Calcined kaolinitic clay as a supplementary cementing material and its pozzolanic effect on concrete blends characteristics (Part I)

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Abstract
The calcined kaolinitic clay (CKC) was investigated as a supplementary cementing material to assess its effect on the characteristics of concrete. The experimental protocol included optimization of the mixes of CKC/OPC containing 5, 10, 15, 20, 25, 30 and 35% CKC for production of hardened concrete. An evaluation of the use of CKC which contains about 79% of kaolinite mineral in south Sinai quarries in concrete fabrication. The objective of this study is to deliver an overview on CKC utilized in concrete modification. The physical and chemical characteristics of CKC were assessed. The compressive strength and slump of concrete was tested and discussed. The findings suggest that adding CKC to concrete enhances certain characteristics, particularly mechanical capabilities. For optimal performance, the right dosage ranged is between 10 to 20% by weight of the binder. The optimal replacement ratios for cement with CKC was 10% and 15%, which improved the compressive strength by an average of 120.8 and 118.7% respectively at the age of 28 days compared to the control mix. Assessment of the optimal dosage out of the two ratios (10 & 15%) requires a study of their fulfillment of many characteristics as well as their effect on durability and sustainability and their efficiency in facing harmful elements and media. And then determine the best ratio from the technical and economic point of view.
Keywords: kaolin, kaolinitic clay, calcined kaolin, metakaolin, pozzolanic materials, concrete mineral additives
Kulcsszavak: kaolin, kaolinites agyag, kalcinált kaolin, metakaolin, puccolán anyagok, beton ásványi adalékok

1. Introduction

Concrete fabrication and application in the building activity have increased due to its durability, and economic characteristics compared with other construction materials [1-3].

One ton of concrete is produced yearly by each human all over the world [4]. The production of Portland cement, has several disadvantages, including intensive energy demand and sharp environment pollution [5]. The calcination process emits huge amount of carbon dioxide due to the heating of limestone and the firing of petrol fuels for production of cement [6, 7]. Cement is the main ingredients in concrete since it needs water to bind the fine and coarse aggregates. Cement production was over 4 billion tons per year in 2018 and the demand is increasing, emitting great quantity of carbon oxides into the biosphere participating in the warming of the earth [8].

The use of supplemental cementitious materials (CMs) such as flied ash [9] fumed silica [10] waste glass [11] and ground granulated blast furnace slag [12] is one solution to this problem while manufacturing concrete or as a partial substitute for cement in the cement industry. High strength, better durability, avoidance of surface cracking of concrete, economic feasibility and sustainability are benefits of adding cementitious ingredients in concrete. The quantity of the cementitious material that replace OPC is limited by their pozzolanic activity [13].

Several researchers have reported that calcined kaolin (CK) called metakaolin can be used as a cementitious additive in concrete [14, 15]. The use of high reactivity CK as a supplemental cementitious ingredient in the concrete industry has gained popularity. Although CK has been known since the sixtieth of the last century. The studies are still interested in application of pozzolanic additives to cement as a cementitious material in concrete for improving its performance [16, 17]. CK is a very fine material fabricated by calcination of kaolinite clay at temperatures between 700 and 800 °C to remove chemically bonded water and disrupt the crystalline structure. CK refines the pore structure and reduces the lime hydroxide of the hardened matrix of the concrete. This is achieved as a result of the fine grained CK.

The reactions of CK with lime hydroxide produced during the hydration reactions of cement forming of secondary binding compounds such as calcium silicate hydrates (CSH) modifying the microstructure of concrete and contributing in improvement of the durability. The improvement can be confirmed by porosity, permeability, and chloride ion diffusivity [18]. CK particles are much smaller than cement particles but not finer than fumed silica [19]. Adding CK to concrete has a significant effect on the mechanical and durability characteristics [20, 21].

The use of calcined kaolinitic clay (CKC) derived from local quarries in south Sinai (low grade kaolin) reduces cement use which can assist to relieve environmental issues. Based on the above, the purpose of this study is to provide an overview of the use of the CKC in concrete. The qualities of CMK are first discussed, which mostly involve physical and chemical characteristics. After that, the hydration, workability, mechanical characteristics, durability and scan electron microscopy of CMC concrete are thoroughly investigated. Furthermore, the most relevant results and recommendations are offered, which will aid future concrete investigations using CKC. This material is also ecologically benign since it helps to reduce carbon oxides emissions into the atmosphere by lowering the amount of ordinary Portland cement (OPC) used [22]. CK can be used in place of ordinary Portland cement (OPC) in fabrication of concrete [23]. The relevant results and recommendations are reported, which will aid future concrete investigations.

2. Material and methods

2.1 CEM II 42.5R (EN 197-1)

Table 1 shows the physical properties OPC, while *Table 2* illustrates the chemical composition of OPC.

The properties	Value		Limits
Specific gravity	2.63		2.5 – 2.75
Bulk density, (kg/m³)	1780		-
The compressive strength for standard mortar (MPa)	2 days	20.8	Not less than 10
	28 days	50.3	Not less than 42.5
Soundness (La Chatelier)	1		Not more than 1
Setting time (min.)	Initial	135	Not less than 60
	Final	180	-

*ESS 4756-1/ 2013

Table 1 Mechanical and physical properties of OPC*
1. táblázat A Portland cement (OPC) mechanikai és fizikai tulajdonságai*

Compound	SiO_2	Al_2O_3	Fe_2O_3	Cao	MgO	SO_3	Na_2O	K_2O	Cl^-	LOI
%	21.16	5.32	3.52	63.8	1.32	2.01	0.2	0.13	0.02	2.52

Table 2 Chemical composition of OPC
2. táblázat Az OPC kémiai összetétele

2.2 Calcined kaolinitic clay (CKC)

2.2.1 The chemical and mineralogical analysis of CKC

CKC was collected from aluminum Sulphate Company of Egypt. Kaolinitic clay (KC) was calcined in vertical fluidized bed kiln at 730 °C for 60 min [24]. The chemical composition based on the XRF was illustrated in *Table 3*. The amorphous silica is about 39% (*Fig. 1*). The XRD pattern displayed the silicate morphism in 20 – 25 2θ complying with that reported in reference [25, 26]. The chemical composition shows that the kaolinitic clay contains about 33% Al_2O_3, while the pure kaolinite contains 42% ($2SiO_2{\cdot}Al_2O_3{\cdot}2H_2O$), therefore KC contains 78.6% kaolinite.

Table 3 shows high content of reactive SiO_2; reactive silica represents the fraction of silica (39%) able to react at the normal conditions with alkalies forming binders. The averaged value of Al_2O_3 in CKC is 33%. CMK was analyzed by XRD analysis using a Bruker D8 Advanced Computerized X–Ray Diffractometer apparatus to show its constituting phases. *Fig. 1* displays that its main crystalline phase is quartz and the amorphous is metakaolin phase.

Compound	Amount (weight%)	
	CKC	CEM 1 42.5
SiO_2	61	21.5
SiO_2 (active)	39 = 0.65 Molar	-
Al_2O_3	33 = 0.32 M	3.5
SiO_2/Al_2O_3	2.3	-
Fe_2O_3	1.3	3.4
TiO_2	2.3	0.15
MgO	0.16	1.2
CaO	0.22	62
Na_2O	0.14	0.28
K_2O	0.13	0.32
SO_3	0.15	2.7
P_2O_5	0.10	0.04
SrO	0.05	-
Cl-	0.05	0.12
L.O.I	1.0	4.5
Total	99.6	99.71

Table 3 The chemical analysis of the calcined kaolinitic clay and CEM 1 42.5
3. táblázat A kalcinált kaolinites agyag és a CEM 1 42,5 kémiai elemzése

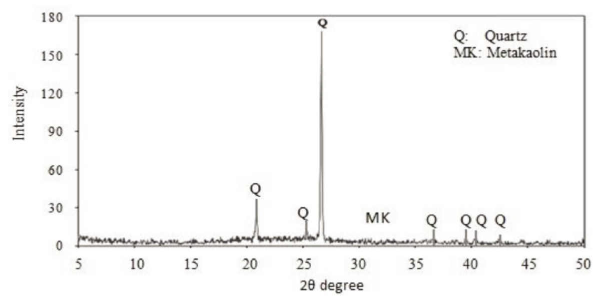

Fig. 1 XRD analysis for CKC
1. ábra Kalcinált kaolinitos agyag (CKC) XRD elemzése

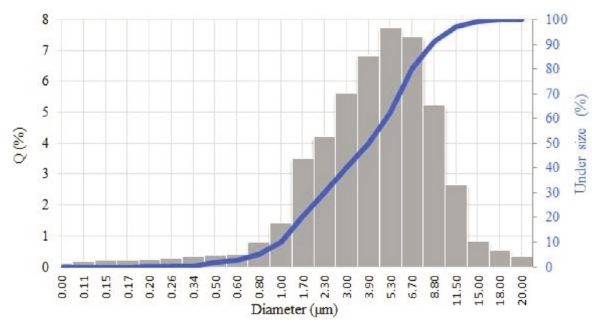

Fig. 2 Grain size distribution of the CKC by laser granulometry
2. ábra A CKC lézergranulometriával mért szemcseméret-eloszlása

2.2.2 The grain size-distribution of CKC

The grain size-distribution of CKC by laser granulometry was plotted in *Fig. 2* which shows that the diameter of CKC in cumulative 90% of below ~35 µm and 10% of below 4.5 µm with an average diameter of ~4.5 µm with a median at 50% = 4 µm and the mode (major) diameter = 5.5 µm.

2.2.3 The pozzolanic testing of calcined kaolinitic clay (CKC)

According to ASTM C 618 the pozzolanic materials reacts with portlandite [$Ca(OH)_2$] resulted from the hydration process of OPC and calcium silicate hydrate compound is formed as a cementing material. The pozzolanic reactivity of the samples was determined chemically by mixing of 2 g CKC with 10% lime and 2 drops of water. The free lime of the mixture was measured directly and after 5 days on another sample well covered and stored at 60 °C. This chemical method is described in detail elsewhere [27].

Table 4. indicates the pozzolanic activity, specific surface area and the bulk density of CKC. The reactivity test after 5 days recorded 120%.

The parameter	Value
The pozzolanic activity	120%
Specific surface area (BET)	20 m^2/g
The bulk density	1.35 g/cc

Table 4 The pozzolanic testing of calcined kaolinitic clay (CKC)
4. táblázat Kalcinált kaolinites agyag (CKC) puccolán vizsgálata

The surface area was measured by means of BET method which shows the adsorption of nitrogen at liquid nitrogen temperature. The specific density was evaluated using Le Chatelier flask according to ASTM C188-84. The mineralogical composition was monitored by means of X-ray diffraction using an automated diffract meter at a scan range from 10 to 50° (2θ). Positive reaction to pozzolana test (EN 196-4) was given by CKC when blended with CEM I 42.5R. Methylene blue method (UNI EN 933/9) 3.85 g/kg for CKC.

The replacement of OPC was made by addition of 5, 10, 15, 20, 25, 30 and 35% CKC in cement paste and the mortar mixes. The details of the mix proportions of the mortars are summarized in *Table 5*. The w/b ratio required to attain the standard consistency of the reference OPC paste was determined using Vicat apparatus according to ASTM C187-92. The initial and final setting times were measured using Vicat test according to ASTM C191-92. The same methods were utilized to investigate the effect of CKC on the water demand and the setting behavior of the pastes. The mixing procedures were carried out according to ISO 9597 (1989) and ASTM C305-82.

2.2.4 Differential thermal analysis (DTA) for Portland cement incorporating CKC

Fig. 3 shows DTA analysis of mixes containing a dosages of 0, 5, 10, 15, and 20% after 28 days of water curing. The main changes revealed by DTA analysis are as follows: An endothermic peak was detected before reaching 100 °C. This peak is related to the moisture absorbed by the sample from the environment. The endothermic peak observed in the 100-200 °C are attributed to the process of calcium silicate hydrate (CSH) formation. The peaks at the temperature range 450-500 °C are due to the dehydroxylation of $Ca(OH)_2$. The endotherms at the temperature near to 700 °C are due carbonation of lime and $CaCO_3$ (CC) formation. It is obvious that, as the cement replacement by CKC increases from 5% up to 15%, CSH content increases at the expense of lime hydroxide. Upon increasing the replacement dose to 20% CKC, insignificant variation in the C-S-H and CH content has recorded relatively to 15% CKC replacement. Increasing the replacement dose to 20% CKC, it seems that, the cementitious materials reactions between CKC and OPC were slowed down. These results are in agreement with previous research studies [28-30].

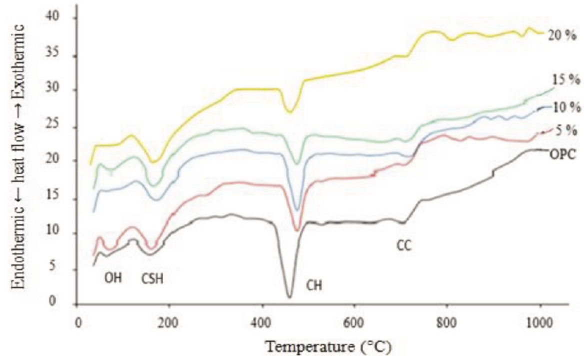

Fig. 3 Thermal properties analysis of CKC containing cement mixes (DTA)
3. ábra CKC tartalmú cementkeverékek (DTA) termikus tulajdonságainak vizsgálata

With replacement ratios of 0, 5, 10, 15 and 20% after 28 days of immersion in water.

2.2.5 Infrared spectrum analysis (FTIR)

This analysis was carried out to identify the structural state after replacement with CKC at 5, 10, 15 and 20% of cement and after 28 days of immersion in water. *Fig. 4* represents the infrared analysis of the reaction products with water after 28 days in the positive range 400-4000 cm^{-1}. There is a band at 3645 cm^{-1} due to the tension fluctuation of the OH group of portlandite $Ca(OH)_2$. The band at 1461 cm^{-1} is due to the presence of the mineral calcite (calcium carbonate) resulting from the interaction of lime with calcium dioxide. Band in the range 900 - 1000 cm^{-1} indicates the presence of a lattice structure of amorphous calcium silicate. The bands agree with that reported in references [31]. We notice from *Fig. 4* that the density of the band at 970 cm-1 in the aqueous mixtures of CKC/OPC increases with increasing the increase of CKC up to 15% of the cement. This behavior indicates an increase in the formation of aqueous calcium silicate compound. The density and pitch of the band begins to decrease with an increase in CKC of more than 15%. This is due to the decrease in pozzolanic reactivity.

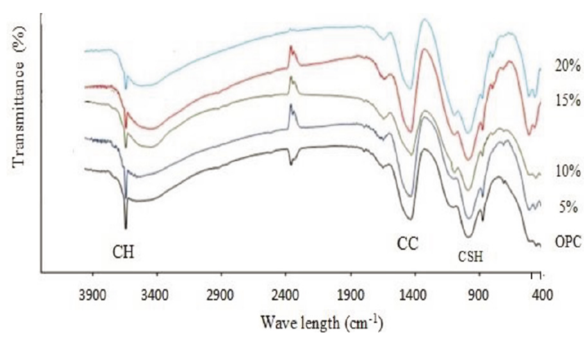

Fig. 4. FTIR analysis of cement mixes containing CKC with a replacement ratio of 0, 5, 10, 15, and 20% after 28 days of immersion in water
4. ábra 28 napos vízbe merítés után a 0, 5, 10, 15 és 20% CKC-t tartalmazó cementkeverékek FTIR elemzése

From the previous experiments conducted on (CKC/OPC) mixture, which contains increasing percentages of CKC, the manufactured product (5-20% CKC/OPC) and after 28 days of curing, to study the behavior of calcined kaolinitic clay as a pozzolanic material, using DTA /IR shows the following:

CKC behaves with cement as a pozzolanic material by interacting with calcium hydroxide resulting from the interaction of cement with water (Hydration) forming more calcium silicate hydrate binder (CSH). Therefore, this product behaves as pozzolanic materials according to the American standard specifications ASTM C618.

2.2.6 The fine aggregate

Using sand as a fine aggregate in both mortar and concrete mixtures. Sand tests were carried out according to the standard specifications (EN 1097, EN 933). The specific gravity = 2.6, the grain size averaged 2 mm.

2.2.7 Coarse aggregates

The chemical composition of coarse aggregates (dolomite) complying with Egyptian code No. 302/2018. The grain size averaged 16 mm.

2.2.8 The chemical additives

In order to achieve good mixing of the concrete mixtures, a water reducing agent, Master Reobuild 3045, was added, which conforms to ASTM 494 Type A.

3. Results and discussion

3.1 Cement mortar mixes

Eight mixtures of cement mortar were implemented using seven different replacement ratios with CKC from cement to test the compressive strength at the age of 7 days as an indicator of the pozzolanic activity of CKC, and based on the results obtained, the best substitution ratios were chosen to test them at the age of 28 days. *Table 5* shows the mixing ratios and the compressive strength resistance results for the different mixtures.

The basic mixing proportions of cement mortar were determined using ASTM C1240 standard, where the compressive strength of the mortar was measured at the age of 7 days using the average value of three cubes with dimensions of 50 x 50 x 50 mm using metakaolin substitution ratios of cement 5%, 10%, 15%, 20%, 25%, 30%, 35% and compared with the control mix that does not contain CKC.

The results of the compressive strength tests of the cement mortar showed that the substitution ratios (20%, 25%, 30%, 35%) achieved the best resistance ratios compared to the control mixture, ranging between 93% and 97.3%. *Fig. 5* shows the relative compressive strength of the mixtures containing CKC and the control mixture at the age of 7 days.

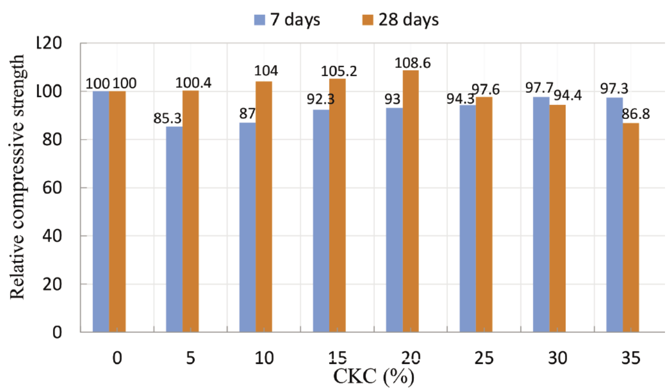

Fig. 5 Relative compressive strength of cement mortars at 7 and 28 days
5. ábra Cementhabarcsok 7 és 28 napos relatív nyomószilárdsága

Mixes	Cement (g)	CKC (g)	Sand (g)	Water (g)	Compressive strength 7 days (MPa)	Compressive strength 28 days (MPa)
Control	500	–	1375	242	35 (100%)	50 (100%)
M-5	475	25	1375	242	30.2 (85.3%)	50.2 (100.4%)
M-10	450	50	1375	242	30.5 (87%)	52 (104%)
M-15	425	75	1375	242	32.3 (92.3%)	52.6 (105.2%)
M-20	400	100	1375	242	32.6 (93%)	54.3 (108.6%)
M-25	375	125	1375	242	33 (94.3%)	48.5 (97.6%)
M-30	350	150	1375	242	34.2 (97.7%)	47.2 (94.4%)
M-35	325	175	1375	242	33 (97.3%)	43.4 (86.8%)

Table 5 The mix proportions and compressive strengths
5. táblázat Keverési arányok és nyomószilárdságok

Mixes	Cement OPC	CKC	Fine aggregate	Coarse aggregate	Water	Chemical additive (liter)
Control	350	–	751	1126	168	6.2
C-5	332.5	17.5	751	1126	168	6.2
C-10	315	35	751	1126	168	6.2
C-15	297.5	52.5	751	1126	168	6.2
C-20	280	70	751	1126	168	6.2

Table 6 Ingredients of concrete mixes (kg/m^3)
6. táblázat Betonkeverékek összetevői (kg/m^3)

In the light of these results, the compressive strength of the four mixtures and the control mixture was measured at the age of 28 days using the average value of three molds with dimensions of 40 x 40 x 160 mm according to the standard specification EN196-1. *Fig. 11* shows that the replacement ratio of 20% of the cement with CKC is considered the best ratio, as the compressive strength was almost the same as the control mixture or slightly superior to it, while the replacement ratios of 25% and 30% gave lower values for the compressive strength reaching 94.4% and 97.7% respectively of the compressive strength of the control mix.

3.2 Concrete mixes

Based on the results of the pozzolanic tests and the compressive strength tests of cement mortar and on the economic consideration of the cost of concrete mixtures, the concrete mixtures were studied with replacement rates of cement with CKC 5%, 10%, 15%, 20% in the fresh state and the hardened state at the ages of 7, 28 and 90 days.

Table 6 shows the components of concrete mixtures per cubic meter. The mixtures were designed using the absolute volume method. The cement content of the control mix was 350 kg/m^3, the ratio of large to small aggregate (1:2.5). The ratio of water/cement materials (0.48) for all mixtures, chemical additives were used at a rate of 1.77 liters per 100 kg of cement materials. *Table 6* shows the results of concrete tests in its fresh and hardened states.

3.3 Concrete mixes tests

3.3.1 Slump test

The settling value of the mixtures was determined immediately after mixing according to ESS 1658. The results indicate that the slump values for the mixtures containing different percentages of CKC increased from the control mixture by a range of 20.6% to 26.3%.

3.3.2 Entrained air

The entrained air determination test was carried out according to ASTM C231. It is clear from *Table 7* that the content of air trapped in the mixtures that contain percentages of CKC was lower than in the control mixture by 6.5 to 21.7% with a replacement ratio of CKC of 5 to 15%.

3.3.3 Compressive strength

The compressive strength of the concrete was measured at the ages of 7, 28 and 90 days using the average value of three cubes with dimensions of 150×150×150 mm for each mixture at each age. The results showed that the early strength at the age of 7 days for the samples of the control mixture and the samples that contained 5% and 10% replacement rates achieved 80.5 and 80% respectively of the pressure resistance at the age of 28 days, while the samples that contained 15% and 20% replacement rates achieved 94.4% and 97.8% respectively of strength at the age of 28 days. Compared to the control mixture, the 5% ratio did not have a significant effect on the compressive strength at different ages. The use of 10% and 15% replacement ratios improved the strength by an average of 120.8 and 118.7% respectively at the age of 28 days.

At the age of 90 days, the results of the mixtures containing 15% replacement showed an improvement of 15% compared to the control mixtures. While the mixtures containing 20% substitution did not show any difference from the control mixtures.

4. Conclusions

In the light of the study of the properties of the local calcined kaolinitic clay using vertical kiln by fluidization CKC, it can be concluded that:

- CKC can be used as a replacement percentage of OPC cement.
- The optimal replacement ratios for cement with CKC 10% and 15%, which improved the compressive strength by an average of 120.8 and 118.7% at the age of 28 days, compared to the control mix.
- The use of CKC at replacement rates of 15% and 20% of the cement caused gaining of early strength at an age of 7 days of 94.4% and 97.8% of the strength of concrete at the age of 28 days.
- Based on the results of the preliminary stage of the study, the following can be recommended:
- Assessment of the optimal dosage out of the two ratios (10 & 15%) requires a study of their fulfillment of many characteristics as well as their effect on durability and sustainability and their efficiency in facing harmful elements and media. And then determine the best ratio from the technical and economic point of view.

Mixes	Slump (mm)	Air content (%)	Compressive strength (MPa) Concrete age (days)		
			7	28	90
Control	160	2.30	30.2	37.5	42.8
C-5	194 (+21.3%)	2.15 (- 6.5%)	31.0 (80.5% of 28 days)	38.5 (102.6%)	43.2 (100.9%)
C-10	198 (+23.8%)	1.90 (-17.4%)	36.3 (80.1% of 28 days	45.3 (120.8%)	46.3 (108.2%)
C-15	202 (+26.3%)	1.80 (-21.7%)	40.1 (94.4% of 28 days	44.5 (118.7%)	50.5 (118%)
C-20	193 (+20.6%)	2.00 (-13%)	40.3 (97.8% of 28 days	41.2 (109.9%)	45.3 (105.8%)

Table 7 Hardened concrete test results
7. táblázat A megszilárdult beton vizsgálati eredményei

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