 3hs, chosen based on the previous studies, Laidani et al. and Rehman, Yaqub, Ali, et al. [44,56] reported dehydroxilation or reactivity of most clay starts after mid calcination temperature 400 °C and at 900 °C for 3 hs reactivity rapidly lessens. Then after, the chemical composition of the raw and different calcined bentonite are conducted by X-ray fluorescence (XRF) as shown in Table 1. Hence, the bentonite oxide composition for SiO₂, Al₂O₃, and Fe₂O₃ exceeds 70% of the minimum requirement of ASTM C618 [59] for pozzolanic materials can be added in cement composite materials.

2.1. Samples tested

Different mortar samples were prepared from raw and calcined bentonite at 400, 600, and 800 °C in addition to the conventional mortar samples. The water to cement ratio was 0.485 for all samples and sand to cement ratio of 2.75 according to ASTM C618 [59]. As presented in Table 2 the cement is replaced by raw and calcined bentonite at 400, 600, and 800 °C by 5%, 10%, 15%, and 20% to the mass of used OPC. The compressive and flexural strength tests were conducted by the dimensions of 50*50*50 mm³ and 40*40*160 mm³ respectively. The 425 cubes of 50*50*50 mm³ casted for tests of compressive strength, strength loss at elevated temperature, acidic, and salt effects, and also 40*40*160 mm³ prism of 102 casted for flexural strength test for both 28 and 56 days age.

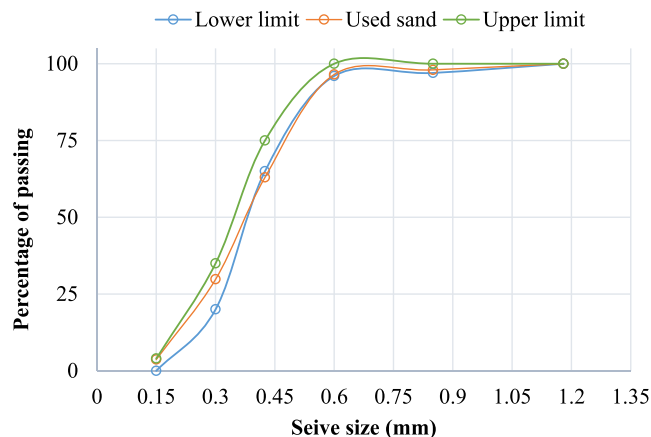


Fig. 2. Gradation of used fine aggregate.

Table 1
Chemical composition of used cement and bentonite.

Composition	OPC	Raw Bentonite	Calcined bentonite		
			400 °C	600 °C	800 °C
SiO ₂	17.57	50.51	52.17	53.50	58.30
Al ₂ O ₃	4.07	12.62	13.29	13.31	10.93
Fe ₂ O ₃	2.63	7.81	7.05	6.95	8.05
CaO	61.48	1.91	3.89	5.19	7.75
MgO	0.41	6.38	5.62	5.79	6.00
SO ₃	1.79	0.13	0.06	0.07	0.10
K ₂ O	0.11	2.02	2.42	2.58	2.36
Na ₂ O	0.04	2.88	3.21	3.29	3.49
TiO ₂	0.29	1.00	0.89	0.93	0.91
P ₂ O ₅	0.13	0.34	0.23	0.25	0.21
LOI	10.74	10.74	9.93	4.30	2.18
Residue at 45µm	12.49	1.72	0.31	1.26	0.80

Table 2
Materials used for the sample preparations.

Sample name	W/C	Cement (gm)	Sand (gm)	Raw bentonite	Calcined bentonite		
					400 °C	600 °C	800 °C
Control	0.485	2400	6600	0	0	0	0
M5Braw	0.485	2280	6600	120	0	0	0
M10Braw	0.485	2160	6600	240	0	0	0
M15Braw	0.485	2040	6600	360	0	0	0
M20Braw	0.485	1920	6600	480	0	0	0
M5B400	0.485	2280	6600	0	120	0	0
M10B400	0.485	2160	6600	0	240	0	0
M15B400	0.485	2040	6600	0	360	0	0
M20B400	0.485	1920	6600	0	480	0	0
M5B600	0.485	2280	6600	0	0	120	0
M10B600	0.485	2160	6600	0	0	240	0
M15B600	0.485	2040	6600	0	0	360	0
M20B600	0.485	1920	6600	0	0	480	0
M5B800	0.485	2280	6600	0	0	0	120
M10B800	0.485	2160	6600	0	0	0	240
M15B800	0.485	2040	6600	0	0	0	360
M20B800	0.485	1920	6600	0	0	0	480

3. Methods

3.1. Reactivity of raw and calcined bentonite

For raw and calcined bentonite Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) characterization was conducted to evaluate the reactivity changes due to calcination of bentonite. The PANalytical X'Pert Pro X-Ray diffractometer machine was used by the potential working setup of 30 mA, 40 kV, which analyzed mineralogical phase by the diffraction peaks covering large 2θ between 10 and 90°. Also, A224159 SHIMADZU Fourier transform infrared spectro-photometer was used to record the percentage of transmittance peaks versus the range of wavelength between 400 and 4600 cm^{-1} . So, for both methods the powder sample of raw and different calcined bentonite were used that passed sieve number 325 as described in Section 2.

3.2. Fresh property

The fresh bulk density of pozzolana incorporation is directly linked with the reactivity of pozzolana to the calcium hydroxide to form hydrated calcium silicate, which is due to the higher surface area of pozzolana the higher pozzolanic reactivity that react with the free calcium hydroxide, hence lower fresh bulk density of cementing materials [60]. Also, fresh bulk density changes can be related to the specific gravity of pozzolanic material compared with the cement specific gravity [61]. The fresh density for both raw and calcined bentonite blended mortar was conducted by taking a volume of the mortar sample to the known volume of a one-litre cylinder.

3.3. Hardened properties

The compressive and flexural strength tests were conducted as ASTM C109 [62] by cube of 50*50*50 mm^3 and 40*40*160 mm^3 prism mortar specimens respectively, by the use of serial number: 1543–08 Zwick-Roell digital compressive strength machine at the

rate of 1800 N/sec and using Model DKZ-5000 Electronic flexural testing machine respectively. The compressive and flexural strength of all samples were measured at the ages of 28 and 56 days. Three samples were casted for each category and a total of 102 samples were exposed for each compressive and flexural strength test for samples cured in water. Also, the strength activity index (SAI) was determined at 28 days of compressive strength in order to rate the pozzolanic activity of both sampled raw and calcined bentonite. So, the strength activity index was calculated by the Eq. (1) as described on ASTM C618 [59].

$$\text{Strength activity index at 28 days} = 100 * \frac{A}{B} \quad (1)$$

A: the compressive strength of mortar containing bentonite at 28 days. B: the compressive strength of mortar containing only OPC at 28 days.

Besides these, the Vicker hardness test is conducted through indenting the test materials by the diamond shaped indenter in the form of a right pyramid with a square base and an angle of 136° between opposite developed the same to a load of 1–100 kgf. So, the Vickers hardness is evaluated by the Eq. (2).

$$HV = \frac{2F \sin 136/2}{d^2} = 1.854 F / d^2 \quad (2)$$

Where: F is load by kgf, d arithmetic mean of the two diagonals of the indented place by mm and HV is Vickers hardness by kgf/mm²

The acid attack test was conducted by mortar specimen of 50 mm³ immersed for 56 days in a 5% acid solution, prepared using 5% hydrochloric acid and nitric acid separately. The weights of all cubes were recorded before the acidic immersion and maintained for a period of 56 days in both acidic solutions. The mortar specimen was taken out after 56 days from both acids and left for 24 hours to dry at room temperature. The compressive strength test was conducted after 24 hours and the resistance to acidic attack of the mortar was established based on the mass loss of the cubes and the strength loss comparing with samples cured in water similar to [63].

Also, the salt attack test was conducted using 50 mm³ two plain mortar cubes and one mortar cube of steel bar embedded sample prepared each by the raw and calcined bentonite blended mortar immersing in 10% of NaCl solution. The samples immersed in the salt solution were taken after 56 days and then permitted to dry at room temperature for 24 hours. Hence, the mass loss due to the salty environment was conducted and the analysis of the steel corrosion resistance due to employment of raw and calcined bentonite by different doses were examined by visual inspection on the surface of the steel bar as [64]. Besides these, the elevated temperature effect was analyzed using a 50 mm³ mortar specimen which was placed in the electronic oven at 200 °C for two hours after its 56 days of curing. The mass change before and after the exposure of elevated temperature was recorded and then the compressive strength test was conducted.

4. Results and discussion

4.1. FTIR and XRD results

The raw and different calcined bentonite sample characterization of the functional group was conducted using Fourier transform infrared spectroscopy. As shown in Fig. 3 the results of both grinded raw and calcined bentonite have significantly different spectrum which can indicates the activation of bentonite by using the calcination method. The spectrum near 619 cm⁻¹, attributed to the coupled Al–O and Si–O vibrations [65]. However, as presented in Fig. 3, the raw and calcined bentonite have most different spectrum between the range of 750–1750 cm⁻¹ and 2500–3750 cm⁻¹. The spectrum between 975 and 1113 cm⁻¹ can indicates Si–O bonds in the SiO₄ molecules [66,67]. At point 1 the peak is 984.14 cm⁻¹ for raw bentonite and slightly stretching to 1040 cm⁻¹ for 800 °C calcined bentonite, which basically shows amorphousness of SiO₂ and the peak heights at this point is slightly reduced with increasing temperature that is due to calcination destructed the crystalline phases of bentonite and changed to the formation of amorphous material

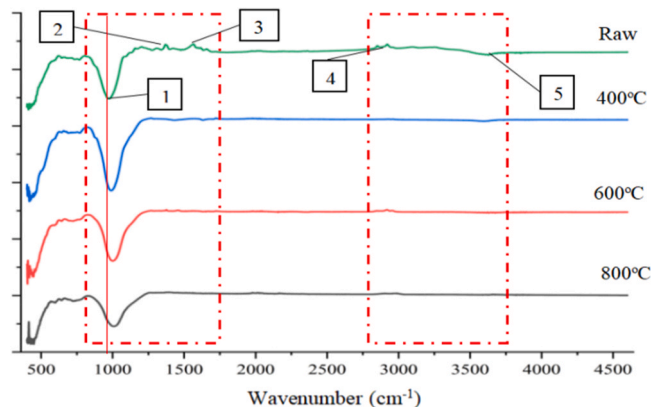


Fig. 3. Result of Fourier transmission infrared spectroscopy radiation for functional group of raw and different calcined bentonite.

[68]. Hence, the increase in amorphous phase can more consume portlandite and improves properties of cement composite materials [57]. Also, point 2 and 3 have spectrum 1375 cm^{-1} and 1558.77 cm^{-1} respectively, both showing H_2O stretching [67], which is slightly disappearing with increasing temperature that shows the reduction of water molecule by temperature and totally disappeared at $800\text{ }^\circ\text{C}$. Besides these, at points 4 and 5, the spectrum's are 2930.97 cm^{-1} and 3620.34 cm^{-1} respectively, reflects the OH^- vibrations that is also slightly reducing its peaks and totally disappeared at $800\text{ }^\circ\text{C}$ due to dehydration by the increased temperature [68], that may indicates the end of crystallization at $800\text{ }^\circ\text{C}$, hence can occur highest reactivity [69].

The raw and calcined bentonite XRD results are shown in Fig. 4, it is observed that the reduction of carbonate mineral because of its decomposition through increasing the calcination temperature, hence calcium carbonate decomposed into CaO and CO_2 at $600\text{--}800\text{ }^\circ\text{C}$ [12]. Also, found that the peak of montmorillonite mineral reduced by thermal activation and almost, the peak destroyed at $800\text{ }^\circ\text{C}$, which shows the dehydroxilation and transformation of bentonite into metabentonite have been performed completely, this indicates the highest pozzolanic reactivity of bentonite [70]. Furthermore, the peaks size of quartz decreased, which is due to loss of crystallization and formation of the amorphous state of calcined bentonite while increasing the temperature [71].

4.2. Physical property test results

4.2.1. Fresh bulk density

The results of fresh bulk density of different substitutions of calcined and raw bentonite in the mortar sample are shown in Fig. 5. The employment of both calcined and raw bentonite in the mortar reduces the fresh bulk density compared to the control mixture. Also, significant reduction of fresh bulk density with increasing the content of bentonite from 5% to 20%. This is mainly due to the specific gravity of bentonite is much lower than cement which can reduce the density of the cement composite materials [61], hence, while increasing the bentonite content it is observed significantly decreases in the fresh bulk density of cement composite materials. Besides these, the decrease in the fresh bulk density may occurred due to rising bentonite replacement dose have caused reduction in the water level, but it was within the acceptable range of water level as ASTM C311 [72]. Furthermore, it have seen increasing the calcination temperature of bentonite lessens the fresh bulk density of calcined bentonite blended mortar. A similar observation as density of cementitious materials reduced by substituting calcined bentonite which is mainly due to the fine particles of calcined bentonite fills and began to take the place of cement particles [73–75].

4.3. Mechanical property test results

4.3.1. Compressive strength

The results of the compressive strength test of mortar at 28 and 56 days for both control and bentonite replacement (raw and calcined) are shown in Fig. 6. At both testing ages, the bentonite replacement revealed higher compressive strength compared to the control mixture. That is mainly due to the pozzolanic reactions of bentonite that consume the free lime and form secondary C-S-H gel which improves the strength of sampled mortar. Besides these, the highest compressive strength has been seen for the employment of calcined bentonite at $800\text{ }^\circ\text{C}$ compared to all the samples. Also, the 20% bentonite calcined at $800\text{ }^\circ\text{C}$ substitution significantly enhanced the compressive strength than the other bentonite replacement. This is mainly due to the activated bentonite at $800\text{ }^\circ\text{C}$ can consume much free lime through increasing the replacement doses which can produce secondary C-S-H gel and participate in strength improvement.

Besides these, the 5% bentonite replacement significantly improved the compressive strength in low calcination temperature and raw bentonite replacement. However, increasing the doses of raw bentonite replacement reduced the compressive strength compared to 5% replacement. However, increasing employment of $800\text{ }^\circ\text{C}$ calcined bentonite from 5% to 20% increases the compressive strength of mortar. This indicates the beneficial effect of calcination of bentonite to replace more cement content with having a safe environment and low cost compared to cement production, which is commonly due to OPC production requires $1450\text{ }^\circ\text{C}$ calcination temperature and one kg of cement unit price is 85.7% more than the same bentonite mass [61,76]. However, bentonite without

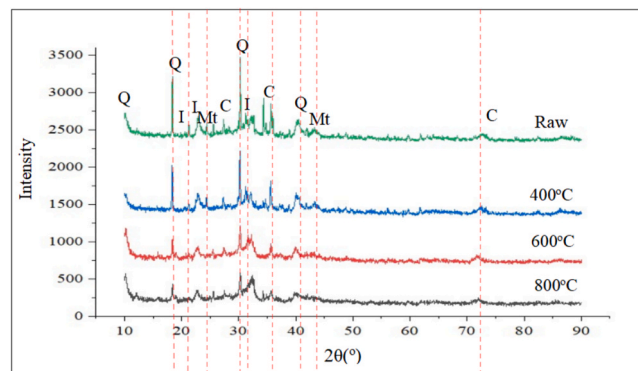


Fig. 4. XRD pattern of raw and calcined bentonite at 400, 600, and $800\text{ }^\circ\text{C}$, where Q-Quartz, I-Illite, C- Calcium carbonate, and Mt-Montmorillonite.

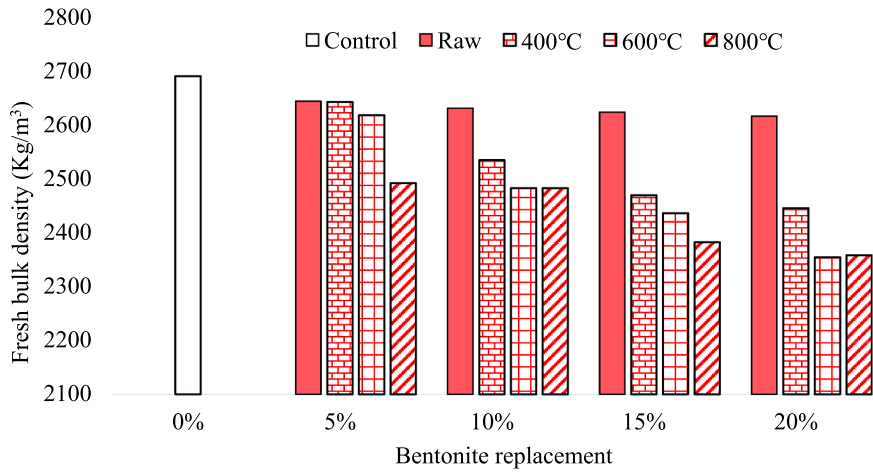


Fig. 5. Fresh bulk density test results for different bentonite replacement.

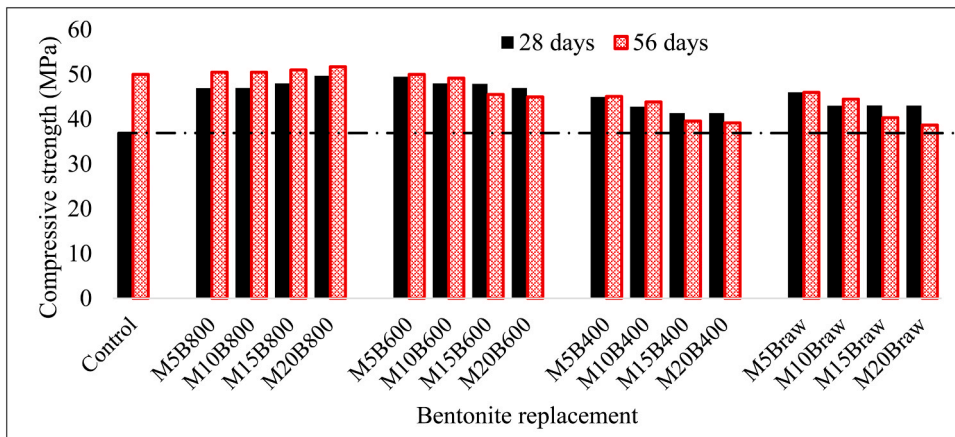


Fig. 6. Compressive strength test results for different bentonite replacements at 28 and 56 days.

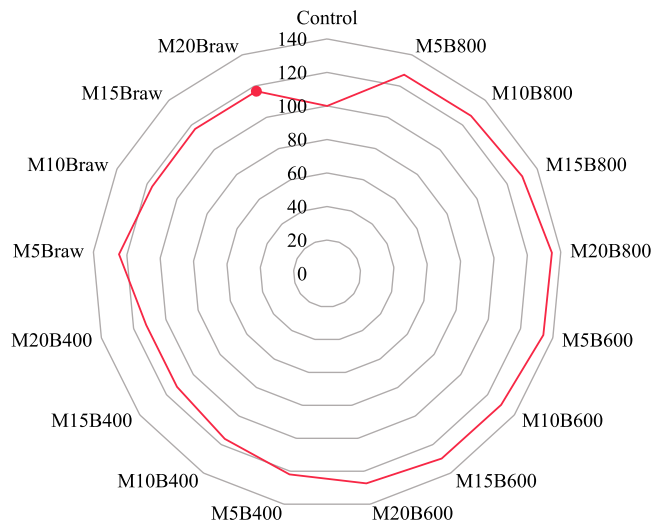


Fig. 7. Strength activity index results for different bentonite replacement at 28 days.

required calcination cannot form a pozzolanic reaction because of it is unreactive without calcination. So, the employment of 800 °C calcined bentonite in mortar improved by 27.24%, 27.37%, 30.08%, and 34.15% at 28 days, and 1%, 1%, 2%, and 3.4% at 56 days respectively by 5%, 10%, 15%, and 20% replacement compared to the control mixture.

Similar observation with Rehman, Yaqub, Noman, et al. [38] studied on the replacement effect of raw grinded bentonite and calcined bentonite (heated at 800 °C). The study used different replacement doses separately for both raw and calcined bentonite and found adding calcined bentonite highly enhances the compressive strength in all sampled replacement levels compared to raw bentonite addition in concrete. That is because of bentonite found in a consolidated form which cannot actively form pozzolanic reactions without calcination which can be used for improvement of strength without calcination [38]. In addition to these, Reddy S. & Reddy M. [58] reported that different calcination temperatures significantly affected the compressive strength of different bentonite replacement doses, and the effects of calcination are mostly reflected at 800 °C of bentonite calcination, which means at this temperature pozzolanic reactivity is high, that is due to increasing the temperature increases the reactivity of calcined clay, especially, while reaching the dehydroxilation stage in which the amorphous phase starts, consequently increases the compressive strength of cementing materials [77]. However, after 900 °C recrystallization of most clay begins and the reactivity decreases [70].

Also, as presented in the Fig. 7 the strength activity index of calcined bentonite replacement increases with increasing the calcination temperature. According to ASTM C618 [59] for replacement of any natural pozzolana at 28 days of curing age, the strength activity index have to be minimum 75% compared to the control mixture, hence, all the raw and calcined bentonite replacement fulfills the requirement of ASTM C618 standard. However, the employment of 800 °C has the highest strength activity index than other calcined bentonite replacements. The 800 °C calcined bentonite employment increased strength activity index by 127.24%, 127.37%, 130.08%, and 134.69% respectively 5%, 10%, 15%, and 20% replacement compared to the control mixture. Hence, the higher value of the strength activity index indicates the higher pozzolanicity of the material which can actively react with free calcium hydroxide during the hydration reactions [78].

4.3.2. Flexural strength

The results of flexural strength test are presented in Fig. 8. The results shows the increasing the dose of bentonite samples raw, 400 °C, and 600 °C reduced flexural strength compared to control mixture at 28 days. However, at 56 days 5% replacement of both calcined bentonite significantly improved the flexural strength. That is mainly because the raw bentonite cannot react actively with free lime to form C-S-H gel to improve flexural strength.

However, the replacement of bentonite in mortar calcined at 800 °C for all sampled replacement doses improved the flexural strength compared to all other samples at both 28 and 56 days of mortar sample. The replacement of calcined bentonite at 800 °C improved flexural strength by 6.61%, 8.21%, 5.37%, 1.24% at 28 days and 12.34%, 8.64%, 5.76%, and 4.94% at 56 days compared to the control mixture, while the raw bentonite replacement improved only by 5% of bentonite substitution which is by 1.23% at 56 days compared to the control mortar mixture. That is mainly due to heat treatment can more activate pozzolanic reaction that can more improves strength than adding raw bentonite. Hence, the study confirmed, that less pozzolanic reaction has been occurred in the raw bentonite than in calcined bentonite replacement in the cement composite materials.

4.3.3. Vicker hardness

The hardness of cement composite material is an important index that shows the mechanical properties of the material [79]. The Vicker hardness test results of the mortar having different contents of raw and calcined bentonite are shown in Fig. 9. The result shows the employment of both raw and calcined bentonite in mortar improves the Vicker hardness compared to the control mixture.

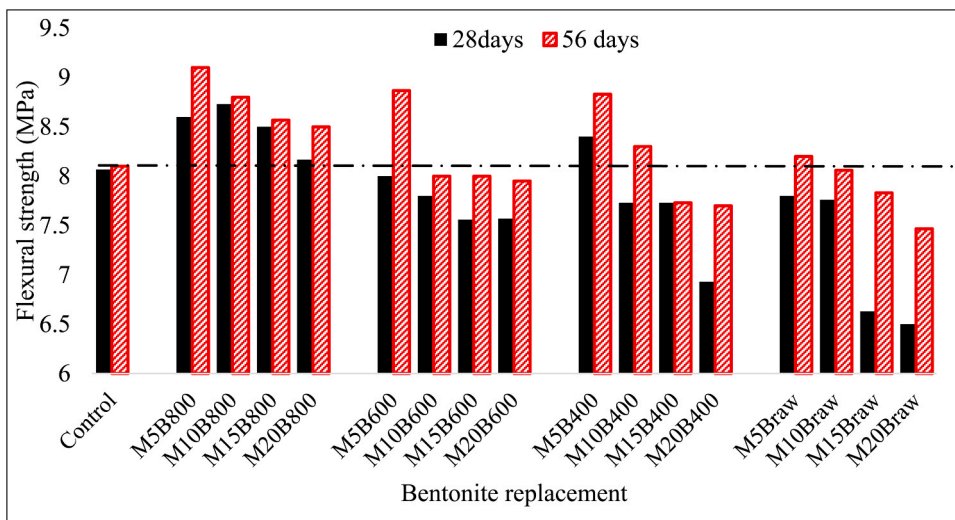


Fig. 8. Flexural strength test results for different bentonite replacement at 28 and 56 days.

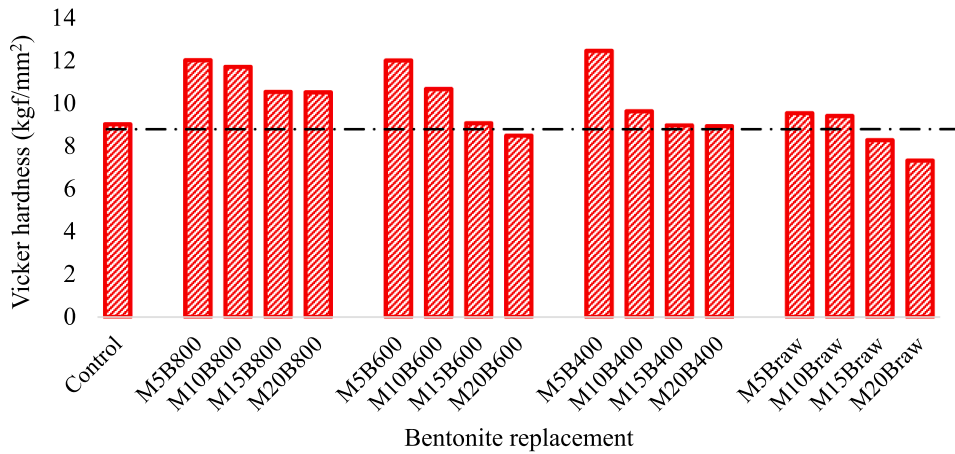


Fig. 9. Vicker hardness test results for different bentonite replacement at 28 days.

However, increasing the doses of bentonite substitution reduces the hardness of the mortar surface, especially, for the substitution of raw, 400, and 600 °C calcined bentonite. But, the employment of 800 °C calcined bentonite improves the surface hardness. That is due to at 800 °C calcination bentonite is highly reactive that can form a pozzolanic reaction. Hence, the employment of the calcined bentonite at 800 °C for 3hs improves the hardness of the mortar by 33.267%, 29.82%, 16.81%, and 16.61% compared to the control mixture. A similar observation with Hakamy et al. [80] found that the calcined clay increased the hardness of cement composite materials. Generally, it is observed that the incorporation of calcined bentonite is more crucial on the improvement of hardness than raw bentonite employment in cement composite materials, which is because, calcined bentonite is highly reactive than raw bentonite that can form active pozzolanic reactions and makes harder surface structure of mortar [79].

4.4. Durability test results

4.4.1. Effect of nitric acid

The results of mortar cubes strength loss due to immersion in 5% nitric acid is shown in Fig. 10. The results indicated the employment of raw, 400 °C, and 600 °C calcined bentonite increased the strength loss with increasing the doses from 5% to 20% bentonite replacement. However, the sampled bentonite substitution both raw and calcined bentonite lessens the strength loss compared to the control mixtures. This is due to the bentonite micro filling ability of pores in the cement composite materials. The employment of 800 °C calcined bentonite significantly reduced the strength loss, even increasing its doses from 5% to 20% reduced the strength loss due to nitric acid immersion. Hence, the reduction are by 11.47%, 11.76%, 13.26%, and 23.67% compared to the control mixtures respective to 5%, 10%, 15%, and 20% calcined bentonite replacement. This indicates the importance of calcined bentonite substitution at 800 °C by 20% can reduce more the effect of acidic adverse environment. That is mainly because of the employment of calcined bentonite at 800 °C has higher reactivity which can interlock the matrix of cement composite materials.

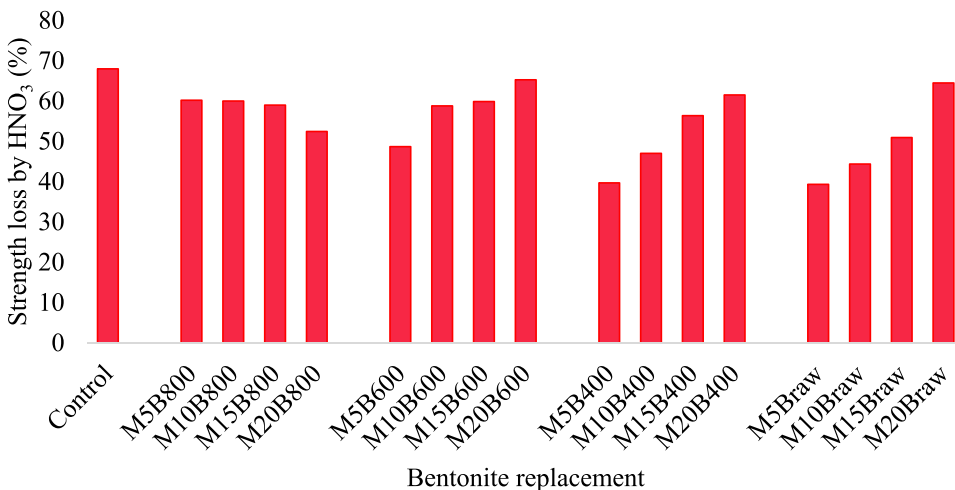


Fig. 10. Compressive strength loss of mortar having different bentonite replacement cured in 5% HNO₃ for 56 days.

Besides these, Fig. 11 shows the mass loss results due to immersion in nitric acid for 56 days and mass loss due to drying after 24hs sample taken from 5% nitric acid solution. The result indicates the bentonite substitution both raw and calcined can reduce the mass loss due to the acid effect compared to the control mixture, especially, mass loss reduced by increasing the replacement of calcined bentonite from 5% to 20%. However, increasing the raw bentonite doses increases the mass loss in the nitric acid solution. That is basically due to the raw bentonite can not actively participate with the calcium hydroxide to form a dense mortar matrix that can reduce the loss of mass and mitigate penetration of acids. Besides these, the specimens after 24hs in atmosphere taken from 5% solution of nitric acid significantly increased the mass loss which is basically because the formation of pores in the mortar matrix by the solution that makes easy to remove water and dry fast, consequently highly loss mass.

4.4.2. Effect of hydrochloric acid

The results of strength loss due to immersion in 5% hydrochloric acid are presented in Fig. 12. The result shows replacement of different calcined and raw bentonite reduced the strength loss of the mortar sample compared to the control mixture. Especially, increasing the dose of 400, 600, and 800 °C calcined bentonite replacement significantly reduces the strength loss due to 5% hydrochloric acid environment. However, increasing the raw bentonite substitution in mortar can not grant the reduction of strength loss, that is mainly because of raw bentonite have low reactivity to produce the pozzolanic reactions which can improve strength loss in the acidic environment.

Also, as shown in Fig. 13 the employment of both calcined and raw bentonite reduced the mass loss of the mortar compared to the control mixture. The calcined bentonite by 15% and 20% replacement significantly lessens the mass loss compared to 5% and 10% in all calcined bentonite replacement samples. That is mainly due to the calcined bentonite replacement highly can participate in the microfilling of pores which can make the mortar matrix denser and mitigate the interference of acids in the mortar structures than the raw bentonite replacement [38]. Similar observation with Lee et al. [46] the higher the calcined bentonite replacement ratio, the smaller the weight loss. Besides these, 20% calcined and raw bentonite employment have low mass loss after 24hs taken from the hydrochloric acid which shows slow drying of the sample due to its dense and low pore structures.

Generally, the dried mass loss after 24hs for mortar specimens cured in 5% of nitric acid is more compared to the mortar specimens cured in 5% of hydrochloric acids, which is basically due to calcium salt formed by the reaction of nitric acid and calcium hydroxide can easily leach and form pores in the cementing materials [81] and hence loss water easily and dried faster.

4.4.3. Effects of different hazards environment by residual analysis

As presented in Fig. 14, the residual of compressive strength test are significantly different for mortar cubes at the age of 56 days cured in 5% hydrochloric acid, 5% nitric acid, normal water curing, and in the elevated temperature of 200 °C for 2hs. The results in Fig. 14 (a-d) shows the fracture pattern of acid and water cured samples are significantly different, which is the acid cured mortar cube has a perpendicular fracture while water cured and elevated temperature have V-shaped pattern. This is due to the place of the existence of maximum stress in each sample is different since the acid cured samples are affected much from the external sides higher stress only occurred in the middle of the samples.

Also, in Fig. 14 (b,d) have observed the different colors of the residual mortar cube having black gray for water cured and white gray for elevated temperature. This is because of the drying effect at 200 °C elevated temperature of the mortar samples. Besides these, as shown in Fig. 14 (e,f) the mortar cube cured in 5% HCl, its residue after compressive strength is more deteriorated than the residue of mortar cube specimen cured in the 5% HNO₃, because of the reaction between HCl and calcium hydroxide forms calcium chloride which is a highly soluble, harmful salt, and cement based material loss binding ability from the affected part due to calcium chloride and therefore easily deteriorate [81,82]. Also, curing the mortar cubes in acids have changed the color of the mortar from black gray to yellow gray respectively from the outer to the inner side of the mortar cubes, similarly as Mohamad et al. [83] reported.

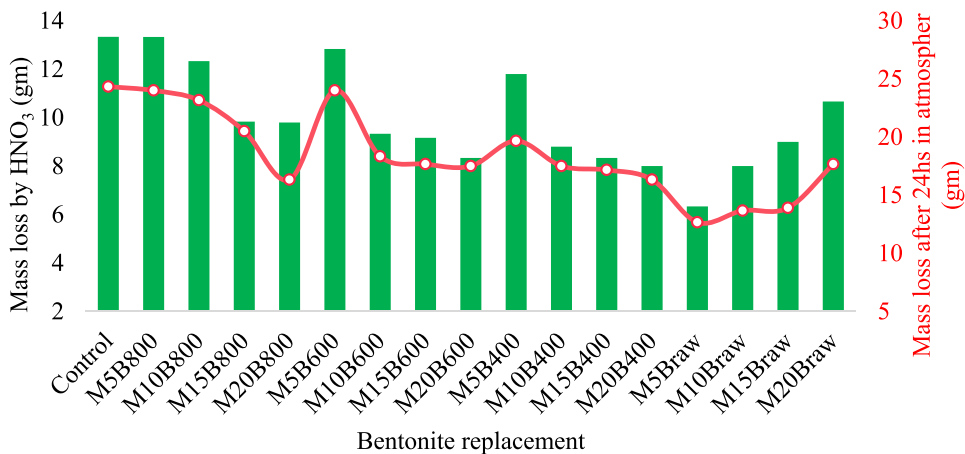


Fig. 11. Mass loss of mortar cube having different bentonite replacement cured in 5% HNO₃ for 56 days and mass loss due to drying rate within 24hs after taken from the solution.

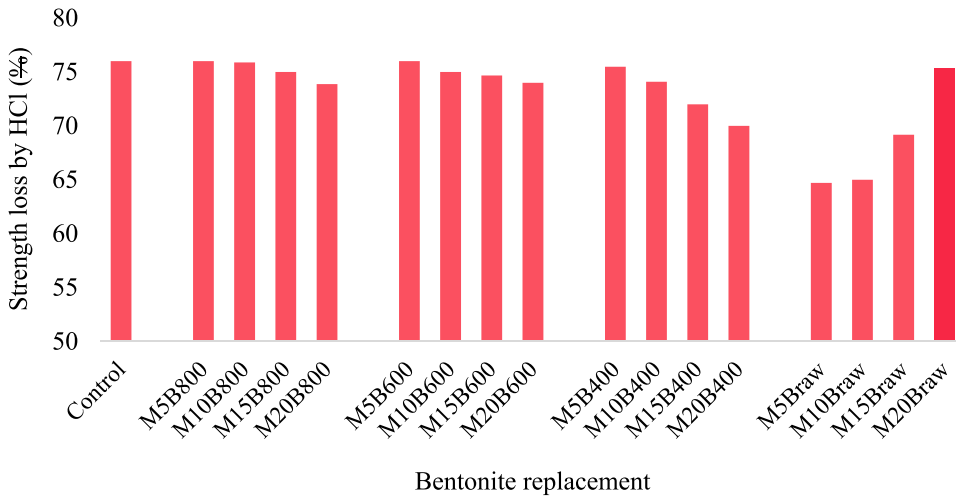


Fig. 12. Compressive strength loss of mortar having different bentonite replacement cured in 5% HCl for 56 days.

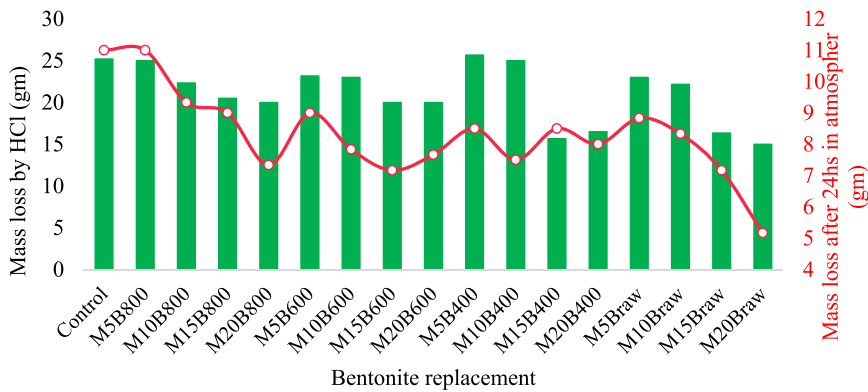


Fig. 13. Mass loss of mortar cube having different bentonite replacement cured in 5% HCl for 56 days and mass loss due to drying rate within 24hs after taken from the solution.

4.4.4. Effect of sodium chloride

The effect of 10% sodium chloride solution on different replacements of bentonite in the mortar is presented in Fig. 15. The result shows the addition of calcined and raw bentonite by 10%, 15%, and 20% significantly reduces the mass loss due to immersion in 10% NaCl solution compared to the control mixture at the age of 56 days. Besides these, the replacement of 800 °C calcined bentonite to the mortar indicated the most reduction of mass loss due to the effect of sodium chloride. This is mainly due to at 800 °C bentonite is highly reactive which can form pozzolanic reactions, interlocks the matrix, and make dense mortar structure that can not easily loss mass in the salt environment [38]. Hence, this is very important which mitigate the entrance of salt to prevent the corrosion of reinforcement steel bars in cement composite materials.

4.4.5. Effect of elevated temperature

The effect of 2hs in 200 °C elevated temperature test results is shown in Fig. 16, the raw bentonite substitution has the lowest strength at the elevated temperature compared to the control and calcined bentonite replacement samples. That is mainly because, the raw bentonite substitution is not very reactive due to bentonite mostly existing as a consolidated form. Also, it is observed the mass loss is higher for raw bentonite employment and reduces with increasing calcination temperature of employed bentonite in cementing materials; which is because of increasing calcination temperature increases reactivity and amorphous phase that leads to an increase in the volume of hydrated phases, which results in higher densification and agglomeration of binder matrix in cement composite materials at elevated temperature [68,84]. Hence, increasing the calcination temperature from 400 °C to 800 °C gradually improved the strength at the elevated temperature of 200 °C. So, all replacement doses at 800 °C calcined bentonite have higher resistance to the elevated temperature than all samples.

Besides these, mass loss increases by increasing bentonite doses with reducing calcination temperature of employed bentonite, which is basically due to a low formation of C-S-H gel structure which can assist to make denser structure of cementing materials to protect fast dehydration while increasing bentonite substitution and reducing calcination temperature.

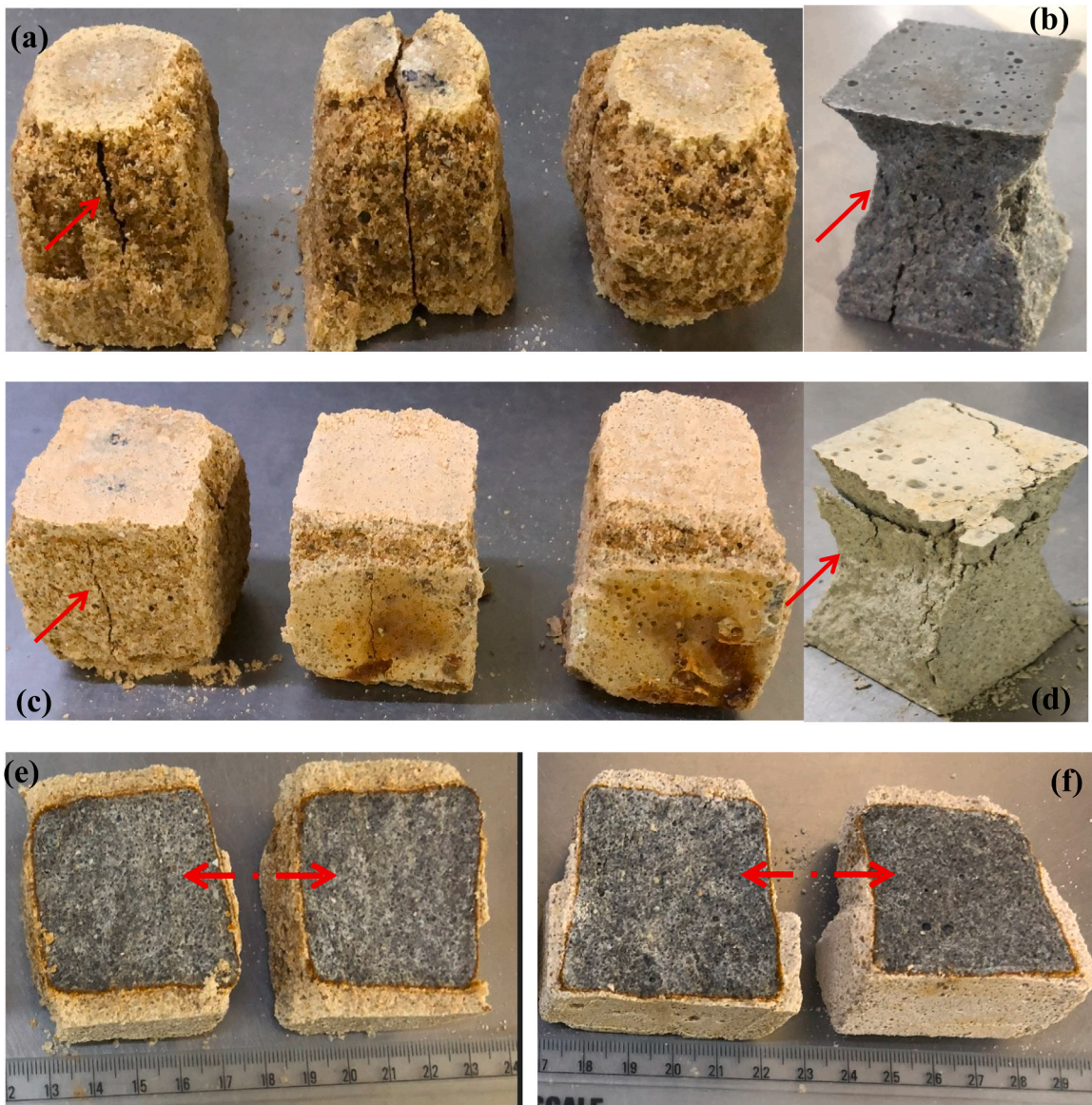


Fig. 14. The compressive strength test mortar cube residue cured (a) 5% of HCl (b) water (c) 5% HNO₃ (d) elevated temperature at 200 °C (e) residue separated into two shown at (a), and (f) residue separated into two shown at (c).

4.4.6. Corrosion analysis

The extent of corrosion in 10% of sodium chloride solution for control, raw, and calcined bentonite blended mortar samples at 56 days are illustrated in Fig. 17. The result shows that mortar embedded with the steel bar for all 5% of raw and calcined bentonite replacements have very active corrosion compared to the higher substitution of raw and calcined bentonite replacement. Also, the study reveals that the corrosion potential is reduced as the proportion of bentonite increases. So, the employment of raw and calcined bentonite is very effective in resistance to the penetration of chloride. The reason behind this phenomenon is indicates the employment of pozzolana grants the pore refinement of cement composite materials [85]. Also, it is observed that calcined bentonite by 15% and 20% have lower corrosion compared to the lower calcined bentonite substitution. That is mainly because more substitution of calcined bentonite can more fills the pore that barriers the penetration of salt to the mortar matrix. However, to observe detail effects of different calcination used isolately on the corrosion resistance, especially, at 15% and 20% substitution its recommended to use detail tests of corrosion analysis that can identify more than the visual inspection method.

Generally, the study revealed that the employment of calcined bentonite improves compressive strength, hardness, and mass loss due to different hazardous environments. Specifically, the replacement of calcined bentonite at 800 °C by 20% significantly enhances the durability and mechanical properties of cement composite materials in addition to lowering the environmental pollution, energy consumption, and production cost of ordinary Portland cement which commonly requires 1450 °C calcination temperature [86].

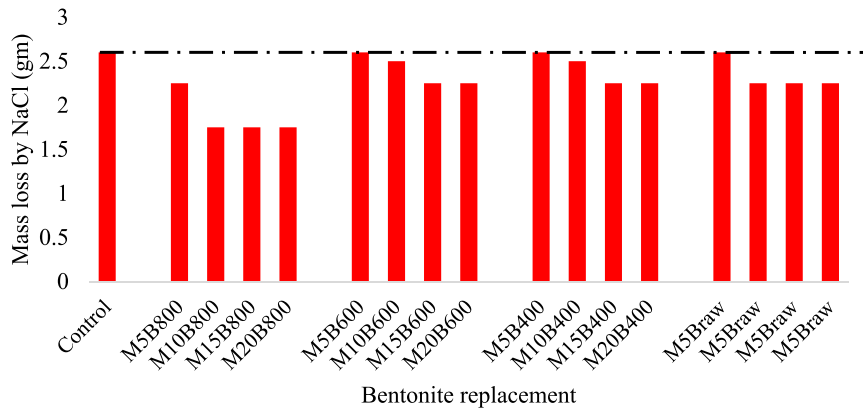


Fig. 15. Mass loss of mortar cube having different bentonite replacement cured in 10% NaCl for 56 days.

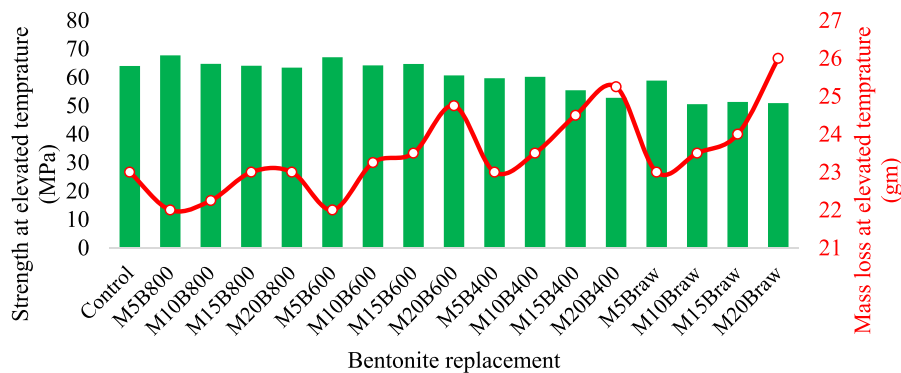


Fig. 16. Compressive strength and mass loss of mortar cube having different bentonite replacement at 56 days of elevated temperature.

5. Conclusions

The present study investigated the effects of raw and calcined bentonite replacement on durability, physical, and mechanical properties of cement composite material. Especially, the resistance in the various hazardous environments were assessed for mortar sample employed raw and different calcined bentonite, and the following conclusions have been reached.

- The employment of raw and calcined bentonite reduces the fresh bulk density of the mortar compared to the control mixture. Also, the fresh bulk density significantly reduced with increasing calcination temperature and replacement dose of bentonite in the mortar.
- At 28 days of curing, the compressive strength of all raw and calcined bentonite replacements are higher than the control mixture. However, the compressive strength increases with increasing the bentonite calcination temperature. Especially, the replacement of 800 °C by 20% has the highest compressive strength than all samples at 28 and 56 days. Also, sample calcined at 800 °C has higher strength activity index than all other samples, which mainly shows its highest pozzolanic reactivity than the other sampled calcined bentonite.
- The replacement of bentonite by 5%, 10%, 15%, and 20% calcined at 800 °C improved flexural strength compared to the respective replacement to all samples. Besides these, the employment of raw and calcined bentonite in the mortar improves the Vicker hardness of the mortar. So, employment of the calcined bentonite at 800 °C for 3hs improves the hardness of the mortar by 33.267%, 29.82%, 16.81%, and 16.61% compared to the control mixture.
- The substitution of calcined bentonite at 800 °C by 20% reflected the lowest strength loss than all the samples after immersion in 5% HCl and 5% HNO₃ for 56 days. That is mainly because, the employment of calcined bentonite at 800 °C has higher reactivity which can interlock the matrix of cement composite materials. Also, the bentonite employment by raw and calcined can reduce the mass loss due to the acid effect compared to the control mixture, especially, the mass loss more reduced by increasing the replacement of calcined bentonite from 5% to 20%. However, the immersion in 5% of HCl solution more affected all the samples by decreasing the strength than 5% HNO₃ solution immersion for 56 days.

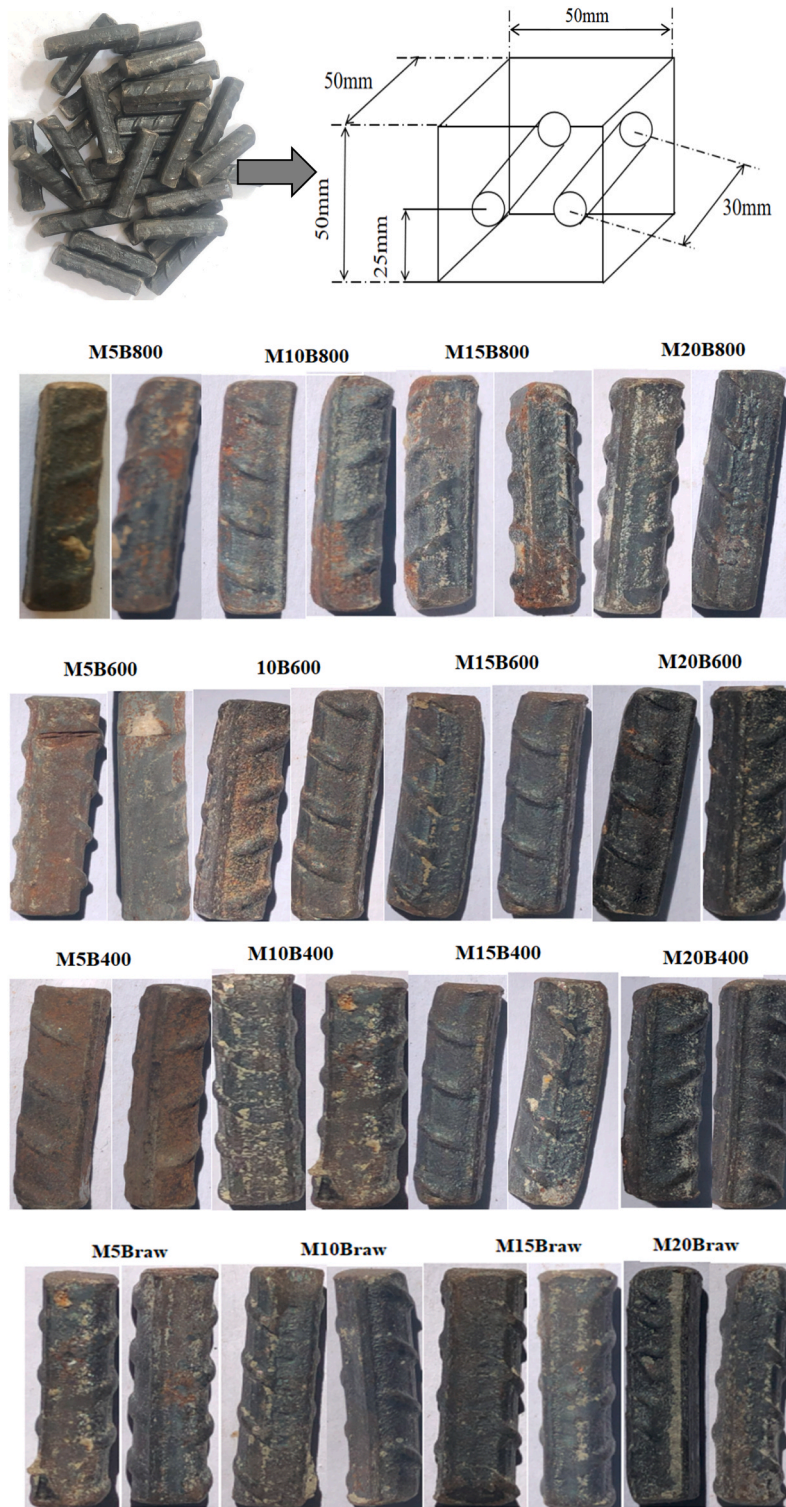


Fig. 17. The corrosion extent of steel bars embedded in mortar having different raw and calcined bentonite replacement cured in the 10% NaCl for 56 days.

- The addition of calcined and raw bentonite by 10%, 15%, and 20% significantly reduced the mass loss due to immersion in 10% solution of sodium chloride compared to the control mixture at the age of 56 days. Besides these, the replacement of 800 °C calcined bentonite to the mortar indicated the most reduction of mass loss due to the effect of sodium chloride.

- The raw bentonite substitution has the lowest strength at the elevated temperature compared to the control and calcined bentonite replacement. All sampled doses of calcined bentonite replacement at 800 °C have higher resistance to the elevated temperature than all samples.
- The fracture pattern of acid and water cured samples are significantly different, which is the acid cured mortar cubes have perpendicular fracture while water cured and elevated temperatures have V-shaped patterns.
- Also, the steel bar embedded for all 5% of raw and calcined bentonite replacement in the mortar have very active corrosion compared to the higher substitution of raw and calcined bentonite replacement. The study reveals that the corrosion potential is reduced as the proportion of bentonite increases. Generally, the employment of calcined bentonite is very beneficial to get improved performance on mechanical and durability properties of cement composite materials.

CRedit authorship contribution statement

Thomas Kivevele: Supervision. **Tsion Amsalu:** Writing – original draft, Visualization, Methodology, Formal analysis, Data curation, Conceptualization. **Yusufu Abeid Chande Jande:** Supervision, Writing – review & editing.

Declaration of Competing 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.

Data availability

No data was used for the research described in the article.

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