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

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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 cement with proportion 5, 10, 15, 20% of two types of cement (OPC and BFSC). Cubical samples were cast, molded and cured under 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 ^{0}C for 2 hrs, then the compressive strength variation of the hardened samples were studied. Weight loss and X-Ray diffraction analysis were also used to investigate the phase transformations in these materials. Visual observation of macro-cracks created due to heating and the carbonation depths were also identified. Results of this investigation showed that, mortar strength is affected and gradual break down is observed, at temperatures up to 600°C, as well as with increasing of the blended cement kiln dust percent. The carbonation depth demonstrated by Ph.Ph. test for OPC-CKD is more than in 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.

يهدف البحث إلى دراسة تأثير درجات الحرارة المرتفعة على الخواص المعدنية والكيميائية والميكانيكية للمونة باستخدام أسمنت مخلوط بغبار حرق الأسمنت، بنسب ٥، ١٠، ١٥، ٢٠% لنوعين من الأسمنت، العادي وأسمنت خبث الأفران بعد معالجة في الماء النقي لمدة ٢٨ يوم، تركت العينات للأحوال المناخية لمدة ٣سنوات. وقد أستهدف البرنامج العملي للبحث دراسة تأثير تعريض العينات لدرجات حرارة مرتفعة ٣٠٠، ٤٠٠، ٥٠٠، و٦٠٠ م لمدة ساعتين وركزت الدراسة علي بيان مدي تأثير العوامل السابق ذكرها علي سلوك الخرسانة تحت تأثير إجهاد الضغط وكذلك الوزن المفقود كما استخدم التحليل بأشعة إكس لاستكشاف التحولات الكيميائية و استخدم الفحص البصري لرصد الشروخ الناتجة عن التسخين كما تم تعين عمق الكربنة. وقد أوضحت النتائج أن مقاومة المونة بدأت في الهبوط التدريجي حتى درجة ٦٠٠ م وكذلك مع زيادة نسبة غبار حرق الأسمنت المخلوط. عمق الكربنة كان أكبر في حالة الأسمنت العادي المخلوط عنه في حالة أسمنت خبث الأفران المخلوط خاصة عند درجات الحرارة المرتفعة. المونة من أسمنت خبث الأفران أظهرت ثبات حراري أعلى من مونة الأسمنت العادي.

Keywords: Cement Kiln Dust, OPC, BFSC, Thermal Stability, Surface Cracking, Phase Transformation.

INTRODUCTION

Cement manufacture discard annually millions tons of dust which collected from exhaust gases of cement kilns, nodulizers and cooling towers. In fact approximately 12% of the kiln feed exists from the kiln with the gas specially with using dry process of cement production as it results in dust three times more than the wet process [1]. The collected dust contains excessive contractions of alkalies that make it unsuitable for return to the cement making process [2]. The effect of substitutions of cement kiln dust (CKD) for preparation of some blended cement pastes were investigated [3], results of addition of cement dust to either Ordinary Portland Cement (OPC) or to blast furnace slag cement (BFSC) indicated that the addition of CKD to some blends of OPC, SRC or BFSC have a limit effect but with increasing dust value, it has adversely affected the physical and mechanical properties of cement paste, mortar and concrete specially with OPC samples.

Cement kiln dust (CKD) represents a mixture of raw material mixed with portions of clinker. The chemical composition of the exit dust in among other factors also influenced by size of particles carried away by the kiln gases. Kiln dust can be used in a number of different ways as land fill, soil stabilizer, sub-base roads, filers for bituminous paving materials, asphalted roofing materials, neutralization of acidic waste of bogs, neutralization of contain industrial wastes and so on [4].

The most popular slag cements are Portland blast furnace slag cement, which contains not more than 65% of granulated slag, and OPC clinker with 5-6 % gypsum. Portland-BFSC has all the physical properties of OPC, but it has much lower heat of

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hydration and is more sulphate resistance. Compressive strength tests of slag mortar indicated that alkali-activated ground granulated blast furnace slag has potential as a replacement for Portland cement in concrete [5,6]. Wang et al. [7], studied the effect of cement-kiln dust (CKD) slag blended cement for durable concrete. Development and performance of cement kiln dust- slag cement was investigated [8]. The hydration, rheology and strength of OPC-CKD-slag binders have been studied by wang et al [9].

Hydraulic reactivity of blast furnace slag in the presence of Portland cement and $Ca(OH)_2$ was studied by determination of the degree of hydration of slag and compressive strength of the hardened specimens [10,11]. The hydraulic reactivity was higher for the slag cement mixture than for the slag lime mixture indicating that the coexistence of such materials that liberate Ca^{2+} during its hydration reaction was necessary and effective to improve the hydraulic reactivity of slag. The effect of pozzolans and slag on the expansion of mortar cured at elevated temperature was studied [12]. The expansive behavior of heat-cured mortars containing pozzolans and slag was investigated, at which in most cases, the addition of any amount of these materials to the mixture typically reduced the long-term expansion. The effect of alkaline carbonate is more marked for pozzolanic cement than Portland ones [13]. The accelerating effect of carbonates is considerable not only at 20°C but also at lower temperatures. Nevertheless they reduce strengths even after 7 days. The effect of potassium salts, including chloride and sulphate on the hydration of alite were studied [14]. It is concluded that the accelerating mechanism of inorganic electrolytes depends on the counter diffusion of Cl^- and OH^- ions, which take place in the CSH surface layer during hydration. The greater the mobility of the anions, the larger accelerating effect.

In mortar and concrete, the aggregate undergoes progressive expansion on heating while the set cement shrinks beyond the point of maximum expansion. The two opposing actions progressively weaken and crack the mortar or concrete. The various types of aggregates used in concrete differ considerably in their behavior on heating. Quartz, the principal mineral constituent of the acid igneous rocks, expands steadily up to 573°C [15]. The major factors responsible for concrete deterioration are permeability, carbonation, chemical attack, alkali aggregate reaction and physical aggression like thermal stock and abrasion. The general deterioration of concrete structures is usually accompanied by cracking and spelling. The effect of temperature on phase composition and microstructure of artificial pozzolanic-cement pastes were studied later [16]. Hydrated Portland cement contains a considerable proportion of free calcium hydroxide, which loses its water above 400-500°C, 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 [17]. When hardened mortars were subjected to high temperatures over the range of 20-1200°C, it was found that drying at 105°C caused a marked increase in strength of 40-55%.

The strength then remained practically unchanged up to 400-500°C. Over the range above 500-550°C, 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 800-1000°C ranges were higher [18]

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. Thus, previous study by the authors [19], was directed to avoid the problems due to direct dust recycling and studying the role of cement kiln dust on activation of slag cements in a comparison to OPC and also predicting the optimum dust percent may be used. 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 also the main goal.

Table (1): The Chemical Compositions of OPC, BFSC and CKD, wt%.

Components / Materials	SiO_2 (%)	AL_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Na_2O (%)	K_2O (%)	Ignition loss (%)
OPC	21.42	3.35	5.23	62.7	2.4	2.35	2.41	0.45	1.22
BFSC	25.8	5.25	7.71	57.4	2.73	3.0	2.13	0.29	1.08
CKD	11.95	1.12	2.45	49.75	1.86	6.35	3.87	2.66	17.92

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 precipitatorsThese materials were provided by Suez Cement Company, Suez, Egypt. 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.

Two series of mortar mix compositions were prepared using 0.5 water/cement ratio and cement blended content of 400 kg/m^3. The sand to cement or cement-blended ratios of 3 was maintained throughout. The mix compositions of the prepared samples are given in table (2).

A small rotating drum mixer was used to mix the contents 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. 75× 75× 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 w ere 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°C for 2 hrs soaking time in semi-open muffle furnace with heating rate at temperature interval of 5-10°C per minute. After heating, the specimens were left to slowly cooling with the furnace switched off, till the sample 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 in Zigzag university [20]. Phenolphthalein (Ph.Ph.) testing as well as chemical analysis of carbonation depth created due to cracks raised from firing was carried out [21]. 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 $CaCO_3$ as the major constituent with different percentages of calcium sulphate $CaSO_4$, NaCl, spurite [2(C_2S). $CaCO_3$], and sulphospurite [2(C_2S). $CaSO_4$][22]. 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 [19].

Differential thermal analysis (DTA) of cement kiln dust represented in Fig. (2), shows two endothermic peaks. The first endothermic peak at temperature about 450°C, which is, corresponds to the decomposition of calcium hydroxide, while the second peak at about 780°C, which is attributed to the calcinations of calcium carbonate into calcium oxide. A slightly observable endothermic peak at about 880°C related to the presence of alkali chloride as K, Na, chlorides.

When building products are heated, their strength will be affected and undergo gradual break down at temperatures lower than 600°C. Up to 300°C the loss in strength is small, but at 500°C it can be 50 percent or more [23].

The variation of the compressive strengths of the two different mortar specimens is graphically represented in figures (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 [24]. 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 hydrates ($3CaO\text{-}Al_2O_3$ -$CaCl_2$. $12H_2O$) and sulphoaluminate hydrates ($3CaO\text{-}Al_2O_3$- $CaSO_4.12H_2O$) [25].

Table (2): The mix composition of the prepared specimens with CKD, constituent wt%.

CKD	0%	5%	10%	15%	20%
OPC	M_1	M_2	M_3	M_4	M_5
BFSC	S_1	S_2	S_3	S_4	S_5

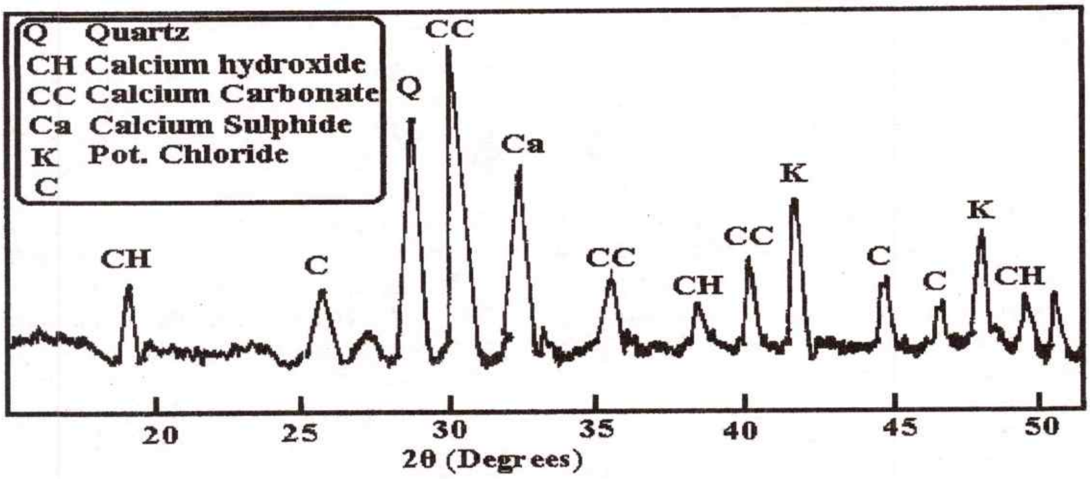


Fig (1): X-Ray Diffraction Analysis (XRD) of Cement-Kiln Dust as received from the cement plant.

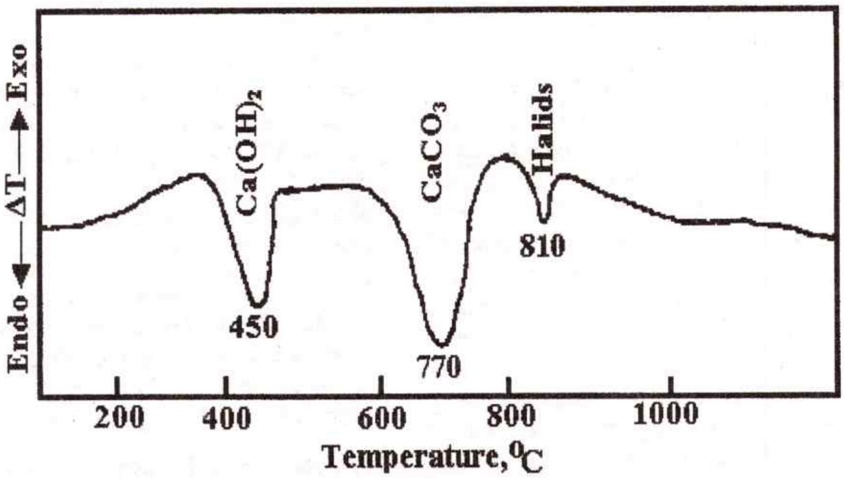


Fig. (2) Differential thermal analysis (DTA) of Cement-Kiln Dust as received from the cement plant.

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 [26]. The decrease in the compressive strength of hardened sample with increasing the CKD substitution is, may be, due to lower amount of CSH, which is responsible for the cementing properties in the hardened samples.

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 compressive strength with the increase in temperature of heating up to 500°C, while significant decrease was observed for all samples when temperature reached to 600°C. 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 accompayining to the high temperature effect are illustrated in Fig. (5,6).

Data listed and represented in these figures indicating that the weight loss due to 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. 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.

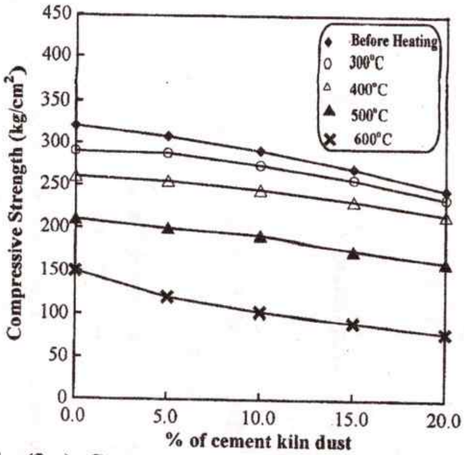


Fig. (3-a): Compressive Strength vs % of Cement Kiln Dust Ratios for Different Heating Temperatures for OPC.

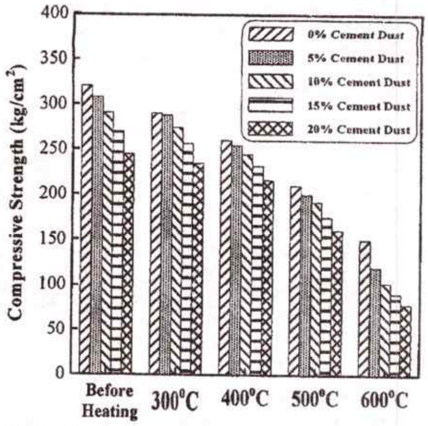


Fig. (3-b): Compressive Strength vs Different Heating Temperatures for different % of Cement Kiln Dust Ratios for OPC.

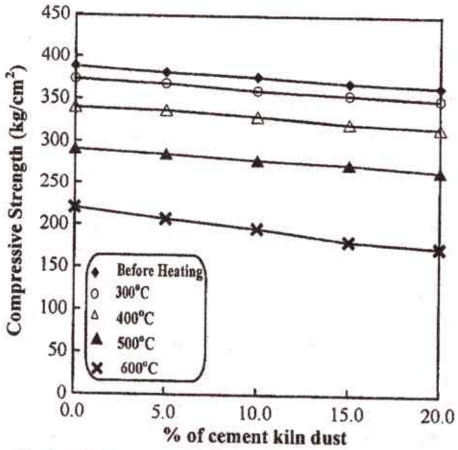


Fig. (4-a): Compressive Strength vs % of Cement Kiln Dust Ratios for Different Heating Temperatures for BFSC.

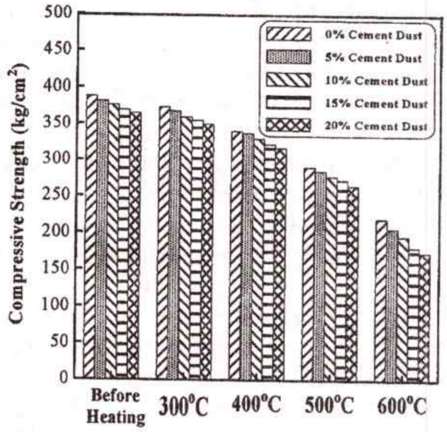


Fig. (4-b): Compressive Strength vs Different Heating Temperatures for different % of Cement Kiln Dust Ratios for BFSC.

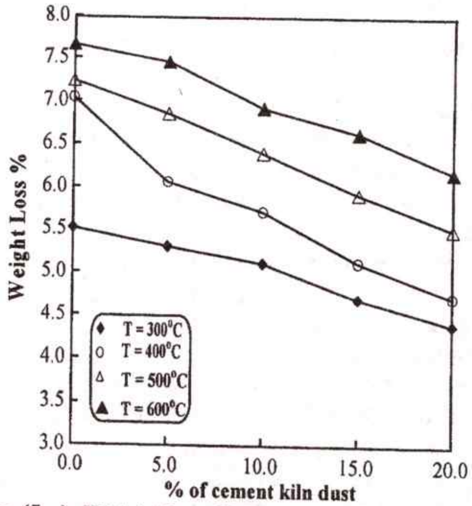


Fig. (5-a): Weight Loss Percent vs % of Cement Kiln Dust Ratios for Different Heating Temperatures for OPC.

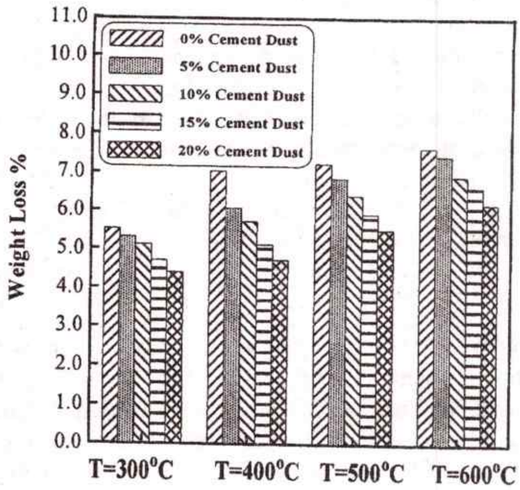


Fig. (5-b): Weight Loss % vs Different Heating Temperatures for different % of Cement Kiln Dust Ratios for OPC.

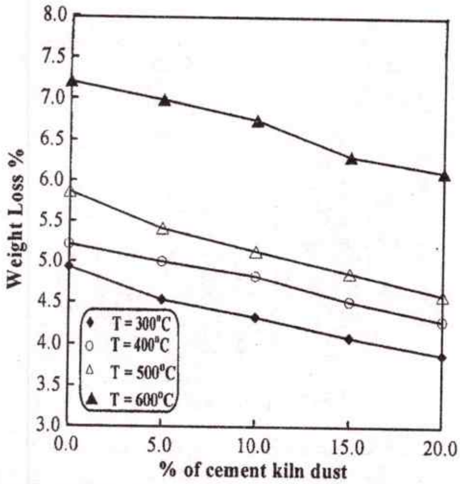


Fig. (6-a): Weight Loss Percent vs % of Cement Kiln Dust Ratios for Different Heating Temperatures for BFSC.

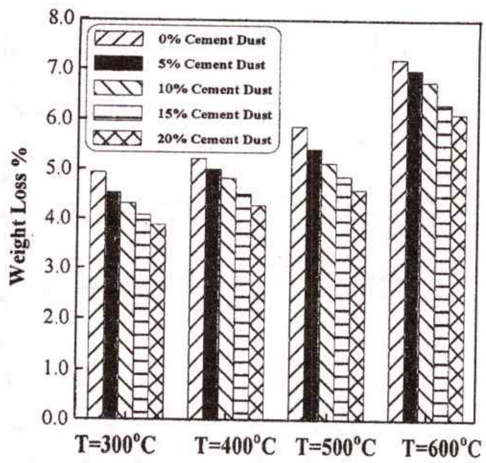


Fig. (6-b): Weight Loss % vs Different Heating Temperatures for different % of Cement Kiln Dust Ratios for BFSC.

The visual observations shown in Figs. (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 °C and 600 °C are shown in Fig. (7a,b). Fig. (8) shows the effect of heating temperature on the surface cracking these mixes at 300, 400, 500, and 600 °C. 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 450°C [17]. At temperature more than this nearly at about 570°C 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 [15]. 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 librated 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.

Figure (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 600 °C 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 percents containing dust as nucleating agent for lime collections or as activator for formation and precipitation the crystalline $Ca(OH)_2$ form. Addition of 5 or 25% limestone to Portland cements enhances the formation of calcium hydroxide at early ages probably because it provides nucleation sites for its growth [27]. 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 C_3A contents the amount of mono-carbonate increased at all hydration ages compared with cements with lower C_3A content with increasing level of dust addition.

Phenolphthalein spraying test [28], 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 percents. The zone in the mortar structure where such phenomena have occurred coincide approximately with the zones which remain uncolored in coloration reaction using 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 $CaCO_3$ through carbonation. It is confirmed that in this discolored portion, C-S-H in hydrated cement has been decomposed into $CaCO_3$, SiO_2 and H_2O [29].

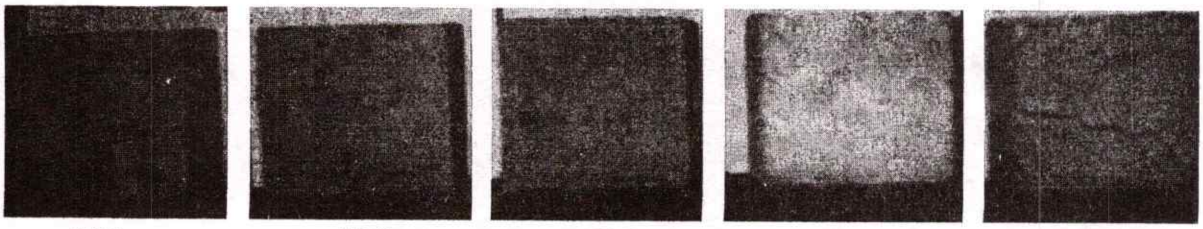


Figure (7,a) : Photographic surface cracking patterns for the OPC mixes treated at 500 °C as start of temperature damage.

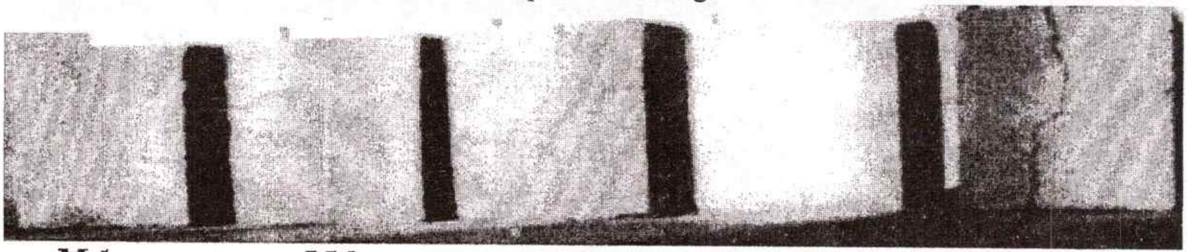


Figure (7,b) : Photographic surface cracking patterns for the OPC mixes treated at 600 °C shows more observable damage.

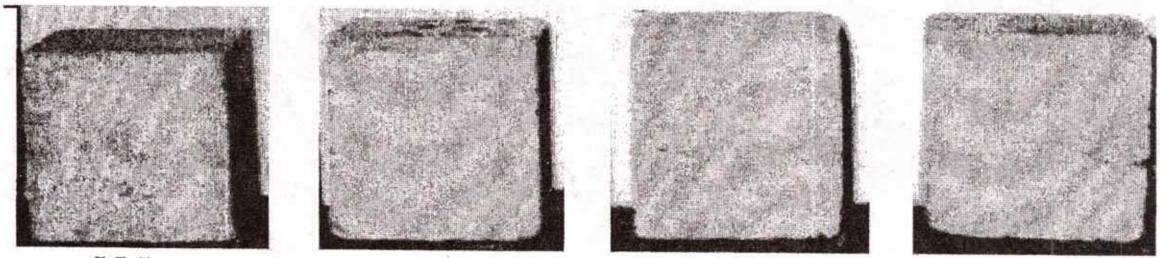


Figure (8) : Photographic surface cracking patterns for the OPC mixes treated at different temperatures.

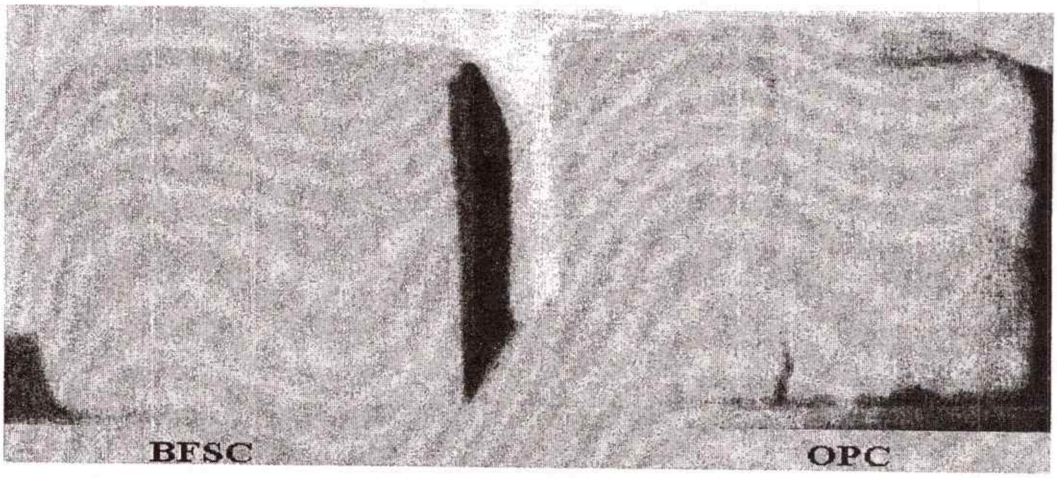
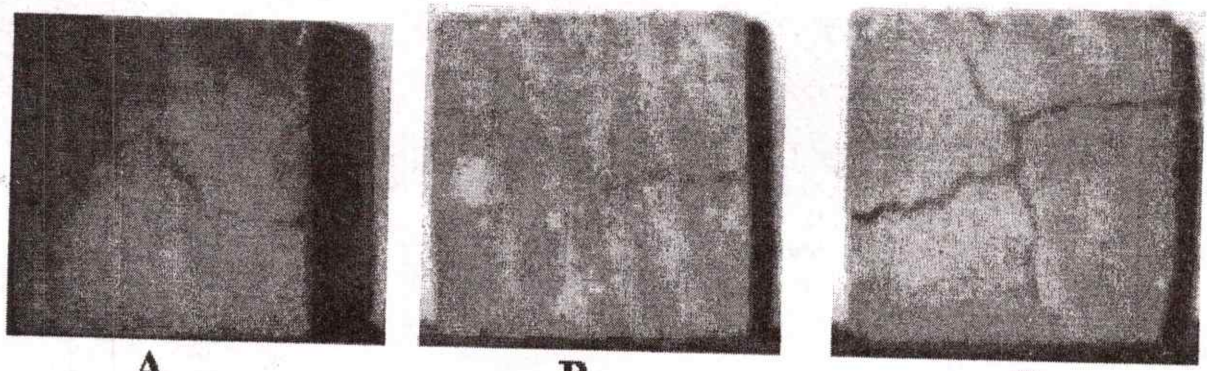
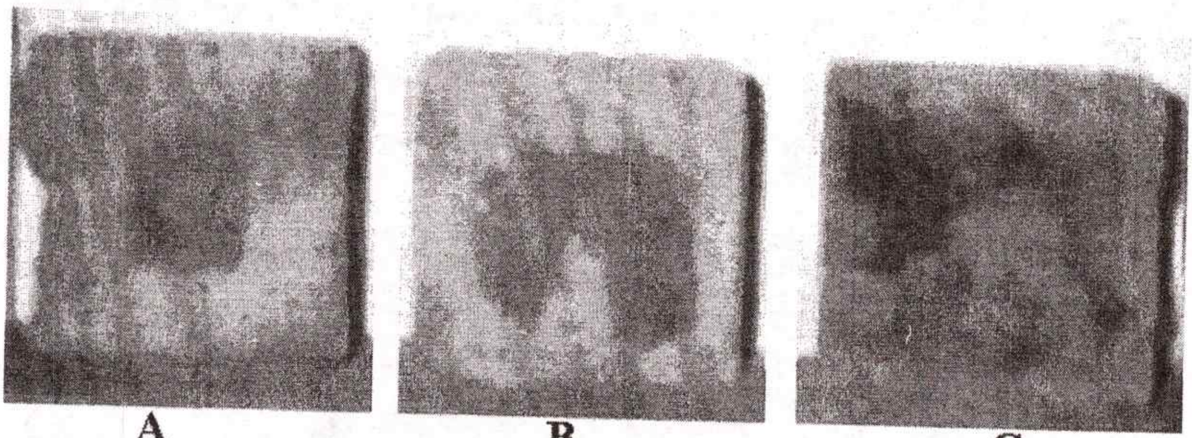


Figure (9) : Photographic surface cracking patterns of (a)- OPC and (b)- BFSC treated at 600 °C.



A B C

Figure (10): Photographic surface cracking patterns of OPC treated at 600 °C with different percents of dust (A)with 10%, (B) with 15% and (C) with 20% CKD.



A B C

Figure (11,a) : Carbonation depth of the thermally treated samples (A)OPC with 20% CKD at 600 °C, (B) OPC with 20% CKD at 500 °C and (C) BFSC with 20% CKD at 600 °C.

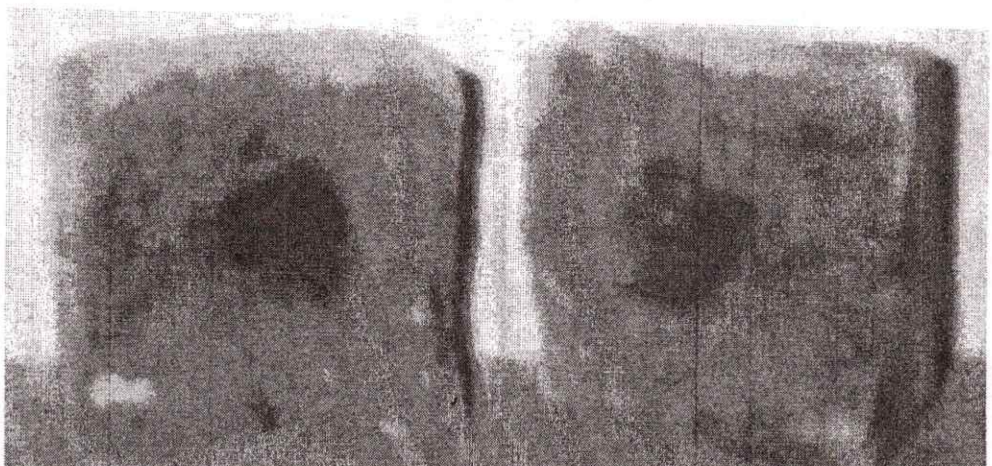


Figure (11,b) : Carbonation depth of the thermally treated samples of OPC-CKD treated at 600 °C.

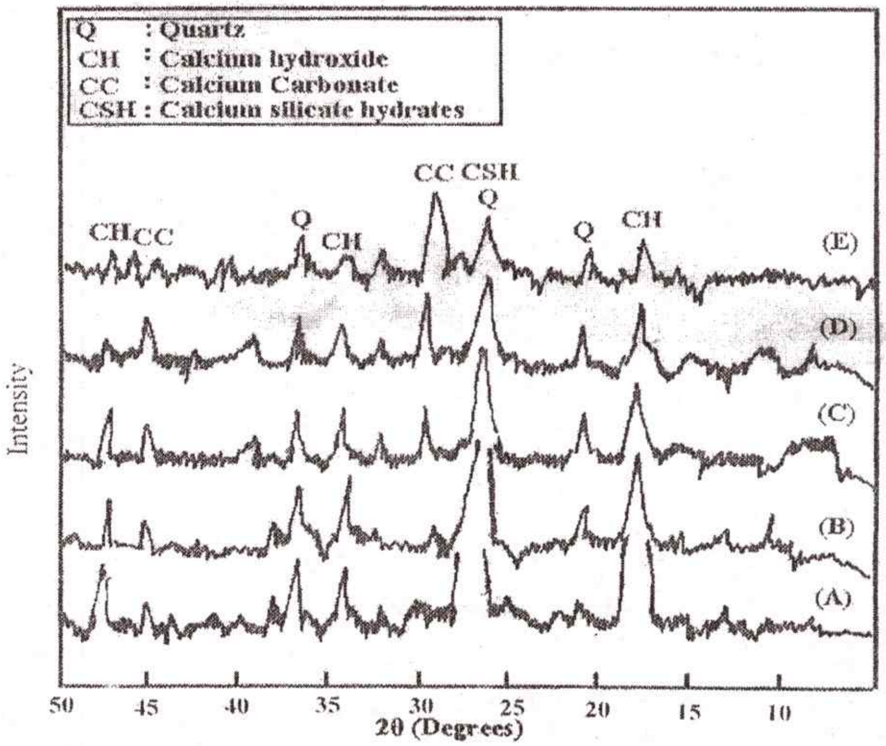


Figure (12): X-ray diffraction analysis patterns of thermally treated OPC-CKD samples at various temperatures A) before, B) at 300, C) at 400, D) at 500 and E) at 600 °C .

Figure (12), shows the X-ray diffraction a nalysis o f 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 hydrate (CSH), calcium hydroxide [$Ca(OH)_2$] and calcium carbonate ($CaCO_3$). It was observed that the peak characteristic for calcium silicate hydrate decreased gradually with increasing the treated temperature up to 600 °C, 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 $(OH)_2$ peak slightly increased at 300°C, this is due to some recrystallization of this portlandite phase, but its decrease up to 500 °C or more is mainly due to thermal decomposition of $Ca(OH)_2$ into CaO and water accompanying by high weight losses. XRD of the thermally treated mortar specimens with BFSC-CKD showed that the Ca $(OH)_2$ peak in these samples are relatively very l ow, t his i s a ttributed t o 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 could to say that BFSC mortar exhibits h igh t hermal s tability t han those made with OPC.

CONCLUSIONS

From the above results of present work, the following conclusions can be written:

1- It is important to reuse cement kiln ecologically as well as economically to overcome the large quantities produced annually.
2- CKD has adversely affected the physical and mechanical properties of cement mortars and concretes specially 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 600oC.
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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