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Kielce University of Technology

# THE INFLUENCE OF THE TYPE OF LOW-EMISSION CEMENT AND AIR-ENTRAINING ADMIXTURES ON THE QUALITY OF AIR ENTRAINMENT OF CONCRETE – UNEXPECTED IMPACT OF GRANULATED BLAST FURNACE SLAG

# WPŁYW RODZAJU CEMENTU NISKOEMISYJNEGO I DOMIESZKI NAPOWIETRZAJĄCEJ NA EFEKT NAPOWIETRZENIA BETONU – NIEOCZEKIWANY WPŁYW GRANULOWANEGO ŻUŻLA WIELKOPIECOWEGO

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## **Abstract**

*The test results indicate that it is possible to obtain the appropriate quality of air entrainment in concrete with low*emission cement with granulated blast furnace slag, such as CEM II/B-S, CEM III/A and CEM III/A-NA. However, *the literature on the subject reports that this is not the rule, and a slag content that is too high is not conducive to the effectiveness of the air-entraining admixture and the stability of the air entrainment. The possible reason for the beneficial effect of slag on the stability of air entrainment was the influence of surfactants used for its grinding. The research also proved that the effectiveness of the air-entraining impact and the stability of air entrainment in concrete with slag cement depend on its type (natural or synthetic) and the alkali content in the cement.*

**Keywords:** low-emission cement, granulated blast furnace slag, alkali, concrete, air-entraining admixture, porosity, frost resistance

## **Streszczenie**

*Rezultaty badań wskazują, iż możliwe jest uzyskanie odpowiedniej jakości napowietrzenia betonu z cementem niskoemisyjnym z żużlem granulowanym wielkopiecowym S, jak CEM II/B-S, CEM III/A i CEM III/A-NA. Jednak literatura przedmiotu donosi, iż to nie jest regułą, a zbyt duża zawartość żużla nie sprzyja efektywności działania domieszki napowietrzającej i stabilności napowietrzenia. Zatem granulowany żużel wielkopiecowy może pozytywnie bądź negatywnie wpływać na efektywność działania domieszki napowietrzającej oraz stabilność napowietrzenia. Za możliwą przyczynę korzystnego wpływu żużla na stabilność napowietrzenia wskazano wpływ środków powierzchniowo czynnych zastosowanych do jego przemiału. Na podstawie badań dowiedziono także, że efektywność działania domieszki napowietrzającej i stabilność napowietrzenia betonu z cementem żużlowym zależy od jej rodzaju (naturalna bądź syntetyczna) oraz od zawartości alkaliów w cemencie.*

**Słowa kluczowe:** cement niskoemisyjny, granulowany żużel wielkopiecowy, alkalia, beton, domieszka napowietrzająca, porowatość, mrozoodporność

#### 1. INTRODUCTION

Low-emission cement is the most effective way to reduce the carbon footprint of concrete, as much as 85% – Figure 1. The European Cement Industry Association CEMBUREAU has prepared a Road Map [1] which assumes that cement and concrete producers will achieve  $CO<sub>2</sub>$  emissions by approx. 40% and achieve climate neutrality in 2050. Since, as mentioned, Portland clinker is responsible for the carbon footprint of cement and concrete, this goal will be achieved, among others, by significantly reducing the amount of clinker in cement [2, 3].



*Fig. 1. Global Warming Potential, GWP, depending on the type of cement of the same strength class (300 kg of cement/ m3 of concrete was assumed) on the basis of [1] [3] and [4]* 

The production of CEM II/A Portland cement with an addition of 18% and CEM II/B with an addition of 33% slag or ash reduces  $CO$ , emissions by 162 kg and 297 kg, respectively, per Mg of cement, compared to CEM I Portland cement. For Portland cement multicomponent СЕМ ІІ/B-М 42.5 R, the total emission is  $562 \text{ kg CO}$ , per tonne of cement, which is as much as 1.54 times lower than in the case of CEM I 42.5 R. Production of metallurgical cements: CEM III/A containing 61% slag, CEM III/B containing 76% slag and CEM III/C with 90% slag content allows for a reduction in  $CO<sub>2</sub>$  emissions by approximately 65, 80 and 95% per Mg of cement, respectively, compared to CEM I Portland cement. However, it should be remembered that currently, slag resources are running out [3, 5], and it will be a component of other cement, such as CEM II/A (S-LL) and others, provided, of course, it is still available. Currently, using low-emission cement is not a choice but a necessity.

The use of the cement mentioned above with reduced  $CO<sub>2</sub>$  emissions has been proven and widely practised on a global scale for decades, and concrete with low-emission cement has advantages in many exposure classes over Portland cement concrete [1, 3, 4] (including better consistency, workability of concrete, resistance to sulfates, protection against alkaline corrosion, long-term strength, higher tightness, low shrinkage). However, concrete with low-emission cement requires special attention due to its durability in the frost and carbonate exposure class [6-13], as well as the protection of steel against corrosion. Problems with their frost resistance may occur even in conditions of moderate frost exposure, especially when exposed to de-icing salts [7, 9, 14, 15, 39].

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The condition for concrete to become frost-resistant is its adequate air entrainment and ensuring sufficient tightness of the concrete [6, 7, 17]. From the point of view of frost resistance, it is not essential to total air content but the quality of air entrainment [16]. Frost resistance is promoted by increased air content and reduced pore size, as both of these factors reduce the distance between pores [13, 18, 19]. Other parameters characterising concrete's air entrainment structure are the surface area of the air pore system  $\alpha$  and the content of micropores  $A_{300}$ . The specific surface area is determined by dividing the total surface of the air pores by their volume, which is expressed in mm<sup>2</sup> . The  $A_{300}$  parameter specifies the content of air pores with a diameter of 0.3 mm  $(300 \mu m)$  or smaller.

As proven in many publications [6, 8, 9, 21], the type of ingredients other than clinker also significantly affects the stability of air entrainment. It is believed that replacing Portland clinker with another ingredient does not promote the effectiveness of air-entraining admixtures and the stability of air-entrainment (Fig. 2) [7, 13, 19, 20, 22, 24]. It is also recommended to increase the amount of air-entraining admixture above the recommended amount in cases where the cement has an increased specific surface. In publications [15, 14] it was proven that granulated blast furnace slag also has a negative impact on the air entrainment of concrete. Another factor that negatively affects the effectiveness of the air-entraining admixture is the low alkali content [7, 22, 24, 25]. According to Committee 225 (Guide to the Selection and Use of Hydraulic Cement) and Committee 201 (Guide to Durable Concrete) of the American Concrete Institute (ACI), different varieties of Portland cement and multi-component cement make it possible to achieve the same level of frost resistance of concrete, provided the correct proportions ingredients and proper airentrainment of the mixture.



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*Fig. 2. The influence of silica fume – D, silica fly ash – V, and slag – S on the air-content of concrete [7]*

The use of cement with a higher slag content, especially with a significantly developed specific surface, may reduce the total amount of air in the hardened concrete (Figs. 3 and 4). Moreover, the presence of slag causes disturbances in the distribution of air bubbles, causing a reduction in the content of pores smaller than 300 µm and an increase in the average distance between air pores [23, 26]. As shown in Figure 10, the increase in the demand for an air-entraining admixture occurs only after exceeding 60% of the amount of slag in the cement, and in the case of CEM III/A, the amount of slag is usually 60- 65%. Between 80% and 90% slag content, twice the amount of air-entraining admixture is required to obtain the same amount of air-entrainment.





*Fig. 3. Impact of the slag content on the effect of airentraining of concrete [27]*



*Fig. 4. Air-entraining admixture demand and air content in concrete relative to the content of slag in cement [28]* 

Obtaining pores with a precisely defined diameter and appropriate spacing in concrete with low-emission cement requires the use of a proper amount and type of air-entraining agents. Agents with a potential airentraining effect can be divided into [6, 14, 29, 30]:

• synthetic – very efficient, requiring short mixing time. Synthetic surfactants have great potential and constitute the largest group of surfactants. It is believed that they create air bubbles with smaller diameters and, therefore, with a more excellent content of micropores.

Natural is the oldest and most well-known group of products, and they are very compatible with other admixtures. It is believed that natural surfactants are less efficient than their synthetic counterparts and are also characterised by limited resistance to the influence of alkalis (which, however, occur to a limited extent in the case of low-emission cement, which theoretically should favour their effectiveness);

• mixed – the synergistic effect of natural and synthetic ingredients allows the creation of more specialised products. Mixtures offer a wider scope for manoeuvre and set new trends in the field.

The research undertaken in this article aims to compare the quality of air entrainment in concrete with low-emission cement and granulated blast furnace slag (S): CEM II/B-S, CEM III/A and CEM III/A-NA, depending on the air-entraining admixture used: natural or synthetic.

# 2. METHODOLOGY OF RESEARCH

#### 2.1. Materials

In the first stage of the study, the effect of the type of cement on the demand of two kinds of admixtures was compared: synthetic (S) based on mixed, synthetic polymers and natural (N), constituting an aqueous solution of a complex mixture of organic acid salts on the air-entrainment of mortar and concrete according to PN-EN 480-1 [31] (Tables 2, 3 and 4, Fig. 5) with slag cement CEM II/B-S 42.5 R, CEM III/A 42.5R and CEM III/A-NA 42.5 R (Table 1) with the characteristics of the components given in Tables 4 and 5.

*Table 1. The percentage amount of granulated blast furnace slag S in the composition of types of cement, %*

| <b>Cement type</b>  | <b>CEM 142.5R</b> | Slag S | Setting regulator* |  |
|---------------------|-------------------|--------|--------------------|--|
| <b>CEM I</b>        | 100%              |        | 0.0                |  |
| CEM II/B-S          | 65.5              | 33     | 1.5                |  |
| CEM III/A           | 45.5              | 53     | 1.5                |  |
| <b>CEM III/A-NA</b> | 37                | 65     | 3.0                |  |

\*amount of  $SO_3$  about 2.8-3.1%.











|   |                    | Average value        | Requirement             |                 |  |
|---|--------------------|----------------------|-------------------------|-----------------|--|
| <b>Compressive</b><br>strength          | <b>Unit</b>        | <b>CEM I</b><br>42.5 | <b>CEM I</b><br>42.5 NA |                 |  |
| after two days                          |                    | 28.2<br>25.0         |                         | >20.0           |  |
|   | MPa                | 57.1                 | 56.2                    | >42.5           |  |
| after 28 days                           |                    |                      |                         | $\leq 62.5$     |  |
| Start of setting time                   | min                | 203                  | 226                     | $\geq 60$       |  |
| Water<br>to the standard<br>consistency | $\frac{0}{0}$      | 27.8                 | 27.9                    | No requirements |  |
| <b>Volume constancy</b>                 | mm                 | 0.4                  | 0.7                     | $\leq 10$       |  |
| Specific surface area                   | cm <sup>2</sup> /q | 4158                 | 4037                    | No requirements |  |
| SO <sub>3</sub>                         | $\%$               | 2.87                 | 2.92                    | < 4.0           |  |
| <sup>-</sup>                            | $\frac{0}{0}$      | 0.076                | 0.041                   | ≤0.10           |  |
| Loss of ignition                        | $\%$               | 2.83                 | 2.61                    | $\leq 5.0$      |  |
| Insoluble residue                       | $\frac{0}{0}$      | 0.51                 | 0.71                    | $\leq 5.0$      |  |
| Na <sub>2</sub> O <sub>eq</sub>         | $\%$               | 0.62                 | 0.56                    | No requirements |  |

*Table 5. Components of slag S [%]*





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*Fig. 5. Gravel aggregate grain size distribution*

#### 2.2. Methods

First, the air-entrainment of the mortars was determined in accordance with the procedure described in PN-EN 1015-7 [32], and then the airentrainment of concrete mixtures according to PN-EN 12350-7 [33] after 4, 20, and 40 minutes. If the mixture was aerated after 5, 20, and 40 minutes and was in the range of 4-7%, the determined amount of air-entraining admixture was left unchanged; otherwise, it was corrected.

After 28 days of concrete maturation in water, the values of concrete air-entrainment parameters according to PN-EN 480-11 [34] were determined by means of a CT scanner and computer image analysis (Fig. 6) in two directions, parallel and perpendicular.



*Fig. 6. View of a) the analysed porosity of concrete using a computer program (air pores are marked in yellow) and b) the concrete core cut out of 15x15x15 cm concrete cubes in the direction perpendicular or parallel to the direction of formation* 



#### 3. RESEARCH RESULTS AND ANALYSIS

### 3.1. Research results

Figures 7 and 8 summarise the amount of natural or synthetic air-entraining admixture necessary, depending on the type of cement, to obtain the air content given in Table 5.







*Fig. 7. Amount of natural air-entraining admixture depending on the type of cement CEM II/B-S, CEM III/A and CEM III/A-NA*



*Fig. 8. Amount of synthetic air-entraining admixture depending on the type of cement CEM II/B-S, CEM III/A and CEM III/A-NA*

*Table 7. Results of the determination of air pore parameters of concrete with synthetic air-entraining admixture (N)*

| <b>Cement type</b>      | <b>Total Pore</b><br>Content<br>in Concrete | Specific<br>surface area<br>of air pores | Pore<br><b>Distribution</b><br><b>Indicator</b> | <b>Micropore</b><br>content | <b>Total Pore</b><br><b>Content</b><br>in Concrete | Specific<br>surface area<br>of air pores | Pore<br><b>Distribution</b><br><b>Indicator</b> | <b>Micropore</b><br>content |
|-------------------------|---|--|---|-----------------------------|--|--|---|-----------------------------|
|                         | parallel direction                          |  |   |                             | perpendicular direction                            |  |   |                             |
|                         | A, %  | $a, \text{mm}^2$                         | L. mm   | $A_{300}$ , %               | A, %   | $a.$ mm <sup>2</sup>                     | $L$ , mm  | $A_{300}$ , %               |
| $CEM II/B-S$ AEA $(S)$  | 5.86  | 42.43                                    | 0.11  | 4.59                        | 5.83   | 40.33                                    | 0.12  | 4.29                        |
| $CEM III/A + AEA(S)$    | 5.65  | 42.99                                    | 0.11  | 4.26                        | 5.81   | 43.99                                    | 0.11  | 4.24                        |
| CEM III/A-NA $+$ AEA(S) | 5.06  | 42.48                                    | 0.12  | 3.48                        | 4.88   | 41.61                                    | 0.12  | 3.50                        |

*Table 8. Results of the determination of air pore parameters of concrete with natural air-entraining admixture (N)*



Tables 7 and 8 summarise the results of determining the parameters of the porosity structure of hardened concrete obtained using a tomograph of samples taken parallel and perpendicular to the direction of formation of a concrete sample with dimensions of  $15 \times 15 \times 15$  cm.

#### 3.2. Analysis of research results

Figures 9 and 10 compare the required amount of synthetic and natural air-entraining admixtures to obtain proper air-entrainment of the concrete mixture according to PN-EN 480-1 (Fig. 11). As evidenced by the test results presented in Figures 6, 7 and 8, the required amounts of air-entraining admixtures are significantly different depending on the type of cement. In the case of cement with S slag (CEM II/B and CEM III/A, CEM III/A-NA), the necessary dosage of air-entraining admixtures to obtain adequate airentrainment of the mortar and concrete is lower than in the case of Portland cement (CEM I). Moreover, the natural admixture was characterised by better air-entrainment efficiency (Figs. 6-8). As can be seen from the data analysis presented in Figures 9 and 10, the amount of the required natural admixture is ten times lower than in the case of a synthetic admixture.



*Fig. 9. Influence of the type of cement on the required amount of natural air-entraining admixture AEA to obtain adequate air-content (Ac) mortar and concrete according to PN-EN 480-1 [31]*



*Fig. 10. Influence of the type of cement on the required amount of synthetic air-entraining admixture AEA to obtain adequate air-content (Ac) mortar and concrete according to PN-EN 480-1 [31]*



*Fig. 11. The relationships between air content in mortar and concrete acc. to PN-EN 480-1 with CEM II/B, CEM III/A and CEM III/A-NA*

As it turns out, CEM III/A also achieved the greatest stabilisation of air entrainment over time (Figs. 12 and 13), also compared to CEM I. Slightly worse airentrainment stability is achieved when the cement is characterised by a low alkali content (CEM III/A -NA).



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*Fig. 12. The stability of air entrainment of the concrete mixture depends on the type of cement with a synthetic air-entraining admixture*



*Fig. 13. The stability of air entrainment of the concrete mixture depends on the type of cement with a natural airentraining admixture*

To protect concrete against the destructive effects of frost, it is necessary to provide [11, 18, 35-38]:

- the total air content will be between 4 and 7%, depending on the size of the aggregate grains [36];
- the air pore distribution index  $\overline{L}$  (spacing factor) will have a value below 0.20 mm;
- the specific surface area of air pores α will be below  $15-20$  mm<sup>2</sup>/mm<sup>3</sup>;
- content of pores with a diameter of less than 0.300 mm:  $A_{300}$  > 1.5-1.8%.

The recommended values mentioned above also depend on the type of concrete (w/c, kind of cement, and others) as well as on external conditions [13, 39, 40, 42]. As evidenced by the test results in Tables 6 and 7 and in Figures 14-17, concrete with slag is characterised by the correct porosity structure. Airentrained concrete with a synthetic admixture is more advantageous regarding frost resistance and mechanical properties; pores are smaller and more closely spaced, as achieved in the publication [41]. However, the test results analysed above have proven that the synthetic-based air-entraining admixture is less resistant to the impact of ingredients other than clinker on the stability of concrete air entrainment (Figs. 9 and 10). Therefore, an increased amount of it should be used at the beginning so that the concrete is properly aerated after a specific time.



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*Fig. 14. Results of determining the air content A in concrete according to PN-EN 480-11 in concrete with CEM II/B-S, CEM III/A, CEM III/A-NA with a natural (N) or synthetic (S) admixture*



*Fig. 15. Results of determining the specific pore surface in the concrete according to PN-EN 480-11 in concrete with CEM II/B-S, CEM III/A, CEM III/A-NA with a natural (N) or synthetic (S) admixture*



*Fig. 16. Results of determining the pore spacing index L in concrete according to PN-EN 480-11 in concrete with CEM II/B-S, CEM III/A, CEM III/A-NA with a natural (N) or synthetic admixture (S)*



*Fig. 17. Results of determining the A300 pore content in concrete according to PN-EN 480-11 for concrete with CEM II/B-S, CEM III/A, CEM III/A-NA with a natural (N) or synthetic (S) admixture*

As shown above, concrete with slag cement may have a porosity structure suitable for frost resistance, even with a higher proportion of slag relative to cement, as in the case of CEM III/A. However, this is not the rule, given the research results mentioned in the introduction to the article (Figs. 2-4). In the case analysed by the author, the reason for the exceptional stability of air entrainment (Figs. 11 and 12 and the negligible demand for an air-entraining admixture, even lower than in the case of CEM I (Fig. 7), is the influence of a surface-active admixture that facilitates slag grinding, cooperating with the air-entraining admixture because both the mortar and the concrete without an air-entraining admixture did not show excessive air content (Table 6). Only introducing an air-entraining admixture caused interactions between the admixtures used for grinding the slag and airentraining the concrete. Although it is mentioned in the publication [43] that the surfactant may cause problems obtaining a low air content in the case of concrete with CEM III/A. Publication [44] also emphasises the need to test the compatibility of surface-active admixtures used for grinding cement or slag and admixtures used for concrete, especially fluidising admixtures.

Admixtures that intensify the slag and cement grinding process are substances that, when mixed with the mill contents, increase the rate of grain reduction, which translates into a decrease in the energy consumption of the process. The most frequently used additives to facilitate grinding are surface-active substances, e.g. glycols, triethanolamine (TEA), and diethanolamine (DEA) [44]. As a result of adding agents that intensify the grinding process, they are adsorbed on the grains. Ethylene glycol distearate (EGDS) or glycol stearate is a chemical compound

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used as an emollient in cosmetics and other consumer products.

Diethanolamine, DEA, is an organic chemical compound from the group of amino alcohols. It belongs to the group of biogenic amines. It is alkaline. Diethanolamine is a solid below the freezing point. Above the freezing temperature, it becomes a syrupy liquid. It has a characteristic, slightly ammoniacal smell. It is a product of the breakdown of natural phosphatides. It is obtained from ethylene oxide and ammonia. Apart from cement production, the primary uses of diethanolamine are in the following areas:

- agrochemicals, herbicides, glyphosate,
- ingredient of surfactants and detergents,
- ingredient of washing agents,
- used in cosmetics,
- absorbent in gas purification scrubbers,
- an ingredient in the paper dyeing process,
- emollient,
- emulsifier,
- plasticiser,
- corrosion inhibitor,
- in the textile industry.

Triethanolamine (TEA), in turn, is a tertiary amine and a triol. It is a bifunctional compound that has the properties of both alcohols and amines. It belongs to viscous, colourless organic compounds. The pH of the compound is alkaline. In addition to its use in cement production, TEA is used. It is widely used as a pH regulator (pH regulation in cosmetic products) and surfactant in industrial and cosmetic products and liquid detergents, etc.

The above-mentioned surface-active admixtures have favourable conditions for air stabilisation in aerated concrete, especially when larger amounts are used in the blast furnace slag grinding process.

Finally, it should be clearly noted that the conclusions obtained regarding the influence of slag and the type of air-entraining admixtures apply only to those used by the author. Other types of air-entraining admixtures, due to their high variability in their elemental composition and polymer structure (in the case of synthetic admixtures – anionic, cationic and others), may give slightly different results compared to the air entrainment of low-emission concrete and require experimental testing before their application.

In addition, it is essential to confirm the frost resistance of properly air-entrained concrete made with low-emission cement such as CEM II/B-S, CEM III/A, or CEM III/A-NA, and other concrete properties after a period stipulated in the recently revised standard PN-B-06265 [44], which is either 56 or 90 days (see Table 9). It is crucial to emphasize that during this period, intensive moisture maintenance is necessary due to the extended development of concrete tightness with hydraulic additives. Figure 18 illustrates the progression of tightness (waterproofness) and strength of concrete with CEM III/A and a synthetic admixture.





*Fig. 18. Permeability test results (W8) of concrete with CEM III/A and air-entraining synthetic admixture after 28, 56 and 90 days of hardening in water*

As mentioned in the introduction, today, using lowemission cement is not a choice but a necessity. So, knowing how to achieve proper air entrainment in lowcarbon footprint concrete is crucial. The composition of low-emission cement will continue to evolve due to the decreasing resources of blast furnace slag and silica fly ash. The problem of low carbon footprint concrete and cement will continue to be relevant. The best way to ensure its frost resistance is to take two parallel paths, improve tightness because of a significant reduction in the amount of water to approximately 0.35 [38] and ensure a dense distribution of air pores with the minor possible diameters in the volume of concrete. To achieve this, it is necessary to take into account not only the type of low-emission cement and air-entraining admixture but also the influence of other ingredients and factors, especially temperature and the concrete production process discussed, for example in publications [15, 16, 25, 39, 46, 47-49].

### 4. CONCLUSION

Based on the obtained research results, it was found that:

• the influence of analysed slag on the air-entrainment of concrete is unstable because depending on the



type of collapse, the method of its preparation, and, more precisely, the admixture facilitating its milling, the effectiveness of the air-entraining admixture and the stability of the air-entrainment are different;

- in the case of the slag analysed in the article, either too much of our factory was used, or such a type was used that it was not recorded that the negative impact of slag mentioned in the literature was noted on the air entrainment;
- an air-entraining admixture is essential from the point of view of efficiency in its operation (airentrainment) and stability of air-entrainment of concrete. On the attitude of the results of airentrainment of air-entrainment during and the results of determining the parameters of the porosity structure, it can be concluded that the natural air-entraining admixture more stabilises the air-entrainment of low-emission binders than a synthetic admixture. To ensure adequate air entrainment of concrete over time, increasing the

content of the synthetic admixture in the concrete mixture is necessary;

- air-entraining admixture is also essential because of its air-entrainment characteristics. A more favourable impact in the scope of obtained parameters of air-entrainment of low-emission concrete is characterised by a synthetic than natural admixture. The air season has smaller sizes and are closer to each other:
- the amount of alkali in cement CEM III/A affects the effectiveness of air-entraining admixture and air-entrainment stability, which also depends on the air-entraining admixture used. The lower alkali content in CEM III/A requires an increase in the amount of air-entraining admixture to obtain the right amount of air-entrainment of concrete. The low content of alkali in cement could also be more conducive to stabilising the air-entrainment of concrete obtained because of the action of synthetic air-entraining admixture instead of natural. This problem requires further explanations and research.

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