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An investigation of the mechanical properties of ternary blend cement pasts containing a large amount of cement kiln dust

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Abstract

Cement kiln dust is a by-product of the cement industry composed of particles of feedstock and condensed volatilized inorganic salts. The effect of Cement kiln dust (CKD) on the mechanical properties of ternary blend cement pastes was studied by preparing different mixes using 40%CKD, 10% OPC and 50% of three different pozzolanic material (ground granulated blast furnace slag (GGBFS), metakaolin (MK), and ground clay brick (GCB)). The mechanical properties were tested via the determination of compressive strength, bulk density, total porosity, and chemically combined water content at 3, 7, 28, 90, and 180 days. The hydration products were evaluated using x-ray diffraction analysis. The result indicated that 40%CKD greatly enhances the activation of GGBFS, MK, and GCB. The paste that contains (10% OPC, 40% CKD, and 50% GGBFS) gives the highest compressive strength, while (10% OPC, 40% CKD, and 50% GCB) gives the lowest compressive strength.

Keywords: Ordinary Portland Cement (OPC), Cement kiln dust (CKD), Metakaolin (MK), Ground clay brick (GCB), Blended cement, X-ray diffraction analysis (XRD).

1. Introduction

Cement kiln dust is a by-product of the cement industry composed of particles of feedstock and condensed volatilized inorganic salts [1]. According to S. Peethamparan cement production produces about 15-20% CKD of all cement production [2]. It cannot be recycled in cement manufacturing because of its high alkalinity [3]. Its chemical composition depends on several factors such as type of kiln,

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raw feed, dust collection system, production technology, type of fuel, and dust removal method [4]. The major constituents of the CKD compound are silica, lime, iron, and alumina. It contains traces of cadmium, lead, selenium, and radionuclides. The pH of cement kiln dust is around 12 [5]. Recycling CKD has been one of the most interesting topics for scientists. It had been used in soil stabilization, waste treatment, production of low controlled cement, replacement of cement [6], and as an activator for some of the pozzolanic materials such as slag and MK, and fly ash.

Blast-furnace slag is formed as a molten stream from the blast-furnace at a temperature of 1400-1500°C. In many researches, GGBFS has been used as a cement substitute to create fresh low-carbon binders. Slag alone reacts with water very slowly but it could be activated by hydroxyl ions (OH) supplied by an activator like the lime in lime-rich sludge (LRS) [7]. The use of CKD as an alkaline activator for slag and fly ash has been established in the previous literature [8-13] and also used as an activator for MK [9]. Additional commercial alkali was used to improve the performance of the alkali-activated slag and fly ash, respectively [14,15]. Ternary blended containing mainly OPC and only small amounts of CKD as alkaline activator was previously reported [16,17]. It was concluded that CKD could effectively activate latent hydraulic properties of some pozzolanic material.

Metakaolin (MK) is produced by calcination of high purity kaolinite at temperature (700 to 850°C). This calcination dehydroxylate the crystalline structure of kaolinite, $\text{Al}_2(\text{OH})_4\text{Si}_2\text{O}_5$, giving Metakaolin (MK), $\text{Al}_2\text{Si}_2\text{O}_7$ [18]. MK contains active silica and alumina which can react with $\text{Ca}(\text{OH})_2$ to produce CSH gel at room temperature. The use of CKD as an alkaline activator for MK has been investigated [9]. Oriola F.O.P used a hybrid mix of CKD and MK to replace OPC and noticed that increasing percentage of replacement decrease compressive strength [19]. When MK was used as cement replacement with different percentages, they obtained that using MK enhances the mechanical properties of hardened cement [20,21].

Homra (ground clay bricks), is a pozzolanic material that consists mainly of silica quartz, aluminosilicates, anhydrite, and hematite. It can react with lime liberated from the hydration of ordinary Portland cement producing additional amounts of calcium silicate hydrates (CSH). Partially replacing cement by GCB gives early strengths that are lower than that of plain OPC. At 90 days, however, the strengths are

the same as or are greater than that of the plain OPC [22]. According to studies take place by [23,24], as the replacement level of RBP (red brick powder) increases the setting time decreases. However, decreasing particle size decrease setting time.

Cement manufacturers produce a huge of CO₂ from fuel combustion and the de-carbonation of limestone. Almost every 1000 kg of cement produces 900kg of CO₂. It is also responsible for 5% of greenhouse gases [25]. CO₂ emissions cause bad weather conditions, climate change, and ecosystem damage, besides the emission of SO_x, NO_x, and particulate matter that harm the health of humans and livestock [26]. So, replacing cement with by-product pozzolanic material reduces CO₂ emission.

This study aims to design friendly cement paste using 10 % OPC, 40% CKD, and 50% of different artificial pozzolana and trying to evaluate the effect of CKD on GBFS, MK, and GCB.

2. Experimental Work

2.1. Materials:

OPC and CKD were supplied from Toura Factory, Cairo, Egypt. While Granulated Blast-Furnace Slag (GBFS) was obtained from Helwan Company of Steel, Cairo. Ground Clay Bricks (GCB) and Clay "Meta Kaolinite (MK)" were supplied from demolished masonry, Egypt, and Kalabsha area, Upper Egypt (Aswan), respectively.

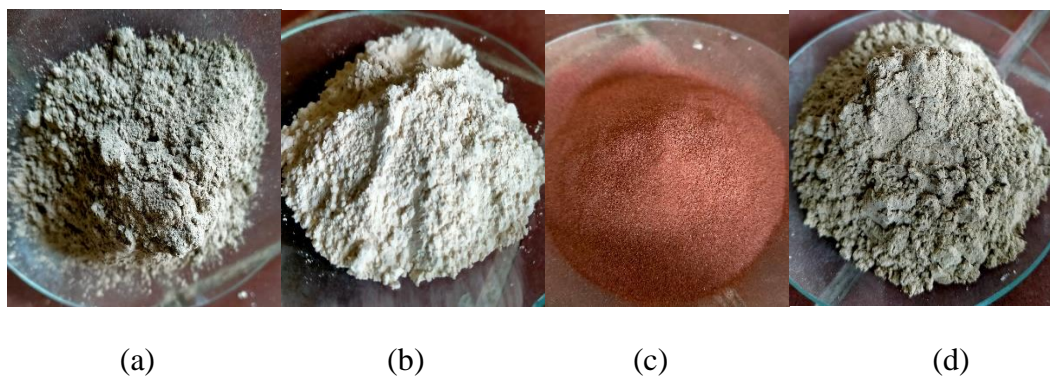


Fig.1. (a) ground granulated blast furnace slag (GGBFS), (b) metakaolin (MK), (c) ground clay brick (GCB) (d) cement kiln dust (CKD)

Table (1): The chemical composition of the starting materials

Oxides, %	OPC	CKD	GBFS	MK	GCB
SiO ₂	20.18	14.16	32.80	64.80	74.8
Al ₂ O ₃	4.67	3.98	7.02	30.10	14.03
Fe ₂ O ₃	3.74	3.42	1.14	0.55	5.04
CaO	61.73	53.87	42.56	0.52	1.25
MgO	1.49	0.86	11.58	----	1.3
SO ₃	3.21	3.68	2.50	0.13	0.8
K ₂ O	0.38	6.62	0.15	----	----
Na ₂ O	0.35	3.01	0.15	0.10	----
Cl	0.03	7.43	----	----	----
TiO ₂	----	----	----	2.70	----
P ₂ O ₅	----	----	----	0.06	----
L.O. I	2.90	2.80	0.93	0.73	2.58
Total	98.83	99.83	99.03	99.69	99.8

2.2. Preparation of mixes:

Each dry mix was prepared, as shown in Table (2) then mixed to obtain complete homogeneity. The blending was done by mixing dry constituent with the required amount of water to attain standard consistency, as given in Table (2) continuously for three minutes. The pastes are molded into cubic specimens of one-inch dimension after complete mixing. The molds were shaken for one minute to remove any air bubbles. The top surface of the pastes smoothed by a thin-edged trowel.

Immediately after molding, the molds were put in 100% humidity for the first 24 hours. Then the samples were demolded and preserved under tap water for hydration ages such as 3, 7, 28, 90, and 180 days to obtain the final setting.

2.3. Methods of investigation

The hydration characteristics of the different blended cement pastes were investigated by:

2.3.1. Compressive Strength

The compressive strength for three cubes was measured using a manual compressive strength machine point load taster (D550-Control type, Milano-Italy) with a maximum load of 60 tons. Then the average of the three results was expressed in MPa. After compressive strength measurements, a few grams were taken from the crushed cubes and stirred with about 100 ml of (1:1 v/v) methyl alcohol and acetone to stop the hydration. After filtration the sample is dried at 50 °C, 100 °C for 24 hrs., and then kept for XRD analysis and combined water measurement respectively.

2.3.2. Combined Water Content

The combined water content was determined using hydration stopped samples after being ignited in porcelain crucibles at 1000 °C for 1 hr. in a muffle furnace. It was calculated according to:

$$W_n\% = [(W_1 - W_2) / W_2] \times 100$$

Where W_1 : the weight of the sample before ignition. W_2 : the weight of the sample after ignition at 900°C.

2.3.3. Total Porosity and Bulk density

Total porosity and bulk density tests are carried out by determining the weight of the samples of the hardened pastes suspended in water, W_2 , and those in air, W_1 . Then dried at 100 for about 24 hours and weighed in air W_3 . The total porosity is calculated according to:

$$\text{Total porosity}\% = [(W_1 - W_3) / (W_1 - W_2)] \times 100$$

The Bulk density of the hardened cement paste was calculated according to:

$$\text{Bulk density} = W_1 / (W_1 - W_2)$$

Where W_1 weight of cube in water and W_2 weight of the cube after drying.

2.3.4. Water for normal consistency

The water for normal consistency was determined according to ASTM C187 [27].

2.3.5. X-ray diffraction analysis

The phase composition for the formed hydrates was investigated by PANalytical XPert Pro MPD using Cu $K\alpha$ -radiation source ($\lambda = 1.54056 \text{ \AA}$) and Ni-filter under working conditions of 40-kilo volt, 40 milliamperes.

To determine the best mix proportion which gives higher compressive strength and before going on with this work we have used different CKD percentage (60,50,40%) to evaluate the effectiveness of increasing CKD amount on mechanical properties of blended cement pastes. We concluded that 10% OPC+40% CKD+50% of different artificial pozzolan (GGBFS, MK, and GCB) give the highest compressive strength as shown in Table (2).

Table (2): composition of the prepared mixes.

No.	Mix ID	OPC %	CKD	GBFS	MK	GBC	W/S %
1	100%OPC	100	-	-	-	-	35
2	MIX I	10	40	50	-	-	47
3	MIX II	10	40	-	50	-	50
4	MIX III	10	40	-	-	50	57

Where W/S is water to solid ratio.

3. Results and discussion

3.1. Water of consistency

Table (2) lists the amount of mixing water needed for standard consistency. it is clear that the standard water of consistency required for blended cement was more than water used to blend plain OPC mix. This is probably due to using a small amount of

OPC (10%) compared with CKD and GGBFS, MK and GCB used. [28] reported that the higher surface area of CKD necessitates more water for lubrication of the particles to achieve better flow. The utilization of CKD requires more water because of the higher ability of CKD in absorbing mixing water due to the free lime (F.L) content which is active and rapidly reacts with water to produce Calcium Hydroxide Ca(OH)_2 [29,30]. It is clear that the system with GGBFS requires less water than MK and GCB pastes for standard consistency. This probably due to the smooth, vitreous, and non-absorbent surface structure of slag particles. Using MK and GCB increase the water of consistency [31]. This is maybe due to amorphous properties, smaller particle size, and larger surface area of MK and GCB which absorb more water [32,33].

3.2. Compressive strength

The compressive strength of 10% OPC–40%CKD–50%GGBFS cured up to 180 days is represented in Fig (2). As the curing age increases the compressive strength increases for hardened cement pastes. This can be attributed to the continuous hydration of OPC as well as the pozzolanic reaction of GGBFS with the liberated CH during the hydration of OPC and CKD leading to the formation of larger amounts of CSH, CAH, and ettringite [8]. The compressive strength of slag pastes is lower than OPC pastes. This mainly due to the dilution effect of OPC and higher amount of CKD which reduce the compressive strength as it contains a high amount of alkalis, sulfates, and chlorides which cause the formation of chloro and sulfoaluminate phases that make hydration products (CSH) softer and expand in comparison with the OPC hydration alone [29,30]. At early ages, the compressive strength of slag blended cement is low compared to the 100% OPC mix. This could be explained by the slower hydration of GGBFS than OPC and the lower rate of strength development of slag [10].

The results of Fig.(2) indicate that the compressive strength increases with the increase of curing age for the 10% OPC–40%CKD–50%MK hardened blended cement pastes. The role of MK in the improvement of the strength results is mainly due to the interaction of the reactive MK with the free lime and alkalis released from OPC and CKD hydration. This leads to the formation of additional amounts of calcium silicate hydrates which increases the total content of hydration product in pastes [34]. lower content of OPC and higher amount of CKD which reduce the compressive strength as it contains a high amount of alkalis, sulfates, and chlorides which cause the formation

of chloro and sulfoaluminate phases that make hydration products (CSH) softer and expand in comparison with the OPC hydration alone [29,30]. Slower hydration of MK and the lower rate of strength development are the main reasons for lower compressive strength values at early ages [35].

The results of compressive strength of 10%OPC–40% CKD–50%GCB blended cement pastes indicate that with increasing age of hydration more hydration products are formed resulting from enhanced activation of GCB in the presence of CKD; these hydrates act as binding centers between the unhydrated parts of cement grains [36]. The pastes containing GCB were less strong compared to plain OPC. This mainly due to the lower content of OPC and higher amount of CKD which reduce the compressive strength as it contains a high amount of alkalies, sulfates, and chlorides which cause the formation of chloro and sulfoaluminate phases that make hydration products (CSH) softer and expand in comparison with the OPC hydration alone [29,30]. The slow rate of pozzolanic reaction and coating effect of GCB on the cement grains [37,38] cause retardation of strength development, especially at early ages.

The dependence of the paste strength on the type of pozzolans can be seen in Fig. (2). It is well known that glassy pozzolans with a low amount of crystalline, non-reactive minerals are considered to be very active, while, on the other hand, weak pozzolans contain low glass and large amounts of crystalline minerals [37]. The hardened blended cement pastes made of 10%OPC–40% CKD–50% GGBFS blend possess the highest strength values. This could be attributed to the large glassy content of GGBFS with only a limited amount of non-reactive crystalline minerals [37] in addition to high alkali and sulphate of CKD that activate a large amount of GBFS producing CSH and ettringite which are responsible for higher strength [39]. MK mix gives higher compressive than GCB because of its higher amorphousness than GCB, which is responsible for more hydration products.

The additions of 50% GCB of the binder mass give lower compressive strength pastes, probably due to lower active aluminosilicate content and the large amount of crystalline quartz which is inactive towards $\text{Ca}(\text{OH})_2$. These results are in harmony with previous research [9,40,33].

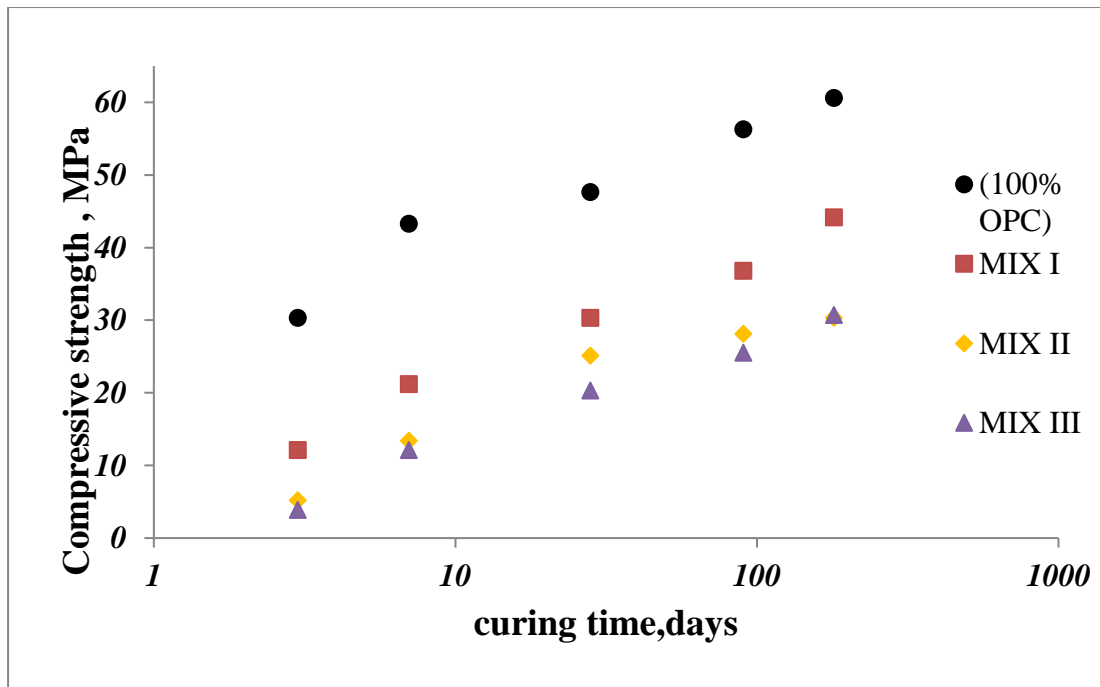


Fig.2 Compressive strength of three mixes cured in H₂O up to 180 days (MPa).

3.3. Chemically combined water contents

The values of the chemically combined water content of the hardened blended cement pastes cured in water up to 180 days are represented in Fig (2). Using the combined water content, we can estimate the degree of hydration of hardened cement pastes. The amount of combined water depends on hydration products type and quantity [22]. As the curing time proceeds for all mixes, the chemically combined water contents increase revealing more hydration products are present and precipitated in the open pores. This hydration product comes from continuous hydration of OPC and the pozzolanic reaction of GGBFS, MK, and GCB [37]. The blended cement pastes give a lower combined water value than OPC pastes because CSH formed from the pozzolanic reaction has lower water content than CSH from OPC hydration. It was found that MixI (50% slag+40% CKD+10% OPC) gives the higher values of chemically combined water because of the large glassy content of GGBFS and rapid reaction with CH forming more CSH [41]. MK shows a higher combined water value than GCB due to its large surface area and very small grain size interacts with the free calcium hydroxide, liberated from OPC and CKD hydration, leading to the formation of excessive amounts of calcium silicate hydrates CSH [42].

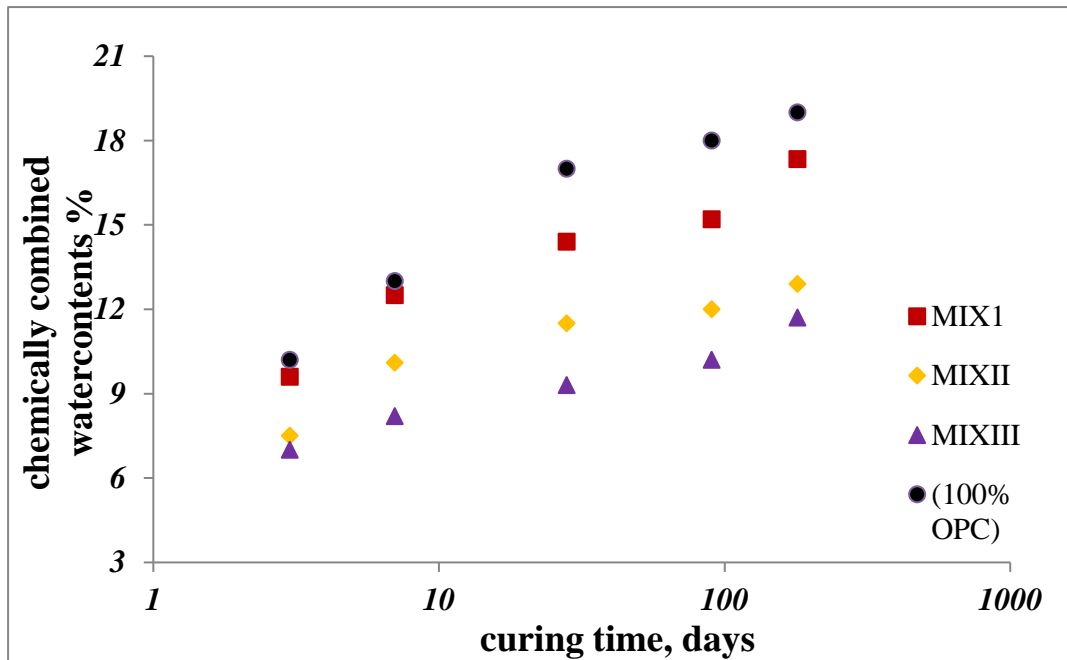


Fig.3 Chemically combined water content of three mixes cured up to 180 days.

3.4. Bulk density and Total porosity:

The results of bulk density and total porosity for all hardened blended cement pastes are given in Table (3) and (4) respectively. Total Porosity is a key property of cement paste that affects the strength of cement pastes and depends on the type of cement. It increases with increasing the water to cement ratio (W/C) and decreases with the curing period [13].

The bulk density increases, and total porosity decreases with curing time up to 180 days for all blended cement pastes. This is due to the continuous hydration and accumulation of hydration products in water which fills pores to form a more compact body. These hydration products have more twice volume than anhydrous cement so they fill the open pores as the hydration proceeds [43].

In the slag system, GGBFS reacts with Ca(OH)_2 liberated from OPC and CKD hydration producing more hydration products as CSH and CASH reducing total porosity and increasing bulk density [11]. MK and GCB reduce total porosity and increase bulk density by producing more hydration products as CSH and CASH when they react with Ca(OH)_2 liberated from OPC and CKD hydration. In addition to unreactive quartz acts as a filler and fill pores [33,35].

The results of bulk density and total porosity of 10%OPC–40% CKD–50%GGBFS blended cement pastes show relatively higher bulk density and lower total porosity than ones contain MK and GCB. This can be attributed to sulphate activation in GGBFS which produces an additional amount of CSH and ettringite [9]. While GBC gives very low density this mainly due to the lower density of GBC compared to OPC and higher water of consistency. These findings match up those data obtained from the compressive strength test and other investigation tests.

Table (3): Bulk density of the three mixes cured up to 180 days (Kg/m³).

Mix ID Days	(100% OPC)	MIX I	MIX II	MIX III
3 days	2670	2570	2520	2400
7 days	2710	2580	2570	2440
28 days	2740	2640	2620	2590
90 days	2760	2690	2640	2610
180 day	2780	2730	2660	2670

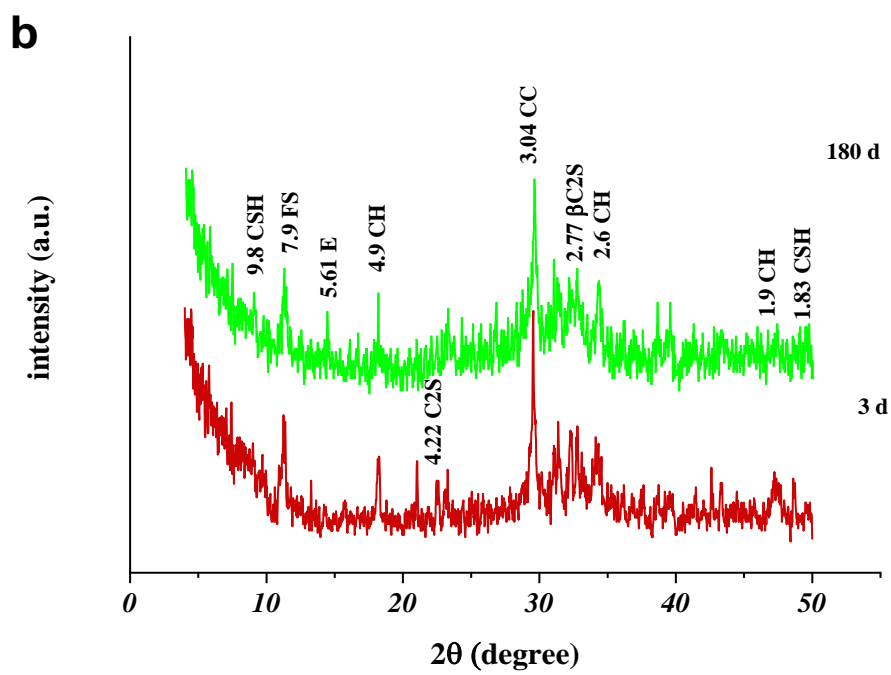
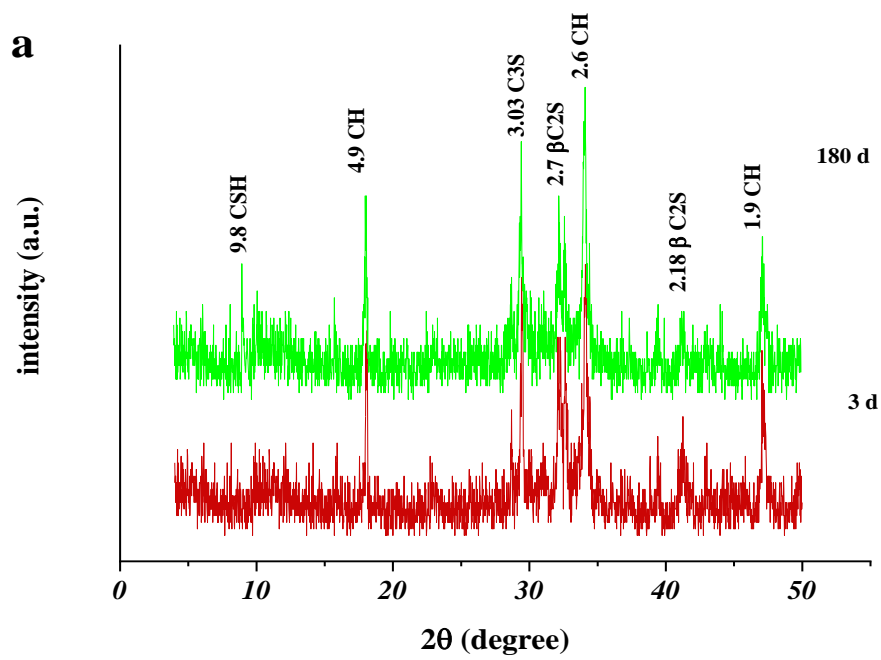
Table (4): Total porosity of the three mixes cured up to 180 days

Mix ID Days	(100% OPC)	MIX I	MIX II	MIX III
3 days	31.4	41.9	49.2	51.4
7 days	30.2	40.1	47.7	49.47
28 days	27.9	39.7	45.8	47.69
90 days	25.8	38.6	45.3	44.4
180 days	23.9	37.9	44	41.2

3.5. X-ray diffraction analysis

XRD patterns of mixes are shown in Figs. 3(a, b, c and d), the main hydration products are CH, ettringite, and CSH. The intensity of CH peak decreased in 180 days than 3 days which is observed at $d = 4.69, 2.64, 1.9 \text{ \AA}^\circ$ due to combustion of CH in the polymerization process which indicates that GGBFS, MK, GCB have been involved in the pozzolanic reaction [10,40,18]. The existence of the weak peaks of the CSH phase at $d = 9.8$ and 1.83 \AA° is attributed to its lower crystallinity. The intensity of unhydrated phases of $\beta\text{-C}_2\text{S}$ and C_3S peaks at $d = 2.7$ and 1.83 \AA° decreases with time due to the continuous hydration of these phases [44]. The attendance of other phases such as the calcium monochloroaluminate hydrate, also known as Friedel's salt at $d = 7.9 \text{ \AA}^\circ$, is a result of the high chloride content of CKD [8] and ettringite which has good space-filling properties because of the compact packing of its needle-like crystals. Ettringite contribute to strength development, however, it has a lower density than other hydration product. It probably forms because of the reaction between the sulfate bearing phases, anhydrite, and arcanite, with the alumina from the slag or ferrite [45,17] or results from the reaction of C_3A in OPC and sulphate in CKD.

In Fig. 3(b,c,d) the intensity of Calcite peaks at $d = 3.04 \text{ \AA}^\circ$ with curing time due to the effect of CO_2 in the presence of H_2O to form $\text{Ca}(\text{HCO}_3)_2$ [18]. Fig.3 (C and d) shows that quartz mineral at $d=3.41 \text{ \AA}^\circ$ is the major crystalline phase and remains unreacted in all hydration ages. This is mainly because of the lower reactivity of quartz to alkali activation during polymerization, this explains why GBC gives lower compressive strength than GBFS and MK [40].



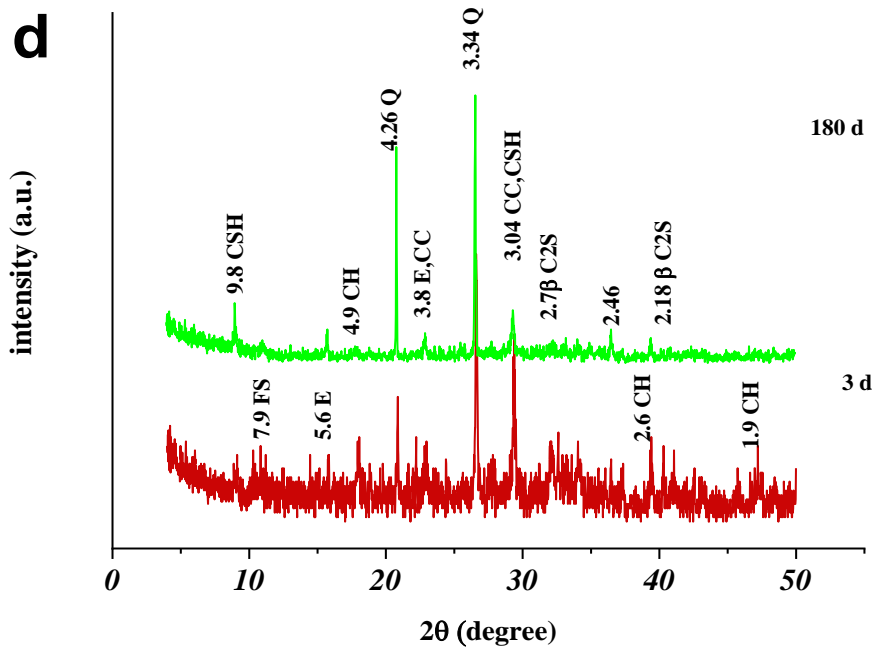
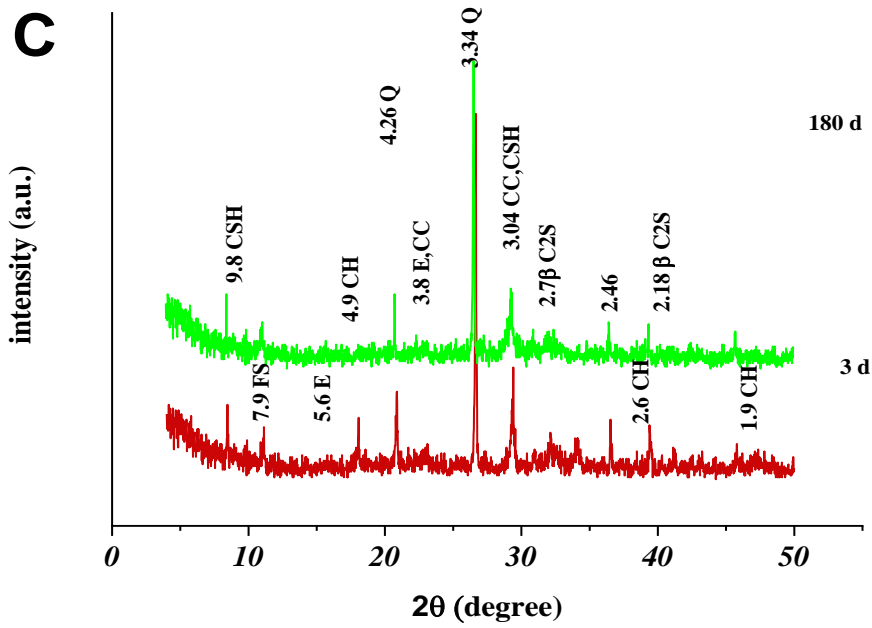


Fig. 3. (a) X-ray diffraction patterns for the mix (100% OPC).

(b) X-ray diffraction patterns for the mix (10% OPC+40% CKD+50% slag) (MIXI).

(c) X-ray diffraction patterns for the mix (10% OPC+40% CKD+50% MK) (MIXII).

(d) X-ray diffraction patterns for the mix (10% OPC+40% CKD+50% GCB)

(MIXIII).

E: Ettringite, CH: portlandite, CC: Calcite, CSH: C-S-H gel, M: Magnesium oxide, Q: quartz, C3S: tricalcium silicate, C2S: dicalcium silicate, FS: Friedel's salt.

Conclusion

Cement kiln dust (CKD) materials were used as alkaline accelerators for latent hydraulic substances and as alkali activators for different aluminosilicate materials, including ground-granulated blast furnace slag, ground clay brick, and metakaolin. and it was observed that:

- Compressive strength and combined water of all the blended cement mix increases with the hydration time.
- The bulk density of all mixes increases with curing time due to the continuous activation and formation of hydrated products. These hydrated products are deposited in the open pores, so the bulk density increases and total porosity decreases.
- 50%CKD greatly enhances the geopolymerization process with the formation of a well-defined and compact matrix, where CKD represents an enrichment source of hydroxide.
- MIX I (10% OPC+40% CKD+50% slag) shows the highest values of compressive strength at all curing ages of hydration. While MIXIII (10% OPC+40% CKD+50% GCB) gives the lowest values of compressive strength at all curing ages of hydration.
- The results of X-ray diffraction analysis indicate that the degree of hydration of samples with GGBFS is higher than that of samples with MK and GCB, which proves that GBFS has higher pozzolanic activity than MK and GCB representatively.

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الملخص العربي

تحقيق في الخواص الميكانيكية لخلطات عجائن الاسمنت الثلاثية المحتوية على كمية

كبيرة من غبار تراب الأسمنت

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يعتبر غبار تراب الاسمنت ناتج ثانوي عن صناعة السمنت. ويتم انتاج كميات ضخمة منه كل عام. لذلك فان استخدام هذه النفايات في صناعة عجائن اسمنت مخلوطة يوفر الكثير من المال ويحمى البيئة. والهدف من هذا البحث دراسة تأثير استخدام 10% فقط من الاسمنت العادي و90% من البوزولانا مثل (غبار تراب الاسمنت، خبث الحديد، الميتا كولين والحمرا) منهم 50% غبار تراب الاسمنت. تم دراسة خواص كل خليط بعد عملية التآدرت في الماء بعد فترات زمنية (3- 7- 28- 90- 180) يوم وذلك بقياس مقاومة الضغط الميكانيكي، الكثافة، المسامية، الماء المتحد كيميائيا ودراسة التركيب الدقيق باستخدام حيود الاشعة السينية من هذه الدراسة نستنتج ان الخليط المحتوي على (أسمنت بورتلاندى 10%+غبار تراب الاسمنت 40% +خبث الحديد50%) هو الخليط الأنسب من حيث جميع الخصائص (هو الخليط الذي يعطى اعلى مقاومة لضغط الميكانيكي واعلى كثافة وأكثر كمية من الماء المتحد كيميائيا واكل مسامية.) ولكنه يعطى مقاومة للضغط الكيميائي اقل من الاسمنت العادي لذلك يمكننا ان نستخدمه في صناعة احجار الرصف في الطرق الجانبية وغيرها من الاستخدامات التي لا تحتاج مقاومة عالية للضغط.