4. STRENGTH APPROXIMATION

4.1 The European Standard EN 206 and strength aspects

According to EN 206 [12], the hardened concrete *is classified* with respect to its *compressive strength* according to Table 4.1.1 (for normal-weight and heavy-weight concrete; for light-weight concrete, see [12]). The characteristic compressive strength at 28 days of 150 mm diameter by 300 mm cylinders ($f_{ck,cyl}$) or the characteristic strength at 28 days of 150 mm cubes ($f_{ck,cube}$) may be used for classification. *Characteristic strength* is the value of strength below which 5% of the population of all possible strength determinations of the volume of concrete under consideration, are expected to fall.

Table 4.1.1 Compressive strength classes for normal-weight and heavy-weight concrete.

Compressive strength class	Minimum characteristic cylinder strength ($f_{ck,cyl}$, MPa)	Minimum characteristic cube strength ($f_{ck,cube}$, MPa)
C8/10	8	10
C12/15	12	15
C16/20	16	20
C20/25	20	25
C25/30	25	30
C30/37	30	37
C35/45	35	45
C40/50	40	50
C45/55	45	55
C50/60	50	60
C55/67	55	67
C60/75	60	75
C70/85	70	85
C80/95	80	95
C90/105	90	105
C100/115	100	115

Where the strength is to be determined, it shall be based on tests carried out on either 150 mm cubes or 150/300 mm cylinders conforming to EN 12390-1 and made and cured in accordance with EN 12390-2 from samples taken in accordance with EN 12350-1.

When compressive strength is to be determined, it shall be expressed as $f_{c,cube}$ where determined using cubical specimens and $f_{c,cyl}$ where determined using cylindrical specimens, in accordance with EN 12390-3. Unless specified otherwise, the compressive strength is determined on specimens tested at 28 days. For particular uses, it may be necessary to specify the compressive strength at ages earlier or later than 28 days or after storage under special conditions. The characteristic strength of the concrete shall be equal or greater than the minimum characteristic compressive strength for the specified compressive strength class, see Table 4.1.1.

The producer shall provide the user the compressive strength class of the concrete and, if requested, information on the *strength development* of the concrete either in terms of Table 4.1.2 or by a strength development curve at 20 °C between 2 and 28 days. The *strength ratio* to indicate the strength development is the ratio of the mean compressive strength after 2 days ($f_{cm,2}$) to the mean compressive strength after 28 days ($f_{cm,28}$), determined from initial tests or based on known performance of concrete of comparable composition.

Table 4.1.2 Strength development of concrete at 20 °C.

Strength development	Estimate of strength ratio ($f_{cm,2} / f_{cm,28}$)
Rapid	≥ 0.5
Medium	≥ 0.3 to < 0.5
Slow	≥ 0.15 to < 0.3
Very slow	< 0.15

On the other hand, EN 206 gives a detailed system for *conformity control*, i.e., the combination of actions and decisions to be taken in accordance with conformity rules adopted in advance to check the conformity of the concrete with the specification. The conformity or non-conformity is judged against the conformity criteria.

The *conformity control for designed concrete* with respect to compressive strength has as follows. For normal-weight and heavy-weight concrete of strength classes from C8/10 to C55/67 sampling and testing shall be performed either on individual concrete compositions or on concrete families of established suitability. A *concrete family* is a group of concrete compositions for which a reliable relationship between relevant properties is established and documented. The family concept shall not be applied to concrete of higher strength classes. A distinction is made for *initial production* (until 35 at least results are available) and *continuous production* (when at least 35 test results are obtained over a period not exceeding 12 months). Sampling of concrete shall be randomly selected and taken in accordance with EN 12350-1.

The *conformity assessment for compressive strength* shall be made on test results taken during an assessment period that shall not exceed the last 12 months. Conformity is assessed on specimens tested at 28 days (or at another specified age) for:

- groups of "n" non-overlapping or overlapping consecutive test results f_{cm} *(criterion 1);*
- each individual test result f_{ci} *(criterion 2)*

Conformity is confirmed if both the criteria given in Table 4.1.3 for either initial or continuous production are satisfied. Where conformity is assessed on the basis of a concrete family, criterion 1 is to be applied to the reference concrete taking into account all transposed test results of the family; criterion 2 is to be applied to the original test results. To confirm that each individual member belongs to the family, the mean of all non-transposed test results (f_{cm}) for a single family member shall be assessed against a specific *criterion 3* [12]. Initially the *standard deviation* σ shall be calculated from at least 35 consecutive test results taken over a period exceeding the 3 last months.

Table 4.1.3 Conformity criteria for compressive strength.

Production	Number "n" of test results in the group	Criterion 1 Mean of "n" results (f_{cm}, MPa)	Criterion 2 Any individual test result (f_{ci}, MPa)
Initial	≥3	$\geq f_{ck} + 4$	$\geq f_{ck} - 4$
Continuous	≥15	$\geq f_{ck} + 1.48\ \sigma$	$\geq f_{ck} - 4$

4.2 Concrete strength approximation using cement's strength class

For a CEM I type of cement, many researchers have shown that the main strength components in hydrated paste are C_3S and C_2S due to CSH production, see section 3 [9,22-27]. However, in the early stages of hydration (0-7 days) the alumino-ferrite phases, 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. As these phases (C_3A and C_4AF) are present at a low concentration in the cement, it is principally the product of C_3S and C_2S, *i.e. CSH,* that is correlated with the total strength of the hydrated cement. Another also strong parameter is the *concrete porosity*, especially in the *transition zone* between cement paste and aggregate surface. Thus, a strength prediction approach could be developed based on fundamental chemical and volumetric characteristics, as these given in the previous section 3, i.e., CSH content, porosity, etc.

However, a reliable prediction of concrete strength based on contribution of each individual compound is very difficult, because this contribution is not simply additive and has been found to depend on age and the curing conditions [9, 25]. Moreover, a generally applicable strength prediction equation is not possible due to interaction between the various compounds, including additions and cement's SCM, the influence of alkalis and gypsum, the influence of the particle size of cement and the influence of particle size and shape of aggregates, etc. Many attempts have been made to generate strength prediction of cement paste, mortar and concrete, but without a generally accepted validity. On the other hand, many *empirical expressions* have been proposed for strength prediction, presenting the most crucial dependences of strength from concrete compositional parameters and calculating the adjustable parameters from experiments [9,25,26,36,42,43].

In all empirical expressions the W/C ratio turns out to be the most important parameter. Probably the first formulation of the relation of strength, (f_c, mean compressive strength, MPa) and the concrete constituents was made by Feret [43,44]:

$$f_c = b\,(C/d_C)^2 / (C/d_C + W/d_W + \varepsilon_{air})^2 \qquad (4.2.1a)$$

$$\text{or}\ \ f_c = b / [1 + (W/C)\,(d_C/d_W) + \varepsilon_{air}\,(d_C/C)]^2 \qquad (4.2.1b)$$

where b is a parameter adjustable from experimental results. Another famous relationship is that introduced by Abrams [43,44]:

$$f_c = b_1 / b_2^{W/C} \qquad (4.2.2)$$

where again b_1 and b_2 are adjustable parameters dependent on cement type, curing and age at test. Also another empirical equation is that deduced by Bolomey [31,45-47]:

$$f_c = p_1 \left(\frac{1}{W/C} - p_2\right) \qquad (4.2.3)$$

where p_1 is a *strength factor* depending on cement type, aggregate type and air content (MPa) and p_2 a *time factor* depending mainly on time, type of curing, and early strength class (cement fineness). All the above rules, as well many others more complicated, require experimental results for the parameter adjustment.

In the lack of experimental results the information from the ***cement strength class*** may be used to estimate a safe lower limit for concrete strength and thus to approach the corresponding value of ***compressive strength class***. European Standard EN 196-1 prescribes a compressive strength test for cement on mortar specimens of fixed composition. The specimens are tested as 40 mm equivalent cubes, and are made with a “CEN standard sand”, natural, siliceous, and rounded. The W/C ratio is 0.5 and the sand/cement ratio is 3. The specimens are cured in water at 20 ^{o}C until testing on 2 or 7, and 28 days. Through this approach the cement strength class is defined [13]. However, when strength results from mortars are compared with ones from concretes made each with the same W/C ratio, a significant difference is observed. The *concrete strength is higher than the mortar strength*, mostly due to greater amount of entrapped air in mortar [9]. Using for example all the above information to the Feret's formula (W/C=0.5, $d_C/d_W \approx 3.15$, $\varepsilon_{air} \approx 0.035$, $d_C \approx 3150$, C ≈490), a lower value for parameter b can be estimated:

$$f_c = b / [1 + (W/C)(d_C/d_W) + \varepsilon_{air}(d_C/C)]^2 \geq SS \qquad (4.2.4a)$$

$$\text{i.e., } b \geq 7.84\ SS \qquad (4.2.4b)$$

where *SS is the standard strength class (at 28 days) of cement (MPa)*. Using Eq. (4.2.4), *the minimum compressive strength class of concrete (at 28 days)* can be estimated at another values of W/C, C or ε_{air} from the following equation:

$$\boxed{f_c \geq 7.84\ SS / [1 + (W/C)(d_C/d_W) + \varepsilon_{air}(d_C/C)]^2 \qquad (4.2.5)}$$

If rounded aggregates are used for concrete the above estimation has to decrease by a factor of 13% [44]. On the other hand, if a strength result from the above mortar specimens is known at another age (2, 7, or 90 days), this could be used in Eq. (4.2.5), as SS, in order to estimate the compressive strength at the same age and for other W/C values. In this way, the *strength development* can be predicted.

Several other empirical expressions may be used as above, i.e., Abrams', Eq. (4.2.2) or Bolomey's, Eq. (4.2.3), etc. However, Feret's formula as contains only one adjustable parameter permits a rather safer approximation from the others models with more adjustable parameters. On the other hand, it contains the effect of air content (ε_{air}) predicting that 1% variation in air content results in a variation of about 4.5% of the compressive strength as many experimental results have shown [48]. As Feret's formula was extracted from mixes of rather high W/C, at modern lower W/C mixes another exponent (than 2.0) may be used in Eq. (4.2.1). In any case, this approach is just *a first rough approximation*, valuable for the initial test proportioning, and a detailed experimental verification is required. It has also to be emphasized that the above approach can be applied for any cement type, but it refers only to *concrete without any active additions such as fly ash or silica fume.* The next section is dealt with strength prediction when active additions are used.

4.3 Strength approximation using SCM efficiency factor

4.3.1 Procedure

When in a concrete, made with CEM I type of cement, a Type II addition is used (silica fume and/or fly ash), the Eq. (4.2.5) is not valid anymore, as it is. The pozzolanic action of addition shall be taken in consideration as it gives strength components. In the previous section 3, a simplified scheme describing the activity of supplementary cementing materials (SCM) in terms of chemical reactions was proposed, yielding quantitative expressions for the estimation of the final chemical and volumetric composition of such SCM-concretes. However, a practical approach to the effect of SCM on the strength of portland cement systems and on their resistance against carbonation and chloride penetration can be achieved, using the concept of the *SCM efficiency factor*. We assume that when active additions are used in concrete a cement of type CEM I is used only.

The *efficiency factor (or k-value)* is defined as the part of the SCM that can be considered as equivalent to portland cement (CEM I), providing the same concrete properties (*obviously k=1 for portland cement*). The quantity of the SCM in the concrete mixture 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-to-cement ratio, minimum required cement content, etc. The compressive strength was so far used as the property for the estimation of k-values [12,49]. In this work, durability properties are also used, such as resistance against carbonation and chloride penetration, and relative k-values are calculated [46,47,50,51]. Knowing these k-values, the mix design for preparation of the building product can be easier and more accurate.

In the case of SCM-concrete, the following expression for compressive strength can be used which involves the concept of k-value in Eq. (4.2.5):

$$f_c \geq 7.84\,SS / \{1 + [W/(C+k_F F_{ACT}+k_S S_{ACT})]\,(d_C/d_W) + \varepsilon_{air}\,[d_C/(C+k_F F_{ACT}+k_S S_{ACT})]\}^2 \quad (4.3.1)$$

where F_{ACT} and S_{ACT} are the active contents of concrete additions fly ash and silica fume (kg/m^3), having an efficiency factor k_F and k_S respectively. These active contents are calculated in the previous section 3. Using this equation, and plenty of experimental results, the k-values for various SCM are calculated and summarized in Table 4.3.1.

For siliceous fly ashes, a k-value of 0.5 was calculated for 28 days' strength [31]. These very low calcium fly ashes are very common in the vast majority of EU, where similar k-values are proposed (0.3-0.5 [12,49,44]). However, as time proceeds, higher k-values are calculated for these fly ashes approaching those of high-calcium fly ashes (0.7 for 91 days and 1.1 for 1 year [31]). For calcareous fly ashes (as well for blast furnace slag [44] and burnt shale), the k-values are around unity (1) at early ages and they exceed it as time proceeds. This means that up to a certain level [33,34], these specific pulverized fly ashes can substitute, equivalently, for portland cement.

The natural SCM exhibit much lower efficiency factors (about 0.3-0.4 for natural pozzolana). This is correlated with their low level of active silica content.

In the case of an artificial pozzolan of low reactivity, very low k-values of 0-0.1 were calculated, proving that the lack of active silica due to slowly-cooled production plays a dominant role in pozzolanic activity. On the contrary, the metakaolin exhibits significant higher strengths, resulting at very high k-values (up to 3 at 28 days and onwards [51]). As this material was treated at high temperatures almost all silica was converted into amorphous and thus reactive. This behavior is similar to that of silica fume, where very high k-values were also calculated (3 at 28 days [31,44]).

Table 4.3.1 Efficiency factors (k-values) for various supplementary cementing materials [31,46,47,50,51]*.

Cementitious/ pozzolanic materials	Strength (2 days)	Strength (7 days)	Strength (28 days)	Strength (90 days)
Portland clinker	1	1	1	1
Silica fume	1	2	3	2.4
Pozzolana (natural)	0.4	0.3	0.3	0.3
Metakaolin	1	1.8	3	3
Siliceous fly ash	0.2	0.3	0.5	0.7
Calcareous fly ash	1.1	1.1	1.2	1

* All these SCM were ground prior to use up to a fineness of 400±20 m^2/kg according to Blaine's test.

According to EN 206, type II additions may be taken into account in the concrete composition with respect to the cement content and the W/C ratio if the suitability is established. The *suitability of the k-value concept* is established for siliceous fly ash and silica fume. If other concepts, e.g., the equivalent concrete performance concept, modifications on the rules of the k-value concept, higher k-values, other additions or combinations of additions are to be used, their suitability shall be established. The establishment of the suitability may result from either a European Technical Approach or a relevant national standard or provision valid in the place of use of concrete.

The EN 206 permits the k-value concept to be taken into account replacing the W/C ratio with W/(C+ k · addition) ratio and in the minimum cement content requirement. The actual value of k depends on the specific addition. EN 206, through EN 450, accepts only siliceous fly ash as type II addition in concrete. The maximum amount of siliceous fly ash to be taken into account for the k-value concept shall meet the requirement:

$$\text{Fly ash / cement} \leq 0.33 \text{ by mass} \qquad (4.3.2)$$

The following k-values are permitted for concrete containing cement type CEM I for siliceous fly ash addition:

CEM I 32.5 $k_F = 0.2$ (4.3.3a)
CEM I 42.5 and higher $k_F = 0.4$ (4.3.3a)

The maximum amount of silica fume to be taken into account for the k-value concept shall meet the requirement:

Silica fume / cement ≤ 0.11 by mass (4.3.2)

The following k-values are permitted for concrete containing cement type CEM I for silica fume addition:

for specified W/C ≤ 0.45 $k_S = 2$ (4.3.3a)
for specified W/C > 0.45 $k_S = 2$ (except exp. classes XC and XF, where k=1) (4.3.3a)

In general, an agreement is observed between EN 206 recommendations and the present work's approach. For example, in the present work only the active parts of fly ash and silica fume are considered (typically for siliceous fly ash: F_{ACT}=0.21C, for calcareous fly ash: F_{ACT}=0.48C, and for silica fume: S_{ACT}=0.14C; when these materials are used alone). When both silica fume and fly ash used then lower active parts are estimated. On the other hand, for the case of siliceous fly ash and silica fume similar k-values are proposed by the EN 206.

The present work is more general from EN 206 giving the dependence of k-values on time, including the case of a combined use of both silica fume and fly ash and introducing also the use of calcareous fly ash as (a future) concrete addition. However, the EN 206 recommendations have to be applied officially without any alteration; the scope of the present work is just on strength prediction and thus it can be used for assistance on initial proportioning.

4.3.2 Experimental estimation of SCM efficiency factor

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 [21]. For the preparation of the control mortar, a reference portland cement is used (CEM I) and a water to cement ratio (W/C) equal to 0.5 and an aggregate (sand) to cement ratio (A/C) equal to 3 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 equal to 75% and 25%, respectively, of the control cement content are specified. 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 the Eq. (4.2.5) and (4.3.1) the compressive strengths of the control and SCM mortars are given, respectively, by:

$$f_{c,c} = b / [1 + (W/C)\,(d_C/d_W) + \varepsilon_{air}\,(d_C/C)]^2 \qquad (4.3.4)$$

$$f_{c,p} = b / \{1 + [W/(0.75C+k0.25C)]\,(d_C/d_W) + \varepsilon_{air}\,[d_C/(0.75C+k0.25C)]\}^2 \qquad (4.3.5)$$

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

$$AI = [(2.8k+8.4)/(k+10.2)]^2 \qquad (4.3.6)$$

$$k = (10.2\,\sqrt{AI} - 8.4)/(2.8 - \sqrt{AI}\,) \qquad (4.3.7)$$

EN-450 specifies that the activity index for fly ash shall be not less than 75% and 85%, at 28 and 90 days, respectively [21]. According to Eq. (4.3.7), a k>0.23 for 28 days and a k>0.53 for 90 days are required.

The Eq. (4.3.7) can be used for a faster estimation of the k-values through activity index measurements.

4.3.3 Theoretical approximation of SCM efficiency factor

a. Active silica

In the literature [31, 52-54], there is concertedness that the activity of SCM is mainly based on the fact that they possess significant contents of active constituents, principally reactive silica, that combine with the CH produced from portland cement hydration and form hydration products with binding properties. It is the reactive silica, which is part of the total silica of the supplementary material, that is involved in the hydration reactions producing calcium silicates upon which the strengthening of cement is attributed (see section 3). Reactive silica is non-crystalline silica glass, more particularly present in the amorphous and mostly vitreous part of the supplementary material [55], which can be combined with the lime formed during cement hydration giving increased contents of C-S-H gel [56], unlike crystalline silica that exhibits very low reactivity [57,58]. Richartz [55] 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 [59] 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.

In earlier methods, reactive silica was estimated as the difference between the total silica and free silica [60,61], which were determined after fusion by gravimetric method before and after treating the fly ash with hydrochloric acid. Sivapullaiah et al. [62] also used the gravimetric method to determine the reactive silica indirectly as acid (HCl 1+1) soluble silica, giving surprisingly low values for different fly ashes. The amounts obtained by this method were even more than those present even in portland cement [60,61] with the difference that the concentration of acid was higher than that used for portland cement, mortar, concrete (where only a dilute HCl of 3N is used) [63]. Another method put forward by Mehta [64], established the concept of the ‘silica activity index’ meaning the percentage of available silica that is dissolved in an excess of boiling 0.5 M NaOH solution during a 3-min extraction period. Simpler methods have been proposed [65], based on the titration of sample suspensions with methylene blue. The amount of methylene blue required to produce a color change in the solution can be used as an index of the amorphousness of the silica contained in the ash. Paya et al. [66] recently proposed a rapid method for the determination of amorphous silica in rice husk ashes, based on bringing the siliceous non-crystalline fraction of the pozzolan into solution as glycerosilicate by treating the test material with glycerol. The results gave satisfactory concordance with the reference method.

b. Determination of reactive silica content

According to European Standard prEN 197-1 [13], reactive silica is defined as the fraction of the silicon dioxide which is soluble after treatment with hydrochloric acid and with boiling potassium hydroxide solution. The European Standard EN 196-2 was used to determine the active silica contents of all SCM 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-hour extraction.

In a brief description, sintering of 1 g of the dried sample with Na_2O_2 is followed by a persistent treatment with hydrochloric acid until the final solution is filtered. The content of the filter paper is burned in an electric furnace for an hour at 1100^0 C giving the percentage of the total silica contained in the pozzolan. The same procedure was followed for determining the silica present in the insoluble residue with the difference that the sintering involves the previous extracted residue.

Although the particular method is considered time consuming and requires increased caution by the analyst, it provides more reliable results when compared with resembling methods. Emphasis must be given in the sample preparation stage, as pozzolans with high carbon content (or higher loss on ignition, LOI) must be sintered in temperatures slightly higher than the reference one. Unsuccessful sintering, accompanied by incomplete nitrate tests (required both in the estimation of total silica and insoluble residue contents) may result in the presence of impurities in the silica sediment. In such cases the burnt sample must be further treated with hydrofluoric and sulfuric acid making the procedure even more tedious.

By following the above method, the reactive silica contents of the various SCM used in this work are determined and given in Table 3.2.2, section 3.

c. Relationship between k-value and active silica

It is evident that although several authors have attempted to connect the pozzolanic effect with a number of parameters such as, fineness [67,68], water to powder ratio [69], curing temperature [69,70] and alkalinity of the pore solution [71], it 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 [72,73] have not produced a relation between the amount of soluble silica of the examined admixtures and their potential activity. The present investigation aims at filling this gap by introducing a relationship between the reactive silica amount of different SCM and their corresponding k-values, estimated both for mechanical and durability properties. This will lead to a more safe 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 (e.g. strength, service life time, etc.).

In an SCM-cement system, the CSH-content will also be the most critical parameter in strength development. In a previous work [46,47,50,51] was fully established that, as time proceeds, i.e., after 1 year, the following expression can be obtained, giving an estimation for the maximum k-value:

$$\boxed{k_{max} = \gamma_{S,P} f_{S,P} / f_{S,K} \qquad (4.3.8)}$$

As a general conclusion, the Eq. (4.3.8) can be applied for a first approximation of the k-value of the artificial SCM, such as fly ash, slag, silica fume, and some thermal treated natural materials, such as metakaolin. In the case of multicomponent use (simultaneous use of various SCM) in the concrete production, the sum of the active silica of the materials can be introduced in Eq. (4.3.8). However, a significant overestimation was observed for the natural materials [47]. This exception can be attributed either to the formation of a weaker CSH component or to errors in active silica measurement.