



TECHLINE1

FOR THE CONSTRUCTION INDUSTRY

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Controlling the Setting Profile of Gypsum Plaster

ABSTRACT

Gypsum is one of the oldest building materials. In the big pyramid in Gizeh, Egypt (2500 BC) the cobble stones which were used are connected to each other through a certain type of mortar from gypsum in interior walls. Today it still plays an important role as gypsum machine plaster and hand plaster in the European construction industry. When applying gypsum plaster on the wall either by spray machine or by hand a series of working steps are required to obtain the desired smooth surface. That makes it very labor intensive. The five to six application steps are done within a time span of about 2 – 3 hours and include spraying, first even-out of plaster, paring of the surface, rough finishing (felting) and the final smooth finishing.

The setting process of gypsum during this process is a very important quality characteristic. The profile of the setting curve must be synchronized with the timely sequence of the individual application steps. It must be well balanced and has to compensate as well for gypsum raw material variations.

Our lab investigations have shown that ultrasonic recording of the setting process is a useful tool to quantify the effects of cellulose ethers, setting retarders and accelerators on the setting profile of gypsum. Calibration of the various application steps with defined sound velocities proved to be a realistic way to predict actual performance based on less labor intensive lab experiments.

INTRODUCTION

One of the most important quality criteria for gypsum plaster is a constant setting profile. Influencing factors include temperature and humidity, the storage time and gypsum raw material variations. The simple, reliable and efficient handling of gypsum plaster requires a controlled and stable setting characteristic. In most gypsum production sites continuous

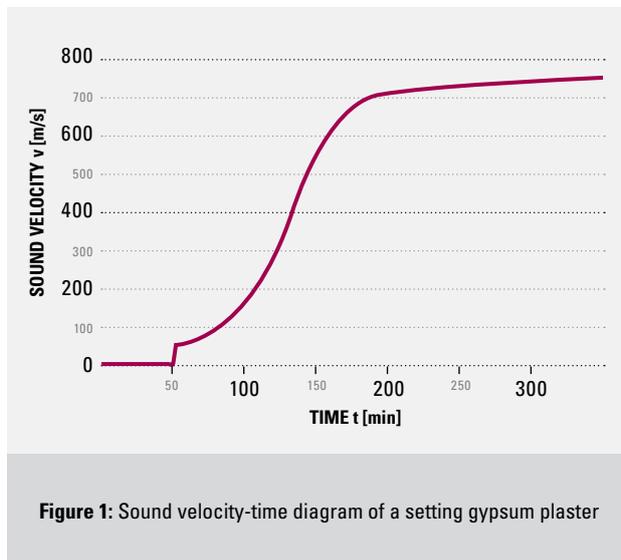
quality control mechanisms ensure that the gypsum setting profile follows the specified limits. The desired setting profile of gypsum plaster can be adjusted using an optimized composition of several different retarders and additional dosage of accelerators.

TEST METHODS

Setting Time/Gypsum Hydration – Ultra Sound Velocity

The principle of the ultrasonic testing device for mortars is based on a transmission measurement where the sound wave velocity and the transmitted energy are continuously monitored.

The operating principle is based on the fact that a pasty material has a lower sound velocity compared to a solid material. Since the signals are recorded continuously in certain intervals, it is possible to observe the complete setting behaviour of gypsum plaster.



The plot in figure 1 is appearing in a velocity-time diagram. With the progressing setting process the sound velocity increases and reaches the maximum after about 5 hours.

Figure 2 shows the measuring cell, the receiver and the transmitter. The distance between receiver and transmitter is very important for comparable measurements and is kept constant at 30mm.



Working Steps

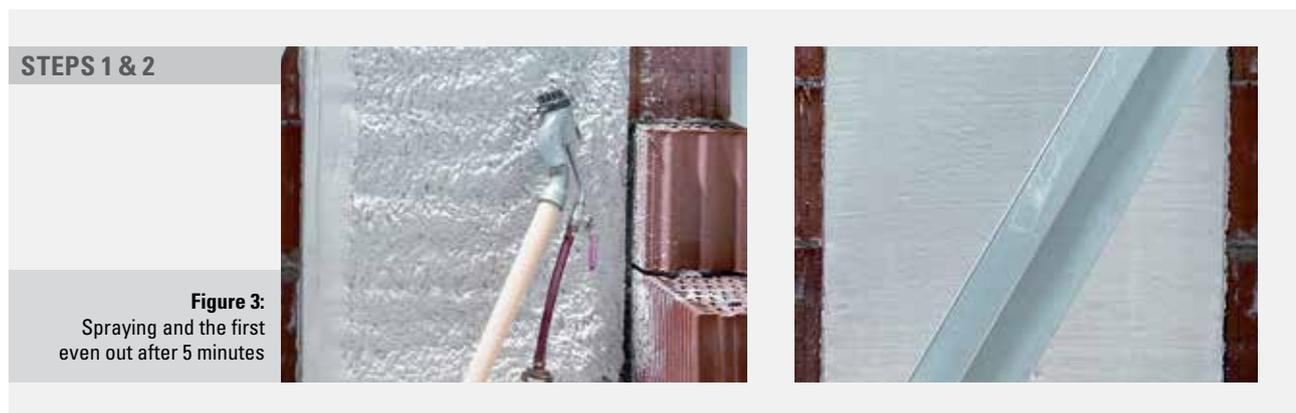
The application of gypsum spray plaster requires a whole series of working steps. It takes about 3 hours of work from the projection of the gypsum mortar onto a vertical wall until a spotless smooth surface is generated.

The timing of these working steps depends on the increasing strength of the plaster paste. In the real application the hardening of the gypsum can have two different origins. One of them is the chemical hydration of the gypsum hemihydrate and the second one is the loss of water to the absorbing substrate and to the atmosphere due to evaporation. Both processes run in parallel and at a first glance, they can not be kept apart. However, the addition of cellulose ether provides water retention to the plaster and prevents loss of water to the absorbing brick substrate.

The remaining experimental error is therefore minimal and mainly based on evaporation loss of water. The calibration of the ultrasound curve was carried out as follows:

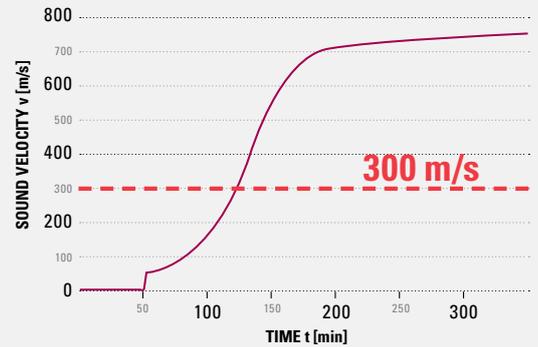
1. GSP was applied of non-absorbent substrate (e.g. concrete)
2. at the same time gypsum plaster was filled into the ultrasonic measuring cell
3. Sound velocity [m/s] was recorded
4. Sound velocities for each working steps were defined

The application steps on the job site were compared to a corresponding ultrasonic velocity as seen in the pictures and graphs below.



STEP 4

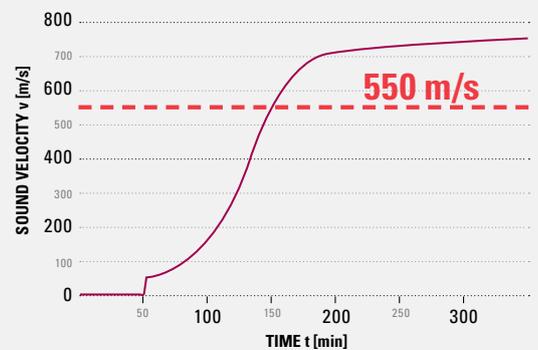
Figure 5:
Rough finishing after 120 min corresponding to sound velocity of 300 m/s



The plaster is treated with a wet sponge or a felt. The top layer will get enriched with a creamy paste of fine gypsum particles.

STEP 5

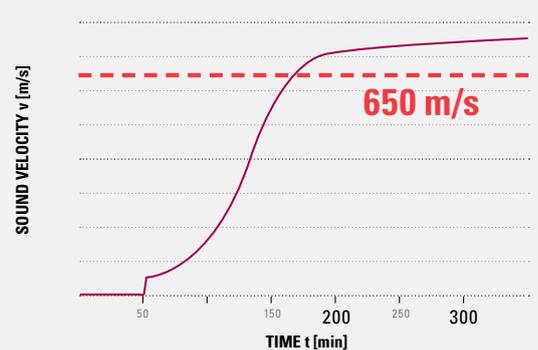
Figure 6:
First smooth finishing after 150 min; sound velocity of 550 m/s



The gypsum plaster has achieved the required stiffness; the smooth finishing is accomplished with a metal trowel applied with the required strength.

STEP 6

Figure 7:
Last smooth finishing after 170 min; sound velocity of 650 m/s



The hydration process continues and the smoothing step is accomplished with a glossy defect free surface. Proper adjustment of the setting curve is necessary to ensure that the working steps on the construction site can be performed within a specified time frame of 2 – 3 hours.

The selection of the right retarder and accelerator is critical to achieve the desired results. The detailed knowledge of their kinetic effects on the gypsum setting process is a prerequisite for successful formulation development. We have studied a number of commonly used additives and their effect on gypsum setting retardation or acceleration.

	Tartaric acid	Malic acid	Polyphosphate
Chemical structure	Dihydroxy succinic acid	Monohydroxy succinic acid	Phosphate condensates
Application	Gypsum spray plaster (GSP)	Gypsum manual plaster	Gypsum plaster
Grain size	100% < 63 µm	100% < 63 µm	–
Effect	Very effective setting retardation	Used in faster setting applications	Delay of finishing steps

Table 1: Retarder properties

Retarder

As retarder, we used in our experiments tartaric acid, malic acid and polyphosphate. The setting time trials were carried out with the two different hydroxycarboxylic acids as setting retarders.

Tartaric acid is widely used in the GSP industry as it delays gypsum setting very effectively. Natural tartaric acid finely milled (100% <63µm) is recommended.

Malic acid delays less in comparison to tartaric acid. The time span between the start of the setting and finish is shorter. Manual gypsum plaster is a typical application. The polyphosphate (Targon G2, BK Giulini GmbH) is in most cases used as an additional retarder. It delays specifically the end of the setting process. The most important retarder properties are summarized in **Table 1**.

Accelerator

Calcium sulphate dihydrate is widely used to accelerate the setting of gypsum plasters. The powder is produced in specially designated gypsum plants with the necessary milling equipment as the particle size distribution is very important for the performance. A typical dihydrate accelerator is Casonic DH 50/45 (VG-Orth, Germany). This product has a very fine granulometry (80% < 20 µm) which causes a high specific surface area of 6000 cm²/g. The calcium sulfate dihydrate particles work as seed crystals during the gypsum setting process. The higher the number of fine particles, the better the accelerating effect will be. However, the finest calcium sulphate dihydrate powders are subject to rapid aging. Longer storage time will result in significant loss of accelerator effectiveness as the fine particles agglomerate under the influence of humidity.

Formulation – Gypsum Spray Plaster

We formulated a gypsum spray plaster using a basic GSP compound and a number of additives. The gypsum consisted mainly of natural gypsum blended with 30 % FGD (flue gas desulphurisation) gypsum. Starch ether, cellulose ether, air-entraining agent and retarder/accelerator system were added. The following formulation was used for our investigations:

GSP compound	99.5 pbw
Starch ether Amitrolit 8860	0.02 pbw
Air-entraining agent	0.012 pbw
Tartaric acid, fine granulometry	0.04 - 0.08 pbw
Malic acid	0.06 - 0.12 pbw
Polyphosphate Targon G2	0.04 - 0.08 pbw
Dihydrate	0.20 - 0.28 pbw
WALOCCEL VP-M 27130, HEMC	0.22 pbw

Table 2: GSP formulation

In subsequent screening tests we varied the levels of tartaric acid, polyphosphate, malic acid and dihydrate powder.

Delay with Tartaric Acid

Typical factors influencing gypsum setting performance are particle fineness, impurities, raw material source (natural or FGD gypsum). They all have an impact on the gypsum crystal nucleation and not all retarders have the same efficiency, depending on the raw material selected. Sometimes, a retarder in certain gypsum qualities shows only an insufficient delay effect caused by less effective prevention of gypsum crystal nucleation. Tartaric acid is a commonly used retarder, but not all gypsum plaster and gypsum based products can be equally well delayed.

Besides gypsum raw material variation the pH value is also very crucial for the delay effect. Tartaric acid is most effective at alkaline pH values greater than 12. The pH value in GSP is adjusted by the addition of hydrated lime.

In figure 9 we see very well the retardation effect of tartaric acid in gypsum plaster as a function of dosage level.

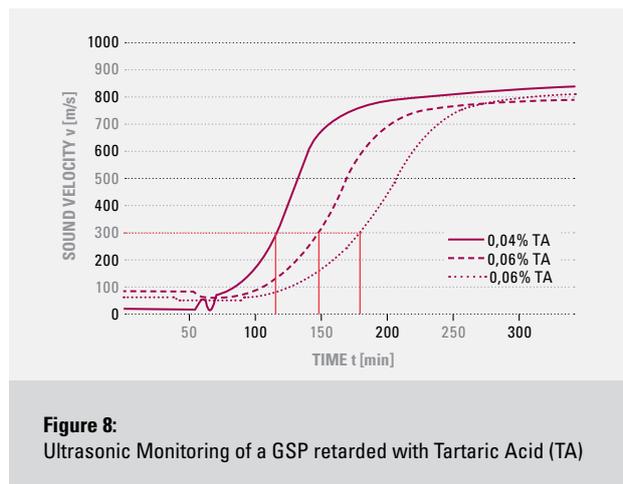


Figure 8:
Ultrasonic Monitoring of a GSP retarded with Tartaric Acid (TA)

The three different curves reflect the setting process of GSP with different amount of tartaric acid varying from 0.04 % to 0.08 %. Increasing the amount of this retarder by 0.02 % shifts the setting curve by 30 – 40 min to later times. The addition of 0.04% of tartaric acid to the gypsum plaster formulation will cause the rough finishing step (at 300 m/s sound velocity) to happen after 120 min. Adding more retarder (0.06%) delays this working step by 30 min. And with 0.08% addition level of tartaric acid the rough finishing will only be reached after 180 min.

We can conclude that in this GSP tartaric acid has a sensitive impact on setting behaviour.

Regulation of the Slope of the Setting Curve

The use of polyphosphate (Targon G2) is the appropriate tool to extend at the same start of initial setting the finishing working steps. This additive influences the slope of the setting curve. Particularly, this is very important during the smooth finishing phase (550 - 650 m/s). An extended time span for smooth finishing can be desirable on the job site.

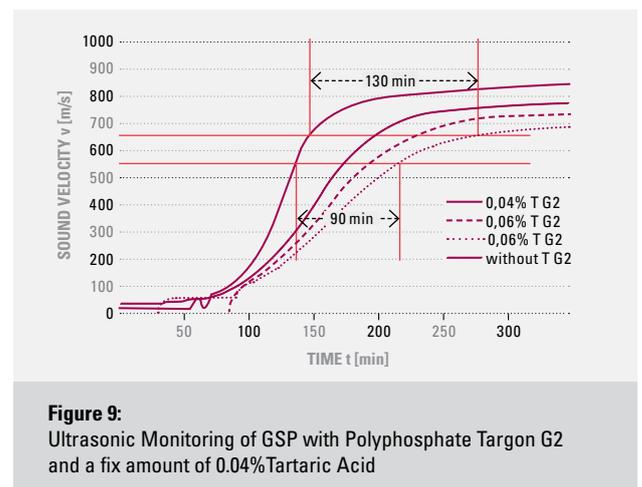


Figure 9:
Ultrasonic Monitoring of GSP with Polyphosphate Targon G2 and a fix amount of 0.04% Tartaric Acid

In figure 10 we see that the slope of the setting curve is decreased with the addition of polyphosphate. With 0.08 % addition level we reach the flattest curve. Comparing this extreme formulation to the system without polyphosphate the smooth finishing steps are extended by 90 min to 130 min (at 550 m/s and 650 m/s sound velocity). The initial working steps 'paring of surface' and rough finishing are much less impacted. The extension of smooth finishing is a quality feature of all GSP's. The applicator has much more time during this last very labour-intensive working step to achieve a high quality, defect-free surface.

Acceleration with Dihydrate

Adding gypsum setting accelerators to the retarded plaster formulation does not seem to make a lot of sense at a first glance. Adding accelerators and retarders to the same formulation appears to increase cost unnecessarily. But the addition of dihydrate stabilizes the setting process. The setting profile without accelerator would appear very instable. Gypsum plaster performance variations are caused by the raw material composition, the impact of humidity and temperature and many different production process parameters. The addition of dihydrate in fact helps to reduce costs because the alternative of daily very labour intensive adjustment of the retarder system can be avoided.

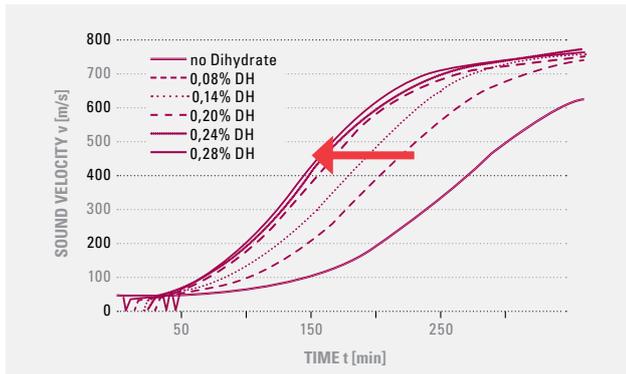


Figure 10:
Ultrasonic Monitoring of GSP with Dihydrate (DH) and a fixed amount of 0.06% Tartaric Acid and 0.07% Targon G2

The strongly retarded plaster formulation containing 0.06% of tartaric acid and 0.07% of polyphosphate is accelerated by the addition of calcium sulphate dihydrate Casonic DH 50/45. Already an addition level of 0.08% of DH allows the rough finishing (at 300 m/s sound velocity) to happen 60 min earlier. Further addition of DH continues to accelerate the setting kinetics of the gypsum until an addition level of 0.20% is reached. More dihydrate would give only incremental acceleration.

Combination of two Retarders

Gypsum manual plaster (GMP) is very much used in Western Europe and Eastern Europe in the renovation sector. The workflow for application of the GMP requires a shorter smoothing phase and therefore faster setting times, i.e. less retardation, are required.

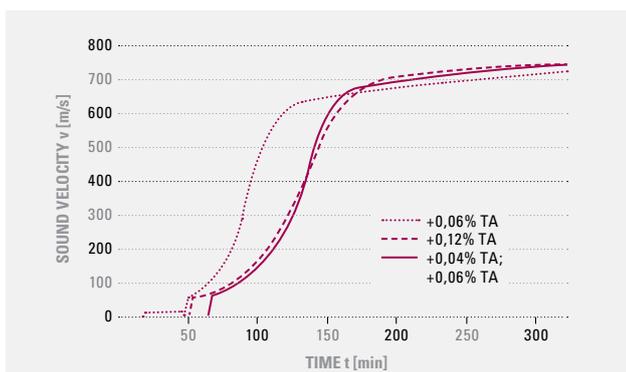


Figure 11:
Ultrasonic Monitoring of GSP with Dihydrate (DH) and a fixed amount of 0.06% Tartaric Acid and 0.07% Targon G2

Tartaric acid (TA) is a very effective retarder and provides long setting time at low addition levels. Figure 8 shows that even with double the concentration (0.12%) of malic acid (MA) less retardation is obtained compared to the system delayed with

0.06% tartaric acid. For manual plaster the faster gypsum setting kinetics are generally a desired attribute. It is typically applied on renovation job sites where the area to be covered with gypsum plaster is usually small. The sequence of working steps is completed in less time in comparison to gypsum spray plaster applied on large wall areas in new buildings.

Curve No.2 (0.12% malic acid) in figure 8 is too fast even for a gypsum manual plaster. Adding more retarder would make the formulation too expensive. However, blending of two different hydroxy carboxylic acids as retarders gives additional freedom to formulate. In our example we have combined 0.04% of tartaric acid with 0.06% of malic acid. Within the first 6 hours, the setting profile is nearly identical to curve No.1 (0.06% of tartaric acid).

The combination of two retarders has other advantages. It is well known that some retarders have a very strong influence on the crystal structure. They will eventually have an impact on the strength properties of the final product. Figure 9 shows the sound velocity of the same test described in Figure 8, however, over 24 hours.

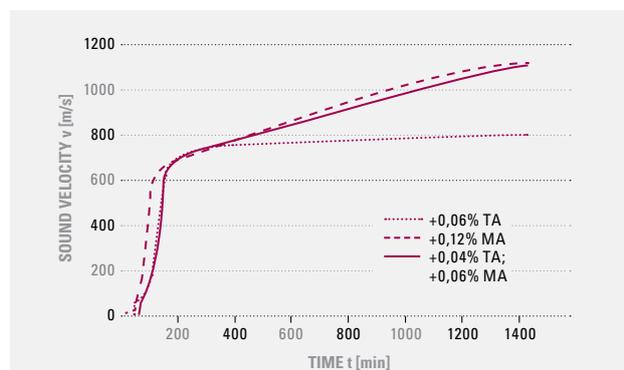


Figure 12
Long term Ultrasonic Monitoring of GSP, comparison Tartaric Acid (TA), Malic Acid (MA)

Curves No. 1 and 3 are almost identical for the first 6 hours. The plaster which was retarded with only tartaric acid (No.1) did hardly show any further increase in sound velocity after the first 6 hours. The plaster formulation with a combination of retarders continued to develop strength for 24 hours. The slope of the increase of sound velocity was similar to plaster No. 2 which contained only malic acid as retarder.

CONCLUSIONS

The use of different retarders and accelerators is state-of-the-art in the gypsum industry for the production of gypsum spray plaster and of other gypsum based products. The use of these additives allows influencing the start and finishing of the gypsum setting reaction and the slope of the setting curve. The setting process can be very well adjusted to the specific application needs. This adjustment is also required to homogenize the effect of gypsum raw material variations which have a strong impact on the setting process.

The finetuning of the retardation/acceleration of the setting profile is typically controlled using automated Vicat test equipment (DIN 1168). This test, however, only gives information over a limited time span of the setting process.

No information about the late setting stages is possible as soon as the mortar is hard enough so that the needle cannot penetrate anymore.

We could demonstrate that the use of ultrasonic monitoring of the gypsum setting reaction is a valuable tool for the gypsum plaster producer to monitor and adjust the setting profile. Information about the late setting stages enables the formulator to optimize the setting profile for the finishing working steps.

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