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Effects of Elevated Temperatures On The Performance of Heat Resistant Mortars

Engineering

KEYWORDS : Refractory mortars, Temperature regimes, Soaking period, Residual compressive strength, Insulation capacity and Cracking potential

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ABSTRACT

Fire has long been a major hazard in our lives. To control the aftermath effects of these fire accidents, flame retardant technology is becoming increasingly important. Various refractory materials are already in use but their life span and economy are questionable. Many refractory concretes are developed but refractory mortars are rarely addressed. Therefore, developing refractory mortars are of prime importance for various industrial applications. In this study, ten different refractory cement mortars are prepared along with 1:3 ratio of cement mortar mix as control and heated in the furnace for elevated temperature regimes from 110°C to 800°C with a soaking period of one hour for each temperature range. Also the insulation capacity and cracking potential of the same mixes were tested for various other applications. The mechanisms for the behaviour of each refractory mix at elevated temperature are discussed elaborately in this paper.

Introduction

High temperature material failures are one of the most important physical deterioration of concrete/mortar that influences the durability of structures in their service life. Fire has long been a major hazard in our lives. To control these fire accidents, flame retardant technology is becoming increasingly important. However, it is very important to note that there is no systematic development of construction material to resist the flame and heat during fire. At present, structures of various types starting from plain cement concrete platforms to industrial chimneys, reactor vessels, furnace linings, coal gasification vessels [1] requires heat resisting mortars to act as a thermal barrier [2-5] without causing any deterioration to the base structure. By choosing, refractory materials as an additive to conventional cement – mortar mix, the determined effects at elevated temperatures can be eliminated. There are several refractory additives are available in the market in the form of chemical and minerals. But each has got its limitation of thermal resistance when mixed with cement mortars [6-8]. Various material properties such as properties of aggregate, cement paste and aggregate cement paste bond, thermal compatibility between aggregate and cement paste affect the behaviour of high temperature mortar [9]. At service temperature regimes, the aggregate, cement – aggregate paste, additives will encounter physical and chemical actions. The complete evaporation of moisture from the mortar mix begins at 110˚C. Micro cracks develop in the area of [Ca (OH_2)] concentration due to evaporation of inter laminar water at 300˚C [9]. At 350˚C, the decomposition of [Ca (OH_2)] into lime and water vapour. The decomposition of [Ca (OH_2)] does not contribute for the strength loss but it may cause a serious damage due to the expansion of lime during cooling period [9]. At 573˚C, the siliceous aggregates containing quartz causes distress in concrete. Further, distress begins due to the decarbonation reaction in carbonate rocks at 700˚C. At 800˚C, all phase transformation from C-S-H and CH begins. At 900˚C, carbonates starts to shrink and the paste changes from grey to buff. Thereby, optimizing the mineral additives in cement mortar mixes for its demand in various industries necessitate manufacturing and processing at elevated temperature. The effects of high temperature upto 900˚C on the mechanical properties and their cracking potential are investigated in the study.

Experimental Part

Materials used

Ordinary Portland cement conforming to IS 12269 (1987) of 53 grade Ultratech brand was used. Screened and washed river sand passing through 600µ sieve was used for casting the specimen. Portable tap water was used for preparing the mixes. The refractory additives such as feldspar obtained from Tiruchengode, Fly ash obtained from Neyveli Lignite Corporation, Micro silica (ELKEM Micro silica), Steel slag obtained from steel foundry, Dindigul, Sodium Silicate of commercial grade purchased from Coimbatore, High Alumina Clay purchased from Madurai, Vermiculite of commercial grade purchased from Chennai, Quartz powder purchased from Madurai, MgO and Al_2O_3 were purchased from M/S Hi-Media, Bombay of laboratory grade grade were used. The oxide composition of each refractory material is shown in Table 1. Oxide compositions were arrived on the basis of X-ray Fluorescence Technique (XRF).

Table 1 Oxide Composition of Refractory Additives by XRF Analysis

S.No.	Oxides	Feldspar	Fly Ash	Micro Silica	Steel Slag	High Alumina Clay	Quartz Powder	MgO	Al_2O_3	Sodium Silicate	Vermiculite
1	SiO_2	49.46	43.54	74.59	9.29	62.03	95.26				11.92
2	SO_3	1.47	1.18	1.12	-	4.77	3.19				-
3	K_2O	46.66	3.46	-	0.68	-	-				-
4	Fe_2O_3	1.08	29.04	21.89	83.33	9.06	1.23				-
5	Rb_2O	1.316	-	-	-	-	-			Commercial	-
6	Al_2O_3	-	10.5	-	-	18.98	-	Laboratory		Grade	43.48

7	TiO_2	-	7.47	0.41	0.27	5.06	-		SiO_2-2.8%	-
8	MnO_2	-	0.25	0.23	0.03	-	-			-
9	ZrO_2	-	0.58	0.61	-	-	-			-
10	CaO	-	-	1.07	1.79	0.002	0.31			-
11	C_2O_3	-	-	0.08	-	-	-			-
12	CuO	-	-	-	2.18	-	-			-
13	ZnO	-	-	-	1.79	-	-			-
14	PbO	-	-	-	0.63	-	-			-
15	SnO_2	-	-	-	-	0.11	0.006			-
16	MgO	-	-	-	-	-	-			14.39
17	FeO	-	-	-	-	-	-			12.82

Mechanical Strength Test

Cylindrical specimens of size 75 mm diameter and 150 mm long as per ASTM C 39 were cast in triplicate. There are ten different composition of refractory cement mortar were prepared with one control mix of ratio 1:3 with W/C ratio of 0.45.The refractory cement mortar compositions are shown in Table 2.

Table 2 Heat Resistant Materials Compositions

S.No	Mix ID	Compositions
1	C	Control
2	1	CM + Feldspar
3	2	CM + Fly Ash
4	3	CM + Micro Silica
5	4	CM + Copper Slag
6	5	CM + High Alumina Clay
7	6	CM + Sodium Silicate
8	7	CM + Vermiculite
9	8	CM + Quartz Powder
10	9	CM + MgO
11	10	CM + Al_2O_3

*CM-Cement Mortar of 1:3 mix ratio with W/C=0.45

b) Relative Residual Strength Test

For heating at elevated temperatures from 110°C, 300°C to 800°C. The cylindrical specimens for all ten mix ratios were cast of same size as in previous test. The cylindrical specimens were subjected to water curing for 28 days. After curing the specimens were placed on the top loading furnace with PID temperature controller. The arrangement of specimen placed inside the furnace is shown in Fig.1.The specimens were subjected to heating at 110°C, 300°C, 400°C, 500°C, 600°C, 700°C and 800°C for one hour soaking period. After one hour soaking period the furnace is turned off for natural slow cooling. The specimens are taken out from the furnace after complete cooling of the specimens and were subjected to visual observations and relative residual compressive strength testing.Fig.2 to Fig.9 shows the specimen subjected to elevated temperature regimes. The Fig.10 shows the compression testing of specimens using 1000 kN capacity compression testing machine of AIMIL make.

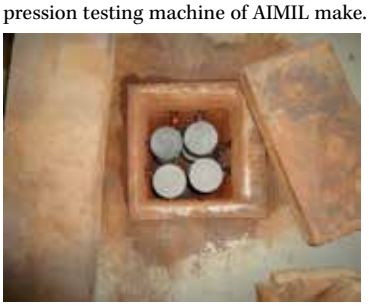

Fig.1 Arrangement of Specimens in High Temperature Furnace

Fig.2 Specimens before heating

Fig.3 Specimens heated to 110°C

Fig.4 Specimens heated to 300°C

Fig.5 Specimens heated to 400°C

Fig.6 Specimens heated to 500°C

Fig.7 Specimens heated to 600°C

Fig.8 Specimens heated to 700°C

Fig.9 Specimens heated to 800°C

Fig.10 Compression Testing of Specimens

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Measurement of Insulation Capacity

The insulating capacities of the material cast were investigated by the steady state temperature study using the top loading muffle furnace. The arrangement of the experimental set up is shown in Fig.11 .Cement mortar mix tile of size 230 x 230 x 20 mm for all refractory compositions were cast. Each tile was then loaded on the top loading furnace and heated to steady temperatures of 100°C, 150°C and 200°C for regular time interval. The temperatures are periodically recorded on the top and bottom of the tile for each steady temperature in regular time interval using the Infra-Red Thermometer as shown in Fig.12. The temperature drop at each elevated steady temperature gives the insulating capacity of the refractory mix at temperature drop. To compare the insulating capacity of refractory mixes a regular conventional tile procured from local shops were also subjected to this study.

Fig.11 Experimental Set up

Fig.12 Temperature measured using Infra Red Thermometer

Infra Red Thermometer

Measurement of Cracking Potential

The cement mortar with different compositions of refractory materials has its own rate of cracking potential. The cracking potential and the influence area due to flame exposure over the material were experimentally investigated using an accelerated flame torch designed and fabricated at CSIR-CECRI is shown in Fig.13. Flame torch held on the tiles of dimensions 100 x 100x 10 mm. The flame intensity exposed over the tile was maintained uniformly throughout the test period until the surface crack emerges out visibly. Also other visual monitoring such as pop outs, discolouration, charring, disintegration, delamination, etc. are examined on the tile surface. The time taken for any damage to occur is called as the "Endurance Limit". This limiting value will vary based on refractory material compositions. Higher the duration to cause any surface damage, the best is the material composition against flame.

Fig. 13 Flame Torch Set up

Results and Discussion

Relative residual compressive strength

Cylindrical compressive strength of specimens was tested for all the eleven specimens and it is given in Table 3. It is found that high alumina clay added specimen showed the highest compressive strength of 28.14 N/mm^2. This may be due to the alumina content (Al_2O_3) reacts with the silicate present in the mortar and the formation of alumina silicate along with calcium silicate provided the highest strength. Next to this specimens with copper slag and sodium silicate provided highest strength of 27.14 and 27.04 N/mm^2 was obtained. The lowest strength was obtained for vermiculite added mix which has 5.19 N/mm^2. When all the specimens were heated from room temperature to 100˚C, 300˚C and 400˚C with one hour soaking period in each temperature regime the strength of the specimens gradually reduced. This is because evaporation of water from the pores and voids of the mix. Thereby minute pores are formed in the places where water was filled. At temperature 105˚C, all evaporable water will be lost and above 105˚C the strongly absorbed and chemically combined water (i.e. water of hydration) are gradually lost from the cement paste hydrates. The dehydration of Portlandite [Ca (OH_2)] is zero upto 400˚C. This is quite in agreement with the findings of Harmathy[10]. When the specimens were heated to 500˚C, 1 hour the chemically combined water gradually lost. At this temperature decomposition of Portlandite takes place [11].i.e.,

$$Ca[OH_2] \rightarrow CaO + H_2O \qquad \text{----------(1)}$$

At this temperature, specimens 2, 3 and 5 has no crack and all other specimens cracked. This can be seen in Fig.6. Drastic reduction in strength is observed in all specimens as indicated in Table 3.

The specimen when heated to 600˚C for 1 hour, ° – Quartz charge to ° – Quartz with the transformation being endothermic and reversible. And because of this reason the micro cracks are formed at this temperature.

Specimens 1, 2, 3, 5, 6, 7 and 8 are found uncracked and rest remained cracked as shown in Fig.7. It is also observed from Table 3 that there is a reduction in compressive strength. Some specimens cracked at 500˚C with hair line cracks are now not showing any cracks at 600˚C. This is due to the fact that the reversible phase transformation of ° and ° quartz respectively. Also it can be attributed to the fact that the materials added for uncracked specimens such as feldspar, sodium silicate and vermiculite are derived from high temperature and therefore no sign of cracking was observed. When the specimens were heated to 700˚C, 1 hour, the specimens 5, 8, 9 and 10 are cracked and specimen 8 is seriously affected as shown in Fig.8 At this temperature decomposition of C-S-H phase transformation takes place and further reduction in compressive strength is observed as shown in Table 3. When the specimens were heated to 800˚C, 1 hour, control specimen, 1, 4, 8, 9 and 10 are cracked with multiple cracks as shown in Fig.9 There is a significant reduction is found in strength as shown in Table 3. This is due to the fact that the lime begins to undergo decarbonation and CO_2 gas is liberated. The rate of decomposition at this temperature is not dependent on temperature and pressure but the SiO_2 content present in the lime.

Table 3 Relative Residual Compressive Strength

S.No.	Mix ID	Cylinder Compressive Strength (MPa) after 28 days water curing							
		Before Heating	After 110°C	After 300°C	After 400°C	After 500°C	After 600°C	After 700°C	After 800°C
1	C	25.49	21.14	18.71	16.23	0.12	-	-	-
2	1	23.14	22.09	20.01	18.62	-	0.08	0.06	-
3	2	24.16	23.18	20.14	19.02	3.48	3.27	2.11	0.95
4	3	26.27	25.11	22.73	20.12	2.94	1.18	0.93	0.12
5	4	27.14	26.18	23.02	20.92	-	-	0.84	-
6	5	27.04	26.07	22.86	21.02	7.31	4.67	-	0.97
7	6	28.14	26.12	21.99	20.86	-	1.94	0.85	1.13

8	7	5.19	4.83	4.11	2.86	-	2.23	0.11	0.34
9	8	21.11	20.17	17.31	15.23	0.18	3.99	-	-
10	9	20.14	19.72	16.93	15.11	-	-	-	-
11	10	24.17	23.81	20.14	16.93	-	-	-	-

Measurement of Insulation Capacity

Insulation capacity of tile specimens was measured with Top Loading Muffle furnace as indicated in Fig.11 These temperature ranging from 100 to 200˚C were selected. The steady temperature ranges was created in the furnace with the help of temperature controller. The temperature of the tile specimens both at top and bottom was measured using IR Thermometer.

A dropping temperature of 3, 6 and 11 was observed for control tile at 100, 150 and 200˚C respectively as shown in Table 4. From this test specimen 3, 5, 8 and 9 performed very well with much higher dropping temperature. The insulation capacity of CM specimen with MgO has performed well at temperature 150 and 200˚C. The mixes containing vermiculite, micro silica and sodium silicate also performed very well. The control specimen and feldspar added mixes has poor performance. Compared to control specimen all other tile specimens performed very well with good range of temperature drop.

Table 4 Insulating Capacity of Tiles

S.No.	Mix ID	At 100˚C		At 150˚C		At 200˚C		Temperature Drop at 100˚C	Temperature Drop at 150˚C	Temperature Drop at 200˚C
		T	B	T	B	T	B			
1	C	69.1	72.1	98.3	104.3	132.6	143.6	3	6	11
2	1	58.2	61.1	79.2	89.5	99	110.1	2.9	10.3	11.1
3	2	67.9	71.6	82.3	89.5	101	113.6	3.7	7.2	12.6
4	3	62.6	65.2	80.4	85.7	97.1	113.7	2.6	5.3	16.6
5	4	70	72.8	90	97.1	104.7	118.1	2.8	7.1	13.4
6	5	73.3	77.6	80.7	90.6	93.4	110.	4.3	9.9	16.6
7	6	67.8	71.6	84.7	90.5	103.3	114.2	3.8	5.8	13.4
8	7	67.8	71.6	84.7	90.5	103.3	114.2	3.8	5.8	13.4
9	8	67.1	72.3	83.3	93.4	106	121.5	5.2	10.1	10.9
10	9	67.8	71.3	84.9	95.9	103.4	117.8	3.5	11	20.6
11	10	68.3	70.6	78.8	84.5	102.8	118	2.3	5.7	15.5

Measurement of Cracking Potential

The cracking potential of each specimen was measured with flame test. The potential of cracking was measured with respect to the formation of crack in front and back faces. Higher the time taken for cracking is the best material. Table 5 shows the observation in the flame test. The area of influence formed by the flame is also showed in Table 5. Lesser the area of influence shows the material's insulation capacity specimens. 10, 11 and 8 has lesser area of influence due to flame as shown in Fig.14. The time taken for the formation of cracks was higher for specimen 7, 6 and 2 for both front and back faces. This is due to the material composition of flyash, micro silica and magnesium oxide. The presence of SiO_2 component derived from higher temperature is the reason for such a higher potential time for cracking.

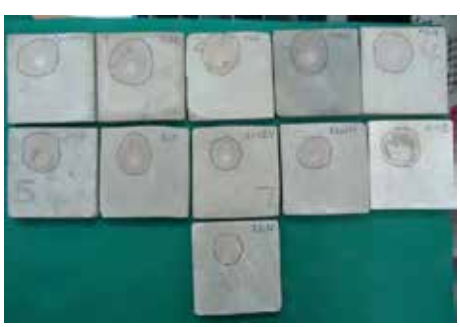

Fig. 14 Influence Area in Tiles after Flame Test

Table 5 Observations of Accelerated Flame Test

S.No.	Mix ID	Influence Area (cm^2)	Cracking Potential (min)		Target Surface Temperature (˚C)
			Front Face	Back Face	
1	C	22.89	4	7	289
2	1	22.05	2	11	217.6
3	2	18.85	4	13	215.4
4	3	19.63	3	10	219.6
5	4	17.34	3	8	234.5
6	5	18.09	2	4	154.6
7	6	18.85	4	10	292
8	7	11.34	5	8	285.3
9	8	12.56	3	6	122.5
10	9	15.20	2	5	299.8
11	10	8.55	2	4	301

Conclusions

- All the specimens upto 400˚C didn't show any sign of cracks. At 500˚C few specimens cracked due to dehydration of Portlandite and also the chemically bound water gradually lost at this temperature.
- Some specimens which cracked at 500˚C performed well at 600˚C without cracking because of significant increase in volume by crystalline transformation of α-quartz to β-quartz.
- Few specimens cracked at 700˚C due to decomposition of C-S-H phase transformation and due to decarbonation of lime.
- At temperature 800˚C, a strong degradation of microstructure results in loss of strength.
- In general, when mortars are heated the compressive strength decreases gradually upto 400˚C and then drastic reduction in strength is found.
- In steady state thermal insulation test mixes with MgO, vermiculite, sodium silicate and micro silica performed well and their insulation capacity is much higher than other mix composition.
- Similar results were obtained in flame test for mixes with same material composition.

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