Characterization of OPC Matrix Containing Dealuminated Kaolin

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Abstract

The suitability of replacing Portland cement by dealuminated calcined kaolin as received waste obtained from an alum production factory through the extraction of aluminium, also by dealuminated samples treated with lime solution, is investigated. The chemical and mineralogical compositions of the samples are measured. Their pozzolanic reactivity and their surface areas were determined. The effect of replacement on setting time, flowability, rate of flowability loss and strength of mortars was tested and compared to control OPC samples and others containing silica fumes. It was found that the as received dealuminated kaolin and that treated with lime possess higher pozzolanic reactivity and show larger surface areas than silica fumes. The incorporation of the as received dealuminated kaolin (DK) in OPC paste accelerates the setting time; while the lime-treated samples lead to retardation. The flowability of the OPC mortar is little affected by the as received DK samples and is strongly reduced by the lime-treated one and silica fumes. The three admixtures cause strong flowability loss with time. The 56d-compressive and tensile strengths of the mortars improve with 5 and 10% OPC replacement by DK.

Keywords: By-product;, Cement replacement material; Dealuminated kaolin; Silica fumes; Pozzolanic reactivity.

1. Introduction

In view of the worldwide interest of having clean and healthy environment, the search of useful application of waste by-product materials from different industrial sectors has become necessary. The manufacture of Portland cement is an energy-intensive process and considerable efforts are being made to find cement substitution materials. Fine pozzolanic materials are known to replace part of cement in concrete due to their ability to increase the bulk density by filling voids as well as their contribution in strength development [1]. According to ASTM C595, a pozzolan is defined as "a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but in the presence of moisture, it reacts chemically with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties". Most of the known

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pozzaolana are waste by-product obtained from industrial and agricultural sectors such as silica fumes, fly ash and rice husk ash. Granulated blast-furnace slag with high percentage of glassy phase, as well as activated clays, are also used as cement replacement materials [2].

Another industrial waste, quite abundant in Egypt is the dealuminated kaolin (DK). It is obtained from the alum production factories and causes a great environmental problem. This waste is produced through the extraction of aluminium from calcined kaolin by means of sulfuric acid. Such treatment leaves behind silicate-rich acidic paste, which upon exposure to air dries intensively. The dealumination process of the calcined kaolin increases the surface area of the resulting material. The amount of increase in the surface area is related to the type of dealuminated chemical reactants used (HCl, H_2SO_4 and Na_2HSO_4), also to the reaction time and the amount of aluminium extracted. Generally the dealumination of calcined kaolin by acid leaching produces amorphous silica with a high surface area expected to be of highly reactive with lime. The main effect of the acid attack on the calcined kaolin is the dissolution of the aluminium from its octahedral and tetrahedral sites and the formation of free silica [3].

The present knowledge indicate that there is few published research in the literature which deals with the chemical, physical, pozzolanic activity and mineral characterization of dealuminated kaolin (DK) as a raw material suitable for use as cement replacement material [4-6]. Most of the studies focus on the DK/lime systems. To gain more understanding on these aspects and the possibility of reuse of this waste as cement admixture, the present study was conducted. The objectives of the study are to characterize the DK and to find out the extent of its suitability for use as a cement replacement material due to its silicate-rich content. The fresh and hardened state related characteristics of OPC matrix containing DK are elucidated.

2 EXPERIMENTAL

Ordinary Portland cement (OPC) and silica fumes (SF) complying with BS 12 (1978) and ASTM C618 (1992a) were supplied from the local market. The chemical analysis of the used materials is summarized in Table 1. Clean siliceous sand complying with ASTM C778-80 and tap water were used for the mortar mix.

The dealuminated kaolin (DK) was collected from large storage areas at the Egyptian alum Company (Abu Zaabal). The sample was sieved on 75 µm sieve to avoid the presence of large particles. The as received specimen denoted as DKut, was acidic in nature with a pH-value equal to 5. Part of the sample was neutralized with limewater up to a pH=8 and was denoted as DKt. All wet samples were dried at $110^{\circ}C$ for one day and then analyzed wet chemically. The results obtained are listed in Table 1.

The pozzolanic reactivity of the samples was determined chemically as follows: 2 gm. from the sample were mixed 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 60ºC. This chemical method is described in detail elsewhere [7]. The initial and 5d-reactivity were expressed in terms of the free lime measured in the sample relative to that of SF i.e. they are formulated as percentage of the lime reacted with the investigated solid compared to the amount of lime reacted with the silica fumes at similar time of investigation. By this means of investigation there is no absolute value for the pozzolanic reactivity of silica fumes.

The surface area was measured by means of BET method which implies 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 50º (2θ).

The replacement of OPC was made by addition of 0, 5, 10, 15, and 20% DKut and DKt in cement paste and the mortar mixes. Another series of OPC mortars containing either 10 or, and 20% SF was also regarded. The details of the mix proportions of the mortars are summarized in Table 2.

   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 DKut and DKt on the water demand and the setting behaviour of the pastes. The mixing procedures were carried out according to ISO 9597 (1989) and ASTM C305-82.

Table 1. THE CHEMICAL ANALYSIS OF THE USED RAW MATERIALS (OPC, DKut, DKt AND SF).

Oxide, %	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	L.O.I
OPC	20.56	5.59	2.65	63.13	1.94	0.43	0.22	2.69	2.61
SF	96.4	0.77	1.05	0.07	0.03	0.06	0.24	0.6	1.21
DKut	72.10	13.0	2.01	2.84	0.3	0.13	0.021	3.08	6.1
DKt	64.8	16.0	1.00	6.63	0.36	0.02	0.024	3.42	7.16

   The effect of the dealuminated kaolin sample on the pH-value of the cement slurry was performed as follows: 20gm of cement were added to 100 ml of distilled water (w/b = 5) in glass bottles, the dealuminated kaolin was added to the cement as a replacement material with the previously mentioned ratios, i.e. with 5, 10 and 20% additions. The components were mixed manually and the change in the pH- values was recorded by means of a digital pH meter after 0, 30, 60, 90 and 120 minutes.

Table 2. THE MIX PROPORTIONS OF THE MORTARS.

Mix No.	Mortar mix code	w/b* ratio	OPC, %	Sand/Cement ratio	Binding materials, % by weight of OPC		
					DKut	DKt	SF
1	OPC	0.5	100	2.25	-	-	-
2	5DKut	0.5	95	2.25	5	-	-
3	10DKut	0.5	90	2.25	10	-	-
4	15DKut	0.5	85	2.25	15	-	-
5	20DKut	0.5	80	2.25	20	-	-
6	5DKt	0.5	95	2.25	-	5	-
7	10DKt	0.5	90	2.25	-	10	-
8	15DKt	0.5	85	2.25	-	15	-
9	20DKt	0.5	80	2.25	-	20	-
10	10SF	0.5	90	2.25	-	-	10
11	20SF	0.5	80	2.25	-	-	20

* w/b = water/(OPC + weight of supplementary material).

   The Mortar Flow Table Apparatus (MFTA) was proposed in this investigation for determining the flowability and the rate of flowability loss of the OPC, OPC/DKut, OPC/DKt and OPC/SF mortars. The details of MFTA are fully described in ASTM C230-80. Immediately after mixing; the flowability of the mortar was measured using MFTA, according to the ASTM C109-99. To examine the rate of flowability loss occurring during the fresh state of mortar mixes, the flow diameters were measured at certain elapsed periods from mixing: 0, 30, 60, 90 and 120 min.

   The processes of casting and compaction of the cube and briquette specimens used in the determination of the compressive and the direct tensile strengths respectively were carried out according to ASTM C109-99. All processes of mixing, casting, curing, and testing were conducted at ambient conditions. After mixing the cubic mortars (5x5x5 cm) and briquette specimens were immediately prepared, covered with plastic sheets for 24 hours, demolded and then cured in water until the age of testing. The compressive strength specimens were tested at various ages of 3, 7, 28, and 56 days according to ASTM C109-99, the specimens prepared to test the direct tensile strength were tested at the age of 56 days according to ASTM C307-99.

3   RESULTS

3.1   Characterization of the Dealuminated Kaolin

The chemical analysis of the raw materials used is illustrated in Table 1. The results show that the amounts of silica reach 72.10%, 64.8%, and 96.4% for the untreated dealuminated kaolin (DKut), treated dealuminated kaolin (DKt) and silica fumes (SF) respectively. The silica content of OPC is equal to 20.56%.

The mineralogical composition of the untreated and treated dealuminated kaolin samples are illustrated in the X-ray diffractograms of Figure 1. The results indicate that the as received sample is composed mainly of quartz with a small proportion of titanium oxide appearing in the form of anatase (A) and small amount of calcium sulfate hemihydrate (H) as well as calcium sulfate hydrate phase appearing in the form of gypsum (G). A considerable amount of amorphous material is present in the sample as indicated from the hump around 20-25 two theta. These results agree with those reported in the literature [4]. The sample treated with lime (DKt) is composed of a higher amount of calcium sulfate hemihydrate (H). The X-ray diffractogram of the silica fumes (SF) illustrated in the same figure is seen to be completely amorphous.
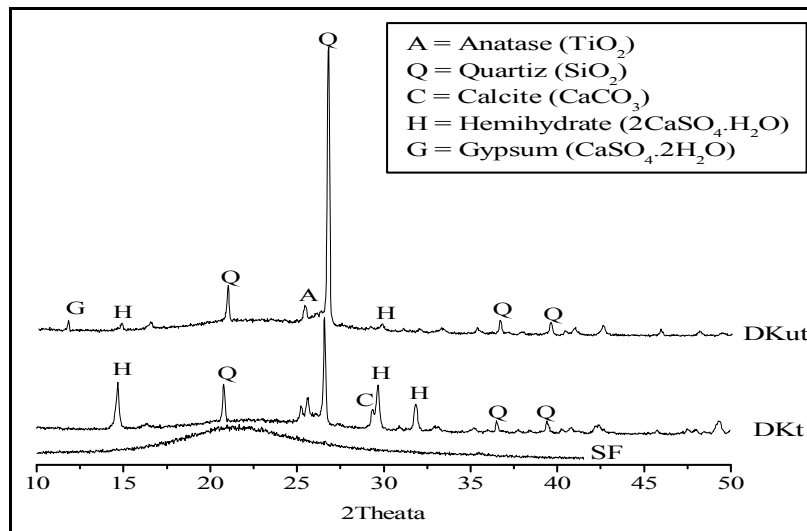

A = Anatase (TiO_2)
Q = Quartiz (SiO_2)
C = Calcite ($CaCO_3$)
H = Hemihydrate ($2CaSO_4.H_2O$)
G = Gypsum ($CaSO_4.2H_2O$)

Fig. 1. X-ray diffraction diagrams of DKut, DKt and SF.

The values estimated for the pozzolanic reactivity and the surface areas of the dealuminated kaolin samples are shown in Table 3. The 5-days reactivity of the untreated and treated samples amount to 127, 117% relative to the values measured for the silica fumes considered as reference. Their BET surface areas are equal to 42 m^2/gm and 40.1 m^2/gm i.e. almost double that of the silica fumes known to be around 20 m^2/g. The densities of the two samples are found to be 1.95 gm/cm^3 and 2.1 gm/cm^3 for the untreated (DKut) and treated (DKt) samples, respectively.

Table 3. THE POZZOLANIC REACTIVITY AND PHYSICAL PROPERTIES OF THE DKut AND DKt.

Property	DKut	DKt
Pozzolanic reactivity, %	127	117
BET specific surface area m^2/gm	42	40.1
Density gm/cm^3	1.95	2.1

Table 4. EFFECT OF DK ON THE Ph VALUE OF THE CEMENT SLURRY.

Slurry No.	Binding materials, % by weight of OPC			Time, min				
	OPC	DKut	DKt	0	30	60	90	120
1	100	-----	-----	12.7	12.7	12.7	12.6	12.6
2	95	5	-----	12.6	12.7	12.6	12.6	12.6
3	90	10	-----	12.5	12.6	12.6	12.6	12.6
4	85	15	-----	12.4	12.6	12.6	12.6	12.6
5	80	20	-----	12.3	12.6	12.6	12.6	12.6
6	95	-----	5	12.6	12.6	12.6	12.6	12.7
7	90	-----	10	12.6	12.6	12.6	12.6	12.7
8	85	-----	15	12.6	12.6	12.6	12.6	12.7
9	80	-----	20	12.6	12.6	12.6	12.6	12.7

3.2   Effect of DK on the pH-value of Cement Slurry

The effect of DKut and DKt on the pH-value of the cement slurry with time is shown in Table 4. The results indicate an instantaneous increase of the pH-value of the as received DK (DKut) in the OPC mixes with all replacement ratios; the instantaneous measurement being considered as the zero time i.e. the starting time of addition. The high pH-value then remains more or less constant for a longer duration up to 120 minutes. The pH-value of the neutralized DK (denoted by DKt) was already alkaline; the respective OPC mixes showed values around 12.6 at duration time equal to and longer than the instantaneous at the four replacement ratios.

3.3   Setting Time

Figure 2 shows the effect of addition of both types of DK (treated and untreated) on the water/binder ratio (w/b) ratio required for the standard consistency of the replaced OPC. It is clear from the figure that the w/b ratio increases with increasing the DK contents. The w/b ratios of DKut mixes are slightly higher than those of DKt for all replacement percentage.
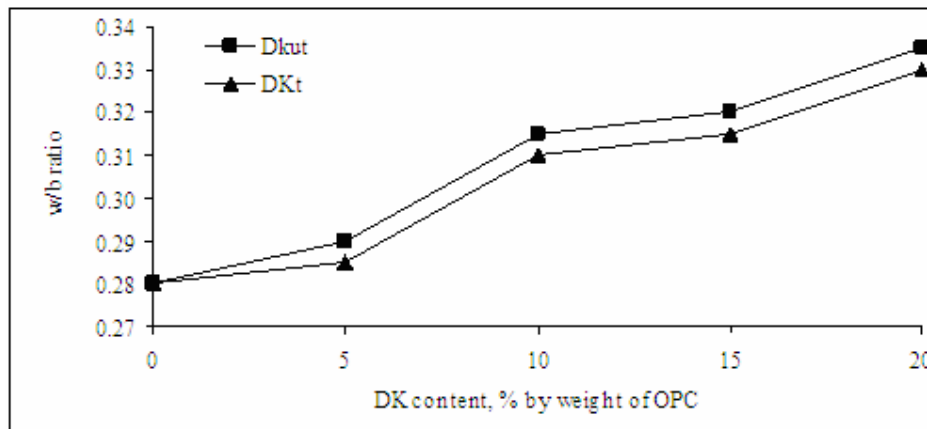

Fig. 2. Effect of DK on the w/b ratio required for standard consistency of OPC paste.

Figure 3 shows the results of the initial and final setting times of the pastes with standard consistency containing the DKut and the DKt at different replacement of OPC. The setting times of the OPC paste without replacement was found to be at 120 and 160 minutes respectively. The addition of 5% DKt leads to a slight decrease of both setting times and is followed by a steady increase at higher replacement ratios up to 20%. In other words, the DKt has a slight accelerating effect at 5% replacement then acts as a retarder.

For the pastes containing the DKut, an opposite effect is produced: the initial and final setting time of OPC/DKut pastes decrease with the increase of DKut, i.e. the DKut behaves clearly as an accelerator. The accelerated effect of DKut appears to be significant on the initial setting time

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where about 25% reduction in the initial setting time was observed at a replacement ratio of 10%. The reduction in final setting time was only 18% at the same replacement percent.

Generally the setting times for both DK pastes are within the recommended range for the OPC paste (initial setting time ≥ 45 minutes and final setting time ≤ 10 hours) according to ISO 9597 (1989) and ASTM C 305-82.
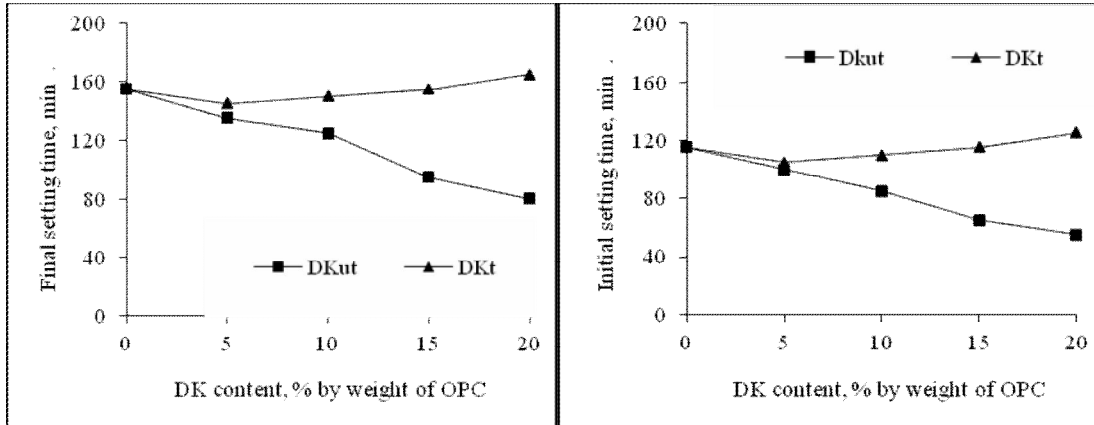

Fig.3 Effect of incorporating DK on OPC cement paste setting times

3.4 Characteristics of Fresh Mortar

Two different parameters of the fresh cementitious materials containing different contents of the dealuminated kaolin were investigated. The parameters included the initial flowability (measured immediately after mixing) and the rate of flowability loss. The OPC mortar mixes made with various contents of DK were prepared and assessed. The previous parameters were also evaluated for OPC mortar mixes containing selected contents of SF (10 and 20%) for the purpose of comparison with the OPC/DK mixes.

Figure 4 illustrates the effect of DKut, DKt and SF on the flowability of OPC mortars. It is clear from the figure that the incorporation of DKut had only a slight effect on the initial flowability of the OPC mortar; the DKt and the SF, however, lead to a significant reduction in the flowability and their effect increases with increasing their contents. The reduction in the initial flowability of OPC mortar reaches about 5, 20 and 30% for 20% of replacement of the OPC by DKut or DKt or SF, respectively. At a constant replacement of the dealuminated kaolin, the initial flowability of the mortars is higher than that in the presence of SF.
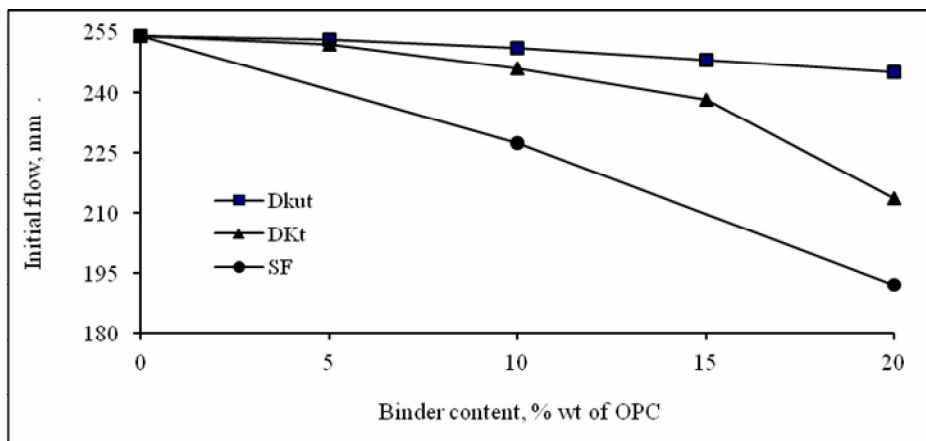

Fig. 4. Flowability of OPC mortar containing different contents of DKut, DKt and SF.

The rate of flowability loss of OPC, OPC/DKut, OPC/DKt, and OPC/SF mortars was performed by measuring the instantaneous flowability at different elapsed periods from mixing (EP), 0, 30, 60, 90 and 120 min. The results are presented in Figure 5 where, the values of the relative flowability (% of instant flowability to initial flowability) against EP are plotted. It is clear from the figures that the relative flowability of the DK-bearing samples decreases with increasing EP. This effect is pronounced with increasing the DK content but is, however, not influenced as a result of introducing SF into OPC matrix. In other words, the rate of flowability loss of the cementitious materials containing the DK is significantly higher than that with SF and the rate of loss of the OPC/DKut mixes is higher than that of the OPC/DKt at all replacement levels. At 90 minutes from mixing, the relative flowability reach around 60, 78, and 83% from the OPC mortars at 10% replacement of either DKut, DKt or SF, respectively, as shown in Figure 6.
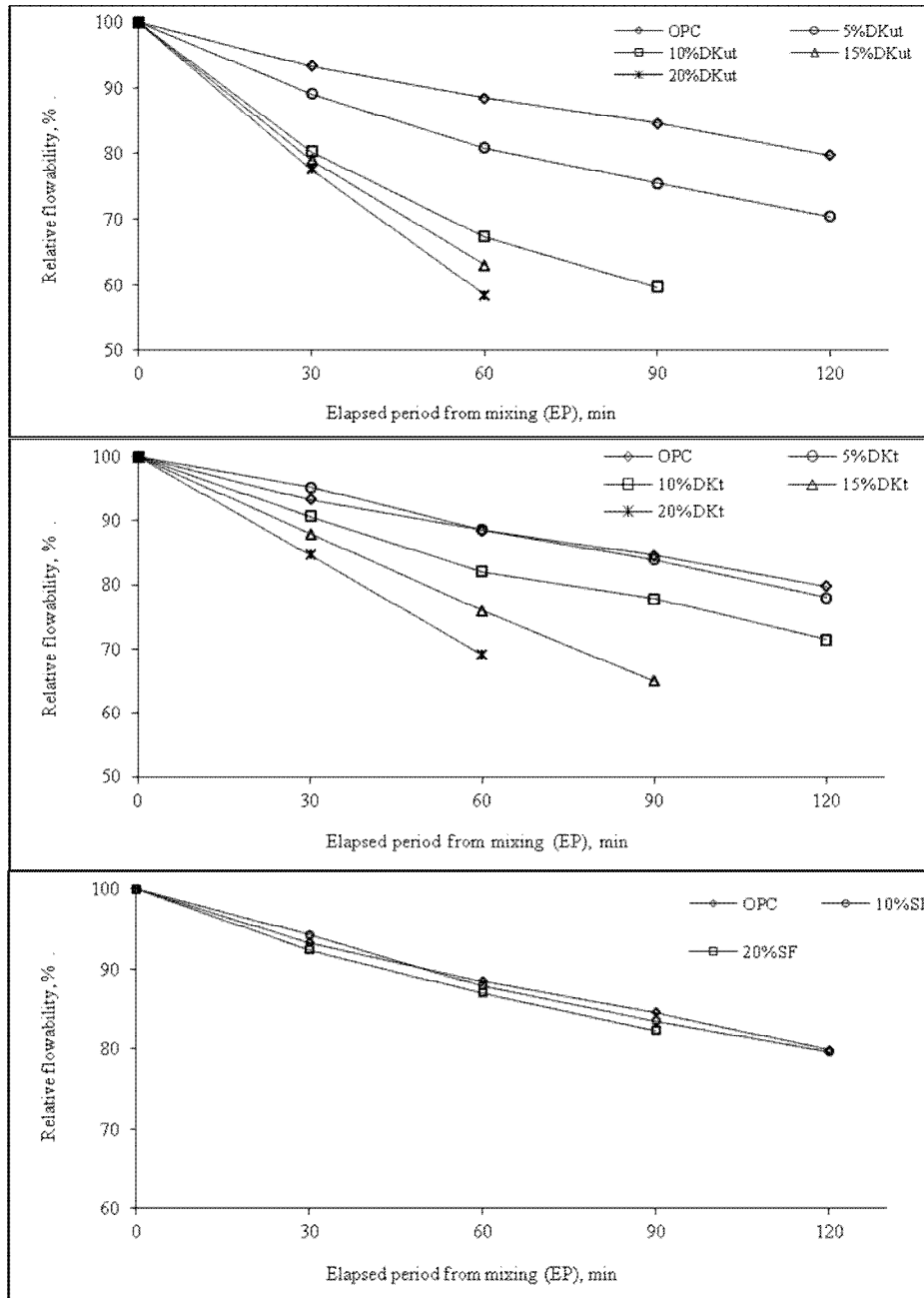

Fig. 5. Flowability loss of OPC mortars containing different contents of DKut, DKt and SF.
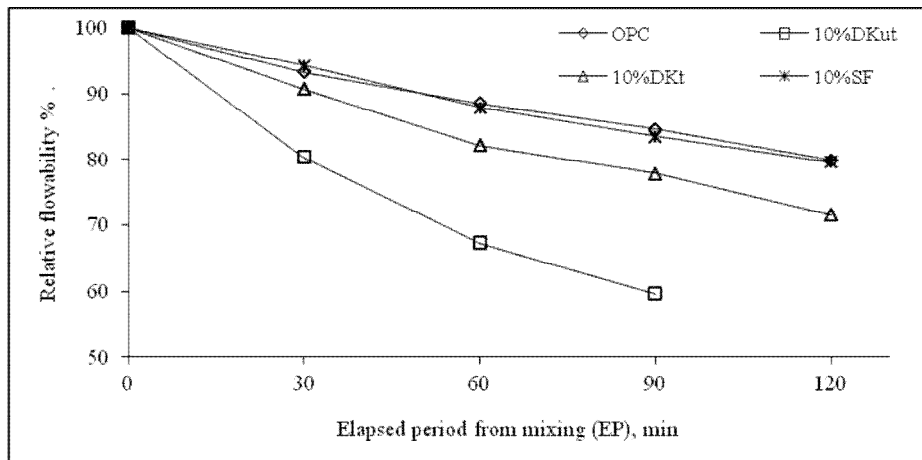
Fig. 6. Flowability loss of OPC/10% DKut, OPC/10% DKt and OPC/10%SF mortars.

3.5 Strength Characteristics

The mechanical properties of the cementitious materials made with DKut, DKt and SF are expressed in terms of their compressive and tensile strengths measured. The results obtained are illustrated in Figures 7 to 9. Figure 7 shows that the 56-days compressive strength of the OPC mortar increases with increasing the replacement ratio of the dealuminated kaolin in the OPC mix. It attains a peak value at 10% DK beyond which the compressive strength started to decrease with increasing the DK content. The effect of both forms of dealuminated kaolin on the compressive strength development of the OPC mortar is similar. The compressive strength of the OPC mortar specimens made with either 5 or 10% DKut are generally higher than that of pure OPC mortar specimens by about 7.0 and 12.0%, respectively.

To study the role of water curing period on the OPC matrix incorporating the DK, the compressive strength of the OPC mortars prepared with different contents of DK and cured in water for various periods was assessed; the results obtained are illustrated in Figure 8. The curing period is seen to have a decisive role in the improvement of the compressive strength of the cementitious material containing dealuminated kaolin: the compressive strength of the OPC/DK specimen's increases with increasing the curing period. Comparing the 56-days compressive strength results of DK with those of the SF specimens it is found that the DK specimens show lower values than SF specimens.

Figure 9 shows the behaviour of the OPC mortars containing the dealuminated kaolin under tensile stresses. The behaviour is similar to that of the compressive stress. The 56-days tensile strength results of the OPC mortar increases with increasing the replacement ratio of dealuminated kaolin. It reaches a peak value at 10% DK beyond which the strength starts to decrease. The amount of improvement in the tensile strength is around 5 and 10% at 5 and 10% of OPC replacement by the both types of dealuminated kaolin.

4 DISCUSSION

Kaolin is a natural clay mineral composed of aluminosilicate with well defined lattice parameters identified by means of X-ray diffraction. The calcination of kaolin at ~700°C leads to its dehydroxylation and leaves behind unstable amorphous structure susceptible to react with basic oxides to form more stable configurations. When properly activated, the calcined kaolin, known as metakaolin, possesses good pozzolanic properties and is a well known artificial pozzolana.

The most important characteristic of this calcined product is the chemical composition (SiO_2 + Al_2O_3 about 90-95%). In the alum industry, aluminium is extracted from the calcined kaolin as soluble aluminium sulfate through treatment with sulfuric acid. The dealumination process is not carried out in the natural mineral because of its stable structure whilst the amorphous state of the

calcined solid facilitates the extraction process. The dealuminated kaolin (DK) waste is a silica-rich solid, acidic in nature and contains some residual aluminium which was have not been extracted. As a result of the acid attack, the silica is present in a disordered non-polymerized weakly- or non-bonded form. The surface area of the multiphase waste is high due to the fine particulates left after the acid treatment in addition to the amorphous nature of the heat treated mineral.
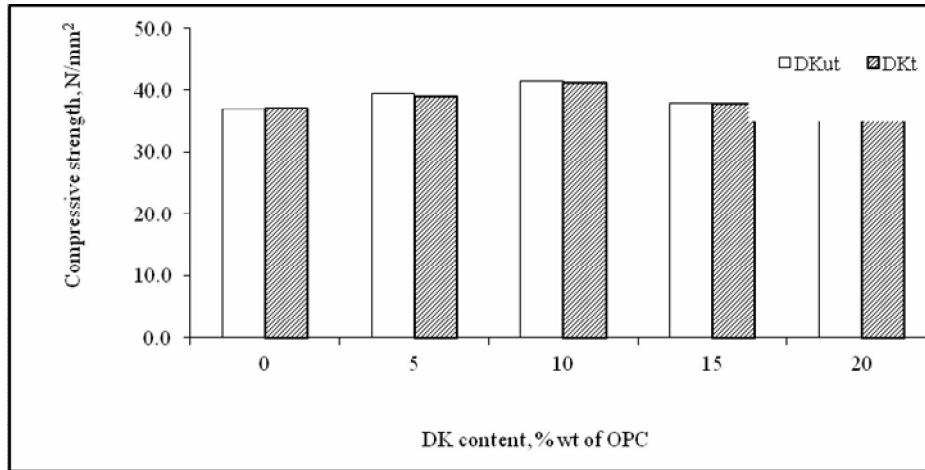

Fig. 7. Effect of DK on 56-days compressive strength of OPC mortar.
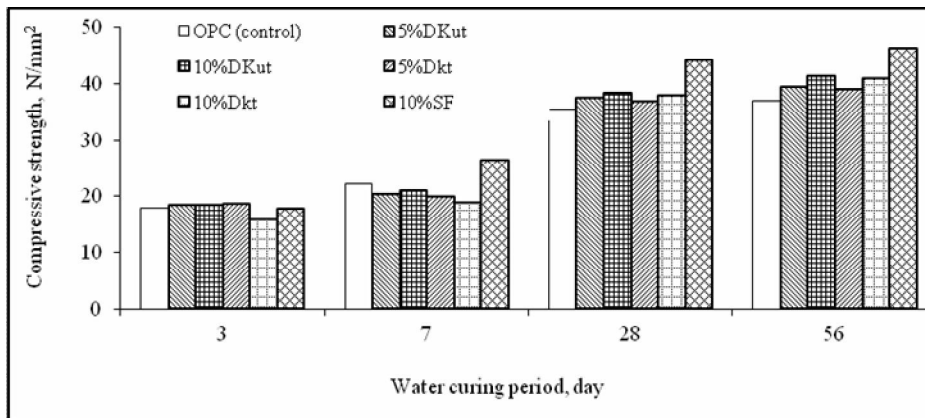

Fig. 8. Effect of binder type and content on compressive strength of mortar cured with water for various periods.
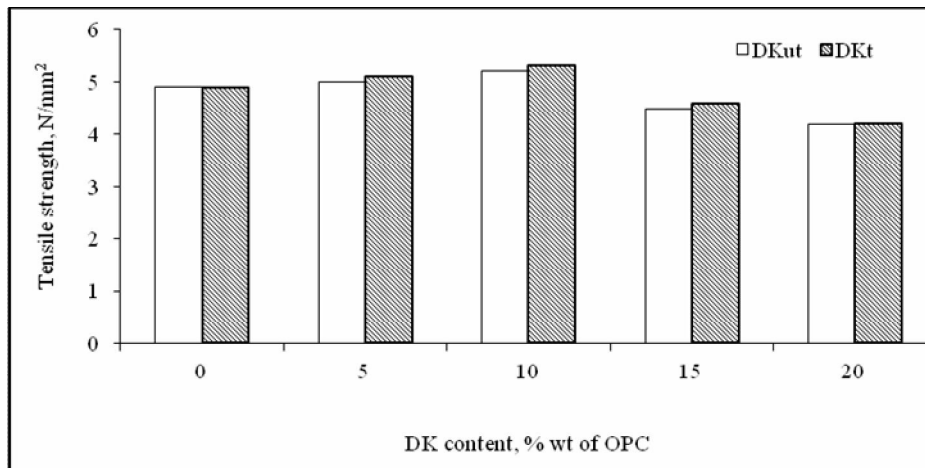

Fig. 9. Effect of DK type and content on 56-days mortar tensile strength.

The potential of the waste to bind calcium and hydroxyl ions is high. The binding capacity follows several mechanisms including neutralization and salt formation. During the neutralization process the hydrogen ions adsorbed on the surface of the acidic particulates are replaced by hydroxyl ions, the trivalent aluminium ions transforms to aluminate and the aluminates react with calcium to precipitate calcium aluminate hydrates of weak cementing properties which in the presence of gypsum they form ettringite. The described mechanism differs totally from that of silica fumes with lime, where the pure condensed fumes of silica react to form calcium silicate hydrate with strong cementing properties. Under these conditions the binding capacity of lime, known generally as the pozzolanic reactivity, cannot be interpreted exclusively as an initiator for the polymerization of the weakly bonded silica to form the calcium silicate hydrates. It involves also the neutralization process as well as the formation of gypsum, calcium aluminate hydrates and ettringite.

The setting time of OPC depends on the phase composition, the concentration of the liquid phase and the pH-value; the amount of alkalis, lime, and soluble alumina play dominant role in the process. Setting is accelerated by alkali hydroxides and soluble alumina; it is retarded by lime, ferric hydroxide, and other anions such as sulfates and borates. Excess sulfates lead to secondary gypsum and cause false setting. Setting is delayed by fly ashes possibly due to the presence of carbon particles in the ashes and is prolonged by silica fumes.

The DK contains a significant content of alumina compared to OPC (13% Al_2O_3 in DKut, 16% in the DKt, 5.59% in the OPC). As previously mentioned, the alumina of the acidic DK (DKut) is in the form of soluble aluminum sulfate which upon replacement of part of OPC it increases the crystalline character of the hydration products and accelerates the hydration and setting. On the other hand, the lime treated DK (DKt) which contains calcium sulfoaluminate hydrates and probably excess lime delays setting.

In general, natural pozzolana accelerates therefore the rate of hydration of the clinker. All pozzolanic properties e.g. BET surface area, chemical composition and physical state of the surface, the more or less rapid release of alkalis contribute to the acceleration of the initial hydration rate of cement. As a result of DK addition the increase in the water demand of the paste due to the higher surface area is expected to contribute to the acceleration of hydration with an increased heat of hydration. The resulting total heat evolved shall be attributed to the heat of reaction and the heat of neutralization.

Pozzolanic materials with fineness similar to that of Portland cement does not change its workability very much; however, very fine substances such as microsilica and diatomaceous earths increase dramatically the water demand and decrease the flowability. The pozzolanic reactivity is indicated by the surface area and potential reactivity with lime. Although the surface area of both types of DK, untreated and treated, is almost double that of silica fumes, the workability of the OPC mortar does not vary with the addition of the untreated but is negatively affected by the lime treated (DKt) and more by SF. This result is explained by the acidic character of the DKut which encompasses fine precipitated salts and grains of high surface area with no cementing properties. The difference in the flowability of mixes with the dealuminated kaolin and that of OPC/SF mixes is attributed to the difference in the structure and the nature of the amorphous silica in these materials [4]. The amorphous silica in the DKut has a lower degree of polymerization than that found in SF and the presence of hydroxilated silica surface in the DKut can hold water as was observed by other researchers in the infrared spectrometer (IR) spectra [4]. The flowability of OPC decreases in presence of the neutralized DK and approaches the performance of SF.

5   CONCLUSIONS

1. DKut is acidic in character. Its potential to react with lime is explained by the affinity of the solid to neutralization, by the formation of the calcium aluminate and sulfoaluminate hydrates

and calcium silicate hydrates. The pozzolanic reactivity of SF is a result of its reaction with lime to form exclusively calcium silicate hydrates.

2. The remarkably high surface area of the dealuminated kaolin (almost double that of silica fumes) is not indicative of a better contribution to the strength development. It is attributed to the presence of fine particulates formed as a result of the dealumination process.
3. The pH value of the OPC-DK mix is alkaline
4. The setting times of OPC is accelerated by DKut incorporation
5. The flowability of OPC is hardly affected by the acidic as received dealuminated kaolin and is strongly reduced by the treated DK and SF
6. The rate of flowability loss of OPC mix is strongly affected by both types of DK and by the SF.
7. 10% replacement of OPC by DK improves the compressive and tensile strengths of mortar.
8. Prolonged water curing period improves the compressive strength of cementitious material containing dealuminated kaolin.

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