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ОПРЕДЕЛЯНЕ НА К-ФАКТОРА НА ЖЕЛЕЗНИЯ СИЛИКАТ, ПРИ ИЗПОЛЗВАНЕТО МУ КАТО МИНЕРАЛНА ДОБАВКА ТИП II В ПРОИЗВОДСТВОТО НА БЕТОН

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DETERMINATION OF THE K-VALUE OF IRON SILICATE FINES WHEN USED AS A TYPE II MINERAL ADDITION IN CONCRETE PRODUCTION

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Abstract:
The growing demand for sustainable construction materials has intensified interest in the utilization of industrial by-products as type II mineral additions in concrete. Iron silicate fines (ISF), generated during non-ferrous metallurgical processes, represent a promising alternative due to its constant physical properties and chemical composition. This study investigates the applicability of ISF within the framework of the k-value concept, as defined in CEN/TR 16639:2014, which enables the incorporation of type II additions in concrete by modifying the traditional water/cement ratio to a water/(cement + k·addition) ratio. The research aims to determine an appropriate k-value for ISF when used in structural concrete. The experimental program is based on continuous testing of compressive strength development for concretes of strength classes C20/25 and C30/37. Mixes incorporating certain proportion of ISF as partial cement replacement are compared with reference concretes with CEM I. The methodology follows the principles outlined in CEN/TR 16639:2014, including the evaluation of strength equivalence and the subsequent assessment of durability-related performance indicators. The k-value concept, being prescriptive in nature, provides a simplified route for standard-compliant use of mineral additions without the need for additional verification procedures. The findings of this study contribute to extending this concept to iron silicate fines, supporting their integration into concrete production. The results demonstrate the potential of ISF as a secondary cementitious material, enabling partial cement substitution while maintaining required mechanical and durability performance. Moreover, ISF has European Technical Assessment for cement substitution from 2023.

Keywords:
iron silicate fines; k-value; cement replacement; sustainable concrete; type II mineral addition; CEN/TR 16639:2014, concrete durability; circular economy in construction.

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1. INTRODUCTION

The construction sector is facing increasing pressure to reduce its environmental impact while maintaining the performance, durability, and economic viability of concrete structures. Concrete remains the most widely used construction material worldwide, and its primary binding component, Portland cement, is responsible for a significant share of global anthropogenic carbon dioxide emissions. Consequently, the incorporation of supplementary cementitious materials (SCMs) especially industrial by-products into concrete has become an important strategy for reducing the environmental footprint of construction materials and promoting circular economy principles.

Among the industrial by-products with potential application in concrete technology are iron silicate fines (ISF), a mineral material generated during the pyrometallurgical production and refining of copper. ISF is co-produced during the treatment of primary and secondary copper-bearing raw materials and consist predominantly of iron silicates, magnetite, and oxide-rich mineral phases. Due to the controlled industrial process from which they originate, ISF exhibits stable physical characteristics and a relatively constant chemical composition over time, which are essential prerequisites for its application in construction materials [1].

In Aurubis Bulgaria, one of the largest copper producers in Europe, iron silicate fines are generated during the slag flotation process. Following copper recovery, the remaining mineral fraction undergoes processing to produce a fine-grained material suitable for various construction applications. The material has been successfully utilized as fine aggregate in concrete and mortar production and is certified according to EN 12620 [2] and EN 13139 [3]. Furthermore, a European Technical Assessment (ETA 23/0286) was issued in 2023, enabling its use in blended cements containing certain amount of iron silicate fines.

The growing demand for alternative cementitious materials is driven not only by environmental considerations but also by the decreasing locally availability of traditionally used SCMs such as fly ash and granulated blast furnace slag. The ongoing transition of the energy sector and changes in steel production technologies have significantly reduced the supply of these materials across Europe. As a result, the concrete industry is actively seeking new mineral additions capable of partially replacing Portland cement while maintaining the required mechanical and durability performance of concrete.

2. DECARBONIZATION OF THE EUROPEAN CONSTRUCTION SECTOR AND THE POTENTIAL OF IRON SILICATE FINES

The European Union has established ambitious climate targets through the European Green Deal and the Climate Law, aiming to achieve climate neutrality by 2050 [4]. These objectives have significant implications for the cement and concrete industries, which together account for approximately 7–8% of global carbon dioxide emissions [5]. The majority of emissions associated with cement production originate from clinker manufacturing, where both fuel combustion and limestone decarbonation contribute to the overall carbon footprint.

As a result, reducing clinker content has become one of the principal pathways for lowering the environmental impact of cementitious materials. European standards increasingly encourage the use of blended cements containing supplementary cementitious materials and other mineral constituents capable of partially replacing clinker. Traditionally, fly ash and ground granulated blast furnace slag have been the most widely used materials for this purpose. However, their availability is steadily decreasing in Europe.

In parallel with clinker reduction strategies, the construction sector is also facing growing concerns regarding the depletion of natural resources. The extraction of natural aggregates and sand has increased substantially during recent decades, leading to environmental pressures and supply constraints in many regions. Consequently, the use of industrial by-products and secondary raw materials has become an important element of sustainable construction practices.

Within this context, iron silicate fines offer a particularly attractive solution. The material is prodcued in significant quantities during copper production. Due to its mineralogical composition, ISF can contribute both as a fine aggregate and as a constituent in blended cement systems. Beyond its application in blended cements, ISF can also be considered within the framework of EN 206 [6] as a type II mineral addition. The relevant provisions are further explained in CEN/TR 16639:2014 [7], which describes the k-value concept, the Equivalent Concrete Performance Concept, and the Equivalent Performance of Combinations Concept. The technical report explicitly allows the extension of the k-value approach to other additions when their suitability has been demonstrated through appropriate testing and performance verification.

The determination of a suitable k-value for ISF would provide a practical and prescriptive route for their use in concrete production. Such an approach would enable concrete producers to account for the contribution of ISF in mix design calculations through the water/(cement + k*addition) ratio, thereby facilitating standard-compliant cement reduction without the need for extensive project-specific verification procedures. Consequently, establishing a scientifically justified k-value for ISF represents an important step toward its broader implementation in sustainable concrete production within Bulgaria.

3. PROCEDURE FOR TESTING A NEW SUPPLEMENTARY CEMENTITIOUS MATERIAL ACCORDING TO CEN/TR 16639:2014

The assessment of a new SCM as a type II mineral addition may be performed using the k-value concept described in CEN/TR 16639:2014. The concept is based on the comparison between a reference concrete produced with cement A only and a test concrete in which part of cement A is replaced by the mineral addition. The comparison is made in terms of compressive strength and, where relevant, durability-related performance.

For concretes containing type II mineral additions, the traditional water/cement ratio is replaced by the equivalent ratio:

$$\omega_0 = \omega_a/(c_a + k.a) \tag{1}$$

here ω_0 is the water/cement ratio of reference concrete without addition; ω_a is the water content of the concrete with addition; c_a is the cement content of the concrete with addition; k is the efficiency coefficient of the addition; a is the addition content.

The k-value expresses the contribution of the mineral addition to the concrete performance compared with cement.

3.1. General principle

The mix design of concrete is normally based on the 28-day compressive strength. Therefore, the k-value determined for a mineral addition is related to the age of 28 days. However, the determination of the k-value shall not be based on strength alone. The durability requirements relevant to the intended exposure classes shall also be considered.

When part of cement A is replaced by the mineral addition, the limiting values applicable to cement A shall remain valid. This means that the same requirements for minimum cement content, maximum water/cement ratio, strength class, and durability performance are used as for the reference concrete.

This process is in two stages:

3.2. Stage 1: Determination of the preliminary k-value from concrete strength tests

In stage 1, k-values are calculated from concrete tests. When testing concrete, all relevant parameters (cement type, strength class, quality of the mineral admixture, water-cement ratio, etc.) must be taken into account to enable the determination of a generally applicable prescribed k-value. For this purpose, test specimens must be prepared: with a percent of additive and

different water-cement ratios, and the percent of additive must be varied until the same strength is achieved in the reference concrete and the tested concrete. Classes C 20/25 and C 30/37 have been developed for this study.

Once the amount of admixture at which equal strength is achieved has been determined, the k-value is calculated using the following formulas:

$$\omega_0 = w_a/(c_a + k.a) \tag{2}$$

$$k = (\frac{w_a}{\omega_0} - c_a)/a \tag{3}$$

here ω_0 is the water/cement ratio of reference concrete without addition; w_a is the water content of the concrete with addition; c_a is the cement content of the concrete with addition; k is the efficiency coefficient of the addition; a is the addition content; ω_a is the water/cement ratio of the concrete with addition (w_a/c_a).

3.3. Verification of concrete performance using the obtained k-value

The objective of Stage 2 is to verify that the concrete designed using the proposed k-value satisfies the relevant durability requirements. At minimum, freeze-thaw resistance shall be considered when relevant. Depending on the intended exposure class, one or more of the following durability aspects shall be tested:

- resistance to carbonation;
- resistance to chloride penetration;
- freeze-thaw resistance with de-icing salts;
- resistance to seawater;
- sulfate resistance.

3.4. Statistical evaluation of durability performance

The evaluation of durability aspects is based on a comparison of n samples (of the cement and mineral admixture) and the test samples made from these samples. Acceptance or rejection is based on the difference in test results between the test samples and the reference concrete. The limit value is based on the following principle: For each durability aspect, a relative difference d [%] (at the population level) is determined, which is considered unacceptable. In other words, an evaluation of the test concrete resulting in an average negative difference d should lead to a high probability of rejection of the test concrete.

The main criteria for the d-values and the required number of samples are:

- For each durability aspect, a fixed difference d [%] that is no longer considered acceptable. The selection of these d-values is based on expert opinion.
- The risk to the consumer is set at 10% of this difference.

The supplier is free to choose the number of samples (at least 3) and can thus limit the risk to the manufacturer.

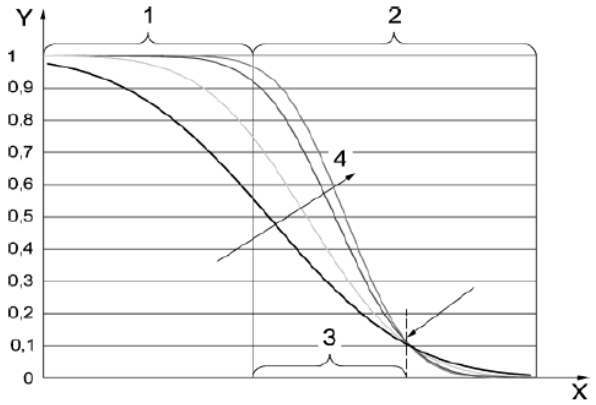

Figure 1. Difference versus probability of acceptance, according to CEN/TR 16639:2014, fig. 8

Legend:
1 test better than reference
2 test worth than reference
3 no longer acceptable difference *(d)*
4 increasing number of samples
X difference test – reference
Y probability of acceptance

For each durability aspect j the assessment is based on the parameter T_j

$$T_j = \frac{[m_r - \frac{m_t}{1 + 0.01 d_j}]}{s/\sqrt{n}} \tag{4}$$

where:

$$s = \sqrt{\left\{ s_r^2 + \frac{s_t^2}{(1 + 0.01 d_j)^2} \right\}} \tag{5}$$

here m_r is the average test result of the n samples of the reference concrete; m_t is the average test result of the n samples of the test concrete; s_r the standard deviation of the averages per sample of the reference concrete; s_t the standard deviation of the averages per sample of the test concrete; n the number of samples; d_j the limit value of the durability aspect j as shown in Table 1.

Table 1. Limit value d (at the population level), according to CEN/TR 16639:2014, table 3.

Durability aspect j	Difference d [%] that leads to rejection with a probability of 90%
Carbonation	+30
Chloride ingress	+30
Freeze-thaw de-icing salt resistance	+30
Seawater resistance	+40
Sulfate resistance	+40
Compressive strength	-30

For approval Tj should be larger than the limit values shown in Table 2:

Table 2. Limit values Tj, according to CEN/TR 16639:2014, table 4.

Number of samples (n)	Limit value Tj
3	1.533
4	1.440
5	1.397
6	1.372
7	1.356
8	1.345
9	1.337
10	1.330
11	1.325
12	1.321

3.5. Acceptance of the proposed k-value

The proposed k-value for the new SCM may be accepted when both of the following conditions are fulfilled:

1. Concrete containing the mineral addition achieves equivalent 28-day compressive strength compared with the reference concrete;
2. Concrete designed using the proposed k-value satisfies the required durability performance for the relevant exposure classes.

4. DETERMINATION OF THE K-VALUE OF IRON SILICATE FINES

4.1. Calculation the k factor by strength

For the purposes of this study, concrete samples corresponding to strength classes C20/25 and C30/37 were prepared. For each concrete class, ISF were incorporated as a partial cement replacement at a dosage level of 5% by mass. All test mixtures contained chemical admixture. The admixture was a liquid, yellow-brown product with a density of approximately 1.04 kg/dm³, designed to maintain concrete workability while promoting high early-age strength development.

Samples were prepared and cured for compressive strength testing at 7 and 28 days. The compressive strength of the concrete samples was determined in accordance with BDS EN 12390-3 [7]. The experimental results obtained for each concrete class are presented in Table 3.

Table 3. Compressive strength measured for classes C20/25 and C30/37

	Concrete with 5% ISF C30/37	Concrete with 5% ISF C30/37	Reference concrete C30/37	Reference concrete C30/37	Concrete with 5% ISF C20/25	Concrete with 5% ISF C20/25	Reference concrete C20/25	Reference concrete C20/25
	28 day	7th day	28 day	7th day	28 day	7th day	28 day	7th day
	55.1	45.6	49.3	48.8	42.8	32.8	39.9	34.8
	53.4	46.0	55.4	48.3	39.3	33.1	40.5	32.7
	54.1	42.0	54.1	48.6	40.6	33.5	39.5	35.0
m_r	54.2	44.5	52.9	48.6	40.9	33.1	40.0	34.2
s_r	0.85	2.2	3.21	0.25	1.77	0.35	0.5	1.27

The results presented in Table 3 demonstrate that the concrete mixtures containing 5% ISF achieved compressive strengths comparable to those of the corresponding reference mixtures. Based on the obtained strength results and in accordance with the k-value methodology, the k-

factors were determined for both concrete strength classes: 0.486 for concrete class C20/25 and 0.476 for concrete class C30/37.

According to the classic formula in EN206 and ACE 211.1 [8] for concrete strength class at 28th day, k can be calculated by the formula:

$$k = \frac{(F_c(concrete + additive) - F_c(reference\ concrete))}{p.F_c(reference\ concrete)} \quad (6)$$

here F_c is compressive strength in MPa; p is percent substitution.

Based on the experimental results, k-values of 0.450 and 0.491 were determined for concrete classes C20/25 and C30/37, respectively.

The close correlation between these values confirms the consistent performance of ISF across different concrete strength classes. Considering the outcomes obtained from both evaluation methodologies, a k-value of 0.5 may be adopted for concrete mixtures incorporating 5% cement replacement with ISF.

4.2. Determination of the freezing-thawing resistance of concrete with ISF

The evaluation of concrete durability was carried out by comparing the test results obtained from three samples: two reference concrete samples and one sample containing ISF. Acceptance of the proposed k-value was assessed based on the difference between the measured test results. For the durability criterion “Resistance to freeze–thaw cycles with de-icing salts,” CEN/TR 16639 specifies the calculation of the statistical parameter T, with a minimum acceptance value of 1.533 for a sample size of three. Accordingly, the proposed k-value may be accepted when the calculated T value exceeds this threshold.

Durability testing was performed on concrete classes C20/25 and C30/37 in accordance with CEN/TS 12390-9 [9], using the test method “Resistance to freeze–thaw cycles with de-icing salts – 56 cycles.” The calculated T values were 2.765 for concrete class C20/25 and 3.763 for concrete class C30/37, both significantly exceeding the required acceptance criterion.

These results confirm that the concretes containing 5% ISF satisfy the durability requirements related to freeze–thaw resistance with de-icing salts. Consequently, a k-value of 0.5 may be adopted for concrete mixtures incorporating 5% iron silicate fines as a Type II mineral addition in accordance with the principles of CEN/TR 16639.

5. CONCLUSION

This present study addressed the growing need for alternative supplementary cementitious materials capable of reducing the cement content and environmental footprint of concrete while maintaining compliance with European standards. Iron silicate fines, a by-product generated during copper production at Aurubis Bulgaria, were evaluated as a type II mineral addition within the framework of the k-value concept defined in EN 206 and CEN/TR 16639:2014. The k-value concept provides a practical and prescriptive approach for incorporating mineral additions into concrete by replacing the conventional water/cement ratio with the water/(cement + k*addition) ratio, thereby accounting for the contribution of the addition to concrete performance. Following the two-stage assessment procedure prescribed in CEN/TR 16639:2014, ISF-containing concretes were compared with reference concretes in terms of compressive strength and durability-related performance. The results demonstrated that a k-value of 0.5 can be assigned to ISF, confirming that the material contributes effectively to the cementitious system and enables partial cement replacement while maintaining equivalent concrete performance. The successful verification of this k-value provides a scientifically justified basis for the use of ISF in structural concrete and complements the existing European Technical Assessment (ETA 23/0286), which already confirms its suitability for use in blended cements. The adoption of ISF as a type II mineral addition offers an additional route for cement reduction,

supports the valorization of industrial by-products in line with circular economy principles, and contributes to the decarbonization objectives of the European cement and concrete industries. The findings of this study therefore demonstrate that iron silicate fines represent a technically viable and sustainable solution for future low-carbon concrete production in Bulgaria.

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