Air Permeability of Air-Entrained Hybrid Concrete Containing CSA Cement

Wojciech Kubissa

Faculty of Civil Engineering, Mechanics and Petrochemistry, Warsaw University of Technology, 09-400 Płock, Poland; wojciech.kubissa@pw.edu.pl; Tel.: +48-24-3672185

Received: 6 June 2020; Accepted: 29 June 2020; Published: 1 July 2020

Abstract: This paper presents the results of research on series of concrete prepared with Portland cement CEM I 42.5R, with partial replacement of this cement with calcium sulfoaluminate cement. In part of the series, an air-entraining admixture was used. It was assumed that the mixture must remain workable for at least 45 min and to ensure that citric acid as the setting retarder was used. Compressive strength tests after 2, 7, 28, 56 and 90 days, tensile splitting strength test and sorptivity test after 28 days were performed. After 56 and 90 days, the moisture content of the specimens was determined, and Torrent air permeability was tested. Freeze-thaw scaling resistance was also investigated. It was found that the addition of 15% and 30% calcium sulfoaluminate cement results in a significant reduction in the relative humidity of the concrete, after storage under air-dry conditions and an increase in air permeability. The series with 30% calcium sulfoaluminate cement in the binder, regardless of aeration, showed significantly lower resistance to cyclic freezing in the presence of salt. A clear correlation between sorptivity, air permeability and surface scaling was not found. On the basis of a multi-criteria evaluation of the tested concrete and performed Performance Index calculations, the best parameters were achieved by concrete series C30-4.5.

Keywords: air enriched concrete; frost resistance; relative humidity; scaling; Torrent air permeability

1. Introduction

Winter in a temperate climate zone is characterized by negative temperatures and large daily amplitudes of air temperature changes. As a result, during the winter months, and sometimes also in autumn and spring, the temperature quite often goes through zero. Concrete structures, unless they are submerged in the ground below freezing point or are roofed and thermally insulated, are periodically exposed to low temperatures under such conditions. It should be noted that concrete is not a low-temperature-sensitive material, but its structure is progressively damaged during the cyclic transition of temperature through zero, which is related to the freezing and thawing of water in its pores.

In Poland, which is located in the temperate climate zone, the total length of roads with concrete pavement has significantly increased in recent years, and there are plans to build further road sections [1–3]. The cement concrete pavement, if properly designed, constructed and exploited, shows a significantly higher durability than the bituminous pavement [4]. However, with the increasing length of the concrete road network, the probability increases that repair works will be necessary in some of its sections during their expected service life. This may result from faults at the execution stage. It may also be a result of random events during operation (e.g., leakage of chemicals harmful to concrete during transport). Another important cause of concrete pavement damages, such as surface scaling and cracking, is the widespread use of de-icing salt in winter [5,6]. They are a source of chloride ions, which penetrate deep into the concrete, reacting with portlandite to form leachable compounds, which weakens the concrete structure [7].
Repair materials using calcium sulfoaluminate cement (CSA) or other types of quick-setting cements are known. However, they are rarely used with aeration, which increases the frost resistance of concrete [8,9]. It is a challenge to develop an optimal material composition, which will enable the quick repair of the concrete surface, and at the same time will be characterized by high frost resistance. The proper direction of the research is the simultaneous use of liquefying, air-entraining and retarding admixtures combined with the use of quick-setting cement, e.g., CSA, as a partial substitute for classical cements based on Portland clinker. The resulting hybrid concrete, i.e., made with more than one type of binder, should have a short setting time and high resistance to cyclic freezing. Due to difficult working conditions, such a concrete should also have a high durability, which is ensured by a properly shaped structure. The measurable features of concrete that correlate well with the quality of its structure include air permeability. Although this parameter is known and quite commonly tested [10–13], and concretes using CSA cement are also not new, the combination of these two elements, i.e., the permeability testing of hybrid concrete, has a novelty value, especially in connection with the use of aeration admixture. Additionally, the attempt to find a correlation between the results of the concrete permeability test and its resistance to cyclic freeze-thawing in the presence of de-icing salts is worth noting. If it is found, such a strong correlation could be an important argument for recognizing the equivalence of both these tests in the assessment of the quality of pavement concrete.

Calcium sulfoaluminate cement was invented in the 1950s by Alexander Klein. During the next decade, the material was improved by the inventor, in collaboration with Ed Rubin and Ed Rice, which allowed for its industrial production at the Kaiser Permanente Cement Plant near Cupertino in USA to begin in the 1960s. Currently, this cement is produced mainly in China [14]. The calcium sulfoaluminate clinker has a different chemical and mineralogical composition than the Portland clinker. Its primary phase component is ye'elimite (C₄A₃$). However, it does not contain alite (C₃S). This has its consequences in the different course of hydration of both types of cement.

Calcium sulfoaluminate cements are characterized by a very fast start of setting, as well as a very fast increase in early strength (several hours). The ettringite, which is the main phase that gives strength to the hardened binder, results in limited shrinkage or even expansion of the material. This feature has determined that CSA cements are used as components of repair mortars and concrete, as well as shrinkage-free screeds and flooring. The production of CSA cements is more environmentally friendly than that of cements based on Portland clinker. This is due to the lower firing temperature (approximately 1250 °C), which results in lower production energy consumption and lower CO₂ emissions to the atmosphere, compared to the production process of both Portland and alumina cements. Moreover, the process of CSA clinker production also enables the use of industrial waste up to 50% of the raw material mass [15]. CSA cements can be used together with Portland cement. The resulting hybrid concrete is characterized by a faster start of setting than the material based solely on Portland cement. With an appropriately selected proportion of both cements, such a hybrid concrete may also show no shrinkage but little expansion. Both these features make it a suitable material for short-term repairs of concrete structures and surfaces [16].

Modification of the concrete structure caused by the use of mineral additives or supplementary cementing materials is reflected, among others, in the results of the air-permeability test. Such tests carried out with Torrent’s apparatus on concrete, in which a significant part of cement was replaced by calcareous fly ash, showed a clear relationship between the amount of the additive and the value of kT coefficient obtained in the test [17,18]. Additionally, the application of siliceous fly ash and granulated ground blast furnace slag was not without influence on air permeability of concrete [19–21]. The introduction into concrete of a binder with a different phase composition, which is characterized by different hydration reactions and different products, also leads to the modification of the structure of the material. Such modification should also affect the air permeability. It can be assumed that this will lead to increased concrete tightness. However, this assumption needs to be confirmed, and it also needs to be established how the kT value changes quantitatively with the exchange rate of Portland cement with CSA cement. The inclusion of air-entraining admixture, which also modifies the structure
of the concrete making it more resistant to cyclic freezing, makes the answer to the question of how the air permeability through the concrete will change less obvious. Thus, it is all the more worthwhile to determine.

The introduction of air-entraining admixtures is one of the most important achievements in concrete technology in the past century. Their application significantly facilitates the production of concrete of high frost resistance [22]. The proper aeration of concrete with the use of admixtures requires some experience and skills, which need to be continuously improved. The success of the aeration process consists not only in obtaining the appropriate structure of air bubbles but also in maintaining their proper content during the transport and placement of concrete and later until its setting. The experience gained in aerating concrete with CEM I Portland cement does not necessarily have to be successful when using a different binder composition. This is the result of the emergence of new types of cement on the market, whose interaction with the aeration mixture may be of a different nature and lead to different effects. Concrete mixtures with cement containing non-clinker main components, generally with a larger specific surface area, are often more difficult to aerate than mixtures containing only Portland cement. An example can be the binder with a large share of circulating fluidized bed combustion (CFBC) fly ash [23]. The results showed that the proper aeration of concrete with the addition of CFBC fly ash could be obtained, however, it required a significant increase in the air-entraining admixture dose. In the case of concrete with CSA cement, the differences in the admixture action proved to be small. Similar aeration was obtained in concrete with CSA as in the reference concrete (with Portland cement only) at a similar dose of air-entraining admixture.

In order to obtain the required frost resistance of the aerated concrete, it is necessary to limit the w/c to 0.50, and even better to 0.45 [24]. If an air-entraining admixture is not used, an effective way to ensure frost resistance is to maximally tighten the concrete structure primarily by even more greatly reducing the w/c ratio. With a very low w/c ratio (about 0.30), high frost resistance of concrete can be expected even without the use of an air-entraining admixture. However, this approach is more expensive and not devoid of additional disadvantages, which are associated, among others, with high self-desiccation shrinkage, characteristic for concrete mixtures of w/c < 0.40. Thus, the risk of early shrinkage cracks increases significantly [4,24]. In the research presented in this article the w/c value 0.40 was assumed in combination with high cement content of 440 kg/m³.

Testing of air permeability of concrete cannot be performed in isolation from the state of humidity of the material. The determination of concrete air permeability in a specified state of moisture content was considered, inter alia in the papers [25–28]. The authors point out that before testing air permeability it is necessary to ensure that the specimens have the same moisture content. Due to the significant influence of concrete humidity on the results of air permeability only after meeting the above condition, the results obtained, and thus concrete quality, can be compared with each other.

One of the methods of testing air permeability is the Torrent method, which is recommended for testing cover aerated concrete on site. The test procedure, maximum kT values depending on the exposure class XC4, XF and XD and compliance criteria are described in Annex E of Swiss Standard SN 505 262/1 (SIA 262/1) [29]. In this document, the conditions that concrete must meet in order to be measured, including the maximum permissible moisture content and the method of its measurement, are also described [12,30]. The standard contains some exemption from Torrent’s instructions, as it recommends determining the moisture content of concrete by means of Tramex apparatus, and not directly by means of the Wenner probe, which is the equipment of Torrent’s set. However, this is not a significant difference, as both devices operate on the same principle, based on the measurement of concrete impedance. The SN 505 262/1 standard is the only standardization document so far containing provisions directly related to the Torrent air permeability test. The results obtained by this method allow to evaluate the concrete quality in a reliable way [10] provided that the requirements for moisture content during the test are met. It should be stressed, however, that although the qualitative influence of the moisture content of the tested concrete on the kT value is generally known, the detailed relations
between these parameters are still being tested, and no unambiguous results have been obtained here so far [26,31,32].

For research purposes, series of concrete were made with Portland cement CEM I 42.5R and with partial replacement of this cement with CSA cement. In part of the series an air-entraining admixture was used, with the target concrete aeration of 4.5% or 8%. It was assumed that the mixture must remain workable for at least 45 min. Citric acid was used as the setting retarder for this purpose, in all batches. Citric acid, apart from phosphoric acid and lactic acid, is one of the most frequently used agents to delay the cement setting [33–35]. Compressive strength tests after 2, 7, 28, 56 and 90 days, tensile splitting strength test and sorptivity test after 28 days were performed. After 56 and 90 days the moisture content of the specimens was determined with Tramex CMEX II apparatus and air permeability was tested with Torrent apparatus. Freeze-thaw scaling resistance was also investigated, according to PKN-CEN/TS 12390-9:2007 [36] standard.

2. Materials and Methods

Portland cement CEM I 42.5R from Górażdże Cement Plant, Chorula, Poland as per PN-EN 197 was used. Calcium sulphoaluminate cement was used as a partial replacement of CEM I 42.5R cement. The ratio of substitution was 15% and 30% of cement amount by mass. Chemical composition of cements, according to articles [15,37], are shown in Table 1.

| Component | Cement CEM I 42.5R [37] | Cement CSA [15] |
|-----------|--------------------------|-----------------|
| SiO$_2$   | 20.04                    | 6.88            |
| Al$_2$O$_3$ | 5.07                    | 34.18          |
| Fe$_2$O$_3$ | 2.35                    | 0.99            |
| CaO       | 63.94                    | 39.94          |
| MgO       | 1.72                     | 0.70           |
| Na$_2$O   | 0.17                     | <0.08          |
| SO$_3$    | 2.81                     | 14.33          |
| K$_2$O    | 0.77                     | 0.35           |
| Cl        | 0.065                    | ***            |
| Na$_2$O$_{eq}$ | 0.68  | 0.31 |

***—no data available.

The basic physical properties provided by the cements’ manufacturers are shown in Table 2.

| Cement Type | Setting Time | Compressive Strength After 28 Days | Specific Surface Area (Blaine) | Specific Gravity | Loss of Ignition |
|-------------|--------------|------------------------------------|---------------------------------|-----------------|-----------------|
|             | Start [min]  | End [min]                          | [MPa]                           | [cm$^2$/g]      | [%]             |
| CEM I 42.5R | 194          | 248                                | 56.9                            | 3721            | 3.10            |
|             | 13           | 21                                 | 76.0                            | 5600            | 2.88            |

All concrete mixes contained 440 kg/m$^3$ of cement and w/c ratio was equal 0.4. As fine aggregate river sand 0–2 mm was used. As coarse aggregate two fractions of granite 2–8 mm and 8–16 mm were used. All aggregates were at laboratory air-dry condition. Superplasticizer according to PN-EN 934-2 was used. Regular tap water was used as mixing water. Recipes of concrete mixes are shown in Table 3.
Table 3. Recipes of concrete mixes [kg/m³].

| Concrete ID:       | C0-1 | C15-1 | C30-1 | C0-4.5 | C15-4.5 | C30-4.5 | C0-8 | C15-8 | C30-8 |
|-------------------|------|-------|-------|--------|---------|---------|------|-------|-------|
| CEM I 42.5R       | 440  | 375   | 310   | 440    | 375     | 310     | 440  | 375   | 310   |
| CSA               | 0    | 65    | 130   | 0      | 65      | 130     | 0    | 65    | 130   |
| sand 0-2 mm       | 622  | 620   | 618   | 589    | 588     | 586     | 556  | 555   | 553   |
| granite aggregate | 533  | 531   | 530   | 505    | 504     | 502     | 477  | 476   | 474   |
| granite aggregate | 533  | 531   | 530   | 505    | 504     | 502     | 477  | 476   | 474   |
| 2-8 mm            |      |       |       |        |         |         |      |       |       |
| 8-16 mm           | 622  | 620   | 618   | 589    | 588     | 586     | 556  | 555   | 553   |
| AE admixture      | 0.0  | 0.0   | 0.0   | 0.6    | 0.6     | 0.7     | 1.1  | 1.2   | 1.3   |
| superplasticizer  | 2.2  | 2.2   | 2.6   | 1.3    | 1.3     | 1.5     | 1.1  | 1.1   | 1.3   |
| water             | 176  | 176   | 176   | 176    | 176     | 176     | 176  | 176   | 176   |
| AE admixture      | 0.00%| 0.00% | 0.00% | 0.14%  | 0.14%   | 0.15%   | 0.26%| 0.28% | 0.30% |
| superplasticizer  | 0.50%| 0.50% | 0.60% | 0.30%  | 0.30%   | 0.35%   | 0.26%| 0.25% | 0.30% |

In order to maintain the desired workability time of 45 min, citric acid was used as a retarder of the setting and hardening of concrete in the amount of 0.2% of cement (both CEM I 42.5R and CSA) mass (in all series). The workability of the C30-1 series was maintained for the shortest time, but the 45 min workability time target was reached. In the remaining series, the mix retained its workability for a longer period of time. In series without CSA cement the effect of delayed setting and hardening was very clear. Especially for the C0-8 series, which had a strength close to zero after two days and therefore was problematic to demould without damaging the specimens. The air entraining admixture was used to aerate the mixtures in the amount of about 0.14% of cement mass for series with the assumed aeration level of 4.5% and 0.26–0.30% admixture for series with the assumed aeration level of 8%.

The components of the concrete mix were mixed in a laboratory mixer with a capacity 100 dm³. Specimens were formed in two layers which were vibrated on the vibrating table for approximately 30 s each. Moulds with the mixture after filling and vibrating were covered with foil. Specimens after demoulding were stored in laboratory air-dry conditions i.e., in temperature 20 °C ± 2 °C and a relative humidity of 55% ± 10%.

The consistency of the mixture was tested before forming of the specimens by the slump test method. The slump test was carried out in accordance with PN-EN 12350-2. The air content of the concrete mixture was tested using a pressure apparatus in accordance with PN-EN 12350-7. The temperature of the concrete mixture was measured before the consistency test using a laboratory thermometer with a resolution of 0.1 °C. Specimens were prepared and cured as per PN-EN 12390-2. Selected properties of concrete mixes are shown in Table 4.

Table 4. Selected properties of concrete mixes.

| Concrete ID:       | C0-1 | C15-1 | C30-1 | C0-4.5 | C15-4.5 | C30-4.5 | C0-8 | C15-8 | C30-8 |
|-------------------|------|-------|-------|--------|---------|---------|------|-------|-------|
| temperature [°C]   | 26.3 | 24.8  | 23.6  | 25.8   | 25.6    | 23.2    | 26.2 | 25.3  | 25.3  |
| slump [mm]         | 160  | 170   | 160   | 180    | 180     | 180     | 180  | 200   | 190   |
| air content [%]    | 1.4  | 1.4   | 1.2   | 4.5    | 4.5     | 5.0     | 7.5  | 7.5   | 7.0   |

Specimens for testing the air permeability after demoulding were left for a week in air-dry conditions. The upper, lower and two lateral opposite surfaces of the specimens were then sealed with an impermeable layer. This ensured a unidirectional flow of moisture in the specimen during drying what was similar to specimen preparation described in [31,38]. After sealing the surfaces, the specimens for testing the air permeability were left in an air-dry condition, until the 56th day after concreting. The specimens for compressive strength, tensile and sorptivity tests were, after demoulding, stored in air-dry conditions, up to the 28th day after concreting.
2.1. Compressive and Tensile Splitting Strength Tests

The compressive strength test was carried out on 100 mm cube specimens on the 28th day of hardening after mixtures preparation with PN-EN 12390-3. The tensile splitting strength test was conducted on the same type of specimens in accordance with PN-EN 12390-6. Both tests were performed using a ToniTechnik, Berlin, Germany equipment with 3000 kN compression force capacity. The rate of increase of the load was maintained at 0.05 MPa/s for tensile splitting strength test and 0.5 MPa/s for compressive strength test.

2.2. Sorptivity Test

The sorptivity test was conducted on the halves of cubic specimens of 100 mm edge by means of mass method. Prior to the sorptivity test, the specimens had been oven-dried to the stable mass at a temperature of 105 °C. The tests were conducted at the temperature of approximately 20 °C. The specimens were weighed before immersing in water and then arranged in a vessel with water. The specimens were immersed up to the height of 3 to 5 mm. In the specific time intervals from the beginning of the test the specimens were weighed again to define their weight gain resulting from water sorption. Subsequent weight measurements were conducted for 6 h. Sorptivity S in cm/h\(^{0.5}\) was defined as a slope of the linear function defining the dependence of the mass of the water absorbed ∆m by the area F on the time root t\(^{0.5}\) [39] as in Equation (1):

\[
\frac{\Delta m}{F} = S \times t^{0.5}
\]  

(1)

2.3. Torrent Air Permeability Test

The air permeability test was carried out by the Torrent method using Proceq, Schwerzenbach, Switzerland equipment. The air permeability test was performed on three cubic specimens with an edge of 150 mm after 56 and 90 days from concreting. The method of specimen surface preparation significantly influences the result of the air permeability test [40], therefore, it was decided to test the two unprotected surfaces of each specimen in their natural state i.e., without any treatment (e.g., grinding). On the specified days both unprotected surfaces of each specimen were tested once. Before the test, the moisture content of the specimens was determined on the tested surfaces using a Tramex CMEX II meter, Tramex Ltd., Dublin, Ireland, which is recommended by the Swiss Standard SIA 262/1 and [12]. Relative humidity (RH) measurements were taken four times on each unprotected side with rotating the Tramex measuring instrument 90 degrees after each measurement. The position of the testing cell of the equipment during the test was chosen in such a way that there were no defects, cavities under the rubber gasket. In some cases, this required the cell to be shifted by a maximum of 2 cm from its symmetrical position, relative to the centre of the wall. The logger of the testing unit automatically reported after the test the value of kT permeability coefficient and the thickness L of concrete layer involved in the measurement. Each measurement lasted a maximum of 720 s, or less in the case of concrete with higher air permeability.

2.4. Freeze-Thaw Scaling Resistance

The surface frost resistance test was performed according to CEN/TS 12390-9 standard except for the slightly different preparation of the specimens. The freeze and thaw cycles were performed in a freeze chamber produced by Unimors, Grodzisk Mazowiecki, Poland. The test consists in determining the mass of scaled material from a specimen surface covered with 3% NaCl solution after a given number of freeze and thaw cycles. The cubes were cut into two plasters of 50 × 150 × 150 mm. Each test specimen obtained in this way was protected on the sides with adhesive tape and sealed with silicone. Then the specimens were soaked with distilled water for three days before starting the test. During the test in freeze chamber specimens were covered with 3% NaCl solution on the exposed surfaces. The remaining surfaces were isolated against humidity and heat transfer. The freezing and thawing
cycles of the specimens lasted 24 h each. The scaled material was collected and weighed after given numbers of freeze/thaw cycles, and the results expressed as mass per unit area have been recorded. The masses of scaling \( m_{14}, m_{28} \) and \( m_{56} \) were determined after 14, 28 and 56 days respectively.

3. Results and Discussion

Table 5 presents the results of tests of the mechanical and physical properties. The mechanical properties include the compressive strength that was tested after 2, 7, 28, 56 and 90 days, as well as tensile splitting strength that was tested after 28 days. The physical parameters, related to durability, are represented by the sorptivity measured after 28 days, the Torrent air permeability coefficient \( k_T \) measured after 56 and 90 days, and RH values measured using the Tramex meter after 28, 56 and 90 days. Each of the presented strength and sorptivity results is a mean of the results of six samples. For Torrent air permeability coefficient \( k_T \), it is the mean of eight results. RH values measured with Tramex are the mean of 32 results—four on each of eight specimen walls.

| Concrete ID: | C0-1 | C15-1 | C30-1 | C0-4.5 | C15-4.5 | C30-4.5 | C0-8 | C15-8 | C30-8 |
|-------------|------|-------|-------|--------|---------|---------|------|-------|-------|
| compressive strength (2 days) [MPa] | 0.5  | 12.6  | 18.2  | 0.9    | 9.9     | 20.5    | 0.2  | 8.1   | 13.2  |
| compressive strength (7 days) [MPa] | 39.5 | 45.6  | 42.6  | 32.8   | 41.4    | 32.3    | 29.5 | 36.0  | 29.8  |
| compressive strength (28 days) [MPa] | 57.9 | 56.0  | 51.1  | 45.7   | 50.5    | 42.7    | 42.9 | 42.7  | 40.1  |
| compressive strength (56 days) [MPa] | 59.5 | 59.3  | 55.8  | 47.9   | 55.2    | 43.9    | 48.2 | 46.0  | 43.1  |
| compressive strength (90 days) [MPa] | 62.0 | 65.5  | 57.7  | 49.0   | 57.3    | 46.1    | 51.1 | 44.2  | 44.0  |
| tensile strength (28 days) [MPa] | 4.06 | 4.29  | 4.37  | 4.37   | 3.91    | 3.66    | 3.98 | 3.87  | 3.81  |
| sorptivity (28 days) [cm/h\(^{0.5}\)] | 0.102 | 0.034 | 0.046 | 0.143  | 0.159   | 0.048   | 0.079 | 0.055 | 0.033 |
| RH Tramex (28 d) | 3.77 | 3.53  | 3.49  | 5.15   | 3.18    | 3.10    | 4.98 | 3.03  | 2.68  |
| RH Tramex (56 d) | 3.60 | 3.00  | 3.06  | 3.67   | 3.06    | 2.93    | 3.61 | 2.98  | 2.80  |
| \( k_T \) (56 d) \[\times 10^{-16} \text{ m}^2\] | 0.048 | 0.116 | 0.111 | 0.068  | 0.395   | 0.276   | 0.075 | 0.320 | 0.396 |
| RH Tramex (90 d) | 3.36 | 2.76  | 2.71  | 3.50   | 2.83    | 2.56    | 3.47 | 2.84  | 2.38  |
| \( k_T \) (90 d) \[\times 10^{-16} \text{ m}^2\] | 0.048 | 0.105 | 0.120 | 0.094  | 0.424   | 0.329   | 0.087 | 0.388 | 0.489 |

Table 6 shows the results of the surface scaling frost resistance test. Each result is an average of the results obtained from two specimens with a total area of 450 cm\(^2\).
Table 6. Results of scaling test.

| Concrete ID: | C0-1 | C15-1 | C30-1 | C0-4.5 | C15-4.5 | C30-4.5 | C0-8 | C15-8 | C30-8 |
|-------------|------|-------|-------|-------|--------|--------|------|-------|-------|
| 14 days [kg/m²] | 0.019 | 0.012 | 0.117 | 0.005 | 0.003  | 0.068  | 0.002| 0.002 | 0.094 |
| 28 days [kg/m²] | 0.013 | 0.007 | 0.094 | 0.007 | 0.005  | 0.022  | 0.004| 0.006 | 0.059 |
| until 28 day [kg/m²] | 0.032 | 0.020 | 0.211 | 0.011 | 0.008  | 0.090  | 0.006| 0.008 | 0.153 |
| 56 days [kg/m²] | 0.007 | 0.010 | 0.020 | 0.003 | 0.003  | 0.009  | 0.001| 0.000 | 0.028 |
| until 56 day [kg/m²] | 0.039 | 0.029 | 0.231 | 0.014 | 0.010  | 0.099  | 0.007| 0.009 | 0.181 |

The addition of a retardant in the form of citric acid resulted in a long delay of setting and hardening, especially visible in the case of series without CSA. The compressive strength of C0-x series specimens was close to zero after two days, and the manner of the specimens destruction was abnormal.

The article [33] also presents the influence of addition of citric acid in the amount of 0.5%, 1% and 3% of binder mass on the mechanical properties of PC/CAC cement mortars. It was found that the compressive and flexural strength of PC/CAC blended cement was greatly reduced by the addition of citric acid. The decrease in compressive strength when dosing 0.5% of citric acid was over 70% after 7 and 28 days of hardening. Larger amounts of citric acid caused a decrease in compressive strength by up to 90% after 7 days and by 80% after 28 days. Taking into account the presented results, it can be concluded that the amount of 0.2% mc. of citric acid used by the authors is appropriate, ensures the expected extension of the workability and does not cause such large decreases in strength as presented in [33].

The highest strength after 2 days was obtained in series with 30% CSA cement content. In case of C30-4.5 series it was 20.5 MPa and in case of C30-1 and C30-8 series it was 11.2% and 35.7% less respectively. After 7 days, the effect of the retarder was already smaller, and aeration started to play a bigger role. The highest compressive strength value of 45.6 MPa was achieved by the series C15-1. The remaining series without additional aeration had strength by 6.6% and 13.3% lower in the case of C30-1 and C0-1 respectively. High strength after 7 days was also achieved by the C15-4.5 series, which was 9.2% lower than the maximum value in case of C15-1 series. The other series had strength from 21.0% to 35.5% lower than the maximum value. After 28 days of curing, the highest compressive strength of 57.9 MPa was achieved by the C0-1 series. The replacement of part of the Portland cement with CSA cement and aeration resulted in reduced strength. In the case of series without CSA cement, aeration 4.5% resulted in a 21.2% decrease and aeration 8% resulted in a 26.0% decrease compared to the result of C0-1 series. The addition of CSA cement without additional aeration resulted in strength lower by 3.4% and 11.8% in the case of C15-1 and C30-1 series, respectively. The lowest compressive strength after 28 days was achieved in the series with the highest content of CSA cement and 8% aeration. The difference was 30.8% compared to the result of C0-1 series. After 56 days of maturation, the highest compressive strength of 59.5 MPa was achieved by C0-1 series. The addition of CSA cement and aeration negatively affected the strength, and the lowest strength, lower by 27.4%, was achieved by C30-8 series. Similar trends were observed after 90 days of maturation. Although this time the highest compressive strength of 65.5 MPa was achieved by the series with 15% CSA cement content and without aeration C15-1, and the lowest by 32.9% C30-8 series. It is worth mentioning different kinetics of strength development of concrete with CSA addition. Since higher values of compressive strength after 2 and 7 days, the long-term (90 days) are lower in comparison with control mixture. Thus, CSA is suitable for application with requirement on the initial strength. This trend is quite visible. However, it should be noted that this concerns a mixture with a low w/c ratio that has been treated in a way that does not provide water for hydration at a later stage. For this reason, the conclusion cannot be considered as general.
The strength increase between 28 and 90 days after concreting was from 3.5% for C15-8 series to 19.3% for C0-8 series. No regular dependence of strength increase on CSA cement content and aeration was found. The average strength gain in all series was 10.9%.

The strength after 28 and 90 days is lower than that given in the literature for concrete with a similar recipe, particularly cement content and w/c ratio [41]. This may be caused, apart from aeration, by the higher water demand of CSA cement and an insufficient amount of water for full hydration for 90 days, as well as an assumed lack of wet curing 2 days after concreting. It is worth checking with such assumed curing (or rather lack of curing) the increase of the w/c ratio in subsequent research. Some authors also state similar strength of pervious concrete of high porosity containing SJ-601 polymer [42].

The article [43] presents the influence of citric acid on the hydration and strength development of a CSA cement pastes with 15, 20 and 25 wt.% of hemihydrate in the binder. Citric acid was added as a retarder at 0.5% mc. The compressive strength of the CSA cements was improved by the addition of citric acid. In [34], the effects of superplasticizers and two retarders: citric acid and sodium gluconate on the fluidity, flow loss and compressive strength of sulfoaluminate cement were studied. It was found that early compressive strength would be slightly decreased by superplasticizer, but the late strength would not.

The tensile splitting strength was 3.66 MPa to 4.37 MPa for the C30-4.5 and C30-1 series respectively. No correlation was observed between this property of concrete and the amount of CSA cement as well as the aeration rate. Only after averaging the results of series with different content of CSA cement but with the same aeration, it was noticed that the highest strength was achieved for series without additional aeration. With an aeration of 4.5% and 8.0%, the strength is 6.1% and 8.4% lower, respectively. The tensile splitting to compression strength ratio after 28 days was between 7.0% for the C0-1 series and 9.5% for the C30-8 series. Averaged over series with the same aeration, the ratio was 11.5% and 19.8% higher for series with 4.5% and 8% aeration, respectively, compared to the series without aeration.

Sorptivity of the reference series C0-1 was 0.102 cm/h^{0.5}. The C0-4.5 and C15-4.5 series had sorptivity values higher by 39.6% and 55.0% respectively. The series with 8% aeration had sorptivity lower than the reference series and it decreased with the increase of CSA cement content. The difference was from 23.2% to 68.2% for the C0-8 and C30-8 series, respectively. The series with CSA cement content equal to 30% had sorptivity significantly lower than the reference series. The difference between the series without aeration and with aeration of 4.5% was about 55%.

The series with 30% CSA cement content in the binder, regardless of aeration, showed significantly lower scaling resistance at cyclic freezing in the presence of salt. Exfoliation values after 56 days of the test are higher by 491% (1% of air), 619% (4.5% of air) and 2531% (8% of air), compared to reference series without CSA cement. The lowest scaling values were achieved for the C0-8 and C15-8 series, 0.007 and 0.009 kg/m², respectively. The series with 4.5% aeration also showed high frost resistance. This was 0.010 and 0.014 kg/m² for series C15-4.5 and C15-4.5 respectively. The amount of scaled material from the non-aerated series specimens without CSA cement and with 15% CSA cement was several times higher than the minimum values achieved by aerated concrete.

When assessing the frost resistance of concrete on the basis of the criteria contained in standard PN-EN 13877-2, it should be assumed that the requirements of the frost resistance category FT2 have been met for all concrete series. The maximum rate of loss of mass after 56 cycles (m₅₆) was 0.231 kg/m² in series C30-1. The maximum rate of loss m₅₆/m₂₈ occurred in series C15-1 and amounted to 1.512.

A similar decrease in resistance to surface scaling was found when changing parts of cement to ash from hard coal combustion in the thermoelectric power station and from brown coal combustion in the power plant [44]. The air content in the mixture, similarly to the studies presented in this article, was from 4.6% to 7.8%. The increase in the amount of scaled material was even about 1300% after 56 days, when comparing the result (1.41 kg/m²) to the result of the reference series (0.11 kg/m²).

Giergiczny [45] concluded that CEM II and CEM III concretes demonstrate more scaling comparing to...
CEM I concrete, while analysing frost resistance in standard time. CEM I cement concrete in most cases showed a minimum surface scaling below 0.05 kg/m$^3$. Similar results were obtained without CSA cement and with 15% CSA cement. In paper [46], the frost resistance tested according to the Polish Standard procedure PN-88/B-06250 of concrete containing fly ash from circulating fluidized bed combustion was presented. Several air-entrained concrete mixes were designed with constant water to binder ratio and with substitution of a part of the cement (Portland cement CEM I 32.5 R) by CFBC fly ash (20%, 30% or 40% by weight). Achieving of the target 5 ± 1% air-content in the mix required eight times as much air-entraining admixture when converting 40% cement to ash, and four times as much air-entraining admixture when converting 20% cement to ash, compared to the amount of air-entraining admixture in the reference series without ash. The compressive strength of concretes of all series subjected to 160 F/T cycles did not change significantly in comparison to the reference specimens.

To assess concrete quality, simultaneously based on selected properties the Performance Index (PI) was used. The method was presented in detail in [47]. An overall EIPI evaluation was not chosen. In the case of the proposed material used, the efficiency and durability of the repair and the reduction of the social impact are much more important than the reduction of CO$_2$ emissions and the use of natural resources. The aim is to enable the repair of the surface in the shortest possible time without causing disruption to traffic.

PI was calculated according to the formula:

$$PI = \frac{20\text{MPa}}{f_{cm,2\text{days}}} \times 0.3 + \frac{KT}{0.5 \times 10^{-16} \text{m}^2} \times 0.2 + \frac{m_{56}}{1.0 \frac{\text{kg}}{\text{m}^2}} \times 0.5$$

(2)

The assessment was based on: compressive strength after 2 days (reference value 20 MPa, weight 0.3), Torrent air permeability (reference value 0.5—limit of exposure class XF4 according to SIA 262, weight 0.2) and weight loss after 56 m$_{56}$ cycles (reference value 1.0—requirements of FT2 category according to PN-EN 13877-2, weight 0.5). The obtained PI values are presented in Table 7. On the basis of multi-criteria evaluation of the tested concrete, the best parameters were achieved by concrete series C30-4.5.

Table 7. PI values calculated on the basis of the assumptions described above.

| Concrete ID | C0-1 | C15-1 | C30-1 | C0-4.5 | C15-4.5 | C30-4.5 | C0-8 | C15-8 | C30-8 |
|-------------|------|-------|-------|--------|---------|---------|------|-------|-------|
| PI          | 11.17| 0.53  | 0.49  | 6.82   | 0.78    | 0.47    | 39.55| 0.90  | 0.74  |

Figure 1 shows the air permeability after 56 and 90 days depending on the relative humidity, measured with a Tramex instrument (RH Tramex), of the tested concrete. It is noticeable that the concrete dries out much faster with 30% CSA. The drying rate increases as the air content of the concrete mixture increases. Between 56 and 90 days of drying the biggest differences in RH can be seen with 30% CSA, and drying is still faster in the concrete with the higher air content. The above observations are probably evidence of the concrete drying itself, which is more intensive with a higher CSA content, and also lasts from 56 to 90 days. Significantly higher RH values in concrete series with 0% CSA confirm more intensive drying of concrete with CSA.

Concrete with a higher air content, apart from drying itself, dries out more intensively in a natural way. Series with 30% CSA had significantly worse scaling resistance. Interpretations of air permeability results including concrete RH allows for a more complete assessment of concrete tightness. Straight lines fitted to the relationship between $kT$ and RH both in the case of results obtained after 56 and 90 days have a determination factor $>0.5$ in spite of significant differences in the recipes of the tested concrete series. Concrete that is not aerated, regardless of the CSA content, is clearly tighter, the point corresponding to the result lies well below the straight line. The most above the straights are the results of the series with 15% CSA and aeration 4.5% and 8%, and these concrete series should be considered as the least tight.
4. Conclusions

The addition of 15% and 30% CSA results in a significant reduction in the RH of the concrete after storage under air-dry conditions, and an increase in air permeability measured by the Torrent method, compared to the results of a series without CSA.

The series with 30% CSA in the binder, regardless of aeration, showed significantly lower resistance to cyclic freezing in the presence of salt. Scaling values after 56 days of testing are even twenty times higher compared to reference series without CSA.

There is no clear correlation between sorptivity, air permeability and surface scaling. These properties probably depend in a different way on the pore structure of the concrete.

The strength after 28 and 90 days reached values lower than those of the reference series without aeration and CSA. This may be due to a higher water demand during CSA hydration and unfavorable conditions of maturation without wet care from day 2.

On the basis of multi-criteria evaluation of the tested concrete and performed PI calculations, the best parameters were achieved by concrete series C30-4.5.

Funding: This research received no external funding.

Conflicts of Interest: The author declares no conflict of interest.

References

1. Rytel, M.; Walewski, M. Road Building in Poland. The Facts and the Myths, Experience and Perspectives; Polski Związek Pracodawców Budownictwa: Warsaw, Poland, 2013.
2. Senderski, M. Concrete roads in Poland: The case for selling non-mainstream infrastructure technologies. In Proceedings of the International Symposium on Concrete Roads, Prague, Czech Republic, 23–26 September 2014; pp. 1–16. [CrossRef]
3. Wasilewska, M.; Gardziejczyk, W.; Gierasimiuk, P. Evaluation of Skid Resistance of Exposed Aggregate Concrete Pavement in the Initial Exploitation Period. Roads Bridg. Most. 2017, 16, 295–308. [CrossRef]
4. Glinicki, M.A. *Inżynieria Betonowych Nawierzchni Drogowych*; Wydawnictwo Naukowe PWN: Warsaw, Poland, 2019.

5. Reiterman, P.; Keppert, M. Effect of various de-icers containing chloride ions on scaling resistance and chloride penetration depth of highway concrete. *Rods Bridg. Dro. Most.* 2020, 19, 51–64. [CrossRef]

6. Wang, K.; Nelsen, D.E.; Nixon, W.A. Damaging effects of deicing chemicals on concrete materials. *Cem. Concr. Compos.* 2006, 28, 173–188. [CrossRef]

7. Shi, X.; Fay, L.; Peterson, M.M.; Berry, M.; Mooney, M. A FESEM/EDX investigation into how continuous deicer exposure affects the chemistry of Portland cement concrete. *Constr. Build. Mater.* 2011, 25, 957–966. [CrossRef]

8. Cai, G.; Zhao, J. Application of sulfoaluminate cement to repair deteriorated concrete members in chloride rich environment—A basic experimental investigation of durability properties. *KSCE J. Civ. Eng.* 2016, 20, 2832–2841. [CrossRef]

9. Guan, Y.; Gao, Y.; Sun, R.; Won, M.C.; Ge, Z. Experimental study and field application of calcium sulfoaluminate cement for rapid repair of concrete pavements. *Front. Struct. Civ. Eng.* 2017, 11, 338–345. [CrossRef]

10. Kucharczyková, B.; Misák, P.; Vymazal, T. Determination and evaluation of the air permeability coefficient using Torrent Permeability Tester. *Russ. J. Nondestruct. Test.* 2010, 46, 226–233. [CrossRef]

11. Ebensperger, L.; Torrent, R. Concrete air permeability “in situ” test: Status quo. *Rev. Ing. Construcción* 2010, 25, 371–382. [CrossRef]

12. Jacobs, F.; Leemann, A.; Teruzzi, T.; Torrent, R.J.; Denarié, E. Specification and site control of the permeability of the cover concrete: The Swiss approach Dedicated to Professor Dr. Bernhard Elsener on the occasion of his 60th birthday. *Mater. Corros.* 2012, 63, 1127–1133. [CrossRef]

13. RILEM Tests for Gas Permeability of Concrete. TC 116-PCD: Permeability of concrete as criterion of its durability. *Mater. Struct.* 1999, 32, 174–179. [CrossRef]

14. Hargis, C.W.; Telesca, A.; Monteiro, P.J.M. Calcium sulfoaluminate (Ye’elimite) hydration in the presence of calcium, calcite, and vaterite. *Cem. Concr. Res.* 2014, 65–69. [CrossRef]

15. Batog, M.; Synowiec, K.; Dziuk, D.; Maurizio Iler, M. Beton zawierający cement wapniowo-siarczanog. *Mater. Struct.* 2010, 46, 1389–1400. [CrossRef]

16. Thomas, R.J.; Maguire, M.; Sorensen, A.D.; Quezada, I. Calcium Sulfoaluminate Cement. *Concr. Int.* 2018, 40, 65–69.

17. Gibas, K.; Glinicki, M.A.; Nowowiejski, G. Evaluation of impermeability of concrete containing calcareous fly ash in respect to environmental media. *Rods Bridg. Dro. Most.* 2013, 12, 159–171. [CrossRef]

18. Glinicki, M.A.; Nowowiejski, G. Strength and permeability of concrete with CEM II and CEM V cements containing high calcium fly ash. In Proceedings of the 3rd International Conference on Sustainable Construction Materials and Technology—SCMT; Kyoto, Japan, 18–22 August 2013; pp. 43–50.

19. Yang, K.; Basheer, P.A.M.; Bai, Y.; Magee, B.J.; Long, A.E. Development of a new in situ test method to measure the air permeability of high performance concretes. *NDT E Int.* 2014. [CrossRef]

20. Adámeck, J.; Juráňková, V. Durability of Concrete as a Function of the Properties of the Concrete Layer. *Trans. Transp. Sci.* 2009, 2, 188–195. [CrossRef]

21. Neves, R.; Da Fonseca, B.S.; Branco, F.; De Brito, J.; Castela, A.; Montemor, M.F. Assessing concrete carbonation resistance through air permeability measurements. *Constr. Build. Mater.* 2015, 82, 304–309. [CrossRef]

22. Glinicki, M.A. Methods of qualitative and quantitative assessment of concrete air entrainment. *Cem. Wapno Beton* 2014, 19, 359–369.

23. Glinicki, M.A.; Zielitiski, M. Air void system in concrete containing circulating fluidized bed combustion fly ash. *Mater. Struct.* 2008, 41, 681–687. [CrossRef]

24. Glinicki, M.A.; Jaskulski, R.; Dąbrowski, M. Design principles and testing of internal frost resistance of concrete for road structures-critical review. *Rods Bridg. Dro. Most.* 2016, 15, 21–43. [CrossRef]

25. Antón, C.; Climent, M.A.; de Vera, G.; Sánchez, I.; Andrade, C. An improved procedure for obtaining and maintaining well characterized partial water saturation states on concrete samples to be used for mass transport tests. *Mater. Struct.* 2013, 46, 1389–1400. [CrossRef]
26. Yang, K.; Basheer, P.A.M.; Magee, B.; Bai, Y. Investigation of moisture condition and Autoclam sensitivity on air permeability measurements for both normal concrete and high performance concrete. *Constr. Build. Mater.* 2013, 48, 306–314. [CrossRef]

27. Kameche, Z.A.; Ghomari, F.; Choisnka, M.; Khelidj, A. Assessment of liquid water and gas permeabilities of partially saturated ordinary concrete. *Constr. Build. Mater.* 2014, 65, 551–565. [CrossRef]

28. Gardner, D.R.; Lark, R.J.; Barr, B. The effect of conditioning to a predetermined weight loss on the permeability of concrete. *Constr. Build. Mater.* 2007, 21, 83–89. [CrossRef]

29. Swiss Association for Standardization. SN 505262/1 (SIA 262/1) Concrete Structures—Supplementary Specifications; Swiss Association for Standardization: Winterthur, Switzerland, 2019.

30. Beushausen, H.; Fernandez Luco, L. (Eds.) *Performance-Based Specifications and Control of Concrete Durability;* RILEM State-of-the-Art Reports; Springer: Dordrecht, The Netherlands, 2016; Volume 18, ISBN 978-94-017-7308-9.

31. Kubissa, W.; Glinicki, M.A. Influence of internal relative humidity and mix design of radiation shielding concrete on air permeability index. *Constr. Build. Mater.* 2017, 147, 352–361. [CrossRef]

32. Romer, M. Effect of moisture and concrete composition on the Torrens permeability measurement. *Mater. Struct.* 2005, 38, 541–547. [CrossRef]

33. Kastiukas, G.; Zhou, X.; Castro-Gomes, J.; Huang, S.; Saafi, M. Effects of lactic and citric acid on early-age engineering properties of Portland/calcium aluminate blended cements. *Constr. Build. Mater.* 2015, 101, 389–395. [CrossRef]

34. Zhang, G.; Li, G.; Li, Y. Effects of superplasticizers and retarders on the fluidity and strength of sulfoaluminate cement. *Constr. Build. Mater.* 2016, 126, 44–54. [CrossRef]

35. Glinicki, M.A.; Zieliński, M. The influence of CFBC fly ash addition on phase composition of air-entrained calcium aluminate blended cement. *Cem. Concr. Res.* 2015, 78, 389–395. [CrossRef]

36. Polish Committee for Standardization. SN 505262/1 (SIA 262/1) Concrete Structures—Supplementary Specifications; Polish Committee for Standardization: Warsaw, Poland, 2017.

37. Brachaczek, W. Study of the Properties of Renovation Plasters as a Function of Air Content and Porosity. In *Proceedings of the Awarie Budowlane 2013,* Międzyzdroje, Poland, 21–24 May 2013; pp. 873–880.

38. Kubissa, W.; Glinicki, M.A.; Dąbrowski, M. Permeability testing of radiation shielding concrete manufactured at industrial scale. *Mater. Struct. Constr.* 2018, 51. [CrossRef]

39. Kubissa, W.; Jaskulski, R. Measuring and Time Variability of The Sorptivity of Concrete. In *Proceedings of the 16th World Congress on Concrete:* RILEM State-of-the-Art Reports; Springer: Dordrecht, The Netherlands, 2016; Volume 18, ISBN 978-94-017-7308-9.

40. Tracz, T.; Śliwiński, J. The effect of concrete surface polishing on permeability evaluated with Torrens’s method. In Proceedings of the Awarie Budowlane 2013, Międzyzdroje, Poland, 21–24 May 2013; pp. 873–880.

41. Pietrzak, A. Wpływ domieszek napowietrzających na wybrane parametry mieszanki betonowej i betonu. Zesz. Nauk. Politech. Częstochowskiej. Bud. Zesz. Nauk. Politech. Częstochowskiej. Bud. 2013, 169, 122–128.

42. Chen, Y.; Wang, K.; Wang, X.; Zhou, W. Strength, fracture and fatigue of pervious concrete. *Constr. Build. Mater.* 2013, 42, 97–104. [CrossRef]

43. Velazco, G.; Almanza, J.M.; Cortés, D.A.; Escobedo, J.C.; Escalante-Garcia, J.I. Effect of citric acid and the hemihydrate amount on the properties of a calcium sulfoaluminate cement. *Mater. Struct. Constr.* 2018, 51. [CrossRef]

44. Marks, M.; Józwiak-Niedźwiedzka, D.; Glinicki, M.A.; Olek, J.; Marks, M. Assessment of scaling durability of concrete with CFBC ash by automatic classification rules. *J. Mater. Civ. Eng.* 2012, 24, 860–867. [CrossRef]

45. Giergiczny, Z.; Boos, P. Testing the frost resistance of concrete with different cement types—Experience from laboratory and practice. *Archit. Civ. Eng. Environ.* 2010, 3, 41–51.

46. Glinicki, M.A.; Zielinski, M. The influence of CFBC fly ash addition on phase composition of air-entrained concrete. *Bull. Polish Acad. Sci. Tech. Sci.* 2008, 56, 45–52.

47. Kubissa, W.; Jaskulski, R.; Chen, J.; Ng, P.-L.; Godlewska, W.; Reiterman, P. Evaluation of Ecological Concrete Using Multi-Criteria Ecological Index and Performance Index Approach. *Archit. Civ. Eng. Environ.* 2019, 12, 97–107. [CrossRef]

© 2020 by the author. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).