Study of dry mixes with aluminic cements for self-leveling floors

Maria Kaddo and Maria Sinotova

Moscow State University of Civil Engineering, Yaroslavskoe shosse, 26, Moscow, 129337, Russia

E-mail: m.kaddo@yandex.ru

Abstract. In this article the results of the research of structure, shrinkage and physical and mechanical properties of concrete obtained from dry mixes based on aluminic cements for self-leveling floors are presented. The significant part of the cost of finishing works is the cost of mounting of the floors, as the quality of the floors determines not only the aesthetic comfort, but also the functionality of the room. The floors (especially, the floors of industrial, sports and so on buildings) must have the different properties: high strength and abrasion resistance, absolute resistance to cracking, dustless, hygienic etc. The main question of possibility of obtaining non-shrinking aluminic concrete is connected with the creation of ettringite using aluminic cement, gypsum and modern superplasticizers. Studies of free shrinkage of complex binder showed that binder could be classified as binder with compensated shrinkage. Cracking resistance of the composition is provided by the fact that the vast expansion in the initial stage of curing and shrinkage of the final stage is in 5...10 times lower than these values for gypsum and Portland cement. The problem of cracking resistance of thin-film coatings of cement compositions can be solved by binders with controlled shrinkage (expandable binders, whose shrinkage is compensated by the expansion). Resistance to cracking would be higher in the case when expansion and shrinkage are close not only in size, but also in the development time. The composition of dry mix for non-shrinking aluminic concrete consists of aluminic cement, gypsum, polycarboxylate superplasticizer and fractioned fine fillers. For experimental verification of the component composition of binder and the type of gypsum studies were conducted in a wide range of ratio of the components at a fixed value of the plasticity of the mixture. The structure of examined concrete is dense and has compensated shrinkage. Physical and mechanical properties of concrete are high enough and suited to the requirements of Russian construction norms (GOST).

1. Introduction
The expediency of using dry mixes of factory readiness is confirmed by foreign and domestic practice of construction. In Western Europe, the production and use of dry mixes in construction is massive. Consumption of dry mixes per capita in Russia is significantly behind the level of Western European countries. It’s about 67 kg per person per year against 90-95 kg. The volume of construction per capita in Russia is greater than in Europe.

The experience of using dry mixtures showed their high efficiency in comparison with the traditional methods of preparation of mortar mixtures. Productivity is increased 2-5 times. The consumption of basic materials is reduced by 3-10 times (with tile works - up to 7 times, with the device floors - up to 10 times). Preparation of the solution is possible in strictly necessary quantities,
which also economize the materials. Dry mixtures can be stored for a long time and transported without loss of quality. While ensuring the properties of the materials obtained on their basis.

Modern dry mixtures are multicomponent specialized systems in which, in addition to the mineral binder and aggregate, there is a complex of chemical additives. Chemical additives providing the necessary rheological properties of the mixture that regulate the setting and setting speed of the binder and provide the necessary physical and mechanical properties of the solution after solidification.

Areas of application of dry mixtures and the variants of their compositions are very diverse: masonry, installation, repair, floor, decorative and others. To ensure the production of dry mixtures for various purposes with the required properties allows the use of various binders: Portland cement, gypsum and aluminate cement.

Regulation of the rheological properties of mixtures is achieved with the help of superplasticizers, which dilute solution mixtures without increasing the water content in them. Increasing the mobility of the mixture often leads to its stratification and deleting water out of it when applied to a porous base. To prevent this fact, water-retaining (thickening) additives are used: mainly water-soluble cellulose ethers, polyvinyl alcohol etc.

To all additives for dry mixtures, a requirement is required - low hygroscopicity. This is necessary to ensure the preservation of the mixture (preventing premature hardening of the binder due to adsorbed moisture). The most important components of most dry mixtures are polymer additives in the form of dry polymer powders, which capable of redisersion. The addition of polymer dispersions improves the workability of mixtures, increases the strength, deformability and water resistance of solidified solutions. The main goal of introducing polymer additives into solutions is to give them a high adhesive capacity. It means that polymer additives are increasing the adhesion properties.

A special place is occupied by dry mixtures for floors. In this group there are two types: floor leveling mixtures (mixes for screeds) and floor mixes (mixes for floor covering).

The tasks carried out by the screed material are to level the base of the floor under the face cover and transfer the load from the cover to the base. Mixtures are used as a face covering. They should have high strength properties, resistance to wear and low porosity.

The self-leveling mixtures are effective for floor screeds and floor coverings. These mixtures after mixing with water and intensive mixing have the properties of a "liquid body". It happens due to the combined action of the system of plasticizing and water-retaining additives. They can spread under the influence of their own weight, forming a flat horizontal surface. At the same time, they do not observe sedimentation (stratification with discharge of water upwards and settling of aggregate). The use of polymeric modifiers provides high wear resistance of the floor. In mixtures for screeds Portland cement and gypsum as binders were used. For facings Portland cement or alumina cement were used. Non-shrinkage dry mixtures are very effective.

From this point of view, self-leveling floors on the base of aluminate cement are the most interesting ones. The production of aluminate cements causes less damage to the environment. Moreover, the use of aluminate cements eliminates losses caused by corrosion of cement stone. Corrosion is probable after a certain period of service in the underground and basement technical rooms of high-rise buildings [2-9].

Shrinkage can be of two kinds when concrete is curing, contractual and humid. The contraction almost has no effect on the volume of construction, and leads to an increase in porosity of the concrete. Humid shrinkage is associated with the evaporation of water from the curing concrete and exceeds the contraction in 5-10 times [10, 11].

For shrinkage compensated concrete can be used organic expanding components in an amount of 0.5-2% [12-15]. More widespread has expanding mineral supplements, the application of which produce products of hydration and cause the expansion of the system and increase the strength of the cement stone. Using the expanding additive requires high production standards. Expansion effect depends on many factors and is not stable [16-18].

The most promising method is the use of binders based on aluminate cements, which expansion is provided by the formation of ettringite 3CaO·Al₂O₃·3CaSO₄·31·32H₂O (CASH) [19].
2. Materials and methods

For the experiment were analyzed compositions and properties of the produced aluminate cements. The data on the chemical composition of aluminate cements are given in Table 1.

### Table 1. The chemical composition of aluminate concretes, (%)

| Composition       | Al₂O₃ | CaO  | Fe₂O₃ | SiO₂  | SO₃  | R₂O  |
|-------------------|-------|------|-------|-------|------|------|
| LUMNITE (Germany) | 38 - 42 | 36-40 | 13-17 | 1 (max) | 0.4  | MgO < 1.5 |
| ISTRA 40 (Germany)| 39 - 42 | 37 - 40 | 14 - 17 | 2 - 5 | 0.4  | MgO 1.2 |
| REFCON (Germany)  | 50-53  | no less than 40 | no more than 3 | 6 | no more than 0.4 | MgO < 1.5 |
| SECAR 51 (France) | 50.8 - 54.2 | 35.9 - 38.9 | 1.5 - 2.5 | 4.0 - 5.5 | - | MgO < 1.0, TiO₂ < 4.0, K₂O + Na₂O < 0.5 |
| CRHAC -70-1 (Russia) | no less than 70 | no less than 20 | no more than 1 | no more than 2 | no more than 0.15 | no more than 0.5 |
| CRHAC -75-0.5 (Russia) | no less than 75 | no less than 20 | no more than 0.5 | no more than 1.3 | no more than 0.15 | no more than 0.35 |

Corrosion resistant high alumina cement (CRHAC), because it contains at least 70% Al₂O₃ was selected for study. The chemical composition of used cement: Al₂O₃ – 70.5%, CaO – 28.1%, SiO₂ – 0.87%, Fe₂O₃ – 0.3%. The mineral composition of used cement: calcium aluminate (CA and CA₂) in the ratio 1:1. As a result of x-ray curing products principally consist of CAH₁₀. In the age of 3 days hydration degree of CA is about 80-85%, CA₂ - about 50%. Selected cement is characterized by high strength. Curing speed is less high compared with other aluminate cements, that corresponds to the task (Table 2). As a second component of binder which necessary for expansion, natural gypsum (CaSO₄·2H₂O) and hemihydrate gypsum (CaSO₄·0,5H₂O) were used.

### Table 2. Technical characteristics of the material

| Value                        | LUMNITE | ISTRA 40 | REFCON | SECAR 51 | CRHAC -70-1 | CRHAC -75-0.5 |
|------------------------------|---------|----------|--------|----------|--------------|---------------|
| Specific surface area, m²/kg | 310-370 | 300 - 340 | 330-380 | 375 - 425 | no less than 450 | no less than 450 |
| Curing time: the beginning -- the end, hour | not earlier than 2, not later than 5 | not earlier than 1, not later than 5 | not earlier than 4 not later than 7 | not earlier than 2, not later than 12 | not earlier than 2, not later than 12 |
Compressive resistance, MPa after a day of curing 35 after a day of curing 65-75 after a day of curing 45 after a day of curing 55-85 after 3 days of curing no less than 50 after 3 days of curing no less than 50

3. Results and discussion

For experimental verification of the component composition of binder and the type of gypsum were conducted studies in a wide range of ratio of the components at a fixed value of the plasticity of the mixture (Table 3).

Table 3. Test results of two-component binders of different composition

| CRHAC, % | 100 | 80 | 70 | 60 | 50 | 40 | 0 |
|----------|-----|----|----|----|----|----|---|
| gypsum, % | 0   | 20 | 30 | 40 | 50 | 60 | 100 |
| CaSO$_4$·2H$_2$O | | | | | | | |
| W/C | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | - | - |
| compression strength, MPa | 54.5 | 50.2 | 53.1 | 48.4 | 34.6 | - | - |
| CaSO$_4$·0,5H$_2$O | | | | | | | |
| W/C | 0.50 | 0.52 | 0.53 | 0.53 | 0.54 | 0.55 | 0.55 |
| compression strength, MPa | 54.5 | 44.3 | 46.5 | 40.5 | 33.0 | 26.8 | 14.7 |

Composition contents 60 ... 80% of CRHAC (the amount of gypsum 40 ... 20%) have a high compression strength. In terms of strength and economic reasons more efficient is to use of natural gypsum.

Composition was fixed after 3, 7, 24 and 72 hours. In first hours of hydration was observed rapid formation of CASH, decaying after 24 hours. Quality of gypsum in curing system decreases in the same regularity (Figure 1).

![Figure 1. Relative intensity of peaks over hydration time, %](image-url)
To study the shrinkage mechanism of curing binders and to evaluate the role of evaporation of water in the development of shrinkage was undertaken experiment with simple unfilled systems: gypsum binder, Portland cement and «CRHAC + gypsum» (70% + 30%). Selection of the first two systems was defined as sufficient of their study, and the fact that in the first is observed a rapid formation of a solid skeleton formed of crystalline neoplasms (CaSO$_4$·2H$_2$O), and in the second - a solid skeleton is formed of several phases, a large share of which is occupied by phase of gel. So, on the example of these two systems are easier to specify the new features of binder deformation «CRHAC + gypsum» (Figure 2).

Deformation of curing two-component (CRHAC + gypsum = 70 : 30) binder developed as a result of the superposition of two competing processes: the expansion due to the formation of CASH and formation on its basis of the cement stone structure and shrinkage caused by the evaporation of water from the curing composition.

As in the initial period is very active the formation of cement stone structure, we observe the rapid expansion, reaching after 4...5 hours – 0.6 mm/m.

As a result, the deceleration rate of hydration, sealing, but not the extension of curing system due to hydration products (indicated by a rapid increase of the strength of the composition after 4...5 hours after mixing) and the evaporation of water from the cured material, we observe a sharp decline in the rate of expansion (90 µm/m·h to 0), and then the resulting transition effect process after 5...6 hours from expansion to shrinkage. Although at this time, continues the process of expansion by hydration of the remaining share of CA and, in particular, CA$_2$. After 3...4 days the rate of shrinkage is stabilized (1...2µm/m·h) and the initial expansion is compensated by shrinkage. The shrinkage of the composition is terminated after 10...12 days at a value of shrinkage of 0.8...0.9 mm/m. (Table 4).
Table 4. Deformation speed of the two-component binder (CRHAC:gypsum = 70:30; W/C = 0.6; start time of measurement - 1.75 hours after mixing)

| Time, hour | Deformation speed, µm/m · h | Time, hour | Deformation speed, µm/m · h |
|-----------|-----------------------------|-----------|-----------------------------|
| 0.25      | -                           | 136       | 1.2                         |
| 0.5       | 240                         | 150       | 1.5                         |
| 2.25      | 90                          | 161       | 1.1                         |
| 3.5       | 2.4                         | 174       | 2.7                         |
| 4.75      | 3.2                         | 198       | 2.1                         |
| 6.25      | 1.0                         | 222       | 0.4                         |
| 7.75      | 0.3                         | 246       | 0.3                         |
| 16        | 7.9                         | 280       | 1.2                         |
| 23        | 17.3                        | 295       | 1.1                         |
| 42        | 2.1                         | 336       | 1.0                         |
| 48        | 0.33                        | 352       | 3.5                         |
| 54        | 0.66                        | 419       | 0                           |
| 63        | 0.11                        | 540       | 2.3                         |
| 89        | 6.3                         | 565       | 0                           |
| 114       | 1.76                        | 590       | 0.02                        |
| 126       | 1.5                         | 725       | 0.01                        |

Using aluminate cement as a main component for dry mixtures for self-leveling floors requires an adequate selection of the superplasticizer.

Commercially available additives for use with aluminate cement to produce dry mixtures for floors were analyzed. Supplements of three main groups were selected for study: polymeric sulfomelamine (Peramin SMF20 Perstorp Construction Chemicals Inc., Sweden) modified with polyethylene glycol (Melflux PP100F, Melflux PP200F, SKV Polymers GmbH, Germany) and polycarboxylate (Sika Viscocrete 105P, Swiss). Evaluating of the effectiveness of the superplasticizer joint venture was carried out in accordance with GOST 30459-2008 “Admixtures for concretes and mortars. Determination and estimate of efficiency” (EN 934-6:2002 Admixtures for concrete, mortars and grout – Part 6 Sampling, conformity control and evaluation of conformity). All the properties were determined on the samples with dimensions of 4x4x16 cm according to ASTM and GOST for the tests. Water-reducing effect is 57...63%. The study works were done by the equipment of MSUCE.

It is known that using aluminate cements mixtures with superplasticizers a rapidly lose flowability. Due to this, studies for saving flowability of mixture after 20, 40 and 60 minutes after mixing were made. Additives were introduced in the amounts recommended by the manufacturer. Saving of flowability (decrease no more than 10% in 60 minutes) was seen only for superplasticizers based on polycarboxylate.
The two-component composition based on a two-component binder (CRHAC + gypsum), filled with quartz sand (Binder:Sand = 1:1.5) and modified by superplasticizers based on polycarboxylates (Sika Visocrete 105P, Swiss) characterized by the following technological and operational properties:
- saving of self-leveling for 30…60 minutes after mixing;
- setting time: beginning - 2 h 00 m ... 2 h 20 m, end - 3 h 00 m ... 3 h 15 m;
- strength of composition: compressive strength after 7 hours - 5 ... 6 MPa, after 1 day. - 23 ... 25 MPa, after 3 days. - 42 ... 44 MPa;
- flexural strength after 3 days. - 11...12 MPa;
- average density - 1750 ... 1850 kg/m³.

4. Conclusions
During the construction of buildings, it is possible and rational to use binders which are alternative to Portland cement.

For construction of floors for technical premises is expediently to use mixes based on aluminate cements.

The production of aluminate cements causes less damage to the environment. Moreover, the use of aluminate cements eliminates losses caused by corrosion of cement stone.

Producing dry mixtures for self-leveling floors became possible after the creation of modern superplasticizers, which provide large shear strength. Analysis of the results shows that for dry mixtures (with the use of Portland cement) modern superplasticizers based on polycarboxylates work effectively. At the same time, there is no consensus about the factors that have influence on the compatibility of superplasticizers with aluminate cement, gypsum and mixtures of these materials.

References
[1] Kyliili A and Fokaides P A 2017 Sust. Cit. Soc. 35 280
[2] Klansek U and. Kravanja S 2006 Int. Jour. of Conc. Str. and Mat. 62 434
[3] Zajac M, Skocek J, Bullerjahn F and Haha M B 2016 Cem. and Concr. Res. 84 62
[4] Winnefeld W and Lothenbach B 2010 Cem. and Concr. Res. 40 1239
[5] Garcia-Mate M, Santacruz I, De la Torre A G, Leon-Reina L and Miguel A G 2012 Cem. and Concr. Comp. 34 684
[6] Pelletier-Chaignat L, Winnefeld F, Lothenbach B, LeSaout G, Muller C J and Famy C 2011 Cem. and Concr. Comp. 33 551
[7] Glasser F P and Zhang L 2001 Cem. and Concr. Res. 31 1881
[8] Pera J and Ambroise J 2004 Cem. and Concr. Res. 34 671
[9] Zhou Q, Milestone N B and Hayes M 2006 Jour. of Heal. Man. 136 120
[10] Zhang T, Gao P, Luo R, Guo Y, Wei J and Yu Q 2013 Constr. and Build. Mater. 662
[11] Bernardo G, Telesca A and Valenti G L 2006 Cem. and Concr. Res. 36 1042
[12] Le-Bihan T, Georgin J F, Michel M, Ambroise J and Morestin F 2012 Cem. and Concr. Res. 42 1055
[13] Collepardi M, Borsoi A, Collepardi S, Olagot J J and Troli R 2005 Cem. and Concr. Comp. 27 704
[14] Rajabipour F, Sant G and Weiss J 2008 Cem. and Concr. Res. 38 606
[15] Rongbing B and Jian S 2005 Cem. and Concr. Res. 35 445
[16] Cao Q and Zhongguo John Ma 2015 Constr. and Build. Mater. 75 450
[17] Sisomphon K, Copuroglu O and Koenders E A B 2013 Constr. and Build. Mater. 42 217
[18] Maltese C, Pistolesi C, Lolli A, Bravo A, Cerulli T and Salvioni D 2005 Cem. and Concr. Res. 35 2244
[19] Popov K and Kaddo M 2006 Constr. Comp. Mat. 2 20