THERMO-CHEMICAL STABILITY AND MECHANICAL PROPERTIES OF MORTAR MADE WITH CEMENT KILN CEMENT DUST- BLENDED IN KSA

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Abstract

The aim of the present investigation is to study the effect of elevated temperature on the mineralogical structure, chemical characteristics and mechanical properties of mortar prepared from cement kiln dust-blended (OPC and BFSC) with proportion 5, 10, 15, 20% of two types of cement. Cubical samples were cast, molded and cured in tap water for 28 days and then left in air under normal atmospheric conditions for about three years. These samples were subjected to elevated temperature: 300, 400, 500 and 600 0C for 2 hrs. then the compressive strength variation of the hardened samples were measured. Weight loss, differential thermal analysis (DTA), and X-Ray diffraction analysis were also used to investigate the phase transformations in these materials. Visual inspection of macro-cracks created due to heating and the carbonation depths were also identified. Results of this investigation showed that, mortar compressive strength is affected and gradual break down is observed, at temperatures up to 6000C, as well as with increasing of the blended cement kiln dust percent. The carbonation depth demonstrated by Ph. test for OPC-CKD is more than in the case of BFSC-CKD blends especially at high temperature. The hardened mortars made with BFSC gives a thermal stability more than that in case of OPC samples.

Key words: cement kiln dust, OPC and BFSC, compressive strength, DTA, XRD, phase transformations, carbonation.

الملخص العربي

يعتبراستخدام الغبار الناتج عن حرق المواد الخام كمادة اضافه الى المونة والخرسانة احدى الوسائل للتخلص من اضراره السلبيه على البيئة. ولهذا السبب يهدف البحث الي دراسة تاثير درجات الحرارة العالية علي الخواص المعدنية والكيميائية والميكانيكية للمونة باستخدام اسمنت مخلوط بغبار حرق الاسمنت، بنسب ٥، ١٠، ١٥، ٢٠٪ لنوعين من الاسمنت، العادي واسمنت خبث الافران بعد معالجته في الماء النقي لمدة ٢٨ يوم، وقد تركت العينات فى الأحوال المناخية لمده ٣سنوات. وقد استهدف البرنامج العملي للبحث دراسه تاثير تعريض العينات لدرجات حراره مرتفعه ٣٠٠، ٤٠٠، ٥٠٠، ٦٠٠ م٠ لمدة ساعتين وركزت الدراسه علي بيان مدى تاثير العوامل السابق ذكرها علي سلوك المونة تحت تاثير اجهاد الضغط وكذلك الوزن المفقود كما استخدم والتحليل الحراري التحليل بحيود الأشعة السينية لاستكشاف التحولات الكيميائيه واستخدم للفحص البصري لرصد الشروخ الناتجه عن التسخين كما تم تعين عمق الكربنه. وقد اوضحت النتائج ان مقاومة المونه بدات في الهبوط التدريجي حتي درجه ٦٠٠ م٠وكذلك مع زيادة نسبه غبار حرق الاسمنت. عمق الكربنه كان اكبر في حاله الأسمت العادي عنه في حاله استخدام اسمنت خبث الافران المخلوط خاصة عند درجات الحرارة المرتفعه المونة المتحزمة من اسمنت خبث الافران اظهرت ثبات حراري اعلي من مونة الاسمنت العادي.

INTRODUCTION

Due to continuous increase in industrial globalization and generation of waste, solid waste management has become one of the major global environmental issue. Cement kiln dust (CKD) is one of such industrial waste or by product which is progressively significant environmental concern related to its emission and disposal. CKD is fine grained, solid, highly alkaline particulate material chiefly composed of oxidized, anhydrous, micron-sized particles collected from electrostatic precipitators during the production of cement clinker. Cement kiln dust so generated is partly reused in cement plants and landfilled. Due to lack of landfilling space and ever increasing disposal cost, utilization of CKD in highway uses, waste treatment, soil stabilization, cement mortar/concrete, CLSM, etc. has become an attractive alternative to its disposal [1].

Several studies have shown that CKD could be used in making cement paste/mortar/concrete. These researches presented the beneficial use of CKD in construction materials, reducing carbon dioxide emissions and CKD leachate characteristics. Effect of CKD on the cement paste/mortar/concrete properties like compressive strength, tensile strength, durability, hydration, electrical conductivity, etc. and leachate test methods and leachate characteristics of cement kiln dust is discussed in these papers[2,3,4,5,6&7].

An attempts are made in utilizing the bacterial (Bacillus sp.) treated cement kiln dust as partial replacement of Portland cement (10, 20 and 30% w/w) and its effect on the normal consistency, setting times and hydration process of blended cement pastes, and on compressive strength (at 7, 28 and 90 days) of blended cement mortars. Test results show increase in water consistency with CKD concentration whereas setting time is decreased up to 10% CKD addition, above which setting time increases due to reduced hydration process. At later curing ages hydration process increases up to 10% bacterial treated CKD–cement paste which later on decreases as CKD content increases. This increase in hydration at later curing ages (91 days) responsible for increase in compressive strength in 10% bacterial treated CKD mortar compared with 0% and 10% untreated CKD mortar, respectively. Scanning electron microscopy (SEM) results exhibits increased calcium silicate hydrate and formation of non-expansive ettringite in pores which dense the mortar structure and increases the compressive strength in bacterial treated 10% CKD mortar [8].

Cement concrete specimens were prepared with 0%, 5%, 10%, and 15% CKD, replacing ASTM C 150 Type I and Type V cement. The mechanical properties of CKD concrete specimens were evaluated by measuring compressive strength and drying shrinkage while the durability characteristics were assessed by evaluating chloride permeability and electrical resistivity. The compressive strength of concrete specimens decreased with the quantity of CKD. However, there was no significant difference in the compressive strength of 0 and 5% CKD cement concretes. A similar trend was noted in the drying shrinkage strain. The chloride permeability increased and the electrical resistivity decreased due to the incorporation of CKD. The performance of concrete with 5% CKD was almost similar to that of concrete without CKD. Therefore, it is suggested to limit the amount of CKD in concrete to 5% since the chloride permeability and electrical resistivity data indicated that the chances of reinforcement corrosion would increase with 10% and 15% CKD [9, 10].

Also CKD was characterized by X-ray diffraction and FTIR analysis.

This byproduct was investigated for its physical–chemical characters, antibacterial activities on sewage water and the presence of nematode, parasites and algae in the treated water. The efficiency of CKD-treated water was also examined. Total bacteria, total and fecal coliform, as well as fecal streptococci were completely inhibited by CKD. Interestingly, zinc, manganese, iron, nickel and lead were completely absent from sewage water as these metals precipitated after treatment with 10 g l−1 CKD. This study highlighted the efficiency of cement kiln dust as an antibacterial agent and its ability of scavenging heavy metals leading to the use of treated sewage water in activities such as crop irrigation [11].

The effect of temperature on phase composition and microstructure of artificial pastes were studied later. Hydrated Portland cement contains a considerable proportion of free calcium hydroxide, which loses its water above 400-5000C, leaving calcium oxide (quick lime). If CaO becomes wetted after cooling as exposed to moist air, it rehydrates to Ca(OH)2 accompanied by an expansion in volume that may disrupt a concrete, which has withstood a fire without disintegration. When hardened mortars were subjected to high temperatures over the range of 20- 12000C, it was found that drying at 1050 0C caused a marked increase in strength of 40-55%. The strength then remained practically unchanged up to 400-5000C. Over the range above 500-5500C, it decreased dramatically by more than 50%, which is more than the Portland cement mortars. The loss in strength then proceeded similarly to that of Portland cement mortars. However, the residual strengths at temperatures within 800-10000C ranges were higher. Utilization of cement dust would eliminate castle pollution that are required to prevent degradation of air, land and water in the vicinity of the dust disposal sites.

Generally, direct recycling of CKD in rotary kiln causes damage in kiln refractories as well as in the formation of phase has different hydraulic reactivity. [12, 13].

In the present investigation cement kiln dust was used as cement blender for production of mortar made from OPC, BFSC and standard sand. The effect of cement kiln dust on the thermo-chemical stability of the prepared mortar was the main goal of the present study.

MATERIALS AND EXPERIMENTAL PROGRAM

Raw materials used in this investigation are ordinary Portland cement(OPC), blast furnace slag cement(BFSC) and raw «untreated» cement kiln dust(CKD) which collected from electrostatic precipitators, these materials were provided by Yanbu Cement Co Ltd - Ras B+aridi, Yanbu, and other campiness in KSA. The chemical analyses as well as X-Ray fluorescence analysis of cement and dust used in the present study are given in Table 1, which reflecting the oxide percent composition of it. Locally available standard sand was used also for preparation of mortar

Table 1 Chemical compositions of OPC, BFSC, and CKD.

Material	SiO_2 (%)	AL_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	NaO_3 (%)	K_2O (%)	Ignition loss (%)
OPC	21.47	3.21	5.22	62.7	2.32	2.36	2.40	0.41	1.23
BFSC	25.83	5.25	7.71	57.4	2.73	3.1	2.13	0.29	1.12
CKD	12.03	1.12	2.45	49.8	1.84	6.35	3.87	2.66	18.2

Two series of mortar mix compositions were prepared using 0.45 W/C ratio and cement blended content of 400 kg/m3. The sand to cement or cement-blended ratios of 3:1 was maintained throughout. The mix compositions of the prepared samples are given in Table 2.

Table 2 Mortar mix of prepared specimens with CKD (wt. %).

OPC	0%	5%	10%	15%	20%
BFSC	M1	M1	M3	M4	M5
CKD	S1	S2	S3	S5	S6

A small rotating drum mixer was used to mix the constituents of mortars. Cement, dust and sand were mixed in dry state and then the predetermined quantity of water was gradually added. The mixing continued until a homogeneous mixture was obtained. 75x 75 x 75 mm cubes were prepared according to ASTM(C 109-80), for all mortar mixes. Samples were demoulded after 24 hrs. and then cured in fresh water at ambient temperature for 28 days. After curing the specimens were kept in the normal atmospheric conditions for about 3 years to attain the ultimate sample strengths and complete phase composition formation due to hydration reactions.

For each mortar mix, three cubes were exposed to heating at temperatures of 300, 400, 500 or 600 0C for 2 hrs. soaking time in semi-open muffle furnace with heating rate at temperature interval of 5-100C per minute. After heating, the specimens were left to slowly cooling with the furnace switched off, till the samples reach the room temperature. The weight loss due to heating was determined. The compressive strength of the three tested mortar samples was measured before and after their exposure to heat. The compressive test was carried out by a hydraulic testing machine of 1000 KN capacity. Phenolphthalein (Ph. Ph.) testing as well as chemical analysis of carbonation depth created due to cracks raised from firing was carried out [14]. The X-Ray diffraction analysis was used to investigate the chemical and mineralogical changes of samples due to firing.

RESULTS AND DISCUSSION

The chemical and mineralogical analysis of the used cement kiln dust with using X-Ray diffractometer are given in Fig. 1 which show that the dust containing calcium carbonate $CaCO3$ as the major constituent with different percentages of calcium sulphate $CaSO4$ $NaCl$, spurite [2(C2S). CaCO3] and sulphospurite [2(C2S). CaSO4] [15]. The influence of CKD substitution on the mechanical properties of OPC, BFSC and SRC concretes, and also evaluation of the relative strengths depending on each of cement type. Inspection of this figure shows that the direct replacement (mixing) of CKD with SRC or BFSC is more effective than OPC, and CKD enhances the hydration reaction of BFSC [16].

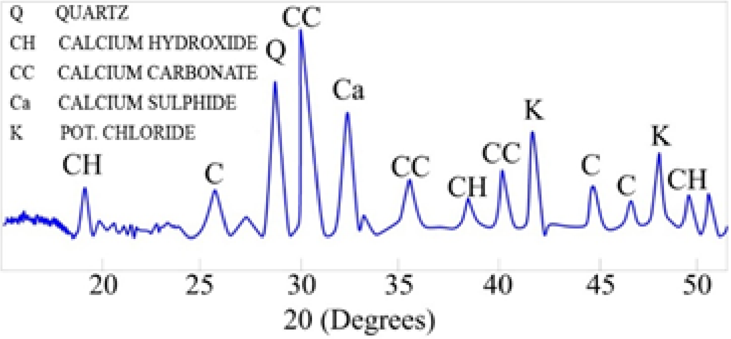


Fig. 1: X-Ray Diffraction Analysis (XRD) of cement-Kiln Dust.

Differential thermal analysis (DTA) of cement kiln dust represented in Fig. 2, shows two endothermic peaks. The first endothermic peak at temperature about 4500C, which is, corresponds to the decomposition of calcium hydroxide, while the second peak at about 7800C, which is attributed to the calcinations of calcium carbonate into calcium oxide. A slightly observable endothermic peak at about 8800C related to the presence of alkali chloride as K, Na, chlorides.

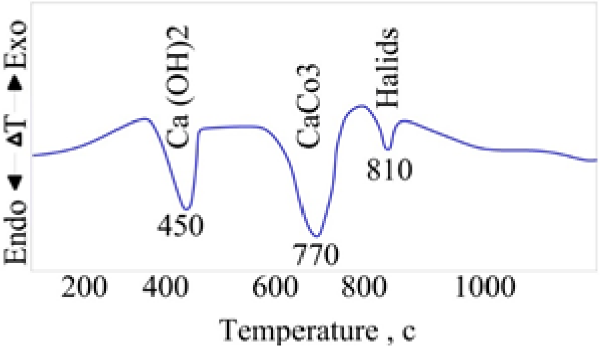


Fig. 2: Differential thermal analysis (DTA) of Cement-Kiln Dust.

When building products are heated, their strength will be affected and undergo gradual break down at temperatures lower than 6000C. Up to 3000C the loss in strength is small, but at 5000C it can be 50 percent or more of its original values [17].

The variation of the compressive strengths of the two different mortar specimens is graphically represented Fig. 3&4. It is indicated that, both mortar types (OPC or BFSC), showed a gradual decrease in the compressive strength with increasing of the blended cement kiln dust percent. This is mainly attributed to substitution of high hydraulically properties materials by CKD which characterized by very lower or no hydraulic properties. Therefore the decreasing in the ultimate compressive strength is due to replacement of the cement phases, which is mainly responsible for strength development. In addition, the relatively larger amount of alkali chlorides and alkali sulphate in dust, which take part of chemical reactions, yielding of chloro-aluminate(3CaO-AL2O3-CaCL2.12H2O) and sulphoaluminate hydrates(3CaO-AL2O3- CaSO4.12H2O) [18].

The measured data corresponds to samples made with BFSC show relatively higher values than OPC samples, this mainly attributed to the influence of alkali CKD in the activation slag. The decrease in the compressive strength of hardened sample with increasing the CKD substitution may be due to lower amount of CSH, which is responsible for the cementing properties in the hardened samples [19].

The effect of elevated temperatures on the compressive strength of OPC-CKD are graphically represented in Fig. 3a, b. All samples showed a gradual decrease in the compressive strength with increase in temperature of heating up to 500oC, while significant decrease was observed for all samples when temperature reached to 600oC. This is primarily due to the thermal decomposition of the hydrated products, as well as some phase transformation as in case of transformation of low α-quartz to high β-quartz at 573°C, which accompanied by a sudden expansion and change in volume. The hardened mortar specimens made with BFSC as shown in Fig. 4a, b, exhibited relatively high strengths indicating the effect of CKD in the activation of slag and also the thermally treated samples shows high stability more than that in case of OPC samples this mainly due to the hydraulic properties of slag which need or consumed some of the liberated Ca(OH)2 due to the hydration of cement phases and no liberated lime raised from the hydration of slag. Therefore the loss of weight is associated with liberation of water from the decomposition of Ca(OH)2, and consequently creating surface and demonstrate micro-cracks. The weight losses accompanying to the high temperature effect are illustrated in Fig. 5, 6.

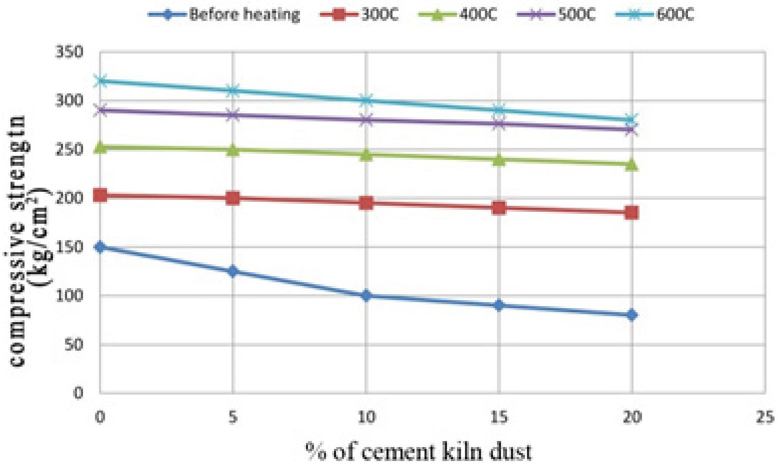
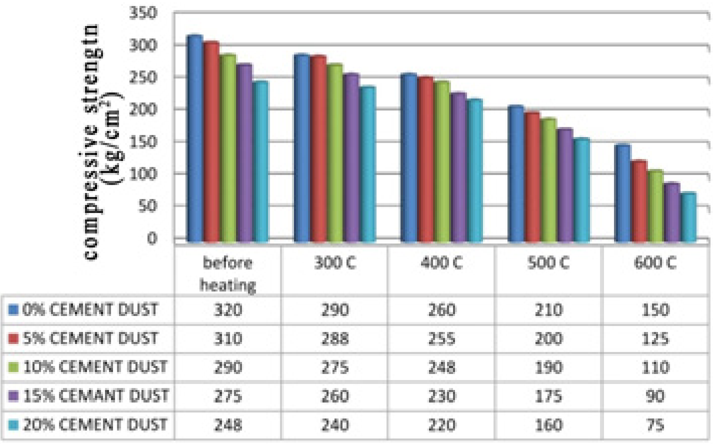


Fig. 3-a: Compressive Strength vs. % of Cement Kiln Dust Ratios for Different Heating Temperatures for OPC.



	before heating	300 C	400 C	500 C	600 C
■ 0% CEMENT DUST	320	290	260	210	150
■ 5% CEMENT DUST	310	288	255	200	125
■ 10% CEMENT DUST	290	275	248	190	110
■ 15% CEMANT DUST	275	260	230	175	90
■ 20% CEMENT DUST	248	240	220	160	75

Fig. 3-b: Compressive Strength vs. Different Heating Temperatures for different Cement Kiln Dust Ratios for OPC.

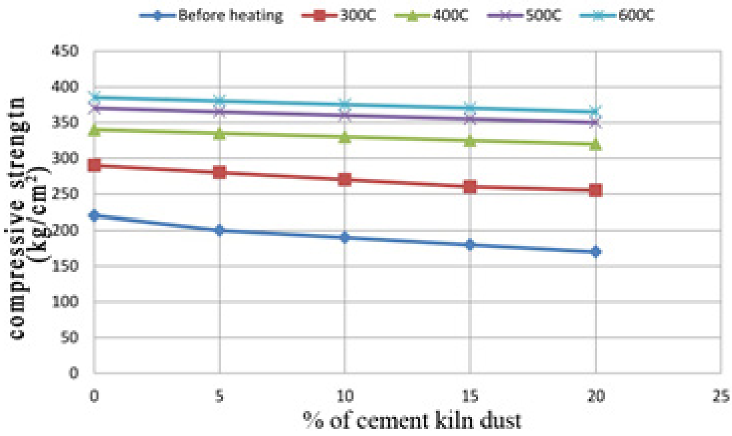
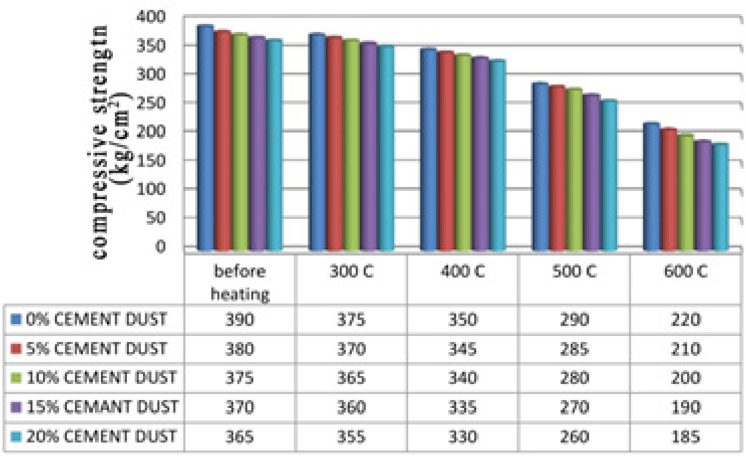


Fig.4-a: Compressive Strength vs. % of Cement Kiln Dust Ratios for Different Heating Temperatures for BFSC.



	before heating	300 C	400 C	500 C	600 C
0% CEMENT DUST	390	375	350	290	220
5% CEMENT DUST	380	370	345	285	210
10% CEMENT DUST	375	365	340	280	200
15% CEMANT DUST	370	360	335	270	190
20% CEMENT DUST	365	355	330	260	185

Fig. 4-b: Compressive strength vs. Different Heating Temperatures for different Cement Kiln Dust Ratios for BFSC.

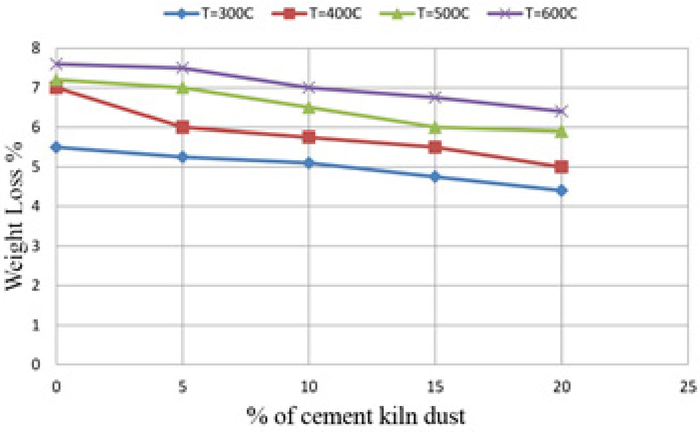
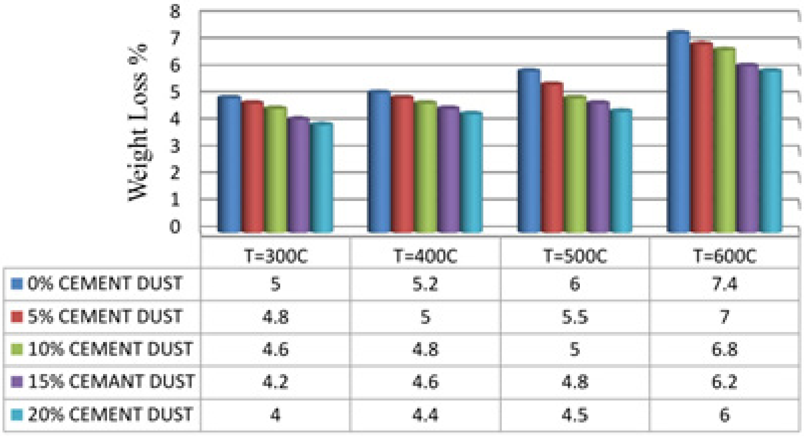


Fig. 5-a: Weight Loss Percent vs. % of Cement Kiln Dust Ratios for Different Heating Temperatures for OPC.



	T=300C	T=400C	T=500C	T=600C
🟦 0% CEMENT DUST	5	5.2	6	7.4
🟥 5% CEMENT DUST	4.8	5	5.5	7
🟩 10% CEMENT DUST	4.6	4.8	5	6.8
🟪 15% CEMANT DUST	4.2	4.6	4.8	6.2
🟦 20% CEMENT DUST	4	4.4	4.5	6

Fig. 5-b: Weight Loss vs. Different Heating Temperatures for different Cement Kiln Dust Ratios for OPC.

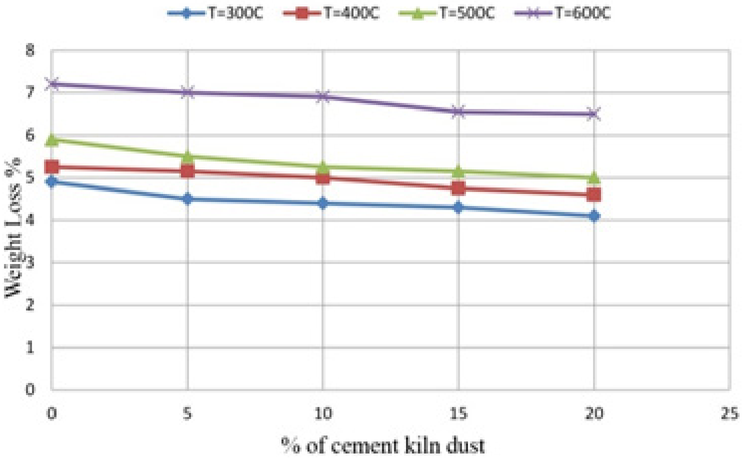
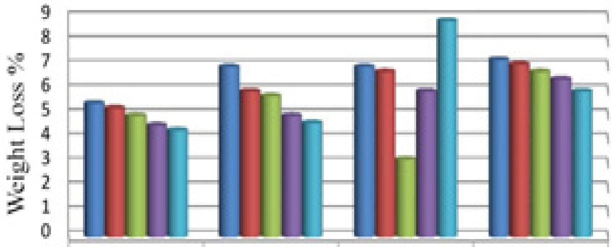


Fig. 6-a: Weight Loss Percent vs. % of Cement Kiln Dust Ratios for Different Heating Temperatures for BFSC.



	T=300C	T=400C	T=500C	T=600C
■ 0% CEMENT DUST	5.5	7	7	7.3
■ 5% CEMENT DUST	5.3	6	6.8	7.1
■ 10% CEMENT DUST	5	5.8	3.2	6.8
■ 15% CEMANT DUST	4.6	5	6	6.5
■ 20% CEMENT DUST	4.4	4.7	8.9	6

Fig. 6-b: weight Loss vs. Different Heating Temperatures for different Cement Kiln Dust Ratios for BFSC.

Data listed and represented in these figures indicating that the weight loss due heating increases gradually with increasing the heating temperature for all samples this is a result for dehydration of free lime as well as calcium silicate hydrate that resulted from the original hydration process. It was observed that the calculated weight loss in case of OPC-blends is greater than that calculated BFSC- blends, this is mainly attributed to the amount of lime contents.

The visual inspection shown in Fig 7-9, give a compatible conformation with that found from compressive strength results. At which the surface cracking increases with increasing the heating temperature and also with increasing of the dust value. The general surface cracking for the OPC mixes treated mortar specimens at 500 0C and 600 oC are shown in Fig.7a,b. Fig. 8 shows the effect of temperature on the surface cracking of these mixes heating at 300, 400, 500, and 600 0C. In general the number and widths of created cracks increased with increasing of the heating temperature. This is mainly attributed to the dehydration of the mortar followed by thermally decomposition of the Ca(OH)2 accompanying by loss of water at about 4500C. At temperature more than this nearly at about 5700C some volume transformation from α-quartz to high β -quartz which accompanied by a sudden expansion and change in specimens volume resulting more observable surface cracks [20].

It was found that the observable cracks in samples prepared with OPC are greater in numbers and depth than those in case of BFSC mortar. This may be due to the role slag cement in consumption of the liberated lime released from clinker hydration in BFS-cement. So, the remaining Ca(OH)2 is less in case of BFS cement, than the losses due to decomposition of Ca(OH)2 and also the volume changes due to carbonation of CaO resulted are smaller than that in case of OPC mortars.

Fig.9 shows the surface cracking width of the hardened samples after leaving it in the atmospheric conditions for one week for carbonation reaction. Fig.10 gives the comparative surface cracking in OPC and BFSC heated at 6000C for 2 hours without subjecting to long time carbonation. It must be mentioned that under the some heating and environmental condition the cracks increases gradually with leaving time after heating in the atmospheric condition. OPC-mortars show more width and depth surface cracking than BFS-cement, this due to the effect of the value of lime in it. The high observable value of surface cracks increases with increasing the amount of dust; this is mainly attributed to the role of calcium carbonate percent's containing dust as nucleating agent for lime collections or as activator for formation and precipitation the crystalline Ca (OH) 2 form. With an increase in hydration age, there is a trend towards the formation of larger amounts of mono-carbonate with increasing level of dust addition. For cement with high C3A contents the amounts of mono-carbonate increased at all hydration ages compared with cements with lower C3A content with increasing level of dust addition.

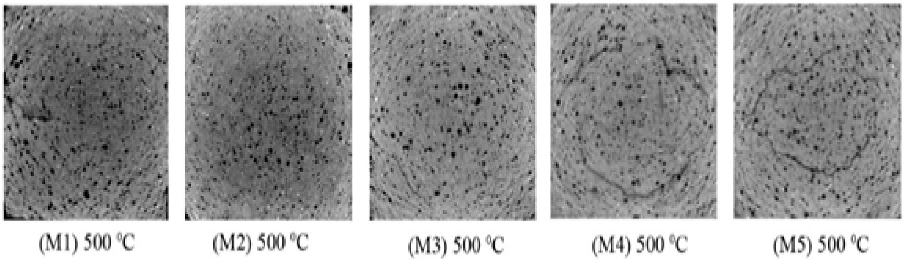
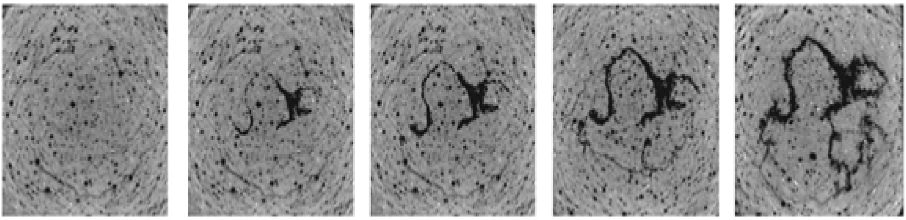
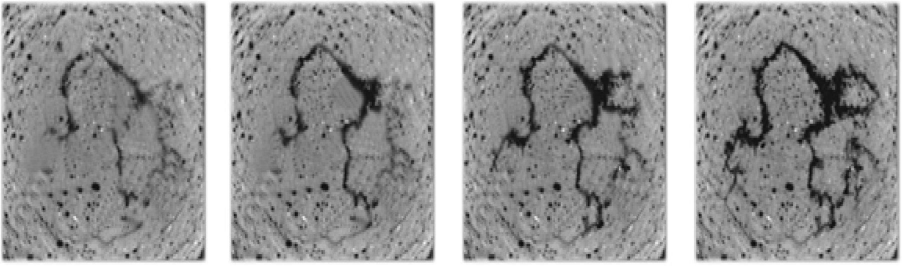


Fig. 7, a: Photographic surface cracking pattern for OPC mixes treated at 5000C



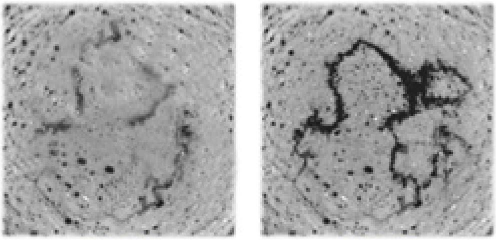
(M1) 600 °C (M2) 600 °C (M3) 600 °C (M4) 600 °C (M5) 600 °C

Fig. 7, b: Photographic surface cracking pattern for OPC mixes treated at 6000C



(M5) 300 °C (M5) 400 °C (M5) 500 °C (M5) 600 °C

Fig. 8: Photographic surface cracking pattern for OPC mixes treated at different temperatures



BFSC OPC

Fig. 9: Photographic surface cracking pattern for OPC and BFSC mixes treated at 6000C

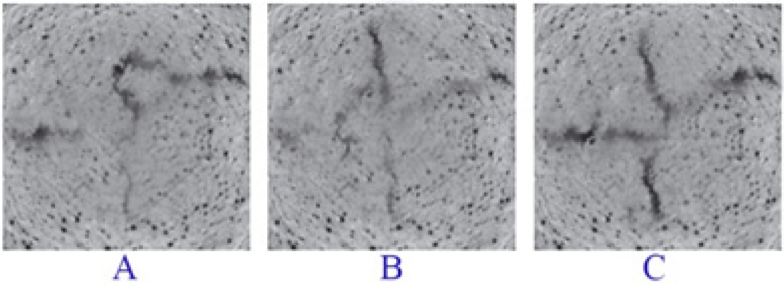


Fig. 10: Photographic surface cracking pattern for OPC mixes treated at 6000C for various percentage of dust (A) with 10%, (B) with 15% and (C) with 20 % CKD.

Phenolphthalein spraying test, for examination of the carbonation depth occupying cracks formation are shown in photograph, Fig. 11a, b, at which the carbonation depth increases towards the bulk of the specimens with increasing the heating temperature as well as with CKD percent's. The zone in the mortar structure where such phenomena have occurred coincide approximately with the zones which remain using un-colored in coloration reaction phenolphthalein, and moreover, the zones when compared with sound zones present a coloring as if bleached. No coloration was observed in this portion at all when the phenolphthalein solution was sprayed. In other words, the discolored zone coincides with the portion where Ca(OH)2 turns to CaCO3 through carbonation. It is confirmed that in this discolored portion, C-S-H in hydrated cement has been decomposed into CaCO3, SiO2 and H2O [21

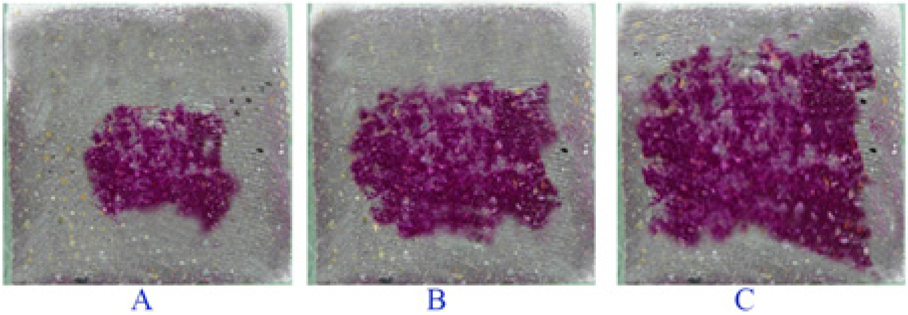


Fig. 11, a: Carbonation depth of thermally treated samples OPC and BFSC mixes (A-OPC, with20%CKD at 6000C), (B-OPCwith20%CKD at 5000C) and (C- BFSCwith20%CKD at 6000C)

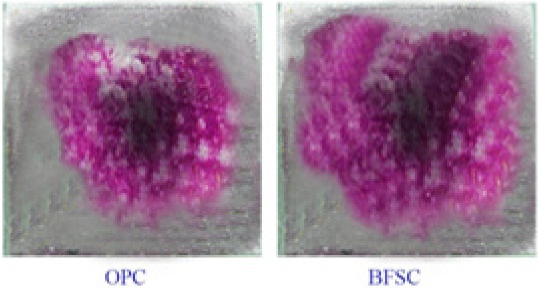


Fig. 11, b: Carbonation depth of thermally treated samples for treated at 6000C.

Fig. 12, shows the X-ray diffraction analysis of some thermally treated samples at various temperatures. XRD patterns of all thermally treated samples with OPC-CKD blended cement exhibits a characteristic peaks for quartz, calcium silicate (CSH), calcium hydroxide [Ca(OH)2], and calcium carbonate (CaCO3). It was observed that the peak characteristic for calcium silicate hydrate decreased gradually with increasing the treated temperature up to 600 0C, this phase is responsible for the mortar strength, so this property decreased with increasing the treated temperature; this is in agreement with measured strength before. It is observed also that the Ca(OH)٢ peak slightly increased at ٣٠٠٠C, this is due to some recrystallization of this portlandite phase, but its decrease up to ٠ ٥٠٠٠C or more is mainly due to thermal decomposition of Ca(OH)٢ into CaO and water accompanying by high weight losses. XRD of the thermally treated mortar specimens with BFSC-CKD showed that the Ca(OH) peak in these samples are relatively very low, this is attributed to low amount of lime released from the hydration of BFSC and also it needs some of lime for activation at the first hydration time. This explains the low weight losses as well as observable cracks and so low carbonation depth. It can say that BFSC mortar exhibits high thermal stability than those made with OPC.

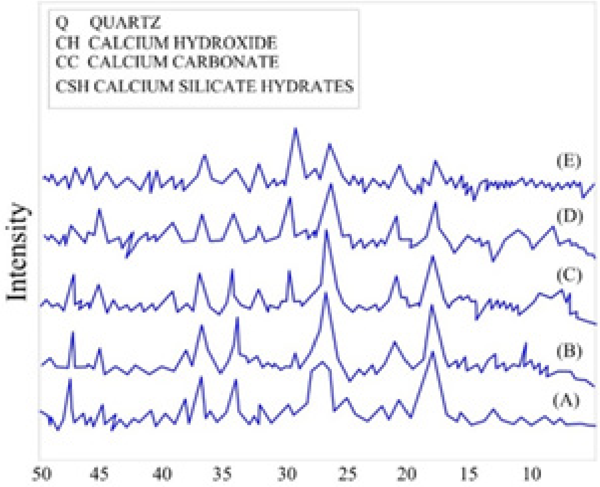


Fig. 12: X-ray diffraction analysis of thermally treated OPC-CKD samples at various temperatures A) before heating, B) at 3000C, C) at 400 0C, D) at 500 0C and E) at 600 0C

Conclusions

Based on the test results, the following conclusions could be drawn:

1- It is important to reuse cement kiln ecologically as well as economically to overcome the large quantities produced annually in KSA.

2- CKD has adversely affected the physical and mechanical properties of cement mortars and concretes especially with OPC samples but gives some enhancement for the hydration reaction with BFSC.

3-All mortar samples strength were affected and undergo gradual break down at temperatures up to 6000C.

4- Mortar types (OPC or BFSC), showed a gradual decrease in the compressive strength with increasing of the blended cement kiln dust percent.

5- The number of the observed cracks in the treated samples prepared with OPC was more than those observed in case of BFSC mortars.

6- The carbonation depth demonstrated by Ph.Ph. test in OPC-CKD is more than in case of BFSC-CKD blends especially at high temperature.

7- The hardened mortars made with BFSC indicating a thermal stability more than that in case of OPC samples.

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