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Supplementary cementing materials in concrete
Part II: A fundamental estimation of the efficiency factor

V.G. Papadakis*, S. Antiohos, S. Tsimas

Department of Chemical Engineering, National Technical University of Athens, 9 Heroon Polytechniou, Zografou Campus, GR-157 73 Athens, Greece

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Abstract

For comparing the relative performance of various supplementary cementing materials (SCMs: silica fume, fly ash, slag, natural pozzolans, etc.) as regards Portland cement, the practical concept of an efficiency factor may be applied. The efficiency factor (or *k* value) is defined as the part of the SCM in an SCM-concrete that can be considered as equivalent to Portland cement. In the present work, an alternative procedure for experimental determination of the *k* value is proposed, using the concept of the pozzolanic activity index. For the first time, also, the *k* value for equivalent strength was correlated with the active silica content of the SCM through analytical expressions. Artificial pozzolanic materials of various compositions and some natural pozzolans were studied. It was found and verified by experimental comparison that these expressions are valid only for artificial SCMs (fly ash, slag), whereas in the case of natural SCMs the *k* value is overestimated. Thus, knowing primarily the active silica content of the SCM, a first approximation of the *k* value can be obtained and, further, the strength of a concrete incorporating artificial SCM can be predicted.

Keywords: Amorphous material; Compressive strength; Fly ash; Modeling; Pozzolan

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1. Introduction

Solid industrial by-products, such as siliceous and aluminous materials, produced during various thermal treatments (fly ash, silica fume, slags, etc.), as well as some natural pozzolanic materials (volcanic tuffs, diatomaceous earth, etc.), may be characterized as supplementary cementing materials (SCMs) as they exhibit cementitious properties. SCMs are increasingly being used in the cement and concrete industry due to technical, economical and environmental reasons [1–4]. The incorporation of various SCMs either as ingredients in blended cements or as separately batched constituents in concrete has been investigated in different countries by a significant number of researchers, giving encouraging results regarding the mechanical and durability properties of concrete [5–11]. Since large quantities of such materials are still unexploited (for instance, in Greece only 10% of the 12 million tons of the artificial SCMs produced annually is utilized), there is an increasing interest in exploring further the parameters that affect the behavior of these materials in concrete.

In previous publications [12–14], a simplified scheme describing the activity of silica fume and fly ash (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. Further, 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 in a companion paper [15], using the concept of the SCM efficiency factor. The efficiency factor (or *k* value) is defined as the part of the SCM in a pozzolanic concrete that can be considered as equivalent to Portland cement, having the same properties as the concrete without SCM (obviously k=1 for Portland cement). The quantity of the SCM in the mixture can be multiplied by the *k* value to estimate the equivalent cement content, which can be added to the existing cement content for the determination of the water-to-cement ratio, minimum required cement content, etc. The compressive

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* Corresponding author. PO Box 5154, GR-26004 Patras, Greece. Tel.: +30-932-327-323; fax: +30-610-436357.

E-mail address: vgp@pat.forthnet.gr (V.G. Papadakis).

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strength is so far used, in practice, as the property for the estimation of *k* values [16,17]. Knowing these *k* values, the mix design for the preparation of the building product can be easier and more accurate.

In the literature [18–20], there is a consensus that the activity of SCMs is mainly based on the fact that they possess significant contents of active constituents, principally active silica, that combine with the calcium hydroxide (CH) produced from Portland cement hydration and form hydration products with binding properties. It is the active silica, which is part of the total silica of the SCM, that is involved in the hydration reactions producing calcium silicate hydrates (CSH) to which the strengthening of cement is attributed. Active silica is noncrystalline silica glass, more particularly present in the amorphous and mostly vitreous part of the SCM [21], which can be combined with the lime giving increased contents of C-S-H gel [22], unlike crystalline silica that exhibits very low reactivity [23,24]. Richartz [21] had focused his attention on soluble silica, stating that the pozzolanic reaction can be expected only from substances or materials whose silica content can dissolve with sufficient rapidity in the alkaline environment of the cement paste, while Bijen [25] noted that in order for the fly ash glass to be activated, the links between Si–O–Si have to be broken as fly ash does not dissolve, contrary to slag, but actually decomposes.

Efforts to relate this critical parameter with cement and concrete properties are limited in the literature. Papadakis [12–14] in previous publications had observed that the final strength gain in SCM-concrete is proportional to the glass phase content, while Sharma et al. [26] proposed an empirical correlation between pozzolanic properties of fly ashes determined by using the compressive strength of mortars and the fineness and the soluble silica. Ranganath et al. [27] investigated the influence of fineness and soluble silica content of Indian fly ashes, concluding that at later ages the soluble silica content of the fly ash becomes more significant than the effect of their fineness. It is evident that although several authors have attempted to connect the pozzolanic effect with a number of parameters, such as fineness [27,28], water to powder ratio [29], curing temperature [29,30] and alkalinity of the pore solution [31], there seems to be a lack in the literature regarding the effect of the reactive silica content on their pozzolanicity and behavior as additives in cement and concrete. Even proposed mechanisms for the quantification of the pozzolanic activity [32,33] have not produced a relation between the amount of soluble silica of the examined admixtures and their potential activity.

The present work aims at filling this gap by introducing a relationship between the reactive silica content of various SCM and their corresponding *k* values. In addition, in the present work a relationship between the *k* value and the activity index [34] is developed, which can be used for a faster estimation of *k* values through activity index measurements. Overall, the approach presented herein can lead to a more precise and rapid prediction of the quantity, but most of all the quality, of the SCM used in the concrete mix design, so that the final product will meet certain specified requirements.

2. Experimental section

Two natural materials and four industrial by-products were used, all produced or located in Greece. The natural materials were: a volcanic tuff from Milos Island (Milos earth, defined as ME), and a diatomaceous earth from Samos Island (defined as DE). Three different fly ashes from Greek power plants (produced by Puplic Power Corporation) were used, i.e., a fly ash of relatively lower calcium content than the other fly ashes (fly ash from Megalopolis plant, defined as FL), a high-calcium fly ash of normal sulfur content (fly ash from Ptolemais plant, defined as FH), and a high-calcium fly ash of high sulfur content (fly ash from Ptolemais plant, defined as FHS). Finally, a nickel slag produced by LARKO was used (defined as SL). All these SCM were ground prior to use up to a fineness of 400±20 m^2/kg according to Blaine's test. A rapid-setting Portland cement of similar fineness was used (CEM I 52.5R according to European Standard EN 197).

Oxide analyses for all materials are presented in Table 1. The fraction of SiO_2 which is active for pozzolanic reactions is also given (active silica). Normal graded limestone aggregates, including fine (37%), medium (21%) and coarse (42%) aggregates, were used. The coarse aggregate maximum size was 31.5 mm. Tap water at 20 °C was used. A common superplasticizer was used at a content of 0.5% of the total cementitious materials in order to retain the slump of the fresh concrete between 8–12 cm. The air content of the concrete mixtures was approximately 1%.

A constant volume unit (1 m^3) of concrete was chosen as a common comparison basis. When an SCM was added to this unit, then an equal mass of another component, either cement or aggregate, was removed in order to keep a similar total volume, in a way described in Table 2. The water content for all specimens was kept constant (175 kg/m^3). The mixture proportions of all concrete specimens are summarized in Table 2. For the control specimen, the water-to-cement ratio (W/C) was 0.5 and the aggregate-to-cement ratio (A/C) was 5.4. The dry materials were mixed for 2 min. Then the water was added, containing the plasticizer, and the mixing was continued for a further 2 min.

The specimens for strength measurements were cast in cubes of 150 mm, 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. The testing age was after 2, 7, 28 and 90 days. For each age, three specimens of each mixture were tested for

Table 1
Chemical (oxide) analyses (%) of cement, low-calcium fly ash (FL), high-calcium fly ash (FH), high-calcium fly ash of high sulfur content (FHS), nickel slag (SL), Milos earth (ME), and diatomaceous earth (DE)[a]

	Cement	FL	FH	FHS	SL	ME	DE
SiO_2	20.73	44.92	33.37	31.33	36.22	58.23	22.33
Al_2O_3	4.78	18.47	17.35	15.89	10.34	14.22	0.96
Fe_2O_3	3.87	7.90	5.57	5.37	40.19	4.31	1.00
CaO	64.73	14.87	25.21	27.38	5.08	7.40	45.89
MgO	2.05	2.22	3.05	3.02	3.12	1.43	1.54
K_2O	0.50	1.71	1.20	1.07	0.47	2.24	0.10
Na_2O	0.10	0.77	0.75	0.53	0.28	1.30	0.32
SO_3	2.47	3.89	5.57	7.90	0.23	1.16	1.24
active SiO_2[b]	–	70	75	73	5	50	50

[a] The methods specified by EN-450, EN-196 and EN-451 were followed.

[b] The fraction of SiO_2 that is soluble after treatment with HCl and with boiling KOH solution (European Standard EN 197-1).

compressive strength and the mean value of these measurements is reported.

3. Results and discussion

3.1. Compressive strength

The experimental results are presented in detail in the previous paper [15]. Briefly, it was generally observed that when SCM substitutes for aggregates, strengths higher than the controls are obtained. When SCM replaces cement, the strength was reduced, at first, but as time proceeds this gap is gradually eliminated. In order to estimate the *k* values, the following procedure was followed.

The compressive strength, f_c (MPa), of a Portland cement concrete can be estimated by the following empirical equation [4]:

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

where *W* is the water content in the fresh concrete mix (kg/m^3), *C* is the cement content in the concrete (kg/m^3), *K* is a parameter depending on the cement type (MPa) and *a* a parameter depending mainly on time and curing. For the Portland cement used in this work, *K* was calculated as 38.8 MPa [15]. Using the mean measured values of the compressive strength of the control specimen, the parameter *a* was estimated as 1.06, 0.72, 0.5 and 0.23 for 2, 7, 28 and 90 days, respectively.

Table 2
Mixture proportions for concrete specimens

Specimen	*C*	*W*	*P*	*A*
Control	350	175	0	1900
P(+10)	350	175	35	1865
P(+20)	350	175	70	1830
P(−10)	315	175	35	1900
P(−20)	280	175	70	1900

C, *W*, *P*, *A*: kilograms of cement, water, supplementary cementing material (FL, FH, FHS, SL, ME, or DE) and aggregate, respectively, per cubic meter of total concrete volume (for zero air content). P(+): the SCM replaces aggregates; P(−): the SCM replaces cement, by weight of the control cement content.

In the case of SCM-concrete, the following expression for compressive strength can be used, which involves the concept of the *k* value [4,15]:

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

where *P* is the SCM content in the concrete (kg/m^3). Using this equation, the measured values of the compressive strength given in Ref. [15], and the *W*, *C* and *P* contents given in Table 2, the *k* values for the SCM of the present work were calculated and are given in Table 3. For fly ashes, the *k* values are around unity (1) at early ages and they exceed it, as time proceeds. The natural SCMs exhibit much lower efficiency factors (0.3–0.4 for ME and 0.2 for DE). This can be correlated with their low level of active silica content (see Table 1). In the case of the nickel slag from LARKO (SL), the strength results were almost identical to the controls, regardless of whether the slag was ground or not. Very low *k* values of 0–0.1 were calculated, proving that the lack of active silica (see Table 1) due to slowly cooled production plays a dominant role in the pozzolanic activity.

3.2. Activity index

Pozzolanic activity is usually determined through an activity index, the ratio of the compressive strength of a pozzolanic mortar to that of a control mortar [34]. For the preparation of the control mortar, a reference Portland cement is used and a water-to-cement ratio (*W*/*C*) equal to 0.5 and an aggregate (sand) to cement ratio (*A*/*C*) equal to 3

Table 3
Efficiency factors (*k* values) for various supplementary cementing materials[a]

Concrete property	FL	FH	FHS	SL	ME	DE
Strength, 2 days	0.8	0.8	1.0	0.0	0.4	0.2
Strength, 7 days	1.0	0.9	1.0	0.0	0.3	0.2
Strength, 28 days	1.1	0.9	1.4	0.1	0.3	0.2
Strength, 90 days	1.2	0.9	1.2	0.1	0.3	0.2

[a] Earlier work [15].

are specified. For the preparation of the pozzolanic (SCM) mortar, the same as above water (W) and aggregate (A) contents are used, and cement and pozzolan contents are 75% and 25%, respectively, of the control cement content. The mortars are cured under water for a certain period of time until testing (at 28 and 90 days).

According to the above mixture proportions and by applying Eqs. (1) and (2) the compressive strengths of the control and SCM mortars are given, respectively, by:

$$f_{c,c} = K[1/(W/C) - a]$$

$$f_{c,p} = K\{1/[W/(0.75C + k0.25C)] - a\}$$

By definition, the activity index (AI) equals the ratio $f_{c,p}/f_{c,c}$, and thus the following relationships between activity index and efficiency factor (k, regarding strength) are observed:

$$\mathrm{AI} = 1 + 0.25(k - 1)/(1 - 0.5a) \quad (3)$$

$$k = 1 + 4(\mathrm{AI} - 1)(1 - 0.5a) \quad (4)$$

EN-450 specifies that the activity index for fly ash shall be not less than 75% and 85% at 28 and 90 days, respectively [34]. According to Eq. (3) and for values of the parameter a equal to 0.5 and 0.23, respectively, at 28 and 90 days, a $k > 0.25$ for 28 days and a $k > 0.47$ for 90 days are required.

The activity index for 28 and 90 days was measured according to EN-450 for the five typical SCM of the present study. The experimental values are shown in Table 4, together with the calculated values by Eq. (3) and an excellent agreement is observed. This comparison proves the validity of Eq. (3) and indicates that Eq. (4) can be used for a faster estimation of k values through activity index measurement.

3.3. Active silica

According to European Standard EN 197-1 [35], active silica is defined as the fraction of the silicon dioxide that is soluble after treatment with hydrochloric acid and with boiling potassium hydroxide solution. The European Standard EN 196-2 [36] was used to determine the active silica contents of all SCMs used in this work. This standard specifies that the reactive silica content in pozzolans is determined by subtracting from the total silica content of the pozzolan the fraction that is contained in the insoluble residue. To be more specific, the percentage of the active silica of a pozzolan was estimated as the difference between the total amount of silica and the silica present in the insoluble residue, as this is determined after treatment with hydrochloric acid and a 25% boiling potassium hydroxide solution in a 4-h extraction. By following the above method, the reactive silica contents of the various SCM used in this work were determined and are given in Table 1.

Many researchers have shown that the main strength components in hydrated cement are the calcium silicates C_3S and C_2S due to CSH production [4,37–41]. However, in the early stages of hydration (0–7 days) the aluminoferrite phases (C_3A and C_4AF), especially in the presence of gypsum, make a significant contribution to the total strength. At an advanced (>28 days) or "complete" hydration level, the strength that the C_3A or C_4AF phase (in the presence of gypsum) can contribute is only 10% of the strength of the C_3S or C_2S phase [4]. As these phases (C_3A and C_4AF) are present at a low concentration in the cement, it is principally the products of C_3S and C_2S, i.e., CSH that is correlated with the total strength of the hydrated cement.

In an SCM–cement system, the CSH content will also be the most critical parameter in strength development. Thus, the strength of a concrete (pozzolanic or Portland cement) should be proportional to the CSH content, and for compressive strength (f_c) the following simple equation is proposed for examination (for ages >28 days):

$$f_c = m\mathrm{CSH} \quad (5)$$

where CSH is the CSH content in kilograms per cubic meter of concrete and m a parameter depending mainly on water content, aggregate content and type, and other compositional parameters of the concrete. The CSH content was calculated previously [12–14] as a function of the cement content (C), SCM content (P), the weight fraction of silica in cement ($f_{S,C}$) and SCM ($f_{S,P}$), respectively, and the weight fraction of the oxide SiO_2 in the SCM, which contributes to the pozzolanic reactions (γ_S: it could be the ratio of active silica to the total silica in the SCM):

$$\mathrm{CSH} = 2.85(f_{S,C}C + \gamma_S f_{S,P}P) \quad (6)$$

and thus from Eqs. (5) and (6):

$$f_c = 2.85m(f_{S,C}C + \gamma_S f_{S,P}P) \quad (7)$$

It should be emphasized that for the SCM content in concrete, there is a maximum value, P_{max}, beyond which the SCM added is inert. As given in previous publications [12–14], the maximum fly ash content in concrete shall be 25–50% of the cement weight depending on calcium

Table 4
Comparison of calculated by Eq. (3) and measured activity indexes

	Activity index, 28 days		Activity index, 90 days	
SCM	Calculated	Measured	Calculated	Measured
FL	1.03	0.99	1.06	0.97
FH	0.97	0.91	0.97	0.96
FHS	1.13	1.00	1.06	0.99
ME	0.77	0.81	0.80	0.83
DE	0.73	0.75	0.77	0.80

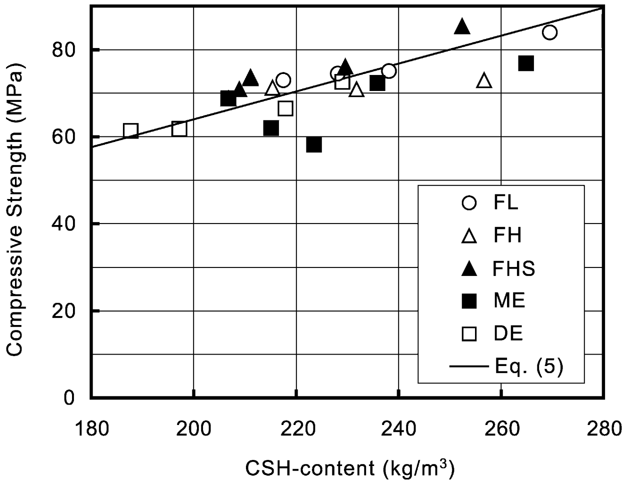

Fig. 1. Experimental values of compressive strength versus CSH content and comparison with Eq. (5) predictions (m=0.32).

content of the fly ash. Especially, for fly ash with almost zero calcium content the maximum content is about 25% of the cement content, for FL (fly ash Megalopolis) the maximum content is calculated as 36% and for FH (fly ash Prolemais) as 50%. The CSH content depends also on hydration time and type of curing. The above equation gives only final values; dependence on time can be estimated from strength results versus time. From the strength results of 90 days, presented earlier [15], and for the material characteristics given in Tables 1 and 2, the CSH contents are calculated and given in Fig. 1. A mean value for m equal to 0.32 was estimated (standard deviation 0.02), which gives calculated strengths in good agreement with the experimental results, as observed in Fig. 1, at least for the artificial SCM of the present work. Thus, this approach to compressive strength by Eq. (7) can be considered as a good first approximation.

By combining Eq. (1) with Eq. (7) for Portland cement concrete (P=0), the following expression for the parameter m is obtained:

$$m = [K/(2.85 f_{\mathrm{S,C}})](1/W - a/C) \tag{8}$$

By substituting the parameters in Eq. (8), a value equal to 0.33 is calculated for the parameter m, very close to the mean experimental value 0.32, proving the validity of this approach.

For SCM-concrete, by combining Eq. (2) and Eq. (7), where m is calculated by Eq. (8), the following relationship for k value is obtained:

$$k = (\gamma_{\mathrm{S}} f_{\mathrm{S,P}}/f_{\mathrm{S,C}})(1 - aW/C) \tag{9}$$

By applying the above equation for the SCM of the present work, k values of 1.3, 1.1 and 1.0 are calculated for FL, FH and FHS, respectively. These estimated values for fly ashes are in a good agreement with the experimental ones given in Table 3, showing the validity and the predictive power of Eq. (9). For the slowly cooled slag (SL), a k value equal to 0.1 is estimated, which agrees with the experimental measurement. However, a significant deviation is observed for the natural materials: for diatomaceous earth (DE), where the estimated k value is 0.5 and the measured 0.2, and for Milos earth (ME) where the estimated k value is 1.2, far enough from the measured 0.3. This exception can be attributed either to the formation of a weaker CSH component or the active silica measurement followed herein is not applicable for natural materials. As a general conclusion, Eq. (9) can be applied for a first approximation of the k value of the artificial SCM, such as fly ash and slag. In the case of multi-component use (simultaneous use of various SCM) in the concrete production, the sum of the active silica of the materials may be introduced in Eq. (9); however, experimental verification is required.

Finally, as time proceeds, i.e., after 1 year, the parameter a approaches zero and thus the following expression can be obtained, giving an estimation for the maximum k value (Eq. (10)):

$$k_{\max} = \gamma_{\mathrm{S}} f_{\mathrm{S,P}}/f_{\mathrm{S,C}} \tag{10}$$

4. Conclusions

In practice [16], the concept of an efficiency factor for the supplementary cementing materials (SCM: silica fume, fly ash, slag, natural pozzolans, etc.) may be applied in order to predict the performance of concrete incorporating SCM. The efficiency factor (k value) is defined as the part of the SCM in an SCM-concrete that can be considered as equivalent to Portland cement. In a companion paper [15], efficiency factors for various SCMs were measured (as summarized in Table 3). These values are valid for a certain amount of SCM in concrete and they are different depending on the property that it concerns (strength, durability). In the present work, first, a faster procedure for experimental determination of the k value is proposed, using the concept of the pozzolanic activity index.

In the sequence, for the first time, the k value was correlated with the active silica content of SCM and an analytical relationship was obtained. This expression was experimentally found to be valid for artificial SCMs (fly ash, slag), while overestimating the k values for the natural materials. This exception for natural materials can be attributed either to the formation of a weaker CSH component or to the fact that the active silica measurement followed herein is not applicable for natural materials. Thus, the analytical relationship can be applied as a first approximation of the k value of the artificial SCM. This approach may be used for a rapid prediction of the quantity, but most of all the quality of the SCM used in the concrete mix design so that the final product will meet certain specified requirements.

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