



Dow Construction Chemicals

TECHLINE 12

Gypsum Spray Plasters

Development of new HEMC Cellulose Ethers for Reduction of Lump Formation in Gypsum Spray Plasters

Spray applied gypsum-based dry mortars became widely used in Western Europe in the 1970s allowing for lower cost and highly efficient plaster application. Their continued commercialization made water soluble cellulose ethers the key additive in gypsum spray plasters. Cellulose ethers impart water retention properties that limit loss of water from the plaster to absorbing substrates allowing for a steady setting and high final strength. Additionally, the very specific rheological profile of cellulose ethers improves spray application and significantly eases subsequent leveling and finishing steps.

In spite of these clear advantages the use of cellulose ethers in gypsum-based plaster has drawbacks- they are also known to potentially lead to the formation of dry agglomerates when working with spray machines. This formation of non-wetted agglomerates, also called clumps or lumps, negatively impacts leveling and smoothing of the plaster: Their formation reduces productivity on site and adds to the cost for high quality plaster application. In order to better understand the impact which cellulose ethers have on the formation of agglomerates in gypsum spray plasters we carried out a study identifying relevant product parameters. Based on our findings new cellulose ethers with a reduced tendency for lump formation were developed and evaluated in application trials.

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Introduction

Water soluble cellulose ethers are successfully employed in gypsum spray plaster to control water demand and improve water retention, as well as to improve the rheology of the mortar [1]. Thus, they help to improve the wet mortar properties while ensuring that the required mortar strength is obtained. In the last 20 years dry-mortar gypsum spray plasters have become widely used throughout Europe as they remain a commercially viable as well as ecologically sound building material for interior use [2]. Spray machines for mixing and application of dry-mortar gypsum spray plasters have been commercially available for decades. Although some of their technical features differ depending on the manufacturer, all commercial spray machines allow very little time for completing the blending of water with gypsum dry-mortar containing the cellulose ether. Typically the whole blending is a matter of several seconds only. Once blended the wet mortar is pumped through the hose and subsequently sprayed onto the substrate. This does not take longer than one minute. However, within this short period of time the cellulose ether needs to dissolve completely in order to reach its full application performance. Complete dissolution of the cellulose ether in the water that is added in the blending step is ensured by the addition of finely milled cellulose ether to the gypsum dry-mortar formulation.

Upon contact with water during mixing in the spray machine the finely milled cellulose ether shows a very quick build-up of consistency. This quick rise in viscosity due to the dissolving cellulose ether can lead to issues regarding the wetting of gypsum binder particles which takes place at the same time: small pores in the binder particles are made inaccessible to water due to its reduced flow ability because of the increasing viscosity. With pores blocked the whole process of the wetting of the binder particles becomes delayed. The mixing time in the spray machines is shorter than the timeframe needed for obtaining a complete wetting of binder particles resulting in the formation of dry agglomerates in the gypsum mortar paste. Once formed, these lumps hinder applicators from effectively performing the subsequent application steps: leveling a mortar that contains lumps is cumbersome and takes more time. But even upon setting an initially formed lump may show e. g. as an unwanted darkish area where a lump was worked into the surface of the plaster. Although cellulose ethers have been used for many years as additives, their impact on the formation of non-wetted aggregates in gypsum spray plasters has not been researched so far. This paper describes a systematic approach to understand cellulose ether related root causes for lumping.

Formation of non-wetted Agglomerates in Gypsum Spray Plasters

Wetting of Gypsum Plaster

When setting up the research program initially, a large number of possible root causes for the development of lumps in gypsum spray plaster was compiled. This exercise was followed by computer assisted analysis with a focus on the question of whether tangible technical solutions were available or not. This resulted in prioritized concepts for tackling the formation of lumps in gypsum spray plaster. Technical as well as commercial considerations ruled out processes that would change the wetting of gypsum binder particles by means of surface treatment. Also not followed for commercial considerations was the replacement of existing gypsum spray plaster machines by new ones with a mixing zone enabling a more thorough blending of dry-mortar with water. Another option was the use of wetting agents as additives in gypsum plaster formulations which is found in a patent [3].

However this concept was not pursued as their incorporation will inevitably result in changes to the workability of the plaster and more importantly change its physical properties especially hardness and strength. Addition of wetting agents may also be considered critical with respect to the environment.

Given that they are part of gypsum plaster formulations already the best option identified was to optimize the cellulose ether, but without compromising water retention performance or impact on application rheology of the plaster. Based on the earlier described hypothesis that non-wetted aggregates in gypsum spray plaster may form due to viscosity increasing too quickly in cellulose ethers during blending we made controlling the dissolution characteristics of cellulose ethers the primary goal of our research.

Dissolution Time of Cellulose Ethers

The obvious approach to slow down the dissolution time of cellulose ethers is to make use of a granular grade. The key disadvantage of doing so in gypsum spray plaster is that this coarse cellulose ether will not fully dissolve within the mixing time in the spray machine that does not exceed 10 seconds. Incomplete dissolution results in loss of water retention performance. Moreover, a subsequent swelling of non dissolved particles may result in post-thickening of the plaster which is unwanted because it negatively impacts workability.

Another option for slowing down the dissolution time of cellulose ethers is to employ grades that have been reversibly cross-linked through reaction with dialdehydes [4]. However, their dissolution is dependent on the pH-value of the surrounding water because the cross-linking reaction is strongly influenced by the pH-value. Gypsum spray plasters that contain lime hydrate have a pH-value so high that any dialdehyde induced cross-linking cleaves upon contact with water resulting in an almost immediate viscosity rise. Therefore this chemical treatment does not allow controlling dissolution characteristics of cellulose ethers in gypsum spray plaster.

The dissolution time of cellulose ethers does also depend on their particle morphology. However, this fact has not been given much attention so far, although the effect is significant. The graph in **Figure 01** depicts results obtained from theoretical calculations for hypothetical monodisperse cellulose ether particles. They have a constant linear dissolution rate [$\text{kg}/(\text{m}^2\cdot\text{s})$], therefore their dissolution and viscosity build becomes proportional to the available surface. This does vary significantly depending on the particle morphology. For our example we assume that full (i. e. 100%) build of viscosity is reached after 5 seconds mixing time.

The calculations for different particle morphologies show that e. g. spherical particles build 35% of the final viscosity after half of the mixing time has elapsed while it is only 10% for rod-like shaped cellulose ether particles after the same period of time. Disc-like formed particles only start dissolving after 2.5 seconds.

The graph also includes the idealized dissolution characteristic for cellulose ethers to be used in gypsum spray plaster. Here the build up of viscosity is delayed for more than 4.5 seconds. This is followed by a very quick viscosity rise in order to reach the final viscosity within the 5 seconds timeframe for mixing. In gypsum spray plaster such long period of non-dissolution would result in keeping viscosity low allowing added water to wet gypsum binder particles pores undisturbed.

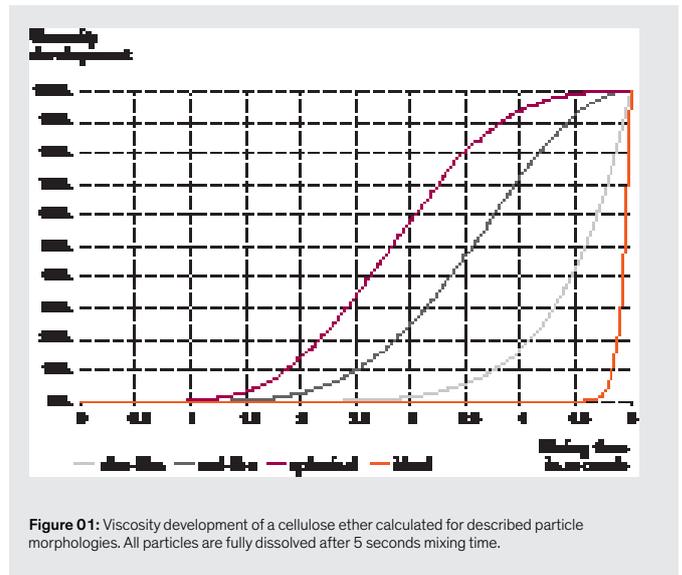


Figure 01: Viscosity development of a cellulose ether calculated for described particle morphologies. All particles are fully dissolved after 5 seconds mixing time.

Particle Morphology of Cellulose Ethers

Particle Morphology Measurement

Acknowledging the significant impact the shape of cellulose ether particles have on dissolution characteristics, it was initially needed to describe morphology parameters for cellulose ethers. Subsequently those parameters were identified that specifically correlated with the formation of non-wetted agglomerates in job-site gypsum spray plaster application trials.

The particle morphology of cellulose ethers was obtained through dynamic image analysis [5]. Using a digital image analysis instrument from SYMPATEC (Germany) and adapted software analysis tools, it was possible to fully characterize cellulose ethers with regard to their particle morphology. The most important particle morphology parameters were found to be the average length of fiber called LEFI (50,3) and the average diameter of fiber called DIFI (50,3). Data obtained for the average length of fiber always consider the full length of a (rolled-out) cellulose ether particle.

Generally data for particle size distribution such as e.g. the average diameter of fiber DIFI might be calculated based on number (designated by 0), length (designated by 1), area (designated by 2), or volume (designated by 3). For all measurements in this paper data was calculated per volume which results in the addition of the suffix 3. In e.g. DIFI (50,3) a 3 indicates volume distribution and the 50 shows that in the particle size distribution curve 50% are smaller than the indicated value and 50% are larger. Morphology data for cellulose ethers are given in μm .

In **Figure 02** examples for morphology parameter calculation are given. The average particle diameter DIFI (50,3) for instance is derived from dividing the projection area (A) obtained from digital image analysis by the length of fiber (l) that is obtained using the analysis tool.

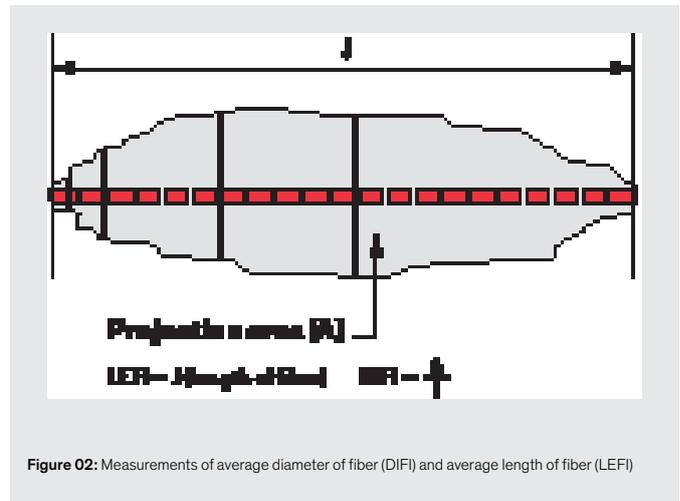


Figure 02: Measurements of average diameter of fiber (DIFI) and average length of fiber (LEFI)

Cellulose Ethers with optimized Particle Morphology

Taking into account the surface of the particles, the dissolution time for cellulose ethers – like for similarly shaped particles with a rod-like morphology – largely depends on the average diameter of fiber DIFI (50,3). Based on this working hypothesis, cellulose ether development work should aim to improve powder dissolution characteristics through providing products with a larger average diameter of fiber DIFI (50,3).

However, the average particle diameter needed to get larger without also having a longer average length of fiber DIFI (50,3). Growing both parameters results in a larger sized particle that would not solubilize completely within the few seconds of mixing time interval in a gypsum spray machine.

Hence, the desired hydroxyethyl methylcellulose (HEMC) needed to have an increased average diameter of fiber DIFI (50,3) while keeping the average length of fiber LEFI (50,3). This optimized HEMC was obtained through a new cellulose ether production process that is breaking the link between the particle morphology of cellulose used as a raw material and the particle morphology of the resulting water soluble cellulose ethers. In other words, this process allows cellulose ether particle morphology design that is independent from that of its raw materials.

Figure 03 shows three scanning electron microscopy pictures: One with cellulose ether obtained from standard production process and a second picture with cellulose ether made with the new process with an increased average diameter of fiber DIFI (50,3) than the regular product. Also shown is milled cellulose the (raw material) in both production processes.

By comparing electron microscopy photographs of cellulose and the cellulose ether that was produced with standard process technology one can easily see that both have similar

particle morphology. In both pictures a large number of the particles exhibit characteristically long and very thin structures indicating that this key morphology feature remained unchanged even after a chemical reaction did take place; clearly the particle morphology of the reaction product remains closely linked to its raw material.

A significantly different result is found for characteristic particle morphology features of a cellulose ether with larger average diameter of fiber DIFI (50,3) that was produced with the new production process. Here the predominant particle shape is rather stocky and roundish while long and thin particles (typical for the raw material cellulose) are almost completely absent.

This picture again indicates that with the new process the particle morphology of cellulose ether becomes independent from that of the cellulose – the morphological link between raw material and finished product does not exist anymore. Morphology parameters compiled in Table 01 for average diameter of fiber DIFI (50,3) and average length of fiber LEFI (50,3) confirm that the new production process not only changes particle shapes visually but also allows for the production of HEMC cellulose ethers with larger DIFI (50,3) without affecting the LEFI (50,3).

Material	Typical average diameter of fiber DIFI (50,3)	Typical average length of fiber LEFI (50,3)
Chemical grade cellulose	30 – 40 µm	350 – 450 µm
Cellulose ether from standard production process	35 – 40 µm	300 – 370 µm
Cellulose ether with improved morphology	47 – 55 µm	300 – 370 µm

Table 01: Morphology data for hydroxyethyl methylcellulose (HEMC)



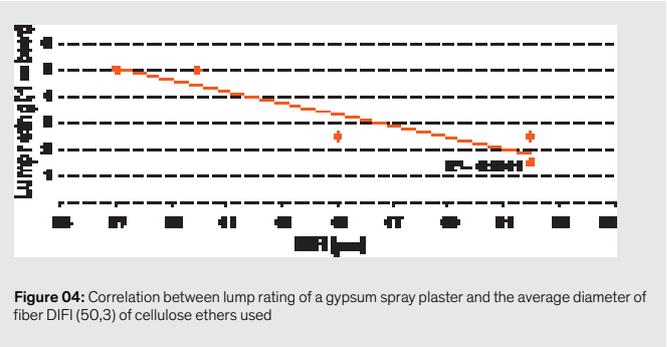
Figure 03: Three scanning electron microscopy pictures of cellulose (A) and cellulose ether with regular (B), and optimized particle morphology (C)

Effects of Hydroxyethyl methylcellulose ether (HEMC) Particle Morphology on the Formation of non-wetted Agglomerates in Gypsum Spray Plaster Application

Gypsum spray plaster trials were accomplished under job-site application conditions in order to validate the working hypothesis that using cellulose ethers with larger average diameter of fiber DIFI (50,3) will reduce the unwanted formation of unwetted agglomerates, i. e. lumps. For these trials hydroxyethyl methylcellulose ethers (HEMC) with an average diameter of fiber DIFI (50,3) ranging from 37 μm to 52 μm were produced. In order to minimize effects other than particle morphology the gypsum plaster base material and all other additives remained unchanged. The viscosity of the tested cellulose ethers was kept constant (60000 mPa-s for 2% solution in water measured with Haake rheometer).

Application trials were carried out using a commercially available gypsum plaster spray machine (PFT G4) focusing on the formation of non-wetted aggregates in the gypsum mortar after the plaster had initially been sprayed onto the wall. Rating of lump formation at this stage in the plastering process best showed any differences in performance. The evaluation was carried out by experienced applicators who rated lump performance with marks ranging from 1 (best) to 6 (worst).

One set of test results is shown in **Figure 04**. Trial results clearly show a good correlation between average diameter of fiber DIFI (50,3) of a cellulose ether and its mark for lumping performance. Following the working hypothesis larger DIFI (50,3) cellulose ethers outperform those having a significantly smaller DIFI (50,3): grades with DIFI (50,3) of 52 μm are rated on average with mark 2 (good) while those with DIFI (50,3) of 37 μm and 40 μm respectively are marked 5 (failed).



As expected, lumping in gypsum spray plaster application is significantly dependent on the average diameter of fiber DIFI (50,3) of the cellulose ether. Furthermore, as discussed earlier among all morphology parameters it is the DIFI (50,3) that strongly influences the powder dissolution time of cellulose ethers. This confirms that the powder dissolution time of cellulose ethers – that is dependent on their particle morphology – ultimately influences the formation of lumps in gypsum spray plasters: a larger DIFI (50,3) resulting in prolonged powder dissolution time leads to significantly lower formation of lumps. However, a too-long powder dissolution time will prevent cellulose ether from complete dissolution within the mixing time window of the spray machine.

Measurement data in **Figure 05** show that the improved powder dissolution behavior of new HEMC grades with their larger average diameter of fiber DIFI (50,3) does not only result in better wetting of gypsum binder – as can be seen from lump rating – but keeps water retention performance unchanged. Water retention measured according to EN 459-2 is constant for HEMC samples having the same viscosity and DIFI (50,3) values ranging from 37 μm to 52 μm . Every measurement after 5 minutes and after 60 minutes meets the required water retention ranges as indicated in the graph.

Yet the data in Figure 05 also confirm that if the DIFI (50,3) becomes too large the cellulose ether particles will not dissolve completely anymore. This is found for the HEMC probe with 59 μm DIFI (50,3); its water retention values obtained after 5 minutes and especially after 60 minutes do not reach the required minimum level.

While spray application trial results indicate that this large DIFI (50,3) is not required for obtaining significantly reduced lump formation, it however underlines the need to control particle morphologies thoroughly in production and ensure correctly set specification limits.

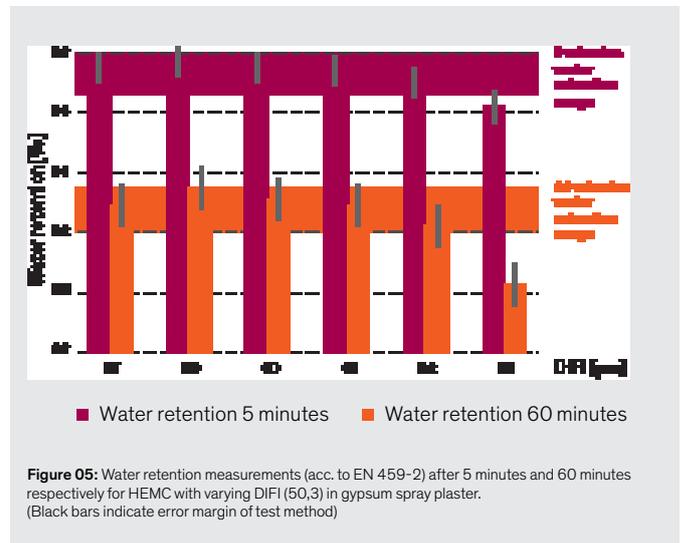


Figure 05: Water retention measurements (acc. to EN 459-2) after 5 minutes and 60 minutes respectively for HEMC with varying DIFI (50,3) in gypsum spray plaster. (Black bars indicate error margin of test method)

Summary

The presented research and development work emphasizes an interdependency between the particle morphology of cellulose ethers that are important additives in gypsum spray plaster formulations and the formation of non-wetted agglomerates (so called lumps) during spray plaster application. It is based on the working hypothesis that the powder dissolution time of cellulose ether influences lump formation as it impacts wetting of gypsum binder. The dissolution time however, depends on particle morphology of cellulose ether which can be analyzed analysis tools. Gypsum spray plaster application cellulose ethers with large average diameter of fiber DIFI (50,3) have best-in-class lump performance because their optimized powder dissolution characteristics allow for more thorough wetting of the gypsum binder particles during the mixing step.

These cellulose ethers are produced with a new production process that allows raw material independent particle morphology control.

The high importance of the average diameter of fiber DIFI (50,3) in lumping performance is validated through job-site plaster application spray trials with commercial gypsum plaster bases. Moreover, these trials confirm laboratory results that best performing cellulose ethers with large DIFI (50,3) still dissolve completely within the mixing time window of gypsum spray machines. Hence, cellulose ethers with improved morphology and best performing DIFI (50,3) do not compromise on water retention.

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