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Effect of ultra-fine fly ash on concrete performance and durability

Patricia Kara De Maeijer[a*], Bart Craeye[a,b], Ruben Snellings[c], Hadi Kazemi-Kamyab[c], Michel Loots[d], Koen Janssens[e] and Gert Nuyts[e]

[a] *EMIB, Faculty of Applied Engineering, University of Antwerp, Groenenborgerlaan 171, 2020 Antwerp, Belgium*
[b] *DUBiT, Department of Industrial Sciences & Technology, Odisee University College, 9320 Aalst, Belgium*
[c] *VITO NV, Boeretang 200, 2400 Mol, Belgium*
[d] *Value Ash Technologies NV, Oosterveldlaan 196, B-2610 Antwerp, Belgium*
[e] *AXES, University of Antwerp, Groenenborgerlaan 171, 2020 Antwerp, Belgium*

*Corresponding author: patricija.karademaeijer@uantwerpen.be (P. Kara De Maeijer)

Abstract
In the present study ultra-fine fly ash as a novel by-product obtained by a dry and closed separation process was investigated as cement replacement in concrete. The impact of ultra-fine fly ash on material properties was investigated following an upscaling as an approach considering paste, mortar and concrete properties. Two types of cement, Portland cement (CEMI) and slag cement (CEMIII), two types of ultra-fly ashes, one with particle size $d_{90} < 9.3$ µm (FA1) and second with $d_{90} < 4.6$ µm (FA2) were used. At paste- and mortar- level, cement was replaced at 0%, 15%, 25%, 35% and 50% with FA1 and FA2. At concrete- level, cement was replaced at 0%, 15% and 25% with different ratios of FA1 and FA2. The results at paste- and mortar- level showed that an increased fineness of the fly ash (FA2) contributes to better workability of the mix. For CEMI, the compressive strength of concrete with FA2 at 25% cement replacement was already equal to the reference 0% replacement concrete at the age of 28 days. For CEMIII, the compressive strength of concrete mix with FA1 with 15% and FA2 with 25 % cement replacements reached the reference concrete value at the age of 91 days. Regarding the durability, replacing cement with ultra-fine fly ash (FA2) had a positive influence on the resistivity, chloride migration coefficient and alkali-silica reaction (ASR), and a negative influence on the carbonation resistance.

Keywords: Concrete; Ultra-fine fly ash; Compressive strength; Corrosion resistance; ASR

Highlights

- Improved reactivity of ultra-fine fly ash;
- Increased workability in case of increased fineness of fly ashes;
- Equal compressive strength of CEMI-FA2-25% to CEMI-REF at the age of 28 days;
- Positive effect on the resistivity, chloride migration coefficient and ASR;
- Negative effect on carbonation resistance.

1. Introduction

Concrete has a dominant role in the modern construction engineering, as second the most-used material in the world after water [1] it serves as a critical component for constructing the infrastructure necessary for the social and economic development, but also brings a negative environmental impact [2]. It is difficult to substitute by other materials due to its mechanical performance, long life and affordable cost on many sites. Nevertheless, the production of concrete is characterized by a considerable demand for energy and raw materials and results in significant emissions of greenhouse gases and has a big impact on the environment [3]. Nowadays, sand and cement are two components in concrete production which are problematic from the point of view of sustainability. For example, Portland cement production is the third-largest contributor of CO_2 emissions after the energy and transportations sectors [4,5]. As of 2018, the annual global cement production is over 4.1 billion tons [6], an increase of 0.3 billion tons is observed within the last five years. Five countries - China (58%), India (7%), US (2.1%), Brazil (1.3%) and Turkey (2%) – produce 70% of the Worlds cement [6]. Cement demand by China has increased exponentially by ~430% in 20 years, while use in the rest of the world increased by 60%. Current policies support material recycling, using non-renewable natural resources more efficiently and minimizing the emission of CO_2 into the atmosphere [7]. At the same time the cement industry is confronted with a continuous increase in cost for energy sources, obligations and commitments to reduce CO_2 emissions and a need for suitable raw materials both in quality and quantity [8]. It is estimated that on average 0.8–1.0 tons of CO_2 are produced for a ton of clinker depending on the type of fuels and kilns used [9]. In total, the cement industry is generating about 8–10 % of the world anthropogenic CO_2 emissions into the atmosphere [10]. This level of emission is becoming increasingly problematic as the demand for construction materials in the developing world continues to grow and alternative technologies are necessary for the construction industry to become environmentally sustainable. The use of recycled materials in sustainable construction applications is one of the most attractive options because of the large quantity, low quality requirements, and widespread sites of construction.

Portland cement partial substitution by solid by-products as pozzolans or supplementary cementitious materials (SCMs) gives reasonable importance for the structural concrete mixture design and practical application in construction although with slightly reduced early age performance in comparison to the reference mixture [11]. Use of pozzolans is known already from antiquity, in combination with slaked lime it formed the binder in Roman concrete [12] and their use is continued to date.

Fly ash (FA), a by-product of coal combustion, along with other pozzolans, is now widely adopted for use as a cement component in concrete by all the major standards, with up to 55% FA content in pozzolanic cement CEM IV 197-1:2011 [13], for example. FA is also recognized as an eco-friendly material as its usage helps to lower the carbon footprint of the cement industry [14]. Based on global coal consumption for electricity generation about 1 Gt/y of fly ashes are generated, of which an estimated third is used in cement and concrete applications [10, 15, 16]. Part of these fly ashes are still landfilled because they do not meet the specifications for use in cement or concrete [17-19]. Further processing of these materials is required to qualify them for use.

Fly ash grain size affects the hydration process in such a way that the whole fine grains turn into hydrates. Though this effect is not only the consequence of fineness but may also result from differences in chemical composition of glass content between size fractions. In general, fly ash inclusion in concrete reduces water demand, improves workability and reduces bleeding and segregation. The benefits associated with these effects enable water contents to be lower and concrete to be designed with reduced water/(cement+fly ash) ratios for equal workability, which is beneficial in terms of strength development, pore structure and durability [20-21]. Investigations into the relationship between a slump and fly ash properties at equal water contents have indicated a strong correlation with fly ash fineness. However, it has been shown that coarse fly ash (fineness > 30%) can be used effectively in conjunction with water-reducing admixtures to achieve equivalent workability and strength to that finer fly ash concrete. Developments in admixture technology have been a boost for developing advanced concrete types, broadening the application field of concrete, allowing concrete solutions for existing problems [22], influence the economics of concrete production.

As the pozzolanic reaction of fly ash is a relatively slow process in general terms, its contribution to concrete strength occurs mainly at later ages, so the early strength (usually considered up to 28 days) is reduced [23-24]. In order to overcome the slow reactivity of fly ashes several approaches were applied previously. On the one hand, one can act on the cement formulation, keeping the fly ash the same: lowering the water/binder ratio [25]; substitution of a high-early strength Portland cement for ordinary Portland cement [26]; replacement of a portion of the fly ash with a more reactive pozzolan such as silica fume or rice husk ash [27]. On the other hand, approaches to increase the reactivity of fly ash by itself by comminution of the fly ash [28-29], addition of chemical activator [30] or elevated temperature curing [31] have been studied. However, the efficiency of some of these methods is debatable because of the high energy consumption and/or the low cost-benefit ratio [20].

In general, post-processing is applied to improve material quality, i.e. it can transform off-spec materials into candidate SCMs that comply with standard specifications, or it can generate SCMs that have superior properties to regular products. In the view of enhancing clinker efficiency, processing of mineral powders to obtain ultrafine materials finer than the cement can enable denser particle packing and can increase SCM reactivity to obtain a denser and more durable concrete microstructure [16].

In this paper ultra-fine fly ash, as a novel by-product, is investigated as a cement replacement in concrete [16, 32]. The ultra-fine fly ash is obtained by post-processing of coal combustion fly ashes by the Dusty Plasma Separation (DPS) technology. A proprietary 0.2 tonne/h prototype DPS was used [33-34]. In the device fly ashes are separated based on particle size in a dry, closed system. The principle of separation uses a combination of air drag and charging forces to separate the particles. Compared to conventional air classifiers the DPS device has a lower per tonne energy consumption of about 15 kWh/t and experience less abrasion during operation. The DPS devices classifies fly ash in four main size fractions: fine fly ash (FA1), ultra-fine fly ash (FA2), and medium and coarse fly ash fractions. In this study both the fine and the ultra-fine fly ash were included to study potential positive effects on concrete properties. Application testing of the medium and coarse fly ash fractions are out of scope of the present paper but include conventional use as fly ash in concrete, or as filler in ceramic or composite materials.

The goal of the current study was to determine the influence of ultra-fine fly ashes of different fineness on concrete workability, mechanical properties (compressive and flexural strength) and durability (alkali-silica reaction (ASR), chloride migration and carbonation resistance). A well-balanced concrete mix composition with ultra-fine fly ashes acting as a cement replacer was developed by means of optimal packing design algorithms.

2. Materials and methods

2.1. Materials

Two types of cement were used: Portland cement CEM I 52.5 R HES (CEMI) and Blast Furnace Slag Cement CEM III/A 42.5 N LA (CEMIII) with a density of 2985 kg/m³ and 2991 kg/m³, respectively. The chemical composition of both cements is shown in Table 1.

Two types of Class F fly ash were used: separated fly ash (FA1) with particle size < 9.3 µm and ultra-fine fly ash (FA2) < 4.6 µm. FA2 is a sub-fraction of FA1 and was obtained by a 0.2 tonne/h prototype DPS [33]. The particle size distribution is shown in Fig. 1. FA1 and FA2 have a density of 2538 kg/m³ and 2524 kg/m³, respectively, defined by helium pycnometry. The chemical composition and loss of ignition of both fly ashes are shown in Table 1. The complete characterization and pozzolanicity of the same enhanced fly ashes used in this paper can be found in [16].

Typical concrete aggregates were used: river sand 0/4, limestone 2/6 and limestone 6/20. Different types of superplasticizer were used: Type 1 – PCE-type (polycarboxylic ether), 30 con%; Type 2 – PCE type, 30 con%; Type 3 – PCE-type, 35 con%; Type 4 – PAE-type (polyacrylether), 30 con%.

Table 1. Chemical composition of cements and fly ashes

Constituent	CEMI [%]	CEMIII [%]	FA1 [%]	FA2 [%]
CaO	66.9	57.8	2.7	2.7
SiO_2	17.2	22.6	53.2	53.6
Al_2O_3	3.8	6.5	26.0	26.4
Fe_2O_3	2.5	1.8	8.4	8.6
MgO	1.9	4.0	1.8	1.8
Na_2O	0.2	0.3	1.6	1.6
K_2O	1.8	1.1	3.7	3.7
Na_2O-eq	1.4	1.0	4.0	4.0
SO_3	5.3	5.1	1.1	1.0
Cl^-	0.1	0.1	-	-
LOI	-	-	4.1 [16]	4.3 [16]

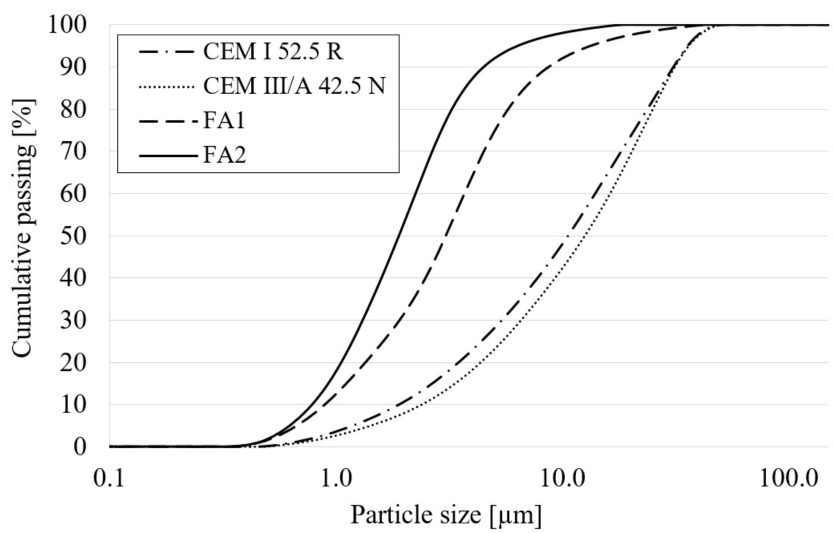


Fig.1. Particle size distribution of fly ashes and cements.

2.2. Experimental program

The behavior of ultra-fine fly ash was investigated by the upscaling principle in cement-rich environments such as paste, mortar and concrete. The term 'upscaling' is used in terms of paste-level as the start of the experimental program, followed by mortar-level and ended at concrete-level (Fig. 2).

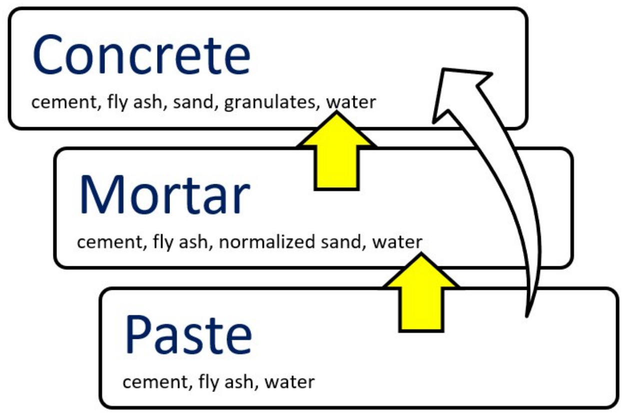


Fig. 2. Upscaling principle of experimental program [35].

2.2.1. Paste level

In the present study the behaviour of FA1 and FA2 was investigated by the upscaling principle in cement-rich environments starting from the paste level. In order to observe the effect on workability of pastes by the compatibility of all superplasticizers, two cements with five cement replacement levels by two fly ashes, the explicit testing program would consist of 100 pastes in total. Based on observations during experiments only 51 pastes with 4 superplasticizers, 2 types of cements and 5 different cement replacement levels (0%, 15%, 25%, 35% and 50%) with FA1 and FA2 were performed: 17 pastes with CEMI and FA1, 8 pastes with CEMI and FA2, 19 pastes with CEMIII and FA1 and 7 pastes with CEMIII and FA2.

The workability of cement pastes was defined by mini-flow table tests in accordance with EN 1015-3 [36] to determine the consistency of mortars. The mini-flow test setup consisted of a truncated stainless-steel cone (h=60 mm, d_{base}=100 mm, d_{top}=70 mm) placed on a flat, smooth base plate with a diameter of 300 mm. Each paste mix consisted of 500 g of binder (cement with or without fly ash, depending on the replacement level) and 225 g of water. The water/binder factor was always 0.45. Superplasticizer was added in varying amounts to reach the flow diameter in the range of 250-300 mm. Considering the results, one of the four superplasticizers was selected for mortars and concrete production at the next steps of the experimental program.

2.2.2. Mortar level

Eighteen mortar compositions were prepared using an automatic mortar mixer in accordance with standard EN 196-1:2016 [37]. In total 54 mortar prisms of 160x40x40 mm^3 have been prepared. After 24h the prisms were demolded and placed under water at a temperature of 20±2°C. The mortar specimens' composition consisted of cement (CEMI or CEMIII), fly ash (FA1 or FA2), normalized sand and superplasticizer (Type 3). Five different cement replacement levels (0%, 15%, 25%, 35% and 50%) with FA1 and FA2 were performed. The choice of dosage of superplasticizer was based on the best flowability of pastes. Mini-slump tests were performed according to the standard EN 12350-2 [38]. The mini-slump test setup consisted of a truncated cone (h = 150 mm, d_{base} = 100 mm, d_{top} = 60 mm) placed on a flat, smooth base plate with a diameter of 300 mm. The flexural strength of the hardened mortar prisms (40x40x160 mm^3) was determined by performing a three-point bending test in accordance with standard EN 196-1:2016 [37]. The flexural strength was determined at the age of 7, 28 and 57 days after production. The compressive strength tests were carried out at the age of 7, 28 and 57 days in accordance to EN 196-1:2016 [37].

2.2.3. Concrete level

A concrete composition with optimal packing density is a very important factor that allows making durable concrete with high strength (above 60 MPa). The concrete mix compositions with optimal packing design was obtained with the software Excel Tool Mix Design (v 1.01) offered by Betonica. This software uses an optimization curve, the Fuller curve, to realize this optimal packing. This curve is suitable for generating a good ratio between the sand and the coarser aggregates. A disadvantage of this curve is the fact that it is less useful to create a continuous distribution for the finest fractions (cement, fly ash). The choice for the Fuller-curve was nevertheless validated: the cement quantity and the percentages of the fly ash replacement were fixed in advance, so no adjustment of the cement/fly ash-ratio was possible. Regarding the packing model (strength model), the choice was made for the Dewar model. The software automatically calculates the best possible concrete compositions. However, the obtained graphs showed that the curve of the solid structure, which represents the particle size distribution of the complete mix, did not approach perfectly the Fuller curve. It was decided to optimize obtained by the software concrete compositions in a manual manner by validating the reference mix experimentally. The proportions between sand and coarse aggregates were adjusted, and then a theoretical, optimal composition was confirmed.

The current experimental planning was based on the logical approach of expected results from the paste and mortar levels to an optimal number of concrete mixes to test which give a high compressive strength and corresponding durability results. In total 8 different concrete mixes were produced (Table 2). Four concrete mixes with CEMI: the reference mix with 0% fly ash (CEMI-REF), the mix with 15% FA1 cement replacement (CEMI-FA1-15%), the mix with 25% FA2 cement replacement (CEMI-FA2-25%) and the mix with 25% cement replacement with FA1 and FA2 (CEMI-FA1+FA2-25%). And, four concrete mixes with CEMIII: the reference mix with 0% fly ash (CEMIII-REF), the mix with 15% FA1 cement replacement (CEMIII-FA1-15%), the mix with 25% FA2 cement replacement (CEMIII-FA2-25%) and the mix with 25% cement replacement with FA1 and FA2 (CEMIII-FA1+FA2-25%). In the present study, each concrete mix composition had a constant water/binder ratio of 0.45.

Fifteen cubes (with side 100 mm or 150 mm) and four cylinders (with height 200 mm and diameter 100 mm) were produced per mix according to EN 206:2013+A1:2016 [39] and resulting in a total of 152 specimens to test mechanical properties and durability. According to research of Van Der Vurst et al. [40], in case of high strength concrete, it is considered that the shape/size factor between cubes with side 100 mm and cubes with side 150 mm is more or less equal to one.

After demolding, the specimens were cured in a water bath at a temperature of 20±2°C until the age of testing. The slump test was performed in accordance with standard EN 12350-2 [38]. The target was to achieve a slump class S3-S4, which should ensure a sufficiently consistent concrete. The flow test was performed to determine the consistency in accordance with standard EN 12350-5 [41]. The volumetric mass (density) of the fresh concrete was determined according to the standard EN 12350-6 [42]. The air content of fresh concrete was determined with the pressure gauge method according to standard EN 12350-7 [43]. The compressive strength of the concrete specimens was determined using an automatic hydraulic press in accordance with standards EN 12390-3 [44] and EN 12390-4 [45].

Table 2. Concrete mix compositions with CEM I and CEM III.

	CEM-REF [kg/m³]	CEM-FA1-15% [kg/m³]	CEM-FA2-25% [kg/m³]	CEM-FA1+FA2-25% [kg/m³]
CEM I 52.5 R (CEM III/A 42.5N)	360	306	270	270
FA1	-	54	-	36
FA2	-	-	90	54
River sand 0/4	471 (514)	471 (514)	471 (514)	471 (514)
Limestone 2/6	663 (641)	663 (641)	663 (641)	663 (641)
Limestone 6/20	755 (733)	755 (733)	755 (733)	755 (733)
Water	162	162	162	162
Superplasticizer (Type 3)	1.2 (0.9)	1.2 (0.9)	1.2 (0.9)	1.2 (0.9)
W/B-factor	0.45	0.45	0.45	0.45

In order to compare the compressive strength results of concrete mixes specimens with different dimensions (cubes with side 100 mm or 150 mm), a correlation of the obtained values was used [46]:

$$(f_{ccub})_{150} = 0.91 * (f_{ccub})_{100} + 3.62\ N/mm^2 \quad (1)$$

Ultrasonic pulse velocity tests were carried out on cube specimens with dimensions 100x100x100 mm^3 using the Pundit Lab from Proceq, in accordance to EN 12504-4 [47]. This test allowed to investigate the uniformity, general quality and durability of the concrete. The ultrasonic tests were carried out 3, 7, 14, 21, 28, 35, 42, 49, 56 and 63 days after concrete production.

The electrical resistivity ρ of concrete was measured using the Proceq Resipod corrosion meter, according to the 'Wenner probe' principle and to AASHTO T 358 [48]. The resistivity tests were performed on cylindrical test specimens with a height of 200 mm and a diameter of 100 mm. For each concrete mix composition, two cylinders were tested at 3, 7, 14, 21, 28, 35, 42, 49, 56, 91 and 263/270 days after concrete production.

The chloride migration was determined by the chloride migration test according to the Nordtest standardized method NT Build 492 [49]. Six concrete specimens (±50 mm in height, ±100mm in diameter) per one concrete mix were tested at a time. The setup for the chloride migration test is shown in Fig. 3.

Fig. 3. The test setup for chloride migration.

Resistance to carbonation was measured on: (1) specimens stored outside protected from rain/snow and exposed to the environment for 13 months and (2) specimens stored for 11 months outside and 2 months in an accelerated laboratory chamber where they were exposed to an environment with 1% CO_2 at a temperature of 20 ± 2 °C and relative humidity of 60 ± 10%. The carbonation depth was measured according to EN 14630 [50] by spraying phenolphthalein (1 %) over the axially split specimens.

The ASR test was performed in accordance with [51]. The mortar bar specimens with dimensions 25x25x285 mm^3 were cast in prismatic plywood molds and demolded 24 h after casting. In total 8 mortars for ASR were prepared (with 3 specimens per each mortar):

- 2 reference mortars: CEMI-REF and CEMIII-REF,
- 6 mortars with cement substitution with fly ash: CEMI-FA1-15%, CEMI-FA2-25%, CEMI-FA1+FA2-25%, CEMIII-FA1-15%, CEMIII-FA2-25% and CEMIII-FA1+FA2-25%), identical to the concrete mix composition cement replacement levels.

The mortars were mixed in accordance with the mixing procedure EN 196-1:2016 [37]. The mortar workability was measured by a flow table test according to EN 12350-2 [38] and a superplasticizer (Type 3) was added to the mortar mixes in order to achieve the required flow of 205-220 mm. The prisms were placed in water and transferred to an oven at 80±2°C for a period of 24 hours. Immediately after this, to avoid shrinkage, the initial length L_0 of each sample was measured. Thereafter, specimens were immersed in a container filled with a 1M NaOH-solution which was already at a temperature of 80±2°C. The container was sealed airtight and placed in a climate chamber/oven at 80±2°C for a period of 28 days (Fig. 4). The length L_n of the mortar prisms was measured at the age of 7 and 28 days.


(a)


(b)

Fig. 4. The ASR samples (a) and curing setup (b).

3. Results and discussion

3.1. Paste level: workability

The results of the slump test showed that the more cement is replaced with ultra-fine fly ash, the better is the workability in case of the reference mixes without superplasticizer for both cement types (see Table 3). Superplasticizer (Type 1) made pastes more viscous with an increasing cement replacement. Superplasticizer (Type 2) at some point didn't have any effect / or had a slight effect on the flowability of the pastes with increasing fly ash amount. Superplasticizer (Type 3) gave a very good flowability and required a smaller dosage in comparison to other superplasticizers. Superplasticizer (Type 4) in combination with FA1 made the pastes more viscous, and in combination with FA2 more flowable. Superplasticizer (Type 3) gave an excellent flow to the paste (and to concrete mix in general) at a smaller dosage in comparison to the other superplasticizers. Selecting the best suitable superplasticizer, the cost of materials can be limited, which is highly important on a large-scale production. In addition, the results showed that the ultra-fine fly ash (FA2) did have a positive influence on the flowability. This advantage enables the use of less superplasticizer or the reduction of the water/binder factor, the latter having a favorable effect on the strength and durability of concrete. FA1, as a slightly coarser ultra-fine fly ash, didn't have such a positive effect on the flowability of the pastes. The higher fineness of the FA2 appeared to have a positive effect on the slump of the pastes.

Table 3. Mini-flow table test results, the superplasticizer dosage (%) is mentioned in brackets.

		Superplasticizer				
		REF	**Type 1**	**Type 2**	**Type 3**	**Type 4**
CEMI-FA1						
Replacement [%]	0	22.2 (0)	27.0 (0.9)	27.8 (0.9)	>30 (0.9)	26.8 (0.9)
	15	24.0 (0)	-	27.8 (0.9)	28.8 (0.8)	-
	25	24.0 (0)	-	-	-	-
	35	23.5 (0)	26.5 (0.9)	-	-	25.6 (0.9)
	50	24.3 (0)	25.3 (0.9)	27.5 (0.9)	27.8 (0.8)	25.8 (0.9)
CEMI-FA2						
Replacement [%]	0	22.2 (0)	27.0 (0.9)	27.8 (0.9)	>30 (0.9)	26.8 (0.9)
	15	23.0 (0)	-	29.0 (0.9)	30.0 (0.8)	-
	25	24.0 (0)	27.4 (0.9)	-	-	28.3 (0.9)
	35	24.3 (0)	-	-	-	-
	50	24.5 (0)	-	-	-	-
CEMIII-FA1						
Replacement [%]	0	22.8 (0)	28.3 (0.5)	27.0 (0.5)	26.8 (0.3)	28.3 (0.5)
	15	24.3 (0)	-	27.8 (0.3)	26.0 (0.2)	-
	25	24.9 (0)	27.5 (0.5)	-	30.0 (0.5)	-
	35	25.5 (0)	27.0 (0.5)	28.0 (0.5)	-	-
	50	24.5 (0)	25.5 (0.3)	27.8 (0.4)	25.5 (0.3)	24.5 (0.5)
CEMIII-FA2						
Replacement [%]	0	22.8 (0)	28.3 (0.5)	27.0 (0.5)	26.8 (0.3)	28.3 (0.5)
	15	25.5 (0)	-	28.3 (0.3)	27.8 (0.2)	-
	25	26.0 (0)	28.0 (0.5)	-	-	-
	35	-	-	27.0 (0.5)	-	-
	50	26.8 (0)	-	-	-	-

***22.2 (0) – flow diameter [mm] (added amount of plasticizer per mortar[ml])**

3.2. Mortar level: workability and strength development

The results of the mini-slump test per cement-fly ash mix per replacement level are shown in Fig. 5. The amount of superplasticizer (Type 3) varied between the different mortar mixes and was based on the results of workability obtained at the paste level.

CEMI-mixes required a higher dosage of superplasticizer than CEMIII-mixes to achieve the same workability. In this respect, working with CEMIII, it has an economic advantage compared to CEMI. In addition, a higher replacement level (more cement replaced by fly ash) resulted in a higher slump value at the mortar level. A remarkable workability trend can be noticed for the mortars with CEMI and fly ashes when with 15% cement replacement with FA2 a reasonable improvement of workability with a lower SP dosage. Through, with a higher cement replacement level with fly ashes and a lower SP dosage, it can be noticed that workability is a slightly lower than for mortars with FA2 than for FA1. The finer fly ash and lower SP dosage provides higher workability for the mortars with cement replacement until 25%.

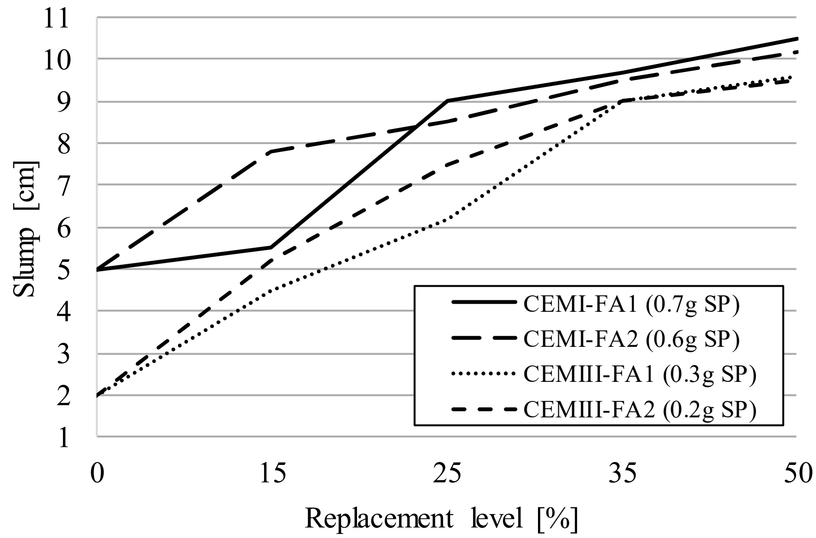


Fig. 5. Mortar mini-slump test results.

The mixes with FA2 at the age of 28 days have a higher flexural strength over the entire time than FA1 (see Fig. 6). This suggests a positive effect of the ultra-fine fly ash FA2 with respect to the less fine fly ash FA1. Compared with CEMI, the CEMIII-mixes showed a similar but slightly different pattern of flexural strength evolution. At the age of 28 days, the mix with 15% FA2 cement replacement obtained almost equal flexural strength in comparison to the reference mix. The reference CEMIII mix has a higher compressive strength than the average of the mixes with fly ash. This is another indication that the optimum replacement level for the flexural strength at mortar-level can be achieved with the lower cement replacement levels. The influence of the fineness of the fly ash is not immediately apparent, because the values of the mixes with the same replacement level roughly correspond.

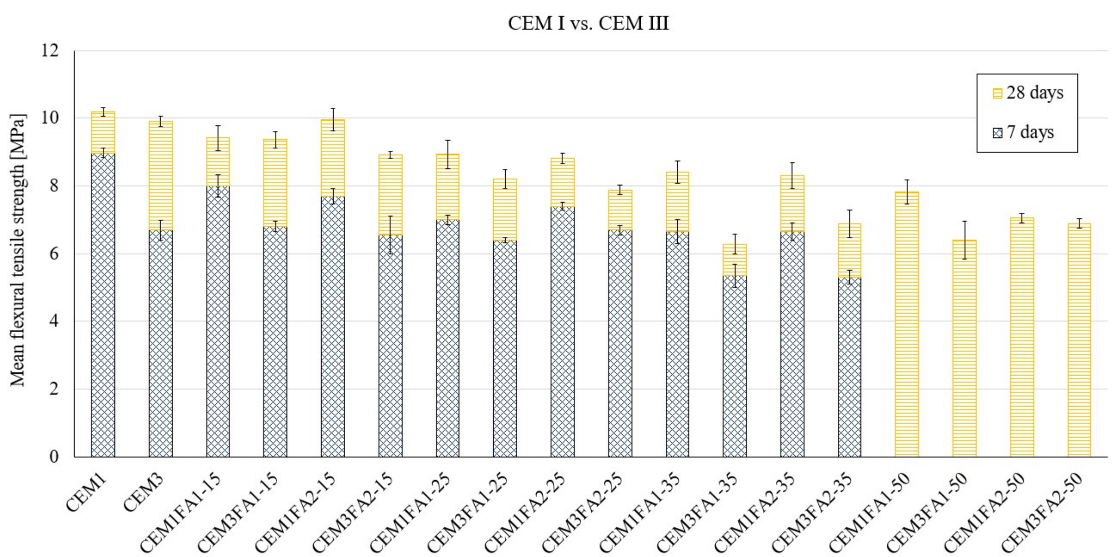


Fig. 6. Mortars flexural strength.

A different situation is observed in terms of compressive strength (see Fig. 7). The compressive strength of CEMI-REF is higher than the fly ash mixes at an early age due to the early start of cement hydration. At a later age, at 57 days, the mixes with 15% and 25% fly ash showed the highest compressive strengths around 80 MPa. The optimal replacement level in terms of compressive strength seems to be close to these two replacement levels in case of CEMI. Replacing cement with fly ash at a higher level than 25% will reduce to the strength of a mortar specimen. The mortars with FA2 show higher results than the mortars with FA1 replacement at the age of 57 days. However, the difference is very small, equal to 2.2%.

The reference mortar mixes with CEMIII achieved higher compressive strength at an early age than mixes with fly ashes. The compressive strength of the mixes with 15% cement replacement at the age of 28 days reached the compressive strength value of the reference mixes and at the age of 57 days exceeded this value with an average value of 10%. The effect of the fineness of the fly ash less obvious as in the CEMI-mortars. At the age of 57 days, the FA2-mixes achieved higher values than the mixes with FA1 with an average improvement of 5%. The general conclusion from the tests at the mortar-level is that in terms of compressive strength the optimal replacement level of cement with fly ash varies between 15% and 25%, and higher results were achieved with ultra-fine fly ash FA2.

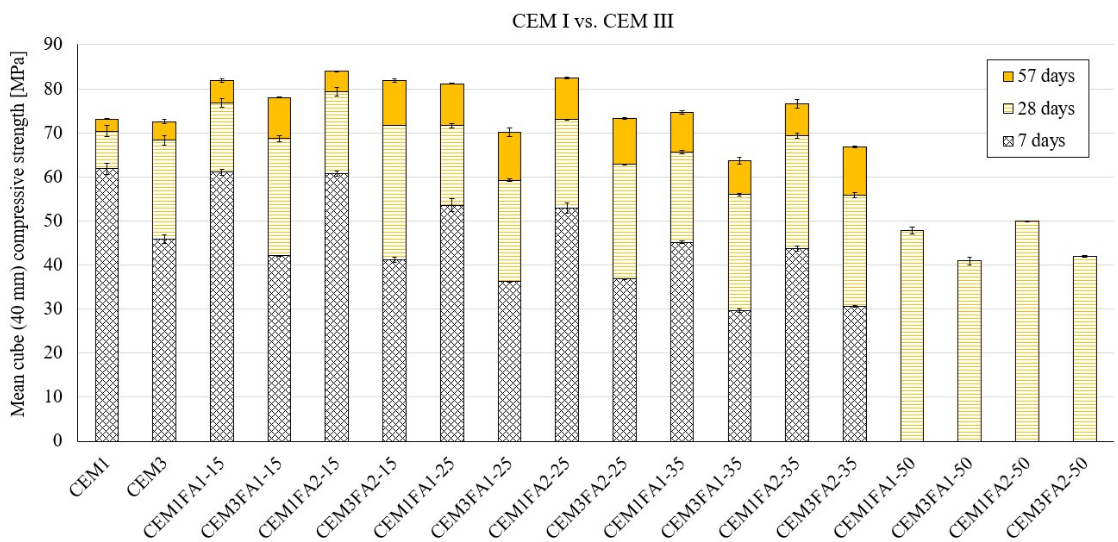


Fig. 7. Mortars compressive strength.

3.3. Concrete level: workability, mechanical properties and durability

3.3.1. Concrete formulation and fresh properties

As soon as slump class S3 or S4 was reached, the flow test was carried out. The results of the slump and flow tests are shown in Table 4. For the concrete mixes with CEMI and CEMIII, a constant dosage of superplasticizer of 1.2 kg/m³ and 0.9 kg/m³ respectively was used. When the cement replacement level increased, the slump increased, and the concrete had a higher flowability. In addition, concrete with 25% cement (CEMI or CEMIII) replaced with FA2 had a greater slump value than concrete with the same level but with a combination of FA1 (10%) and FA2 (15%). This indicated that the use of fly ash with a higher fineness (FA2) results in better workability. The densities of the compacted concrete specimens were relatively constant, around 2400 kg/m³ (Table 4). Both concrete mixes with CEMI and CEMIII contained a smaller amount of air when more cement was replaced with fly ash (Table 4). The air content decreased with an increasing replacement level, which is also confirmed by literature [52]. The results of compressive strength are shown in Fig. 8.

Table 4. Workability, density and air content of concrete mixes.

Concrete mix	Slump [mm]	Slump class	Flow class	Density [kg/m³]	Air content [%]
CEMI-REF	120	S3	F3	2396	2.00
CEMI-FA1-15%	160	S4	F2	2397	1.60
CEMI-FA2-25%	190	S4	F3	2408	1.40
CEMI-FA1+FA2-25%	185	S4	F3	2433	1.50
CEMIII-REF	120	S3	F2	2400	2.20
CEMIII-FA1-15%	160	S4	F4	2407	1.40
CEMIII-FA2-25%	160	S4	F3	2404	1.15
CEMIII-FA1+FA2-25%	150	S3	F4	2396	1.10

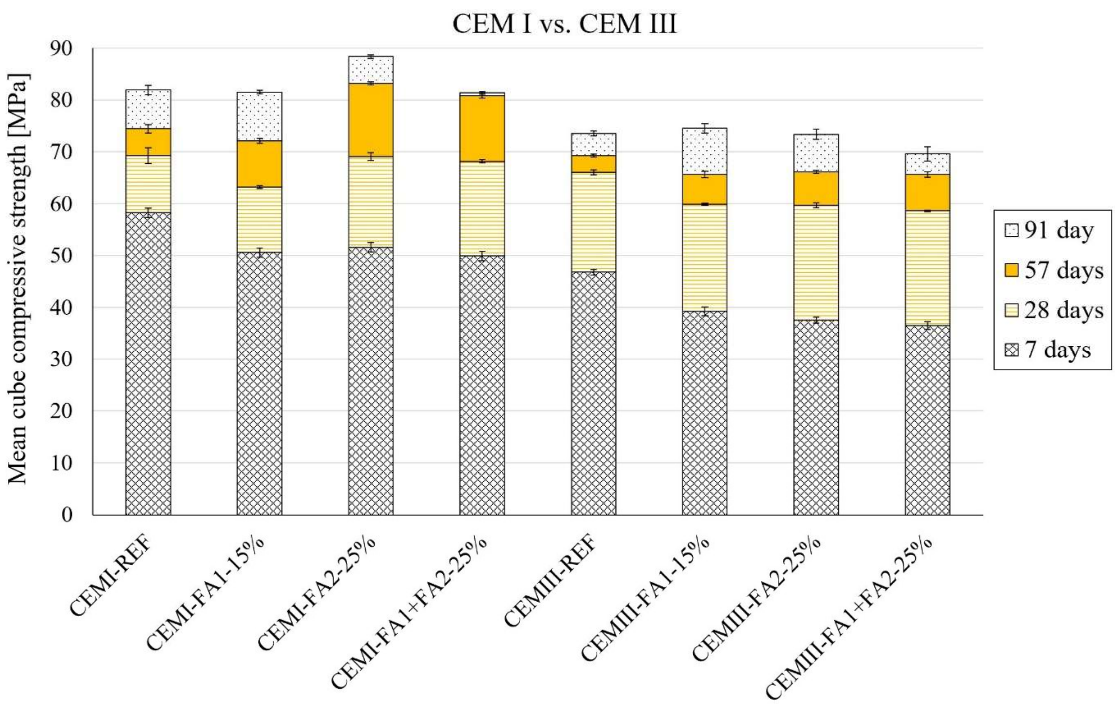


Fig. 8. Compressive strength development of concrete mixes.

3.3.2. Concrete mechanical performance

At the age of 28 days, two mixes with 25% cement substitution (CEMI-FA2-25% and CEMI-FA1+FA2-25%) approached the reference mix CEMI-REF compressive strength value (70 Mpa). From this, it can be inferred that a higher fineness results in a higher pozzolanic reactivity and the reference compressive strength is reached at the age of 28 days and not at the later ages like for conventional fly ash as mentioned in the literature [53]. It is known that the pozzolanic activity of a fly ash depends upon many parameters such as fineness, amorphous content, chemical and mineralogical composition, and the unburned carbon content or loss on ignition of the fly ash [54]. In an earlier study on the same fly ashes, it was shown that both FA1 and FA2 were enriched in amorphous phase compared to medium and coarse fractions resulting from the post-processing. However, it was concluded from reactivity modelling that at least for early age reactivity fineness was a predominant factor when considering fractions of the same initial fly ash [16]. This is explained by only partial reaction of the fly ash at early age, resulting in the total amorphous fraction not being a limiting factor, while at the same time the composition of the glassy phase is usually homogeneous over different fly ash size fractions [55]. Concrete mix CEMI-FA2-25% obtained a compressive strength of 88 Mpa at the age of 91 days (7.8% higher in comparison to CEMI-REF value). The same compressive strength value was obtained for CEMI-FA1-15% in comparison to CEMI-REF at the age of 91 days.

It was observed that at the age of 28 days the combination of CEMIII with fly ashes gave lower results in comparison to the reference mix. Considering that there is less Portland cement clinker in CEM III producing $Ca(OH)_2$ is needed for the pozzolanic reaction of fly ash, lower results in comparison to concrete mixes with CEMI and fly ashes were noticed. The compressive strength of CEMIII concretes with fly ash was almost equal at the age of 28 and 57 days, despite the use of different fly ashes and a different replacement levels. An increase of compressive strength was observed at 91 days when concrete mix CEMIII-FA1-15% reached value of the reference mix of 74 Mpa and CEMIII-FA2-25% of 73 Mpa. The fineness of the fly ash and the replacement percentage did not affect the compressive strength of the concrete with CEMIII.

The ultrasonic measurements are shown in Fig. 9 and Fig. 10. It can be seen that at the age of 3 days, the ultrasonic pulse velocity of each concrete mix is at least 4900 m/s. At a later age, the pulse velocity increases to a maximum of ± 5600 m/s. The velocity of this ultrasonic wave gives an idea of the quality of the concrete. An ultrasonic pulse velocity above 4000 m/s indicates a more compact concrete composition [47], which proves the necessity of the application of optimal packing design for concrete mix design. Though, the effect of fly ash and its fineness on the results of the ultrasonic tests is limited.

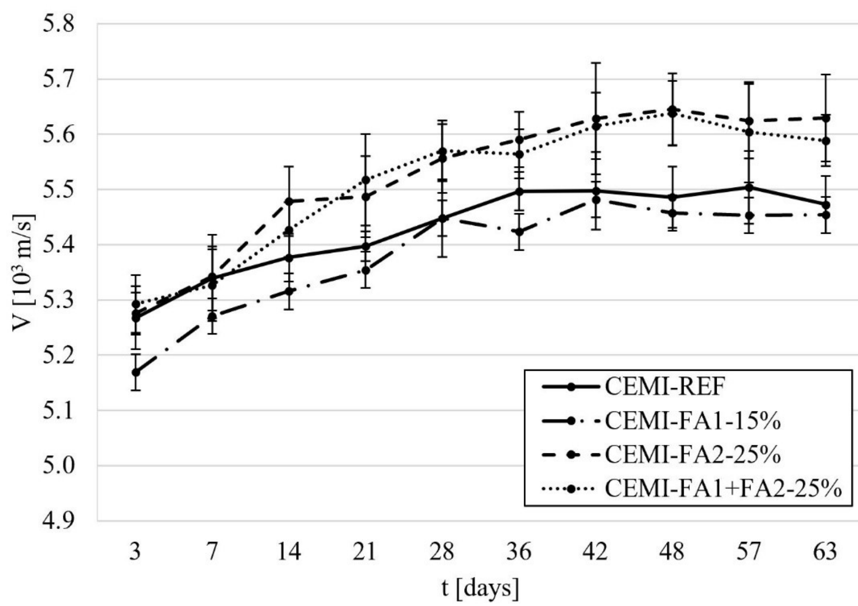


Fig. 9. Ultrasonic pulse velocity concrete CEMI-mixes.

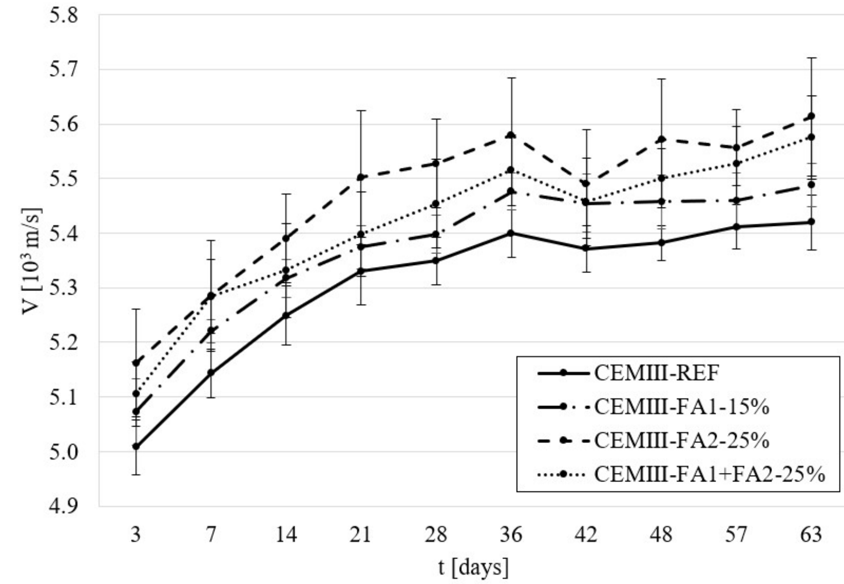


Fig. 10. Ultrasonic pulse velocity concrete CEMIII-mixes.

3.3.3. Concrete resistivity

At an early age, after 3 and 7 days, the reference mixes of CEMI and CEMIII have slightly higher resistivity than the CEMI- and CEMIII-mixes with fly ash (see Fig. 11 and Fig. 12). This is due to a denser, less porous or less connected microstructure of the reference mixes, which results in a faster strength development at early age. From 14 days the resistivity of the mixes with fly ash exceed the values of the reference concrete mixes.

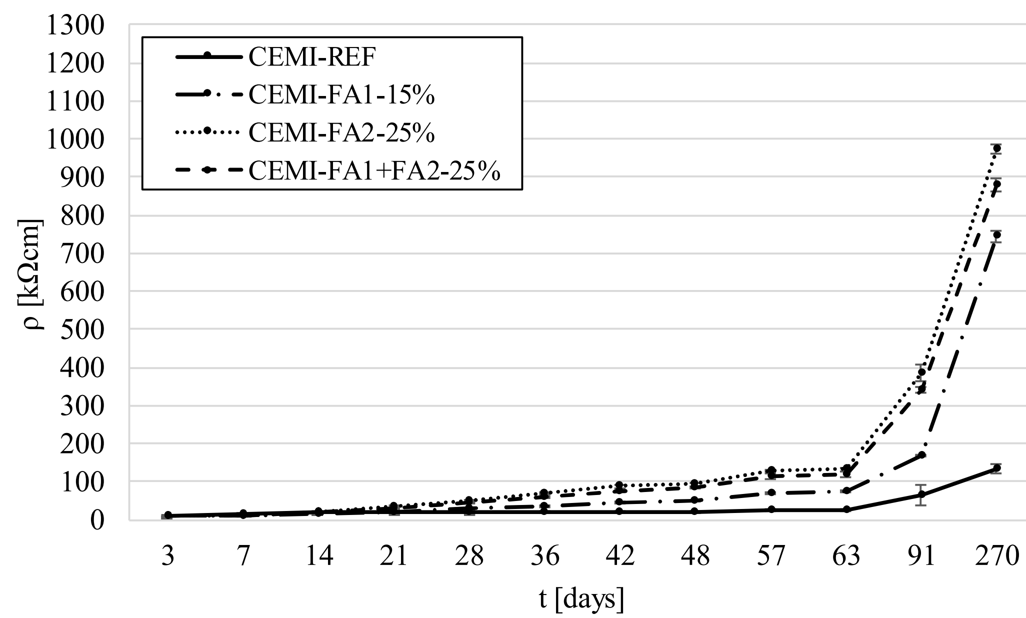


Fig. 11. Resistivity of concrete CEMI-mixes.

The reason for this is the pozzolanic reaction of the fly ash which starts later than the cement hydration. From 21 and 28 days, there is a separation between the different mixes. The concrete mixes with 25% substitution have the highest resistivity, followed by the mix with 15% and below that the reference mix, both for CEMI and CEMIII. This could be related with the well-known pore structure refinement, which increases tortuosity and increases the path electrons/ions need to travel through the specimen.

At the age of 270 days, CEMI-FA2-25% has a resistivity of 974 kΩcm. This value is 7.3 times higher in comparison to CEMI-REF at the same age. For CEMIII, CEMIII-FA1-15% obtains the best value of 1211 kΩcm after 263 days, which is 3.1 times higher than CEMIII-REF (Fig. 11 and Fig. 12). It is notable that all mixes with fly ash rise rapidly, in particular at a later age. However, that is not the case with the reference mixes. If to compare increase of resistivity between CEM I and CEM III concrete mixes with fly ashes within time period of 63 days and 270 days, it can be seen that resistivity increases for CEM I in 7 times and for CEMIII in 3 times faster in comparison to cement types and reference mix values. The following conclusion is valid for both CEMI and CEMIII: the higher the replacement level, the greater the resistivity and thus the corrosion resistance at a late age. This is the result of the pozzolanic reaction that starts later but continues longer than the cement hydration. In addition to the replacement level, the fineness of the fly ash also plays an important role in the development of resistivity and the associated corrosion resistance. Concrete mix CEMI–FA2-25% has a higher resistivity than concrete mix CEMI–F1+F2-25%. So, a higher fineness results in a greater electrical resistivity and a better resistance to corrosion. The finer fly ash particles result in a stronger pozzolanic reaction because of the higher specific surface area and greater content of particles in the vitreous phase.

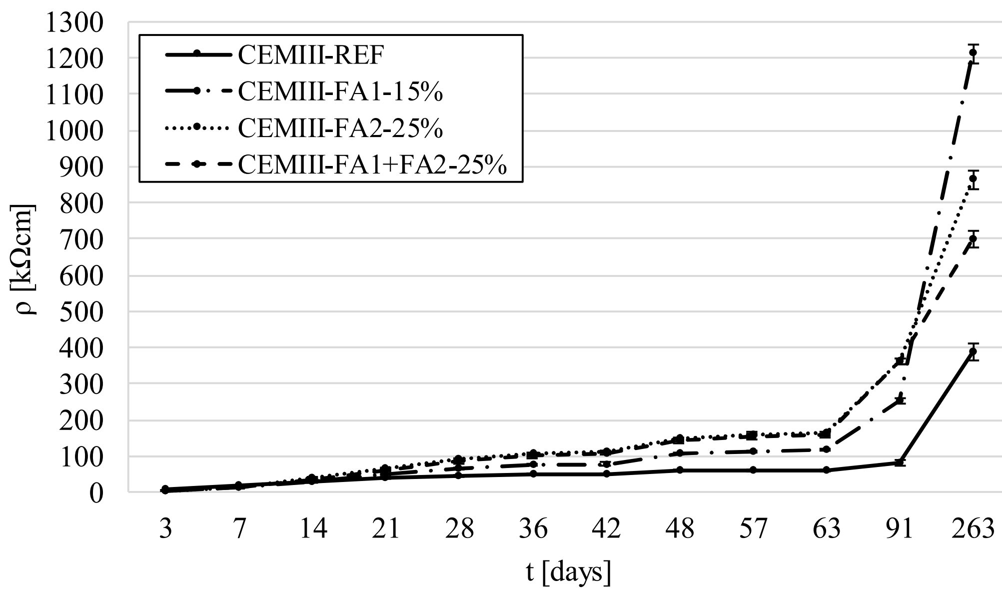


Fig. 12. Resistivity of concrete CEMIII-mixes.

3.3.4. Concrete alkali-silica reaction expansion

ASR expansion tests showed that replacement by ultra-fine fly ash strongly reduced expansion (see Fig. 13 and Fig. 14). From Fig. 13 it appears that the replacement of 25% cement by FA2 is the most optimal to control the expansion after 28 days of immersion. For the mixes with CEMIII (Fig. 14) the replacement of 25% cement by the combination of FA1 (10%) and

FA2 (15%) performed best. It can be concluded that among the test replacement levels, 25% is the most effective cement replacement to limit the risk of harmful ASR in the current study. The results for the CEMI- and CEMIII-mixes did not clearly show that the fineness of fly ash influences the resistance to ASR. After 28 days of immersion in the NaOH-solution, the expansion of CEMI-REF was significantly greater than the expansion of CEMIII-REF, e.g. four times higher. This expansion difference was due to the larger amount of alkalies in CEMI (Portland cement) than in CEMIII (Blast Furnace Slag cement) (see Na_2O-eq in Table 1) [56]. It can be concluded that the choice of cement is an important factor in limiting the expansion and consequently preventing harmful ASR.

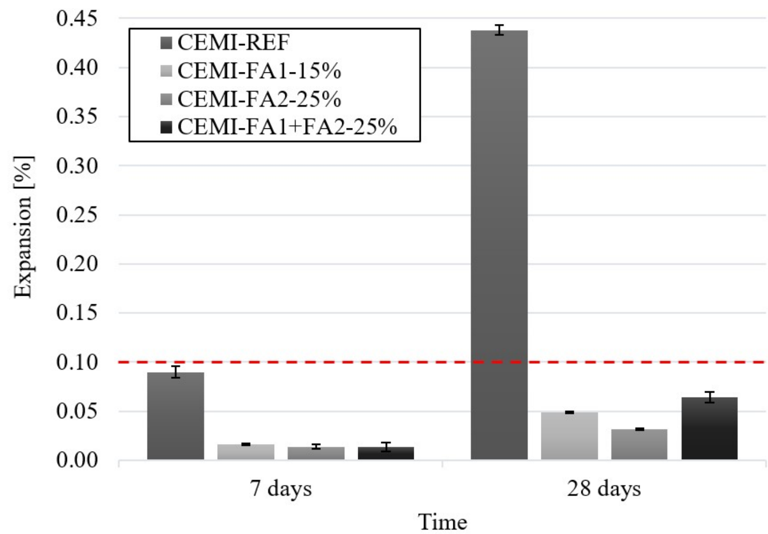


Fig. 13. Expansion of the mortar CEMI-mixes.

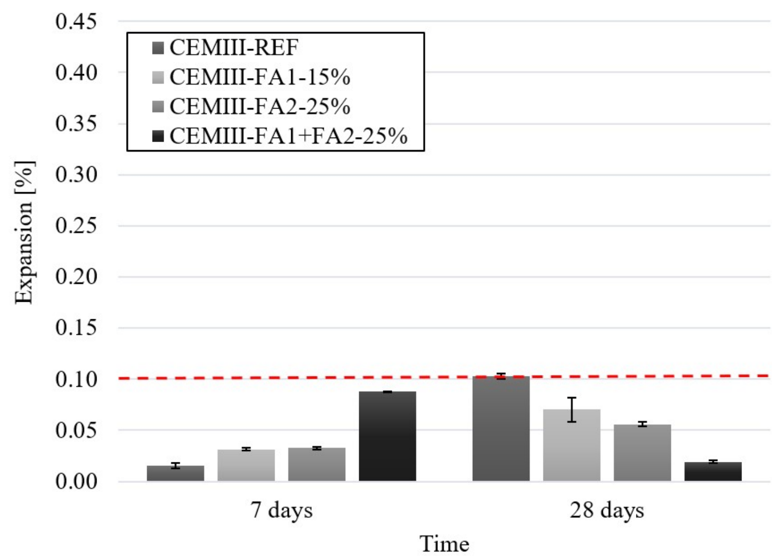


Fig. 14. Expansion of the mortar CEMIII-mixes.

According to RILEM recommended test method AAR-2 [51], a threshold expansion of 0.10% after 16 days of storage in a NaOH-solution is recommended (red line in Fig. 15 and Fig. 16). If the expansion exceeds this threshold, precautions in terms of aggregate selection will need to be taken to minimize the risk of ASR damage to any concrete. The results after 28 days of storage show that the expansion of CEMI-REF after 16 days will be much higher than the limit value, which makes it almost impossible to avoid harmful ASR. On the other hand, the expansion of CEMIII-REF will probably not exceed this maximum value after 16 days. The use of FA reduces the expansion of the mortar bars and makes sure that the limit value is not approached. This exemplifies the well-known durability impact of cement replacement by FA [57].

3.3.5. Concrete chloride resistance

The addition of FA in concrete had a positive influence on the chloride migration coefficient (Fig. 15). Usually, the chloride migration coefficient of the FA concrete is much smaller than that of the reference concrete after 91 days [58]. The porosity of the concrete also has a major influence on chloride penetration. A lower porosity results in a slower chloride penetration. Furthermore, the binding capacity is very important. The better cement is able to bind chlorides, the smaller the free chloride content is and therefore the longer it will take before the critical chloride content is reached. A higher Al_2O_3 or Fe_2O_3 content of the fly ash will show a higher binding capacity. FA has a positive effect on the time-invariant chloride binding capacity of concrete and the binding capacity increases with the increase of FA replacement ratio [59]. Determination of chloride penetration depth can mainly be applied to concrete compositions that are exposed to thawing salts, seawater, brackish water and chloride-containing solutions. In the current study, chloride migration was considerably higher for the reference concrete with CEMI. In the case of fly ash concrete compositions, the migration coefficient was higher for combinations with CEMIII (Al_2O_3 – 6.5%) than for CEMI (Al_2O_3 – 3.8%). Concrete specimens with FA2 had a lower chloride migration coefficient (Fig. 16 and Fig. 17). This can be due to the fineness of fly ash that ensured a denser packing. Replacing cement with fly ash was the most effective for CEMI and replacing this cement with 25% FA2.

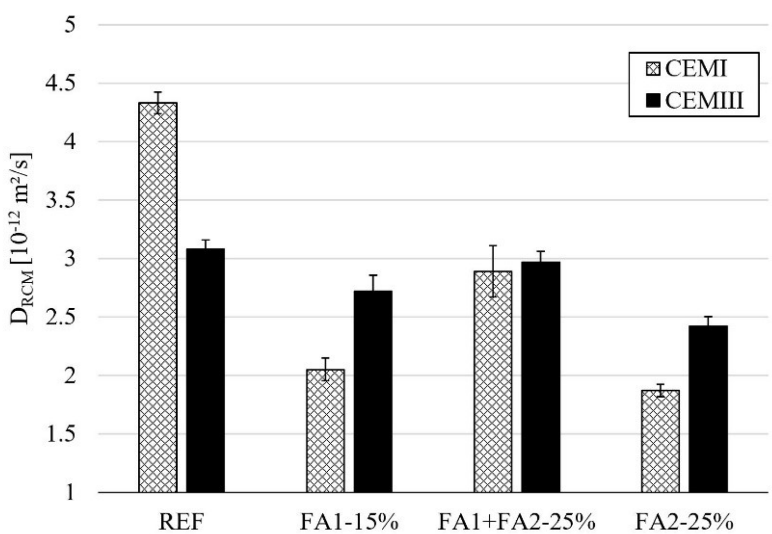
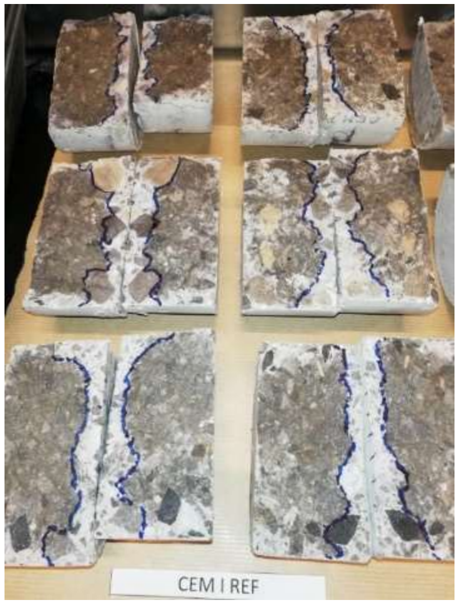


Fig. 15. Chloride migration test results.



(a) (b)

(b) (d)

Fig. 16. Chloride migration specimens: (a) CEMI-REF, (b) CEMI-FA2-25%), (c) CEM III-REF and (d) CEMIII-FA2-25%).

3.3.6. Concrete carbonation resistance

Reaction of concrete with atmospheric CO_2, carbonation, reduces the internal pH of the concrete and can cause reinforcement corrosion. In general terms, there is consensus in the literature published over the last 35 years that the use of FA as a cement component in concrete gives rise to increased carbonation rate of the concrete, the extent of which in general increases with FA content. The extent of this increase varies depending upon a series of factors associated with the mix design parameters such as FA characteristics and water/binder ratio and the concrete curing conditions and the age upon exposure to CO_2 [14]. In terms of concrete microstructure, mainly the $Ca(OH)_2$ content of the cement and to a lesser extent the pore structure affect the carbonation resistance. The pozzolanic reaction of FA consumes $Ca(OH)_2$, and therefore, also reduces the cement carbonation resistance. The use of FA as a cement component requires careful consideration in the design of concrete mixes and effective curing in order to produce FA concrete of similar carbonation resistance to reference concrete. It should be noted that atmospheric conditions at which carbonation rates are high (RH 60-70 %), do not coincide with conditions at which steel corrosion occurs (RH > 95%). Therefore, only for exposed elements that undergo cyclic meteoric conditions such balconies or facade elements, carbonation induced corrosion occurs. FA fineness and cement content replacement were found to influence the carbonation of FA concrete, becoming more visible at FA contents of about 25%. The carbonation rate was found to increase with increasing fineness of FA and cement replacement level, in line with a higher pozzolanic reactivity of ultra-fine FA2. It was observed that the carbonation depth was greater for CEMIII compared to CEMI (CEMI - 66.9% CaO and CEMIII - 57.8% CaO) (Fig. 17 and Fig. 18), confirming the dominant impact of $Ca(OH)_2$ content (lower in CEMIII than in CEMI) over binder microstructure. If the test specimens with the same composition and different age are compared, the carbonation depth also increased with increased age. The carbonation was higher for CEMI FA2 25% and CEMIII-FA1+FA2 25% by means of accelerated carbonation test (2).

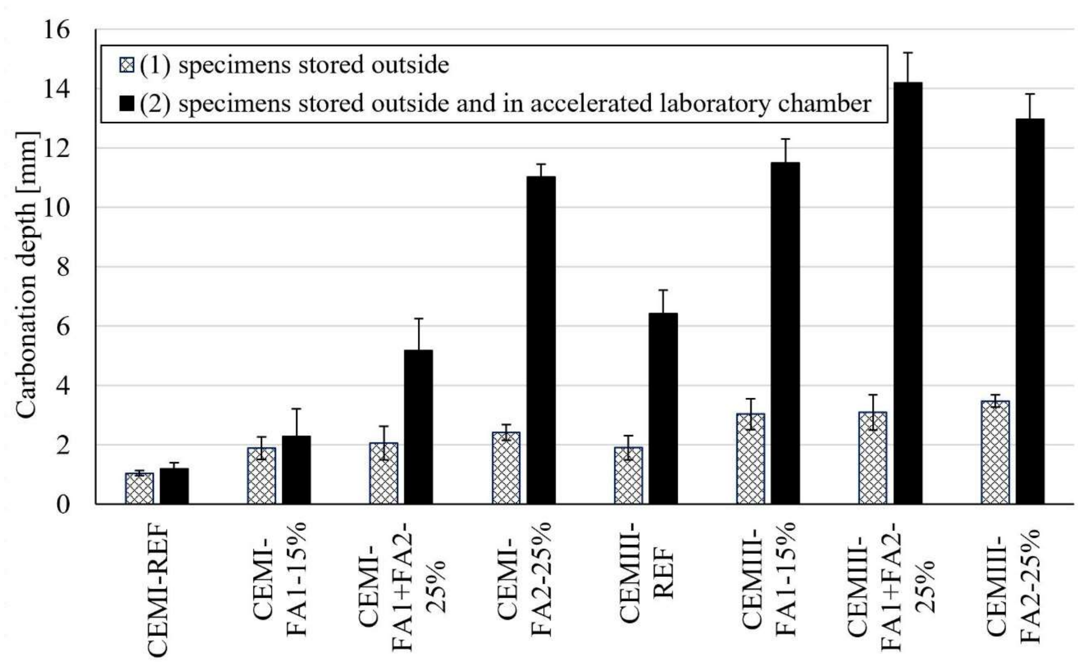
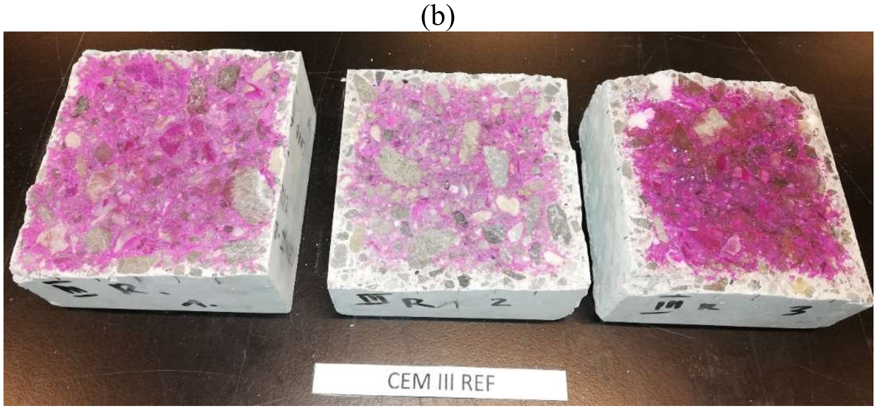


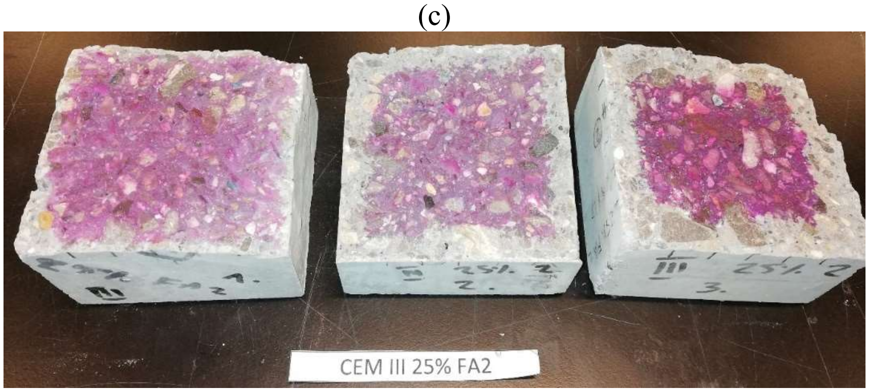
Fig. 17. Carbonation test results.

(a)

(b)



(c)



(d)

Fig. 18. Carbonation (2) test specimens: (a) CEMI-REF, (b) CEMI-FA2-25%), (c) CEM III-REF and (d) CEMIII-FA2-25%).

4. Conclusions

The results at the paste- and mortar- level showed that an increased fineness of the fly ash (FA2) contributed to better workability of the concrete mix. For CEMI, the compressive strength of concrete with FA2 with 25% cement replacement was already equal to the reference mix at the age of 28 days. However, for CEMIII, the compressive strength of concrete mix with FA2 with 25% cement replacement was still below the reference mix value at the age of 57 days and reached it at the age of 91 days. Regarding the durability, replacing cement with ultra-fine fly ash (FA2) had a positive influence on the resistivity, chloride migration coefficient and ASR, and a negative influence on the carbonation resistance.

CRediT authorship contribution statement

Patricia Kara De Maeijer: Data curation, Methodology, Investigation, Formal analysis, Writing - original draft. **Bart Craeye:** Methodology, Writing - review & editing, Project administration. **Ruben Snellings:** Methodology, Investigation, Writing - review & editing, Project administration. **Hadi Kazemi-Kamyab:** Methodology, Investigation. **Michel Loots:** Resources, Project administration. **Koen Janssens:** Investigation. **Gert Nuyts:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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