

ESTIMATION OF CHLORIDE MIGRATION COEFFICIENT IN AIR-ENTRAINED CONCRETES CONTAINING FLUIDIZED BED COMBUSTION FLY ASH

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The objective of this investigation was comparing the penetration of chloride ions in ordinary and air-entrained concretes containing a waste material Fluidized Bed Combustion Fly Ash (FBCFA). All concretes were tested with 15% and 30% cement replacement by FBCFA, with the same water-binder ratio of 0.45. Two kinds of fly ash coming from fluid bed combustion in two power plants in Poland have been used.

In this study the rapid chloride permeability test – Nordtest Method BUILD 492 method – was used. The microstructure of the concrete was analyzed on thin polished sections and the measurement of air voids sizes and their distribution, using digital image analysis, was carried on according to PN-EN 480-11:2008.

Obtained results have shown a significant influence of partial cement replacement by FBCFA on the chloride ions movements in concrete. It has been found that this kind of addition reduced considerably the chloride ion penetration. The influence of air entrainment on the chloride diffusion coefficients was also measured and it was shown that application of air-entraining admixture for concretes with FBCFA reduce the chloride diffusion coefficient but it should be used with caution.

Key words: Fluidized Bed Combustion Fly Ash (FBCFA); pore size distribution; chloride permeability; concrete; durability.

1. INTRODUCTION

Various kinds of civil engineering concrete structures are exposed to chloride ingressive environment; there are road pavements in cold regions where de-icing agents are used, structures in marine regions, harbour and offshore structures, etc. Reinforcement in these structures is exposed to chloride-induced corrosion, which destroys the passivated film on rebars embedded in concrete, subsequently cracking occurs due to corrosion products and finally the service live of reinforcement concrete structures is reduced, [1]. A chloride resistant concrete is a high durable concrete whose chloride diffusion coefficient is very low, i.e. below 2×10^{-12} m²/s according to Nordtest Method BUILD 492, [2]. Such concrete may be produced by rationally chosen suitable materials and mix proportions. It is well known that the addition of silica fume in binary blends with

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Portland cement positively contributes to reduce the permeability of chloride ions into concrete, [3]. Studies in the early 1970s [4], demonstrated that the addition of natural pozzolana to Portland cement (PC) may reduce the chloride diffusion coefficient of concrete by three times. Addition of fly ash has an even more significant effect on the diffusion coefficient [4], due to the reduction of permeability and diffusivity of concrete. When mineral additions are used as substitutes for cement, the pore size becomes smaller. In concretes with partial cement replacement when compared with the ordinary concrete, a higher total volume of mercury intrusion (MIP) is observed. However, most of this volume is concentrated in pores with size smaller than 50 nm, [5], that are not suitable for penetrating of chlorides. At early ages, the cement paste is more porous when the mineral addition is used but later in hardened concrete the transition zone between aggregate and matrix becomes denser as a result of the use of pozzolana. The concrete containing mineral addition is therefore finally less susceptible to the ingress of harmful chloride ions, [6][7].

Since the sustainability development is more and more important for our infrastructure, the new by-product Fluidized Bed Combustion Fly Ash (FBCFA) is taken into consideration as a concrete ingredient. Due to the limestone in the combustion process and the low temperature of combustion (appr. 850°C) FBCFA differs distinctly in its physical, chemical and mineralogical properties from the ash commonly used as mineral additive in concrete industry, [3]. Several investigations on application of FBCFA were already carried out, with sometimes contradicting results, [8-13]. The amount of $\text{Ca}(\text{OH})_2$ in cement pastes is reduced in case of cement pastes with FBCFA addition compared with control one. Therefore FBCFA shows better pozzolanic activity compared to traditional fly ash, [9]. Havlica et al. [10] have found that FBCFA exhibited different hydraulic properties, yielding C-S-H phase and ettringite as main product of hydration. Conn et al. [11] have shown that FBCFA cannot be used as a cement replacement in concrete due to its unacceptably high sulfur content, while later Zieliński [11], Chugh et al. [13] and Glinicki [14] proved that FBCFA can be used in concrete application if rational proportions are maintained.

Mechanically activated FBCFA was found to be more effective in increasing the compressive strength of mortars and concretes. At the content of 35% in case of mortars as well as at the 40% in case of concretes good effects were observed at ages of 7, 28 and 90 days, [11]. The influence of FBCFA on both the fresh mix properties and concrete strength was tested in [14]; the results showed benefits in strength and durability of FBCFA applications as concrete component. Chugh et al. [13] built piers to support photo-voltaric arrays and three years later the piers showed no signs of deterioration of any kind and the achieved strength values exceeded the design expectations.

The FBCFA is a relatively new by-product and its influence on the concrete durability, e.g. on chloride ions diffusion was never before tested in a systematic way. In this paper the influence of FBCFA on the chloride migration coefficient in air-entrained and non air-entrained concrete is analyzed on the basis of test results. Two kinds of

FBCFA coming from selected power plants in Poland were studied. The effects of cement replacement by fluidized fly ash on the migration of chloride ions in concretes were investigated during 360 days and the air-void microstructure were determined. Additionally the dependency between compressive strength and chloride diffusion coefficient was studied.

2. EXPERIMENTAL PROGRAM AND DETAILS

2.1. MATERIALS AND MIXTURE PROPORTIONS

Two series of concretes were made; 'B' – ordinary concrete and 'C' – with air-entraining admixture. Ordinary Portland cement type I from Małogoszcz cement plant was used. Gravel from "Niwka" deposit, fractions 2÷8 mm and 8÷16 mm, and river sand fraction 0÷2 mm, were used and constant water to binder ratio $w/b = 0.45$ was maintained for all mixes. To keep the relatively constant slump and a porosity of fresh mix around 5%÷6% in air-entrained concretes (class XD3 according to PN-EN 206-1:2003, [15]) different amounts of superplasticisers were applied. Fluidized fly ash from hard and brown coal combustion in the thermal-electric power station denoted respectively "K" and "T" were used. The reference concretes were made without FBCFA. The Portland cement mass was replaced by FBCFA in 15% and 30%. Chemical and physical properties of Portland cement type I, ordinary fly ash and CFBC fly ash are shown in Table 1. Table 2 presents the mix design proportions for 10 different concretes with different cement replacement by two kinds of FBCFA.

For each mix three cylindrical specimens ($\varnothing 100 \text{ mm} \times 200 \text{ mm}$) for chloride migration tests were cast and cured. One test was performed on three specimens ($\varnothing 100 \text{ mm} \times 50 \text{ mm}$). The compressive strength f_c , was determined on concrete cubes 150 mm (six specimen for each mix) and air-voids content – 100 mm (two specimens for each mix). All the specimens were demoulded after 48 h and stored in water at room temperature until the testing day.

2.2. DESCRIPTION OF TEST PROCEDURE FOR NT BUILD 492 TEST

The chloride penetration test for this study was based on the standard of NordTest Build 492 – Non-Steady State Migration Test, [2]. The principle of the test is to subject the concrete to external electrical potential applied across the specimen and to force chloride ions to migrate into it, [16]. After the specified period of time, depending of the initial current intensity, the specimen is split open and sprayed with silver nitrate solution, which reacts to give white insoluble silver chloride on contact with chloride ions. This provides a simple physical measurement of the depth (Fig. 1) to which the sample has been penetrated and has the advantage of being unaffected by the chemistry of the pore solution within different varieties of concrete, [17].

Table 1

Chemical composition and physical characteristics of cement, siliceous fly ash, and FBCFA, [20], [21].
Skład chemiczny oraz fizyczna charakterystyka cementu, popiołu lotnego krzemionkowego i popiołu fluidalnego, [20], [21]

Chemical compounds	CEM I	Ordinary fly ash	FBCFA	
			from hard coal K	from brown coal T
SiO ₂	21.4	50.8	47.18	36.47
Fe ₂ O ₃	3.5	8.6	6.8	4.4
Al ₂ O ₃	5.7	23.9	25.62	28.4
TiO ₂	NA	1.11	1.08	3.84
CaO	64.1	4.0	5.84	15.95
MgO	2.1	2.8	0.15	1.65
SO₃	2.1	0.8	3.62	3.8
Na ₂ O	0.5	0.8	1.18	1.64
K ₂ O	0.92	2.9	2.36	0.62
Cl ⁻	0.029	0.02	0.1	0.03
CaO_{free}	0.9	0.6	3.4	4.75
Specific gravity [g/cm ³]	3.15	2.16	2.68	2.75
Los on ignition, 1000°C/1h	1.1	2.9	3.4	2.73

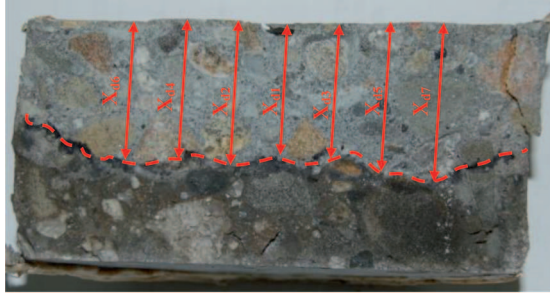
Table 2

Composition of the concrete mixes in kg/m³ and results of concrete compressive strength.
Skład mieszanek betonowych w kg/m³ oraz wyniki wytrzymałości na ściskanie

Mix	Cement	Additive		Aggre- gate	Water	Plasti- cizer	HRWR	AEA	Compressive strength, [MPa]			
		T	K						28 days	90 days	180 days	360 days
Content [kg/m ³]												
B0	360	-	-	1859	162	3.2	4.3	-	55.0	70.0	72.1	73.9
B15K	306	-	54	1854	162	3.2	3.2	-	56.2	64.3	64.9	69.9
B30K	252	-	108	1847	162	3.2	3.2	-	51.6	61.0	63.0	67.2
B15T	306	54	-	1850	162	3.2	4.7	-	60.3	70.4	72.3	73.8
B30T	252	108	-	1841	162	3.2	5.6	-	58.7	66.3	69.1	72.0
C0	380	-	-	1822	171	3.4	2.7	0.4	46.3	49.8	54.2	54.8
C15K	323	-	57	1813	171	3.4	2.5	0.6	47.2	48.4	49.6	57.3
C30K	266	-	114	1803	171	3.4	3.4	0.6	46.8	56.4	60.1	64.8
C15T	323	57	-	1810	171	3.4	3.8	0.6	45.3	50.1	53.8	60.9
C30T	266	114	-	1800	171	3.4	4.8	0.6	46.3	47.7	49.3	49.9

HRWR – high range water reducer, AEA – air-entraining admixture

The conformity criteria for concretes according to Non-Steady State Migration Test, [2] are based on the voltage magnitude, temperature of anolite measured on the



$$x_d = \frac{1}{7} \sum_{i=1}^7 x_i = 29.8 \text{ mm}$$

Specimen \varnothing 100 mm, depth – 50 mm

Fig. 1. Example of the depth of chloride ions penetration in a concrete B0, specimen tested after 28 days of curing.

Rys. 1. Przykład głębokości wniknięcia jonów chlorkowych w beton B0, próbka badana po 28 dniach dojrzewania

beginning and end of test and the depth of chloride ions penetration, are shown in Table 3. The non-steady-state migration coefficient, D_{nssm} , is calculated from equation derived from the second Fick's law:

$$(2.1) \quad D_{nssm} = \frac{0.0239(273 + T)L}{(U - 2)t} \left(x - 0.0238 \sqrt{\frac{(273 + T)Lx}{U - 2}} \right),$$

here:

D_{nssm} – non-steady-state migration coefficient, $\times 10^{-12}$ [m²/s],

U – absolute value of the applied voltage [V],

T – average value of the initial and final temperature in the anolyte solution [°C],

L – thickness of the specimen [mm],

x – average value of the penetration depths [mm],

t – test duration [h].

Table 3

Estimation of the concrete resistance to chloride ions penetration, [22].
Określenie odporności betonów na wnikanie jonów chlorkowych, [22]

Diffusion coefficient	Resistance to chloride penetration
$< 2 \times 10^{-12}$ m ² /s	Very good
$2 - 8 \times 10^{-12}$ m ² /s	Good
$8 - 16 \times 10^{-12}$ m ² /s	Acceptable
$> 16 \times 10^{-12}$ m ² /s	Unacceptable

2.3. DESCRIPTION OF THE AIR-VOID ANALYSIS

Air-content A , specific surface α , spacing factor \bar{L} and the content of micropores below 0.3 mm A_{300} in the hardened concrete specimens were measured with the PN-EN 480-11, [18] method on plane sections after 28 days. Technique of careful preparation of specimen surface was used and described in [19]. Flat specimens of planar dimensions 100×100 mm were polished with SiC powders of different gradation. A special program for automated air void analysis was used based on the linear traverse method was used. Each specimen was tested using 45-traverse lines. Digital image analysis of plane sections was performed using stereomicroscope Nikon SMZ 800 and Image Pro Plus image analysis software.

Additionally, the evaluation of the concrete microstructure was performed using thin sections prepared from concrete before the tests. The fluorescent epoxy impregnated thin sections used for this study were prepared according to [23]. The concrete thin section was ground to thickness of 25-30 μm . Thin section analysis were prepared using optical polarizing microscope Olympus BX51 connected with digital camera. The thin sections were examined in ordinary light, crossed polarized light (also with lambda plate) and fluorescent light.

3. RESULTS AND DISCUSSION

Compressive strength

The values of the compressive strength are shown in Table 2. The results show that different behavior is observed in the concretes of series B and series C. This can be explained by the different grain size compared to the Portland cement and also the content of CaO_{free} in FBCFA was 5-7 times higher than in traditional fly ash, Table 1, which can influence the cement hydration process.

In ordinary concretes the most rapid increase of compressive strength was noticeable between 28 and 90 days of maturity period, further up to 360 days the increase of compressive strength was negligible. After one year the similar values of compressive strength achieved on two concretes, the reference concrete without addition of FBCFA – B0 and the concrete with 15% of FBCFA from brown coal – B15T. Non air-entrained concretes with FBCFA from brown coal exhibited slow hardening but achieved comparative values to the reference concrete only after 360 days.

The air entraining admixture (series C) decreased the compressive strength compared to non air-entrained concretes (series B). On the contrary to the series B, in air-entrained concretes the highest values of the compressive strength were achieved for concretes with 30% of FBCFA from hard coal (C30K) – at 60 days and at 90 days. All values of compressive strength obtained after 28 days in series C were comparable, but after one year the air-entrained concretes with addition of FBCFA showed higher compressive strength than the reference concrete C0.

Chloride ions migration

Fig. 2 and Fig. 3 present the effect of FBCFA content on chloride migration coefficients in non air-entrained (B) and air-entrained (C) concretes. There was a decrease in non-steady-state migration coefficient D_{nssm} in all series as the curing period increased up to 360 days. The results show also the general trend in almost all concrete mixtures that value of D_{nssm} decreased with increased FBCFA content because of the changes in concrete microstructure.

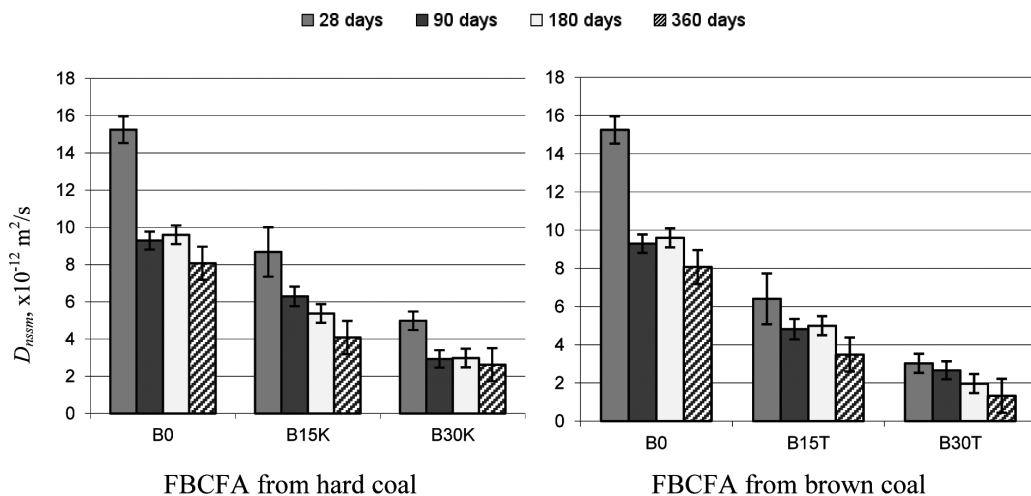


Fig. 2. Chloride migration coefficients for non air-entrained concretes, series B.

Rys. 2. Współczynnik migracji jonów chlorkowych w nienapowietrzonych betonach, seria B

The concretes without FBCFA were the ones that showed the highest values of D_{nssm} (only acceptable resistance to chloride penetration). In all concrete series the chloride migration coefficient tested after 90 days showed relative stabilization, Fig. 2 and Fig. 3. In almost all concretes the differences in chloride migration coefficients tested after 90 and 180 days are small and their values are smaller than standard deviations value. Concretes in series B with FBCFA from brown coal showed lowest values of D_{nssm} compared to concretes with FBCFA from hard coal, both 15% and 30% of cement replacement by FBCFA. In non air-entrained concretes – series B – with FBCFA both from hard coal and brown coal, showed decrease in D_{nssm} proportional to the FBCFA content.

In air-entrained concretes – series C, better resistance to chloride penetration is achieved in concretes made with FBCFA from hard coal. The differences in D_{nssm} between concretes C15K and C30K are small and can be neglected. The reference concrete C0 and concretes made with FBCFA from hard coal showed systematic decreasing of chloride migration coefficient during the maturity period. In concrete with FBCFA from brown coal with 30% cement replacement (C30T) it was found that the

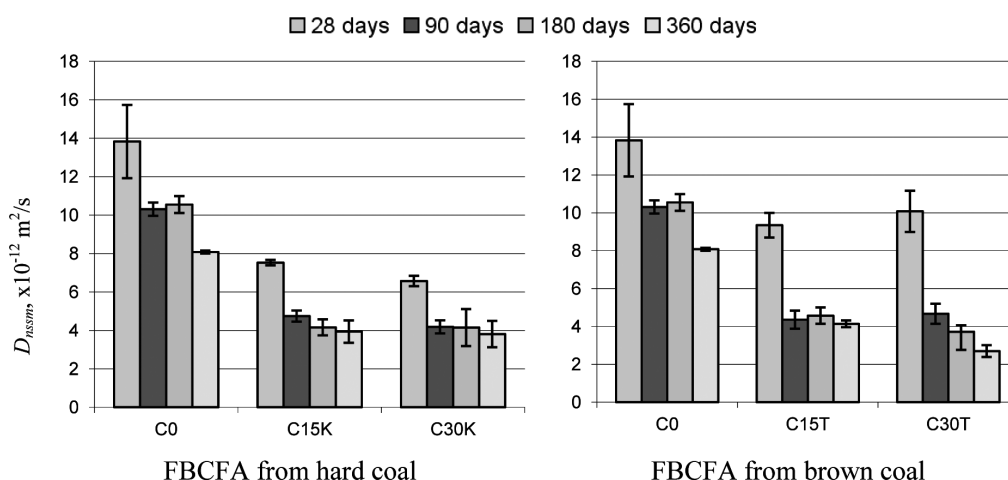


Fig. 3. Chloride migration coefficients for air-entrained concretes, series C.

Rys. 3. Współczynnik migracji jonów chlorkowych w napowietrzonych betonach, seria C

chloride migration coefficient was lower than for concrete C15T not before 180 days of curing. The chloride resistance of concrete C30T tested after 90 days is equal to those for ordinary concrete C0 tested after 28 days.

In air-entrained concretes with FBCFA replacement the differences between D_{nssm} tested after 28 and 90 days are higher than for concretes without air-entraining admixture, especially for concretes with FBCFA from brown coal.

The correlation between chloride migration coefficient and compressive strength after 28, 90, 180 and 360 days, for series B is shown in Fig. 4. In all concretes a decrease in D_{nssm} is observed as compressive strength increases. This happens because the microstructure of the cement paste becomes denser during the curing period. For concretes series B, without air-entraining admixture the relationship between chloride resistance and compressive strength in time is almost linear (R^2 – squared of the coefficient of correlation nearly 1). The greater decrease of chloride migration coefficient with increase of compressive strength is observed for concretes without FBCFA. Higher content of FBCFA results also in decreasing of D_{nssm} with increase of f_c but the tendency is less visible. In air-entrained concretes this tendency is not so clear. The chloride migration coefficient decreases with increase of compressive strength but the differences between concretes with 15% and 30% of FBCFA are not noticeable.

Porosity and microstructure of air-voids

Fig. 5 shows the porosity of fresh mix in non air-entrained and air-entrained concretes. Series B varied from 1.3% (B30K) to 2.1% (B0), in series C from 5.8% (C30K) to 6.8% (C15K). For concretes series B the values of porosity were slightly decreasing with increase of FBCFA content. For series C the highest results were obtained for

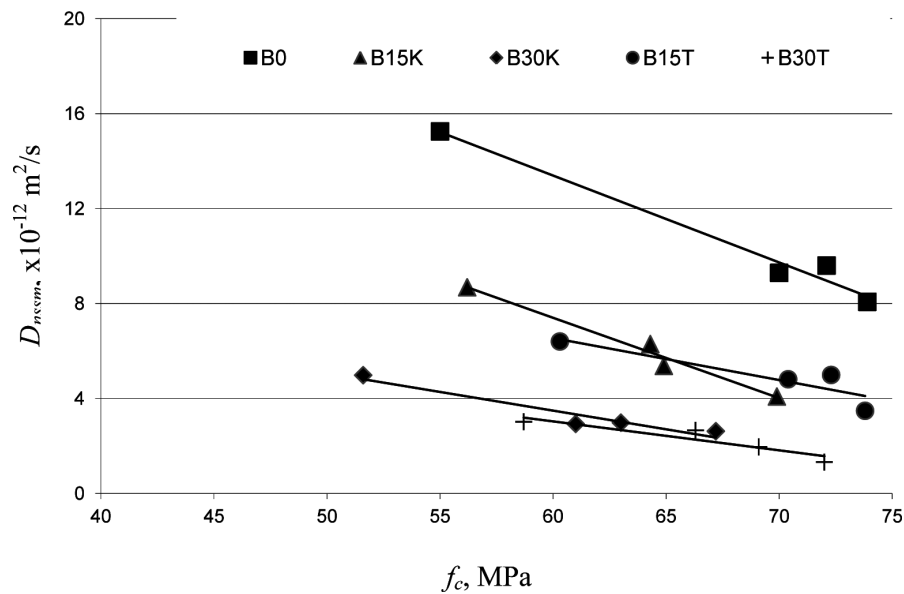


Fig. 4. Correlation between chloride migration coefficient and compressive strength after 28, 90, 180 and 360 days of curing, non air-entrained concretes, series B.

Rys. 4. Zależność między współczynnikiem migracji chlorków i wytrzymałości na ściskanie po 28, 90, 180 i 360 dniach dojrzewania, nienapowietrzony betony, seria B

concrete with 15% of FBCFA, but the differences in results were contained in standard deviation. The use of FBCFA did not deteriorate the porosity of fresh mix.

Similarly, the influence of FBCFA on total air content in hardened concrete was negligible. In air-entrained concretes the values of air content A , and consequently that of specific surface α , spacing factor \bar{L} and content of micropores A_{300} were improved by FBCFA content, Table 4, e.g. the total air-content of concretes varied from 4.59% to 5.13%. The difference in A_{300} value between FBCFA from hard coal and brown coal are visible only for concretes with 30% of replacement, but it is similar to measurements errors. The concrete without FBCFA addition did not show higher porosity than other concretes. The percentage of micropores below 300 μm increased with increasing of FBCFA content, Fig. 6, what is important due to the ingress of harmful chloride ions.

The particle size distribution, the content of unburned carbon and the chemical composition (CaO , CaO_{free} and SO_3) influences the concrete properties. In fly ash with low content of loss on ignition the spherical particles dominate but when the content of unburned carbon is high (like in FBCFA) the conglomerate both, spherical and irregular shape is visible. The non spherical grains and high content of CaO in FBCFA from brown coal would influence on the activity of the air-entraining admixture. The relation between the content of unburned carbon and the chemical admixtures,

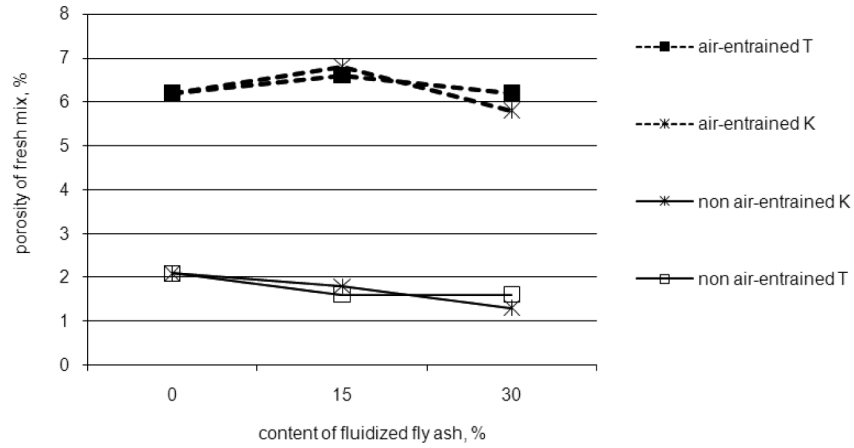


Fig. 5. Porosity of fresh mix in non air-entrained (B) and air-entrained (C) concretes vs content of FBCFA.

Rys. 5. Zawartość powietrza w mieszance betonowej nie napowietrzanej (B) i napowietrzanej (C) odnośnie do zawartości popiołu fluidalnego

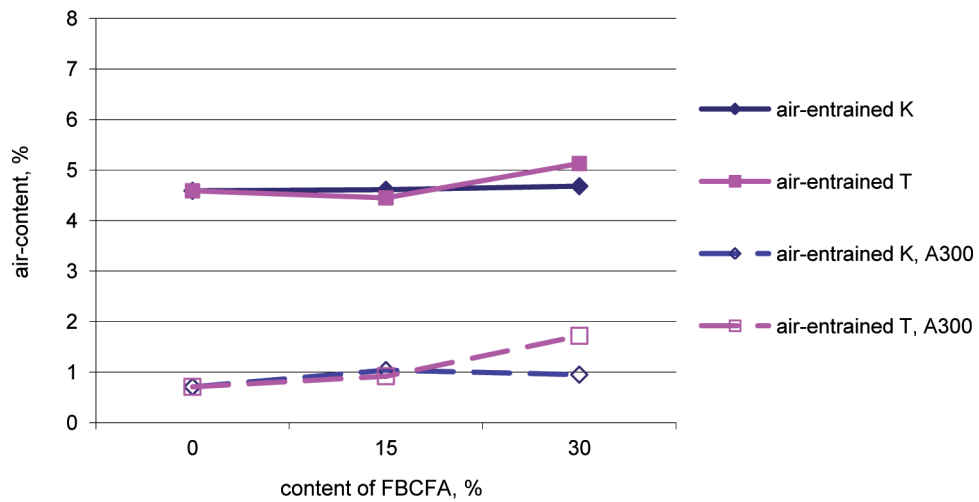


Fig. 6. Total air content and content of micropores below $300 \mu\text{m}$ – A_{300} in concretes series B and C.

Rys. 6. Całkowita zawartość porów powietrznych i zawartość mikroporów poniżej $300 \mu\text{m}$ – A_{300} w betonach serii B i C

Table 4

The characteristic of the air-voids microstructure.
Charakterystyka mikrostruktury porów powietrznych

Mix	Air content A [%]	Spacing factor \bar{L} [mm]	Specific surface α [mm^{-1}]	Micropores below $300 \mu\text{m}$ A_{300} [mm]
C0	4.59	0.317	14.37	0.71
C15K	4.61	0.259	17.52	1.04
C30K	4.68	0.297	15.86	0.95
C15T	4.45	0.310	19.74	0.92
C30T	5.13	0.199	20.51	1.72

especially air-entraining admixture, seems to play a principal role in chloride resistance of concrete.

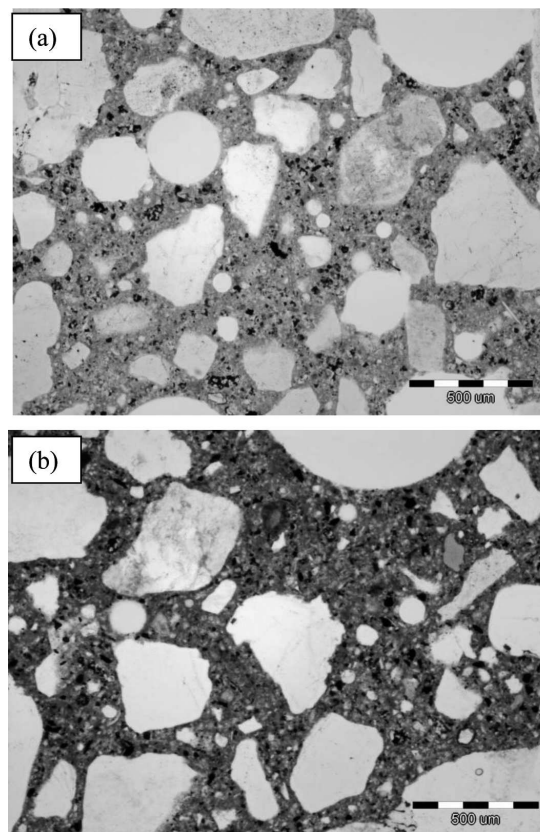


Fig. 7. Microstructure of the air-entrained concretes C0 (a) and C30K (b), ordinary plane polarized light, magnification $\times 40$.

Rys. 7. Mikrostruktura napowietrzonych betonów C0 (a) i C30K (b), światło przechodzące, równoległe nikole, powiększenie $40\times$

The results of thin section analysis of tested concretes revealed that all concretes appeared sound with a dense dark paste. The reference concretes, both series B and C, contained the higher amount of the cement grains relicts with visible alite, belite and ferrite grains than concretes made with FBCFA. The content of the unburned carbon particles increased with the increasing of the addition of the FBCFA, Fig. 7, so the color of the paste becomes darker. Fly ash particles are observed in the paste in concretes with FBCFA. In addition, numerous zones of less-dense paste were observed at the aggregate-paste interfaces (especially below the aggregate particles) in the plain concrete mixes. The air content of the samples were visually estimated and all in all seemed to correlate with the plane section analysis. The volume of air-voids decreased with the content of the FBCFA.

4. CONCLUSIONS

Based on the results and discussion, the following conclusions are proposed:

1. The chloride migration coefficient in all tested concretes decreased with their age.
2. In non air-entrained concrete the decreasing of D_{nssm} was proportional to the content of both kinds of FBCFA. It means that for higher FBCFA content lower values of D_{nssm} have been obtained. The chloride resistivity for concretes with FBCFA from hard coal was slightly higher than for FBCFA from brown coal.
FBCFA improved resistance against chloride ions intrusion in non air entrained concretes.
3. In air-entrained concretes with FBCFA from brown coal chloride diffusion coefficient D_{nssm} :
 - was higher than in non air-entrained concrete,
 - was higher at early stage of hydration (up to 180 days) for 30% replacement than for 15% replacement.
4. The chloride resistance of air-entrained concretes with FBCFA replacement was higher than for air-entrained concrete without addition of FBCFA, but the value of D_{nssm} for 28 and 90 days for brown coal have been considerably higher than for non air-entrained concretes.
5. In air-entrained concretes FBCFA should be applied as partial cement replacement with caution.

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OCENA WSPÓŁCZYNNIKA MIGRACJI JONÓW CHLORKOWYCH W BETONACH ZAWIERAJĄCYCH POPIÓŁ FLUIDALNY

Streszczenie

Celem przedstawionych badań było porównanie stopnia przenikania jonów chlorkowych w betony zarówno nienapowietrzone jak i napowietrzone, zawierające materiał odpadowy jakim jest popiół fluidalny pochodzący z dennego złoża (Fluidized Bed Combustion Fly Ash, FBCFA). Badane betony zawierały 15% lub 30% FBCFA, przy stałym współczynniku wodno-spoiwowym równym 0,45. W badaniach użyto dwa rodzaje popiołu fluidalnego pochodzące ze spalania węgla kamiennego i brunatnego.

W przeprowadzonych badaniach zastosowano metodę migracji jonów chlorkowych – Nordtest Method BUILD 492. Mikrostrukturę betonów analizowano na cienkich szlifach betonowych, natomiast obliczenia dotyczące mikrostruktury porów powietrznych, ich wielkości oraz rozmieszczenia przeprowadzono na zglądach betonowych przy pomocy cyfrowej analizy obrazu wg. PN-EN 480-11:2008.

Otrzymane wyniki uwiarydliły znaczący wpływ częściowego zastąpienia cementu przez popiół fluidalny na przepływ jonów chlorkowych w betonie. Zauważono, że tego rodzaju dodatek do betonu diametralnie obniża wnikanie jonów chlorkowych do betonu. Badano również wpływ napowietrzenia na wielkość współczynnika migracji jonów chlorkowych. Zastosowanie środków napowietrzających do betonów zawierających popiół fluidalny powoduje obniżenie współczynnika migracji jonów chlorkowych jednak proces napowietrzania powinien być przeprowadzany rozważnie i z dużą uwagą.

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