SUPERPLASTICIZER COMPATIBILITY PROBLEM WITH INNOVATIVE AIR-ENTRAINING MULTICOMPONENT PORTLAND CEMENT

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Abstract

When increasing the degree of fluidity of previously aerated cementitious mixtures, there is a problem of maintaining their correct aeration. Most of the available superplasticizers cause a significant increase of the air content of concrete mixtures. The problem of compatibility of superplasticizer and air-entraining admixture increases in case of multicomponent Portland cement, due to different effects of these additives. It comes down to achieving a compatibility of the three variables mentioned in the title of paper, due to the required air entrainment and consistency of mixture. Achieving compatibility of such a system requires a series of experimental studies that were presented in the paper together with their resulting indications.

Originality

The research results conducted by the authors proved that in case of previously air-entrained concrete, i.e. performed with the use of that is made with innovative air-entraining multicomponent Portland cement, after the addition of new generation superplasticizer occurs very large increase in air entrainment. The problem of compatibility of superplasticizers with innovative air-entraining multicomponent Portland cement is very important and new. Compatibility testing of superplasticizers with the air-entraining cement with were not conducted. The authors did not find similar results of studies in the literature.

Keywords: air-entraining cement, fly ash, air-entraining admixture, superplasticizer, compatibility, air-content.

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1. Introduction

In order to maintain better durability performance and extended service life in freezing and thawing environments, concrete must consists of a proper air-void system. For this, a suitable amount of entrained air-voids with accurate specific surface and spacing factor should be maintained in concrete. Commonly, limits on volume of air-voids or air content are specified although the role of spacing factor is important. This is due to the fact that the air content can be determined more effortlessly and immediately than spacing factor (Safiuddin Md., et al. 2006). It may happen that the variation in cement's type while keeping all other technological and material parameters, may decrease concrete's frost resistance (Gebler S. and Klieger P., 1983), (Freeman E., et al. 1997), (Kjellsen K. and Atlassi E., 1999). This issue is clearly formulated by the opinion of Committee 225 (Guide to the Selection and Use of Hydraulic Cements) and Committee 201 (Guide to Durable Concrete) of American Concrete Institute (ACI). It is stated that: "variations of Portland cements and composite cements, allow to obtain the same level of concrete's frost resistance, providing the correct proportions of components and correct mixture's air-entrainment. Crucial, from the point of final air-entrainment effect view apart from the graining, is the type and amount of mineral additives included in cements (blast furnace cement CEM III, pozzolanic cement CEM IV and composite cement CEM V). Mineral additives activate beneficial changes in the structure of paste's porosity (concrete) resulting in decrease of capillary pores and increase of gel pores. The test results of concrete on cements with mineral additives show that despite enhanced sealing and higher resistance, there are problems with frost resistance of non-air-entrained concretes, even under conditions of moderate frost effect.

One solution to the problem is the creation of possibilities of the use of cement with mineral additives containing in the composition of which air-entraining admixture (AEA): air-entraining multicomponent Portland cement. The goal of the project supervised by the authors is to establish the innovative technology of the aerating cements production for designing and production of freeze and thaw resistant concrete. Aerating cements technology concludes dosing aerating additives in the grinding or blending process of cement production. Project focuses principally on cements containing large numbers of non-clinker constituents, i.e. Portland-composite cements CEM II (CEM II/B-V, CEM II/B-S), blast furnace cement CEM III (CEM III/A, CEM III/A NA) and composite cements CEM V (CEM V/A, CEM V/B). This choice of aerating cements for implementation by cement industry fits the sustainable development philosophy. The another favor of the project will be original contribution to the new cement and concrete scientific approaches. However, the results of studies conducted by the authors have shown that the use of new generation superplasticizer with the previously air-entrained concrete mixture causes a problem of maintaining the proper air entrainment. The research results (Mosquet M. 2003), (Sakai E., et al., 2006), (Szwabowski J. and Łaźniewska-Piekarczyk B., 2008) show that the new generation of SPs have side effects manifested by increasing the air content in the mixture (Table 1). The air content in the hardened concrete, which is a side effect of SP, may be higher than 8% (Szwabowski J. and Łaźniewska-Piekarczyk B., 2008). The solution to the present problem is the condition of compatibility of system: innovative air-entraining cement and superplasticizer (SP), due to the presence of the air in the mixture and its consistency.

Table 1	Influence	of superp	lasticizers	type on	air-content	of mixture	(Mosquet N	1. 2003)
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Type of SP	Lignosulfonate	Naphthalene	Melamine	A new generation of superplasticizers		
	LS	SNF	SMF	Polycarboxylate	Aminophosphonates	
				PCP	polyoxyethylene	
					AAP	
Air content	++	+	0	++	++	

The present study is an experimental analysis of the impact of the type of plasticizing and superplasticizing admixtures on the air content and consistency of cement based mixtures made with the participation of the air-entraining innovative CEM II/B-V. Unfortunately, fly ash which contains carbon can attract and absorb the surfactants in AEAs (Kqlaots I., et al., 2004). As model cement

based mixture adopted the reference cement mortar according PN-EN 480-1. Four types of airentraining innovative cement were used, diversified manufacturing technology (jointly mixed and ground together), with the participation of two different types of air-entraining admixture: natural and synthetic. The consistency of mortars was changed with different types of plasticizing and superplasticizing admixtures.

2. Research significance

In case of previously air-entrained concrete that is made with an air-entraining cement, after the addition of new generation SP occurs very large increase in air entrainment. The air-content of mixture according PN-EN 480-1 may be higher than 13%.

3.

Experimental procedure

Materials used to prepare mixtures of cement based mortars, which are the subject of the study were: air-entraining innovative CEM II/B-V (Table 2), normalized sand, distilled water and different types of plasticizing and superplasticizing admixtures (Table 3). The content of siliceous fly ash cements V in all cements is 30%. With these materials, standardized cement mortar with w/c = 0.50 was prepared, in accordance with the guidelines of EN 480-1. Reference mortar are also made, i.e. air-entraining and superplasticizing without admixtures.

Symbol of cement	Type of cement; method of preparation the cement	Air-entraining admixture	Participation of admixtures, % mass of cement.
М	CEM II/B-V; cement mixed together	-	-
G	CEM II/B-V; common ground cement	-	-
M+S	CEM II/B-V; cement mixed together	synthetic	1.70
M+N	CEM II/B-V; common mixed cement	natural	0.12
G+S	CEM II/B-V; cement ground together	synthetic	1.70
G+N	CEM II/B-V; common ground cement	natural	0.12

Table 2. Properties of the air-entraining innovative cements CEM II B-V

Table 3 Characteristic of plasticizing and superplasticizing admixtures

Basic chemical base of SP	Symbol	Type of admixture
polycarboxylate ether	PCE-1	III generation
polycarboxylate ether	PCE-2	III generation
polycarboxylate ether	PCE-3	III generation
polycarboxylate ether	PCE-4	III generation
modified polycarboxylates	PCP-1	III generation
modified polycarboxylates	PCP-2	III generation
modified naphthalenes	MN	III generation
substances from the group of polycarboxylates	РС	III generation
cross-linked polymers, acrylic	CLAP	III generation
modified amino phosphonates	AAP	III generation
sulfonated naphthalene-formaldehyde resins	SNF-1	II generation
sulfonated naphthalene-formaldehyde resins	SNF-2	II generation
sulfonated naphthalene-formaldehyde resins	SMF	II generation
modified lignosulfonates	MLG-1	I generation
modified lignosulfonates / carbohydrates of natural origin	MLG-2	I generation

In the first step of the test, the goal was to analyze four available AEAs for their air entraining compatibility, the air void system and the air stability. EN 206 for XF classes has specified ranges of air content between 4% and 7%. Commonly, around 4.5% to 6.5% air content is required in concrete for freeze-thaw resistance in Poland. Nonetheless, in mortar only fine aggregates are used, because of the absence of coarse aggregates the air content in mortar is roughly twice of that in concrete at the same condition. Thus, in the mortar test air content of about 10-11% was aimed at as the normal condition. After trial and error tests, it was concluded that at a dosage around 0.12% and 1.70 by weight of cement all of these two AEAs can entrain a total air content of around 10-11% (Table 2).

The aim of the second stage of the study was the choice of the type and the quantity of plasticizers and superplasticizers to the air content of mortar was approximately similar to that of the reference mortar, i.e. without plasticizing and superplasticizing admixtures. The liquid plasticizing and superplasticizing admixture dosed with the mixing water, in accordance with recommendation of EN 480-1. While plasticizing and superplasticizing admixture in powder form were dispensed with cement, according to the manufacturer's recommendations. Table 4 summarizes the required amounts of admixtures necessary to liquidate the mortar a comparable degree. In most cases the greatest degree of fluidity of mortars was obtained which is important, maintaining the stability of the mortar. The consistency of the mortars was determined according to EN 1015-3, while the air-content according to EN 1015-7. Ambient temperature during testing mortar was $20^{\circ}C\pm1^{\circ}C$. The relative air humidity was about 50%.

Symbol of	М	G	M+S	G+S	M+N	G+N
cement						
Method of	Cement	Common	Cement	Common	Cement mixed	Common
preparation	mixed	ground	mixed	ground	together	ground
the cement	together	cement	together	cement		cement
AEA	-	-	synthetic	synthetic	natural	Natural
PCE-1	0.44	0.44	0.86	0.81	0.87	1.20
PCE-2	0.45	0.53	2.00	2.00	2.00	2.06
PCE-3	0.46	0.56	1.61	1.47	1.62	1.33
PCE-4	0.44	0.44	3.00	2.66	2.74	2.56
PCP-1	0.46	0.45	1.25	1.45	1.25	0.97
PCP-2	0.44	0.46	1.70	1.65	1.63	1.62
MN	0.32	0.44	0.62	0.62	0.62	0.44
PC	0.44	0.44	1.08	0.90	1.00	0.90
CLAP	0.46	0.46	1.63	1.02	1.64	1.04
AAP	0.46	0.45	3.08	3.10	3.11	3.05
SNF-1	0.96	1.72	2.15	2.19	2.19	2.44
SNF-2	0,92	1.80	1.87	2.26	1.90	2.34
SMF	1.62	1.69	3.15	3.26	3.45	3.44
MLG-1	1.60	1.72	4.44	4.24	4.08	4.27
MLG-2	1.63	1.64	4.51	4.27	4.71	4.22

Table 4 The amounts of admixtures used in research; % mass of CEM II/B-V

4. Test results and discussion

Surface-active agents can be classified according to the type of polar (hydrophobic) group in their molecules: anionic agents, cationic agents, amphoteric agents (Cullum D.C., 1994). There are three groups of AEAs: wood-derived products, vegetables acids, and synthetic detergents. Air entraining agents (AEAs), which can be based on natural resins (for example vinsol) or synthetic surfactants, were added to the concrete mix to enhance the controlled quantity of air in the form of microscopic bubble in cement paste. From the first usage of air-entertainers until now, many various types of air-entertainers have been established. However, disregarding of what kind of air-entertainers, they all have similar properties or functions, that is, all of them are influential surfactants. Research results presented in publication (Kqlaots I., et al., 2004) show that the natural admixture aeration is more powerful than synthetic one because the natural admixture participation is much smaller than the synthetic in cement. Commercially available air-entraining agents are generally manufactured from chemically complex raw materials, and the final products may consist of blends of these raw materials plus other raw materials or chemicals, hence it is challenging to define air-entraining agents chemically except by rather broad classification. Wood-derived products and synthetic detergent, two types of most frequently used airentraining agents are described here. The aim of using AEAs is to get more balanced and uniform air bubbles with small sizes homogeneously distributed in the cement paste. Synthetic detergents allow for quick production of air bubbles in concrete, these bubbles tend to be coarser than those produced using wood-derived materials. While their primary application has been for foaming agents, some are also used as air-entraining agents. Generally speaking, the synthetic ones produce air quicker than the organic ones; yet, the organic ones have better compatibility with other admixtures than the synthetic ones (Whiting D.A., et al., 1998). The synthetic detergents have been blended with water-reducing agents to generate water-reducing/air-entraining agents. The synthetic agents were more active in lowering the surface tension of cement filtrate than their vinsol resin counterparts. The synthetic agents decrease while surface tension plays an important role in production of all sizes of bubbles, other factors may be responsible for formation of large bubbles and prevent formation of smaller bubbles in synthetic admixtures. More comprehensive research is needed to thoroughly understand this phenomenon.

Figures 1 and 2 show the results of measurements of the air content in mortars. These results show that some of the new generations of superplasticizers significantly increase the volume of air in the previously air-entrained cement based mortar. This is probably perhaps the particles of slag are finer than cement. Replacing cement with SCMs is kind of improving the fineness of the cementitious materials which stabilizes the air bubbles better. The SCM particles usually have higher surface areas than Portland cement grains.



Figure 1 Comparison of air content in the non-air-entrained mortar containing superplasticizers according to the type of cement and admixtures.



Figure 2 Comparison of air content in the air-entrained mortar containing superplasticizers according to the type of cement and admixtures.

Especially the latest generation admixtures significantly increase air content. It happens that the air content increases three times compared to previously aerated mortar (Figure 2). Many inorganic electrolytes and polar organic materials effect the foaming ability of surfactants. The impact of other chemical admixtures for air entrainment is complex. Generally, most organic chemical admixture can enhance the air entrainment. The test results showed in 错误! 未找到引用源。 show that the superplasticizer can reduce the surface tension of water in a similar way as admixture (AEA).





The results of tests (Kobayashi M., et al. 1981) indicated that the different types of high-range waterreducing admixtures influence surface tension, foaming, and stability of air bubbles in a different way. Some types of superplasticizers effect on the surface tension of the liquid phase of the cement paste. The presence of functional groups (oxygen in form of etheric group (-O-), hydroxyl group (-OH) and carboxyl group) produce water surface tension decrease, producing flocculation of associated molecules and increase in moisture of not only grains of cement but also the whole mineral framework (Kucharska L., 2000). The research (Reknes K. and Peterson B.G., 2003) results show that the surface tension changed considerably with time depending on the combination of powder and superplasticizer. The change seems to be caused by the sorption, which includes chemical adsorption, physical adsorption and absorption. Among three kinds of sorption, the absorption of superplasticizer by powder obstructs the function of superplasticizer. The tendency was indicated that the absorption could occur in paste according to fluidity test of paste.

The addition of a superplasticizer (SP) can destabilize the air-void system, but this effect is immensely fluctuating and is especially related to the cement and the air-entraining agent used. The results show

that melamine and naphthalene-based superplasticizers can form essential discrepancies in the air content versus the spacing factor relationship. It is concluded that concrete producers must be very careful when using SPs in air-entrained concrete because significant air-void system destabilization can occur without any significant air content variation. In-plant testing is highly recommended to verify the effect of a given combination of cement-air-entraining agent-SP (Saucier F., et al., 1990).

Experimental results (Baalbaki M. and Aitcin P.C., 1994) performed on 12 various combinations of admixtures with a Type 10 (ASTM Type I) Portland cement show that the addition of superplasticizer practically always enhanced the air content without changing the bubble spacing. The only case in which the air bubble spacing was somewhat altered was when the air content of the concrete was lower than 4.5 percent 70 min after batching. In this case, the total air content decreased after the introduction of the superplasticizer, while the spacing factor increased notably. Second Type 10 cement was used to duplicate these results. No significant change was found between the results of the two sets of experiments.

Results (Plante P.K., et al., 1989) demonstrate that superplasticizers can create a significant boost of the value of the spacing factor. The impact of superplasticizers was found to differ significantly with the characteristics of the cement and also with the type of air-entraining admixture.

In the SPs group there are ones that show only dispersion functioning not decreasing surface tension (Ley M.T., et al., 2009). They are: hydrocarboxylen acid salts, sulphonic melamine-formaldehygenic resins, formaldehygenic picodensats salts of beta-naphtalensulphonic acid.

Analysis of the results listed in Figure 2 mixture made of participation of the innovative, air-entraining multi-component cement, first second generation superplasticizers based on modified naphthalene, and then modified phosphoramidate should be used. Action of admixtures mentioned in the second order involves only the steric dispersion - and so the "natural" blockade of polymer without using deflocculating electrostatic phenomena. This results in compatibility with cements that have extremely different properties and compositions, including air-entraining cements. The analyzed results show that the superplasticizers based on polycarboxylate, polycarboxylic ether and acrylate cause a significant increase in the air-content of the air-entrained mortars. In certain cases of cement mortars and less dosage of SP, up to three times.

Many research reports show that high-range water reducers can make some loss of the air content during occasional agitation (Johnston C.D., 1994), (MacInnis C. and Racic D., 1986). The effects of research analyzed in the publication (Sakai E., et al., 2006) suggests that the type SP is also important because of the size and proportions of air pores in air-entrained concrete. This is perhaps attributed to the generation of greater large-size bubbles, which could quickly disappear with time. Also, the large dosage of high-range water reducer could induce extra fluidity and segregation, and thus may cause some loss of entrained air. Higher dosages of SP can drive some of the entrained air-voids to coalesce resulting in enhanced spacing factor for a given air content (Pigeon M., et al., 1989), (MacInnis C. and Racic D., 1986). Therefore, the air-void system in hardened concrete could be influenced, and the freeze-thaw durability would be decreased (Safiuddin Md., et al. 2006).

Superplasticizers exclude large air voids visible under the microscope but do not greatly interfere with the action of air-entraining admixtures in the creation of small pores. The paper (MacInnis C. and Racic D., 1986) deals with the effect of two different superplasticizers (Mighty and Melment) on the air-void system parameters generated by two various air-entraining agents (NVR and Darex), when the superplasticizer is added about 40 minutes after the concrete has been batched and mixed. A notable drop in air content was noticed with corresponding reductions in total air-void surface areas and increases in air-void spacing factors.

As presented by the research (Mather B., 1979) the addition of high range water reducers to air entrained concrete enhances the spacing factor and reduces the specific surface area of the air void system. Yet, present study's results suggest that the effects of synthetic agents on the air void system are independent of the effects of high range water reducers.

Tests (Gorzelańczyk T. and Hoła J., 2011) have shown that the pore structure evidently depends on the superplasticizer added to the concrete mix. When superplasticizer, based on a combination of polycarboxylans and viscosity, bonding and hardening regulators, is added to concrete mix, the structure of the achieved concrete is characterized by more beneficial parameters than those characterizing the structure of the concrete made from the concrete mix to which superplasticizer based on polycarboxylic ether was added.

The results show that the distribution of the air-void diameters is effected by the nature of the airentraining agent but not by the usage of a superplasticizer (Pleau R. et al., 1990). Hence, clashing information on the freeze-thaw resistance of superplasticized concrete is given in the literature. For this reason, the Research Centre of the German Cement Industry instigated a broad research program. Superplasticizers based on melamine and naphthalene sulfonates were used in the main program and lignosulfate-based products in a subsidiary program. Some of these consisted of de-airing agents. The air-void distribution and the spacing factor were measured as well as the freeze-thaw resistance with deicing chemicals. When superplasticizers were used in a high-workability air-entrained concrete, the amount of pores with a diameter up to 300 µm declined, while the content of pores larger than 500 µm and the bubble spacing factor enhanced. Small pores coalesced and created larger pores. Although the air content of the fresh concrete was satisfactory, the superplasticized concrete sometimes had a spacing factor above 0.20 mm. Due to this fact, concrete with superplasticizers did not always have decent freeze-thaw resistance. An effect due to the type of superplasticizer could not be distinguished, while the de-airing agents of the superplasticizers had a noticeable effect. Some further tests with plasticizers and retarders show that these admixtures also alter the air-void distribution or air-entrained concrete.

The analysis results shown in Figure 2 indicates that, as recommended admixture of the second generation in the case of air-entraining cement admixture based on the naphthalene and melamine can be identified. The results (Saucier F., et al., 1990) show that melamine and naphthalene-based superplasticizers can form crucial discrepancies in the air content versus the spacing factor relationship. It is concluded that concrete producers must be very careful when using SPs in air-entrained concrete because serious air-void system destabilization can occur without any significant air content variation. In-plant testing is highly recommended to verify the impact of a particular combination of cement-air-entraining agent-SP, and a general procedure to do it is proposed.

Comparison of the results from Figures 4-6 demonstrates that the third generation SPs based on modified naphthalenes (MN) provide a good workability of the mortar, no worse than SPs on the basis of a polycarboxylate, polycarboxylate ether, acrylate, or a phosphoramidate.



ure 4 Comparison of diameter of the initial flow the non-air-entrained mortar containing superplasticizers according to the type of cement and admixtures. 1 cm = 0.0109 yd.







Figure 6 Comparison of diameter of the final flow the non-air-entrained mortar containing superplasticizers according to the type of cement and admixtures. 1 cm = 0.0109 yd.



Figure 7 Comparison of diameter of the final flow the air-entrained mortar containing superplasticizers according to the type of cement and admixtures. 1 cm = 0.0109 yd.

Serious advances have been made in process, production, and application of LS based admixtures. There is a broad range of lignosulphonates available and the performance in concrete differs from basic water reduction and strong retardation to high range water reduction (Reknes K., 2004), (Jun S.D., 2008). With the progress of a new modified lignosulphonate superplasticizer (PLS), it is possible to produce self-compacting concrete (SCC) with such an admixture. With modified lignosulphonate superplasticizers entering the market, its primaryperformance, including workability, retardation and strength, have been researched. However, there is not much information available in the literature on the effect of thesefreshly developed modified lignosulphonate superplasticizers on cement hydration, workability retention and pore structure, in comparison to those of other types of superplasticizers such as naphthalene and polycarboxylate based admixtures and to those of traditional lignosulphonate water reducing admixtures. Thus, the ongoing research was conducted (Reknes K., 2004). Nevertheless, do not combine lignosulfonates (first generation of admixtures) with some air-entraining admixtures.

Certain producer's admixtures in their commercial offer of admixtures plasticizers are also made based on polycarboxylate ether with the addition of a large amount of antifoaming admixture, especially recommended in prefabrication. However, as shown by the analyzed results, the addition of antiadmixtures not always prevents excessive growth of the pre-air entrainment of previously air-entrained mixture.

Research results summarized in Figures 8 and 9 show the lack of relationship between the consistency and air content of the air-entrained and not air-entrained cement based mixtures. According to the authors, to such a wide diversification of commercial superplasticizers, unfounded is to build a mathematical model that binds air content in the mortar and its propagation.



Figure 8 Relation between flow diameter and air content in air-entrained mortar. 1 cm = 0.0109 yd.



Figure 9 Relation between flow diameter and air content in non-air-entrained mortar. 1 cm = 0.0109 yd.

To finish it can be added, it is impossible to speculate the effects of admixtures interactions with surfactants on air entrainment. The compatibility of the admixtures should be experimentally tested if the effects of such combinations are unknown in advance. Most organic chemical admixtures like superplasticizer can enhance the air entrainment since it can slightly reduce the absorbed AEA molecules on the solid surface by competing with them. Additionally, macromolecular materials may help stabilize the dispersed air bubbles. Moreover, some high-range water reducers themselves may have the air-entraining potential. However, the adding of straight calcium chloride may tend to limit the number of entrained air due to the precipitation of surfactants in the solution by creation of insoluble salts (Lianxiang D., Folliard K.J., 2005). Other admixtures like retards, accelerators, etc., have negligible effect on the air entrainment. Yet, today there are many types of AEAs like wood-derived acid salts AEA, vegetable oil acids AEA and synthetic detergents AEA, which may react with chemical admixtures. This adds the difficulty on the study of the impact of chemical admixtures on the air entrainment. The compatibility of the admixtures should be experimentally tested if the effects of such combinations are unknown in advance. In view of the obtained results of the research it can be concluded that the compatibility of admixtures SP and the AEA and cement can be checked only when they occur together. Checking each individual admixture does not take into account their interaction.

5. Conclusions

Within the scope of the research and examined admixtures, it was found that in order to increase the degree of fluidity of previously aerated concrete, made with the participation of innovative air-entraining CEM II/B-V, should be:

- For each air-entraining cement the choice of amount and type of plasticizer or superplasticizer, due to the required aeration and consistency of mixture, can be successfully carried out only on the basis of experimental comparison. The condition of compatibility of entraining and superplasticizing admixtures with cement should be verified taking into account their mutual influence of both the consistency and the content of the air in the mixture. It is important to verify their interactions and possible consequences for air-entrainment and mortar consistency, as well as concrete.
- The recommended admixtures in case of the air-entraining CEM II B-V are second generation admixture based on the naphthalene and melamine. In case of a significant increase in the degree of liquidity of concrete mixture, first second generation superplasticizers based on modified naphthalene, and then modified phosphoramidate should be used. The new generation superplasticizers based on polycarboxylate, polycarboxylic ether and acrylate cause a significant increase in the air-content of the air-entrained mortars. In certain cases of cement mortars and less dosage of SP, almost three times.

Acknowledgements

The present study was funded by the National Centre for Research and Development Project PBS1/A2/4/2012 pt. "Innovative Cement Concrete Aerating".

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