

POWDER ADDITIVES FOR DRY MIX MORTARS WITH RISING DEMANDS

The requirements for mineral based mortars have been steadily increasing in the last decades. Without additives it is hardly possible to cope with these technical challenges. The content of additives in dry mixes normally ranges between 0.1 and 10%. Nevertheless these additives do have a crucial influence on the properties of the mortar. There are several kinds of additives used in dry mix mortars, e.g. air-entraining agents, accelerators, retarders, superplasticizers and rheology modifiers. In this paper three other product groups are discussed more in detail: shrinkage reducing agents, powder defoamers and powder dispersants.

SHRINKAGE REDUCING AGENTS

Excessive shrinkage of cement and the resulting crack formation is one of the most severe problems in the field of cement based construction materials. The cracks do have negative influence on several properties of the applied material, such as visual appearance, usability and durability.

Theory of Shrinkage

Shrinkage is defined as the load independent volume reduction during the drying process of hardened cement paste. This effect is caused by a reduction of the moisture content of the hardened cement paste. There are four different types of shrinkage, depending on the time and reason of appearance[1].

The early shrinkage (plastic shrinkage, capillary shrinkage) takes place in the plastic phase from the beginning of the hydration until the start of the solidification process. It results from capillary forces arising from the withdrawal of water from the fresh mortar. The reason for this is e.g. evaporation of water from the mortar or water absorption of the aggregates. Depending on the formulation the early shrinkage varies in value but the resulting cracks are quite large.

The early shrinkage is the only type of shrinkage that can be reduced by timely curing or decelerated hydration. A reduction can be achieved by covering the fresh mortar with plastic foil, spray with water, curing additives or paraffin based dispersions. The chemical or autogenous shrinkage takes place in the first days of the hardening process. It is based on the fact that the volume of the cement gel (hydrate phase) is smaller than the combined volume of mixing water and cement. In the case of complete hydration the volume of the hardened cement paste (water/cement ratio = 0.4) is about 92% of the volume of the hydrate phase. In the hydration process the amount of free flowing water is reduced and the cement is using water from the capillary pores. This leads to self-desiccation of the pores with the chemical fixation of water in the cement gel. For this reasons the process of autogenous shrinkage depends on the w/c value. Especially low w/c values promote autogenous shrinkage. Drying shrinkage is to be understood as the loss of free, chemically uncombined water from the hardened cement paste. The hardened cement paste yields water until it is balanced to the ambient moisture.

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Table 1. Guide formulation for a cement based self-leveling compound.

Component	Function	Weight %
Silica sand (0.1-0.5 mm)	Filler	41.24
Portland cement (42.5)	Binder	35.00
Limestone (0.05 mm)	Fine filler	10.00
High alumina cement	Binder	6.00
Anhydrite	Binder / shrinkage compensation	4.00
Redispersible polymer powder	Flexibility / adhesion	3.00
Plasticizer		0.40
Defoamer	Air and foam control	0.10
Na-citrate / Na-tartrate	Retarder	0.10
Li-carbonate	Accelerator	0.10
Cellulose ether	Rheology	0.06

This process is reversible and diffusion controlled and for this reason very slow. It depends on the ambient moisture, the composition and the dimension of the surface. Like the autogenous shrinkage the mechanism of drying shrinkage is based on the fact that water is dispensed from the capillary pores. In the case of autogenous shrinkage this process is caused by a physicochemical process (hydration) and not by a simple evaporation to the ambience. Another slow shrinkage process is the so-called carbonation shrinkage. This irreversible process is caused by a chemical reaction between the ambient atmosphere (carbon dioxide) and the calcium hydroxide of the hardened cement paste. In this reaction calcium carbonate and water is formed. The water is dispensed from the hardened cement paste to balance to the

ambient moisture. The carbonation shrinkage is a very slow process that lasts from months to decades and the resulting shrinkage effect is quite small.



Figure 1: Length determination of the specimen with an electronic dial gauge

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Composition and Mechanism of Action

To understand the mechanism of action of shrinkage reducing agents it makes sense to take a closer look on the mechanism of cement hydration and hardening. The literature discusses several models for this process. In the context of this article it is useful to focus on one model that is feasible to explain the process of shrinkage reduction. The classical assumption is that cement and water form a network of colloidal hydration products that is called cement paste or gel[2]. This paste mainly consists of calcium silicate hydrates, where water is bound in different modes. On one hand as chemically bound water of crystallization in the calcium silicate hydrates, and on the other hand adsorbed to the gel particles or finally as free flowing capillary water. In the course of the hydration process the distance between the gel particles is getting smaller and the layers of adsorbed water are no longer developed properly and water condenses in these areas. This so called capillary pore water creates pressure onto the gel particles. This pressure deforms the gel particle and the pore structure is enlarged. In the end this process is the reason that leads to shrinkage of the cement paste in the ongoing desiccation. There are several options to reduce shrinkage in the hardened cement paste. This can for example be achieved by a volume expansion in the early phase of the hydration by adding sulphate (gypsum)[3, 4] or formation of hydrogen cavities (addition of aluminum powder). As mentioned earlier the evaporation of water from the fresh mortar can be reduced by covering with plastic foil, sprinkling with water or paraffin based dispersions. Especially in the field of dry mix mortars with high quality demands

powder shrinkage reducing agents are used often, e.g. in systems with large surfaces such as self-leveling compounds, anchoring mortars and also repair mortars. Shrinkage reducing agents contain surface active components to reduce the surface tension of water in capillary pores. Experience has shown that a lot of nonionic surfactants with distinct hydrophobic character the conventional used substances have a big drawback. They are not VOC-free. Because of a constantly growing demand of the markets, low emission systems will become more important (e.g. EMICODE). For this reason modern shrinkage reducing agents are formulated VOC-free to meet these requirements. For easy handling the active ingredients are applied onto specific carriers with high adsorption and fast desorption capacity.



Figure 2:
Shrinkage reduction in a self-leveling compound by using a shrinkage reducing agent (SRA)

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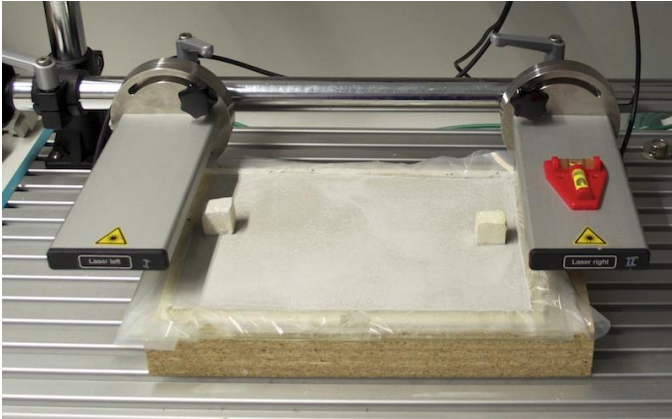


Figure 3: Monitoring of early shrinkage using laser distance measurement

Different mechanisms of action are discussed for shrinkage reducing agents. On one hand hardly soluble calcium hydroxide, which is generated by hydration of the cement, is complexed by the active ingredients of the shrinkage reducing agent and kept in solution. Therefore the hydration process is decelerated. On the other hand because of their surface active properties the ingredients are reducing the water loss from the capillary pores which leads to a decreased capillary tensile stress. These processes lead to larger distances between the cement paste particles and as a result the autogenous and drying shrinkage is reduced[5].

Experimental and Results

To show the mode of action of shrinkage reducing agents a guide formulation for a self-leveling compound is used (see Table 1). The mixing is done by using a Hobart mixer with a short mixing time of 1-2 minutes to simulate the processing at the construction site. The shrinkage is measured using DIN

52450. To do so three standardized specimens are produced in a special casting mould.

After 48 hours the specimens are removed from the mould and the length is measured (see Figure 1). The specimens are stored for three months under standardized conditions (23°C and 50% rel. air humidity) and their length is determined in periodical intervals. The value of shrinkage is given in millimeters per meter (mm/m) in relation to the reference value. Early shrinkage cannot be observed with this method because the reference value is measured after drying of the specimen. As shown in Figure 2 shrinkage is reduced by over 20% after 2 months. The early shrinkage can be detected by using laser distance measurement. The self-leveling compound is applied to a mould build from plastic foil and window sealing tape. The base is a leveled glass plate. Two polystyrene based reflectors are positioned on the self-leveling compound and adjusted perpendicular to the laser beam. The change in length during the hardening process is recorded. At the end of the test the final length is measured using a precision sliding caliper and calculated back to the starting distance (see Figure 3).

The length reduction is calculated in mm/m. The result of the experiment is shown in Figure 4. Straight after the application the self-leveling compound expands excessively. This effect changes into a massive shrinkage after 4-5 hours. By using a shrinkage reducing agent both effects are reduced significantly.

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Powder Defoamer

Powder defoamers are used in a lot of dry mix mortar formulations. These are for example systems based on cement, gypsum, limestone and redispersible polymer powders (leveling compounds, screeds, tile adhesives, joint fillers, powder paints, plasters, repair mortars).

Function and Composition

Powder defoamers are used to reduce and control the air content in wet mortars (see Figure 5). In general this leads to an increased stability of the mortar. Not in every case low air content is preferred. Tile adhesives are formulated with higher air content to achieve a better workability and accurate adjustment of tiles. Also in lightweight screeds higher air content is important to save weight. Powder defoamers consist of a liquid phase which is applied on solid carriers with high adsorption and fast desorption capabilities. Powder defoamers should provide free-flowing properties and a low tendency to form lumps during storage. The liquid phase of the defoamer consists of compounds with influence on the surface tension of the wet mortar, e.g. hydrocarbons, polyglycols or polyethersiloxanes. The wetting properties of the liquid components are crucial to achieve a homogeneous and bubble-free surface of the mortar. More hydrophobic formulations provide more defoaming effectivity, but tend to generate surface defects like stains and pinholes (Figure 6). Sometimes interactions between defoamers and plasticizers could be observed which also lead to an inhomogeneous visual appearance. Therefore it requires a skilled selection of the defoamer composition for a given dry mix

mortar. Often intensive empiric defoamer screenings are needed to achieve the best compromise between defoaming power and visual appearance of the applied material.

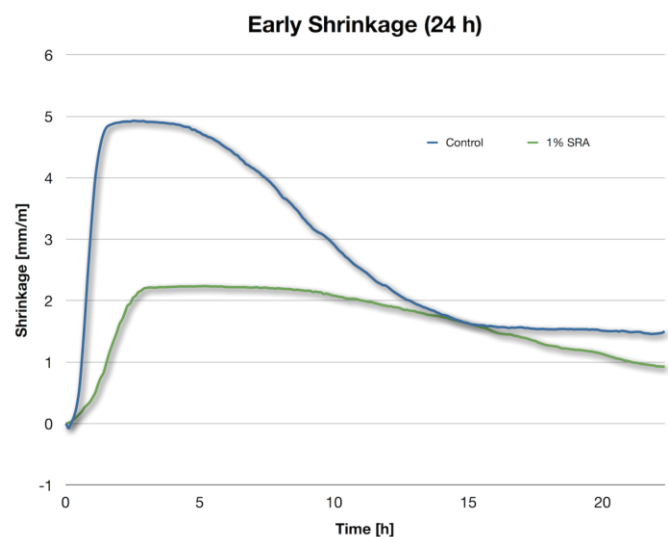


Figure 4: Reduction of early shrinkage in a self-leveling compound

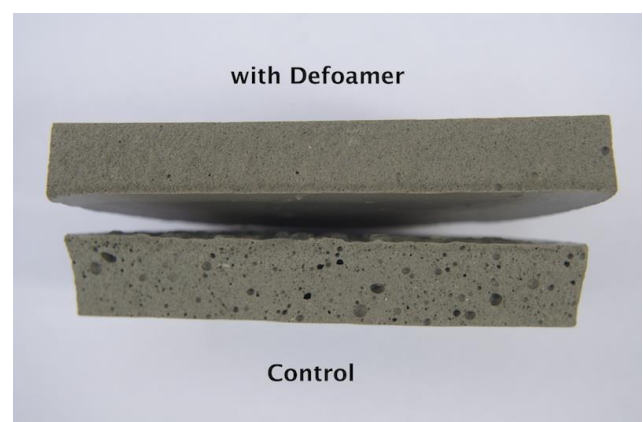


Figure 5: Reduction of air content with a powder defoamer

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		Without defoamer	Defoamer 1	Defoamer 2	Defoamer 3	
Wet density [g/cm³]		1.863	1.915	1.975	1.982	
Dry density [g/cm³]		1.645	1.716	1.785	1.786	
Flow rate [cm]		24.5	25.5	25.5	26.0	
Specimen	Appearance	Grey, no spots	Grey, few spots	Grey, some spots	Grey, few spots	
	Air	Surface	1	6	2	8
		Inside	1	1	9	9
		Bottom	1	1	6	8

Table 2: Defoaming properties in a self-leveling compound

Experimental and Results

To illustrate the effect of powder defoamers a guide formulation for a self-leveling compound, containing 0.3% defoamer is blended in a Scandex mixer. The compound is then mixed with water by using a kneading machine for 30 seconds. As mentioned earlier short mixing times and low shear rates simulate the processing at the construction site. To check the influence of the defoamer on the properties of the self-leveling compound different parameters are measured. The flow rate is obtained with a Hagermann table and the wet density by using a pycnometer. The dry density is measured by producing a specimen that is coated with a protective lacquer after drying. The density of the specimen is obtained by weighing in water and air. The optical appearance is judged with the help of a petridish casting. The results are shown in Table 2. In this case defoamer 3 shows the best results.

Powder Dispersants

Powder dispersants can be used to accelerate and improve wetting of hydrophobic components in mineral mortars during mixing with water. These hydrophobic components can be for example fibers, pigments or silica sand. For this reason powder dispersants are mainly used in colored joint fillers, fiber-reinforced mortars and machine mixed mortars.



Figure 6: Optical impairment due to wrong defoamer selection

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	Density [g/cm ³]	Colour strength		L- value
		abso- lute	[%]	
W/o disper- sant	1.778	10.32	100	40.59
Disper- sant 1	1.808	13.32	129.1	36.95
Disper- sant 2	1.759	15.03	146.6	35.18
Disper- sant 3	1.795	7.16	69.4	46.11

Table 2. Physical properties of the analyzed joint filler.

Powder dispersants increase colour strength in pigmented systems, reduce floating of pigments to the surface and attain a homogeneous surface (see Figure 7). The wetting of reinforcement-fibers is improved and floating to the surface can be suppressed. This leads to a higher stability of the mortar. In machine mixed mortars the mixing time is reduced and therefore higher delivery rates can be achieved. In some cases combining powder defoamers and dispersants generate synergetic effects that result in more smooth and homogeneous surfaces (see also Figure 7). The surfactant molecules are wetting the surface of fibers, pigments and aggregates. One can distinguish two different mechanisms of action: electrostatic and steric stabilization. In the case of electrostatic stabilization the surface of the particle is covered with ionic additives. If all particles are charged identical, electrostatic repulsion takes place.

If the repulsive forces are stronger than the opposite attractive forces, reagglomeration of the particles is prevented. In the case of steric stabilization high-molecular polymers stick to the particle surface. The polymer branches are covering the particle surface to inhibit the approach of other particles and reagglomeration is prevented. It is possible to combine both stabilisation types in one surfactant molecule.

Experimental and Results

The mode of action of powder dispersants is demonstrated by using a black joint filler. The colour is generated by incorporation of black iron oxide pigments. The black joint filler is blended with 0.5% of powder dispersant.

To avoid negative impacts of the dispersant regarding workability and consistency, the same parameters are measured as mentioned in the previous paragraphs. To evaluate the dispersing properties of the joint filler, colour strength and L-values are measured using a Datacolor colorimeter. The colour strength describes the capability of the pigments to tint the filler.

In this example the dispersant increases the colour strength by 45% (see Table 3). The L value is a measure of the brightness of the surface and the L axis describes the achromatic colours in the L*a*b* colour space. The scale ranges from 0 (black) to 100 (white). A decrease of the L value in this specific example means that the joint filler appears more black.

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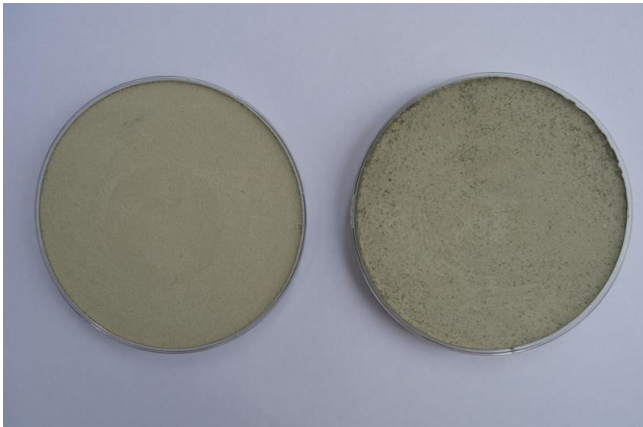


Figure 1. Creating a homogeneous surface and improve pigment distribution by using a powder dispersant.

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