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Exploitation of poor Greek kaolins: Strength development of metakaolin concrete and evaluation by means of *k*-value

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Abstract

In this paper, the effect of metakaolin on concrete properties is investigated. A poor Greek kaolin was thermally treated at defined conditions, and the produced metakaolin was superfine ground. In addition, a commercial metakaolin of high purity was used. Eight mixture proportions were used to produce high-performance concrete, where metakaolin replaced either cement or sand in percentages of 10% or 20% by weight of the control cement content. The strength development of metakaolin concrete was evaluated using the efficiency factor (*k*-value). The produced metakaolin as well as the commercial one imparts a similar behavior with respect to the concrete strength. Both metakaolins exhibit very high *k*-values (close to 3.0 at 28 days) and are characterised as highly reactive pozzolanic materials that can lead to concrete production with an excellent performance.

Keywords: Concrete; Kaolin; Metakaolin; Compressive strength; *k*-Value

1. Introduction

The worldwide demand for high-performance, cement-based materials has increased, and predictions are that it will reach a major industrial dimension during the early 21st century. Economics and environmental considerations have had a role in the mineral admixture usage as well as better engineering and performance properties.

The cementitious materials that are widely used, as concrete constituents, are fly ash, ggbs, and silica fume [1]. Metakaolin, produced by controlled thermal treatment of kaolin, is the most recent mineral admixture to be commercially introduced to the concrete construction industry. It has been claimed that concrete containing metakaolin exhibits premium-level engineering properties comparable to silica fume concrete [2–4].

According to the literature, the research work on metakaolin is focused on two main areas. The first one refers to the kaolin structure, the kaolinite to metakaolinite conversion, and the use of analytical techniques for the thorough examination of kaolin thermal treatment [5–13]. The second one concerns the pozzolanic behavior of metakaolin and its effect on cement and concrete properties [3,4,14–32]. Although there is a disagreement on specific issues, the knowledge level is satisfactory and continuously extended.

The concrete performance depends mainly on the environmental conditions, the microstructure, and the chemistry of the concrete. The two latter factors are strongly affected by the concrete components. It is obvious that the metakaolin presence affects the concrete performance. In particular, this effect on concrete properties can be practical, approached by the supplementary cementing materials (SCM) efficiency factor (*k*-value).

The *k*-value is defined as the part of the SCM in a pozzolanic concrete which can be considered as equivalent to Portland cement, having the same properties as the concrete without SCM (obviously, $k = 1$ for Portland cement) [33]. The quantity of the SCM in the mix can be multiplied by the *k*-value to estimate the equivalent cement content, which can be added to the cement content for the determination of the water–cement ratio, minimum required

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Table 1
Chemical analysis of kaolins (%)

	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	LOI	SO_3
KC	47.85	38.20	0.03	0.04	1.29	12.30	–
K	65.92	22.56	0.36	0.02	0.90	8.60	2.00

The methods specified by EN 450, EN 196, and EN 451 were followed.

cement content, etc. The property used for the estimation of *k*-values is the compressive strength [33,34]. However, durability properties can also be used, and relative *k*-values can be calculated. Knowing these *k*-values, the mix design for the preparation of the building product can be easier and more accurate.

In previous publications, a simplified scheme describing the activity of silica fume and fly ash (of low and high calcium) in terms of chemical reactions was proposed, yielding quantitative expressions for the estimation of the final chemical and volumetric composition of such SCM concretes [35–38]. Furthermore, a practical approach to the effect of SCM on the strength of Portland cement systems and on their resistance against carbonation and chloride penetration was presented using the concept of the SCM efficiency factor [39,40].

This work forms part of a research project, which aims towards the exploitation of poor Greek kaolins in concrete technology. Up to now, the optimization of the kaolin to metakaolin conversion [11,30,41], the study of the CH–metakaolin system [30], the effect of the crystallinity of the original kaolinite on the pozzolanic activity of metakaolinite [12,30], and the properties and behavior of metakaolin cements [42] have been carried out.

The present work deals with the behavior of two metakaolins: a produced metakaolin that originated from poor kaolin and a commercial one of high purity. More specifically, the strength development of metakaolin concrete, the evaluation of metakaolin activity according to accepted quantitative criterion (*k*-value), and the comparison of the produced metakaolin with the commercial one are studied.

2. Experimental

2.1. Materials

A poor Greek kaolin (K), which originated from Milos Island, was used. In addition, a commercial metakaolin (MKC) of high purity was also used as a reference material. The chemical analysis of the materials is given in Table 1.

Table 2
Mineralogical analysis of kaolins (%)

	Kaolinite	Alunite	Quartz	Illite
KC	96	–	–	3
K	52	5	41	–

Quartz (mainly) + cristobalite.

Table 3
Chemical analysis of OPC and characteristics of clinker

Cement		Clinker	
Chemical analysis (%)		Mineralogical composition (%)	
SiO_2	21.54	C_3S	57.8
Al_2O_3	4.83	C_2S	18.1
Fe_2O_3	3.89	C_3A	6.2
CaO	65.67	C_4AF	11.8
MgO	1.71	Moduli	
K_2O	0.60	LSF	0.949
Na_2O	0.07	SR	2.47
SO_3	2.74	AR	1.24
Cl^-	0.00	HM	2.17

Concerning the commercial metakaolin, for comparison reasons, the characteristics of the commercial kaolin (KC), instead of MKC, are given.

The semiquantitative mineralogical estimation of the materials is presented in Table 2. The estimation is based on the characteristic X-ray diffraction (XRD) peaks of each mineral, in combination with the bulk chemical analysis of the samples and has been presented in details in a previous work [12]. The Greek kaolin K mainly consists of kaolinite ($Al_2O_3·2SiO_2·2H_2O$) and quartz. K also contains K-alunite [$KAl_3(SO_4)_2(OH)_6$]. KC contains kaolinite and a detectable amount of illite.

Ordinary Portland cement (OPC; I/55) of industrial origin was used for the production of the mixtures. The chemical analysis of OPC and the characteristics of clinker are given in Table 3.

2.2. Metakaolin production

The optimum conditions of thermal treatment have been reported in previous works [11,30]. The kaolin K was thermally treated in a propilot plant furnace at $T = 650$ °C for 3 h. The complete transformation of kaolinite to metakaolinite was confirmed by XRD. The metakaolin that originated from K is referred to as MK. The metakaolinite content of the used metakaolins is 49% and 95% w/w for MK and MKC, respectively (Table 4). The estimation is based on the chemical and mineralogical analysis of the kaolins (Tables 1 and 2). In Table 4, the SiO_2 content (estimated from Table 1 data) and the active SiO_2 (measured according to EN 196-2) of the metakaolins are also given. The active silica is defined as the fraction of the SiO_2 that is soluble after treatment with hydrochloric acid and with boiling potassium hydroxide solution (EN 197-1).

The produced metakaolin MK was superfine ground, using the AJ100 Aerojet Mill Minisplit Classifier of British

Table 4
Metakaolinite, SiO_2, and active SiO_2 content of metakaolins

	Metakaolinite (%)	SiO_2 (%)	Active SiO_2 (%)
MKC	95	54.6	53
MK	49	72.1	30

Table 5
Metakaolin fineness characteristics

Sample	Fineness characteristics			Rosin–Rammler parameters	
	d_{20} (μm)	d_{50} (μm)	d_{80} (μm)	n	pp (μm)
MK	13.6	7.5	3.4	1.42	9.7
MKC	10.3	5.1	1.9	1.18	6.9

Rema. The fineness characteristics of the ground metakaolin as well as the MKC are given in Table 5.

2.3. Concrete preparation and properties

The concrete production was carried out in a mixer of a 50-l capacity. A constant volume unit (1 m^3) of concrete was chosen as a common comparison basis. When metakaolin was added to this unit, an equal mass of another component, either cement or sand, was removed to keep a similar total volume. In this way, four concrete mixtures were prepared for each metakaolin, where metakaolin replaced either cement or sand in percentages of 10% or 20% by weight of the control cement content. The water content (tap water at 20 °C) for all specimens was kept constant (175 kg/m^3). Normal graded calcareous aggregates, including fine (37%), medium (21%), and coarse (42%) aggregates, were used. The coarse aggregate maximum size was 31.5 mm. For the control specimen, the water–cement ratio (W/C) was 0.5 and the aggregate–cement ratio (A/C) was 5.5. A common superplasticizer was used at appropriate percentages to retain the slump of the fresh concrete between 50 and 90 mm (class S2 of EN 206). The mixture proportions of all concrete specimens are summarized in Tables 6 and 7 for cement and sand replacement, respectively.

The slump (ASTM C 143-90a), the density (BS 1881-103/1993), and the air content (ASTM C 138-92) of the fresh concrete were tested. Concerning the hardened concrete, the compressive strengths after 2, 7, 28, and 90 days (ASTM C 39) were measured. More specifically, the specimens for strength measurements were cast in 150-mm cubes, vibrated for 20 s on a vibration table, and then covered to minimize water evaporation. The molds were stripped after 24 h, and the specimens were immersed in lime-saturated water at 20 °C until testing. For each age, three specimens of each mixture were tested for compressive strength, and the mean value of these measurements is reported.

Table 6
Concrete mix proportions (cement replacement)

Sample	Content (kg/m^3)					Superplasticizer (%)	W/C	W/B
	C	P		Aggregates	W			
		MKC	MK					
OPC	350	–	–			0.057	0.50	
MKC-CR10[a]	315	35	–	Fine: 720		0.140	0.56	
MKC-CR20	280	70	–	Medium: 400	175	0.170	0.63	0.50
MK-CR10	315	–	35	Coarse: 800		0.181	0.56	
MK-CR20	280	–	70			0.400	0.63	

[a] CR10—Cement replacement, 10% by weight of the cement.

Table 7
Concrete mix proportions (sand replacement)

Sample	Content (kg/m^3)							Superplasticizer (%)	W/C	W/B
	C	P		Aggregates			W			
		MKC	MK	F	M	C				
MKC-SR10[a]	350	35	–	685	400	800		0.145	0.50	0.42
MKC-SR20	350	70	–	650	400	800	175	0.207	0.50	0.45
MK-SR10	350	–	35	685	400	800		0.222	0.50	0.42
MK-SR20	350	–	70	650	400	800		0.357	0.50	0.45

[a] SR10—Sand replacement, 10% by weight of the cement.

3. Results and discussion

3.1. Concrete properties

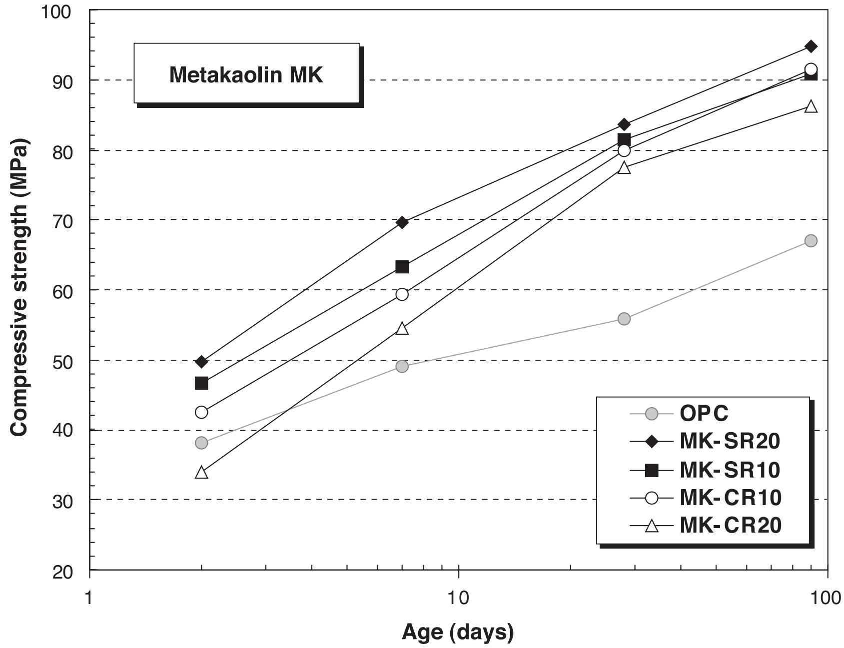
Table 8 presents the properties of the fresh concrete. The slump of most mixes is in the range of 50–90 mm (class S2 of EN 206-1) with the appropriate use of superplasticizers (Tables 6 and 7). The density of the fresh concrete varies from 2427 to 2453 kg/m^3. The concrete is well compacted as it is shown from the air content values.

The experimental results from compressive strength tests are summarized in Fig. 1. It is generally observed that when metakaolin substitutes either sand or cement, higher strengths than the OPC concrete are succeeded at all ages up to 90 days (with the exception of MKC-CR10, MKC-CR20, and MK-CR20 for the age of 2 days). There is a reasonable distribution of the strength increase according to the metakaolin content. As far as the metakaolin type is concerned, it is observed that MK has a more considerable effect on strength development than MKC (Fig. 1; strength at 90 days).

More specifically, the compressive strength at 28 days varies from 77.4 to 83.7 MPa for the MK concrete and from 74.0 to 91.6 MPa for the MKC concrete, while the OPC concrete presents a strength of 55.8 MPa. The compressive strength at 90 days varies from 86.3 to 94.8 MPa for the MK concrete and from 80.5 to 91.0 MPa for the MKC concrete, while the OPC concrete presents a strength of 67.0 MPa.

Table 8
Properties of fresh concrete

Sample	Slump (mm)	Unit weight (kg/m^3)	Air content %)
OPC	70	2.453	1.4
MKC-CR10	90	2.434	1.5
MKC-CR20	60	2.440	1.0
MK-CR10	50	2.433	1.5
MK-CR20	70	2.427	1.0
MKC-SR20	60	2.443	1.0
MKC-SR10	100	2.449	1.1
MK-SR20	40	2.439	1.0
MK-SR10	70	2.452	1.1



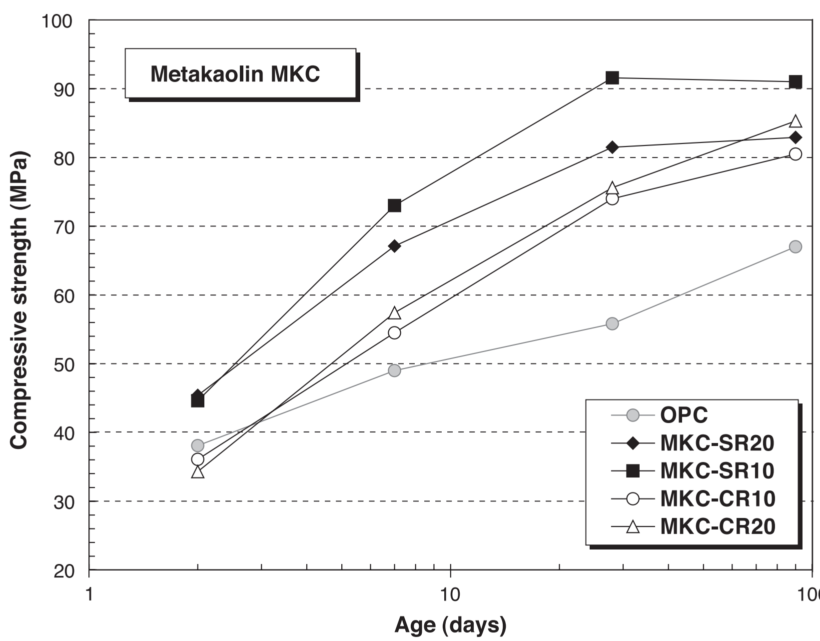


Fig. 1. Compressive strength development for concrete incorporating metakaolins MK and MKC.

Fig. 2 presents the relative strength of metakaolin concretes in relation to curing age, the metakaolin type, and the replacement level. Relative strength is the ratio of the strength of the metakaolin concrete to the strength of the control (OPC) concrete at each particular curing time. The rate of strength development in control concrete is mainly dependent on the hydration rate of OPC, while in OPC–metakaolin systems, it depends on the combination of OPC hydration and the pozzolanic reaction of metakaolin. Therefore, the relative strength–time plots provide an insight into the rates of reaction in the blended system relative to the plain OPC system [29]. According to the literature, the main factors that affect the contribution of metakaolin in strength are (a) the filler effect, (b) the (OPC) dilution effect, and (c) the pozzolanic reaction of metakaolin with CH [29].

For metakaolin concrete and sand replacement, the increased relative strength at 2 days (Fig. 2) is mainly attributed to the filler effect that leads to an initial acceleration of OPC hydration. For metakaolin concrete and cement replacement, at 2 days, the filler effect is surpassed by the dilution effect, and this leads to lower relative strength (with the exception of MK-CR10) than that observed in the case of control concrete. The high relative

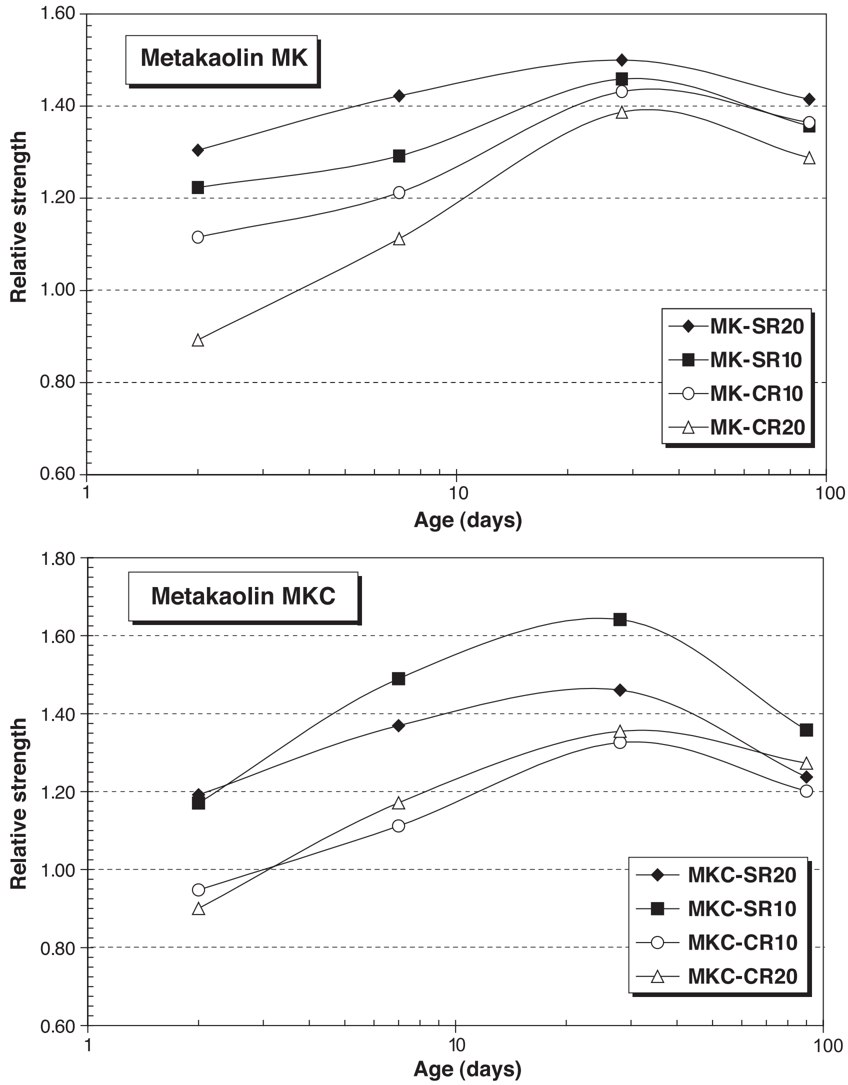


Fig. 2. Relative strength of metakaolin concretes in relation to curing age, metakaolin type, and replacement level (relative strength—ratio of the strength of metakaolin concrete to the strength of OPC concrete).

strength of MK-CR10 concrete must be attributed to its reactivity. As it is established elsewhere, kaolinite in kaolin K is less ordered than kaolinite in KC, and this has a positive effect on MK reactivity [12]. Between 2 and 28 days, the relative strength shows a clear increase due mainly to the pozzolanic reaction of the metakaolin. The increase of the relative strength is greater in the case of sand replacement due to the avoidance of the dilution effect. Between 28 and 90 days, there is a decrease in the relative strength due to the completion of the pozzolanic reaction. It must be noticed that in all metakaolin concretes, with 10% or 20% metakaolin addition, the relative strength at 90 days is greater than 1. It means that even long-term strength is affected by the metakaolin addition in a positive way.

Taking into account the strength development of the metakaolin concretes (Fig. 1) as well as their relative strength (Fig. 2), it is shown that the produced MK, which originated from poor Greek kaolins, indicates a similar behavior with MKC of high purity with respect to the concrete strength development. Both metakaolins affect the concrete strength in a very positive way.

3.2. Evaluation of strength development using k-value

To estimate the *k*-values, the following procedure was followed. The compressive strength of a Portland cement concrete can be estimated by the following empirical equation [1]:

$$f_c = K\left(\frac{1}{W/C} - a\right) \tag{1}$$

where f_c is the compressive strength of Portland cement concrete (MPa); W is the water content in the initial concrete mix (kg/m^3); C is the cement content in the concrete (kg/m^3); K is the parameter depending on the cement type

(MPa); and a is the parameter depending mainly on time and curing.

For the Portland cement used in this work, the K was calculated as 37.2 MPa. Using the values of the compressive strength of the control sample (Table 6), parameter a is estimated as 0.98, 0.68, 0.50, and 0.20 for 2, 7, 28, and 90 days, respectively.

In the case of metakaolin concrete, the following expression for compressive strength can be used, which involves the concept of k-value:

$$f_c = K\left(\frac{1}{W/(C+kP)} - a\right) \tag{2}$$

where P is the metakaolin content in the concrete (kg/m^3) and k is the k-value.

Using Eq. (2), the measured values of the compressive strength (Figs. 1 and 2) and the W, C, and P contents given in Tables 6 and 7, the k-values for MK and MKC were calculated and are given in Table 9.

From Table 9, it is shown that both metakaolins (MKC and MK) are highly reactive as the k-values are close to 3.0 at 28 days. MK exhibits higher k-values than commercial MKC at 2 and 90 days. This phenomenon needs further investigation and is one of our future aims. As it is established elsewhere, kaolinite in kaolin K is less ordered than kaolinite in KC, and this has a positive effect on MK reactivity [12]. In addition, the role of alumina and SO_3 must be investigated to explain the contribution of MK at early and long-term strength.

The effect of active silica on the strength development can be initially evaluated based on Eq. (3), as given in a previous paper [43].

$$k = (\gamma_S f_{S,P}/f_{S,C})(1 - aW/C) \tag{3}$$

where k is the estimated (approximate) k-value; $f_{S,C}$ is the weight fraction of silica in cement; $f_{S,P}$ is the weight fraction of silica in SCM; γ_S is the ratio of the active silica to the total silica content in the SCM; a is the parameter depending mainly on time and curing; W is the water content in the concrete mix (kg/m^3); and C is the cement content in the concrete (kg/m^3).

Eq. (3) is based on two assumptions: (a) In an SCM–cement system, the CSH content is the most critical parameter in strength development. Thus, at an advanced (>28 days) or complete hydration level, the strength of a concrete (pozzolanic or Portland cement) should be proportional to the CSH content; (b) The CSH content is proportional to the active silica content of the SCM [43].

Table 9
Efficiency factors (k-values) of metakaolins

Concrete property	MK	MKC
Strength, 2 days	1.1	0.7
Strength, 7 days	1.8	1.9
Strength, 28 days	3.0	3.1
Strength, 90 days	2.9	2.3

By applying Eq. (3), for the metakaolins of the present work (data from Tables 4 and 6; $a = 0.20$), the estimated k-value (90 days) are 2.2 and 1.3 for MKC and MK, respectively. With respect to MKC, the estimated k-value is very close to the measured one (2.3). On the contrary, for MK, the estimated k-value (1.3) is far from the experimentally determined one (2.9). Eq. (3) describes satisfactorily the behavior of MKC, which is a high-purity metakaolin with all silica being amorphous and thus reactive. In the case of MK, which is a metakaolin originating from a poor Greek kaolin, the hydration process is more complex, and the active silica does not seem to be the only factor affecting the strength development. In any case, the behavior of MK needs more investigation.

All the above results show that MK, derived from a poor Greek kaolin, shows a similar behavior with MKC of high purity with respect to the concrete strength development. Although a more thorough investigation is required, its exploitation seems to be very promising.

4. Conclusions

The following conclusions can be drawn from the present study:

- □ The produced metakaolin, which is derived from a poor Greek kaolin, imparts similar behavior to that of the commercial metakaolin, with respect to the concrete strength development.
- □ When metakaolin replaces sand, higher strengths than the OPC concrete are succeeded at all ages up to 90 days. When metakaolin replaces cement, its positive effect on concrete strength generally starts after 2 days.
- □ Both metakaolins exhibit very high k-values (close to 3.0 at 28 days) and are characterised as highly reactive pozzolanic materials that can lead to concrete production with an excellent performance.

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