TECHLINE6
FOR THE CONSTRUCTION INDUSTRY

Controlling the Application Performance of Cement Renders with Cellulose Ethers

ABSTRACT
Cellulose ether is an indispensible component of a cement render formulation. This highly functional additive helps to adjust the performance profile of the render at various levels. The main role of the cellulose ether is to provide sufficient water retention to the render so that the cement can set and develop strength before it dries out. The solubilized cellulose ether in the mortar matrix determines to a large extend its rheological properties and subsequently the workability of the render. Depending on the substitution level cellulose ethers have a delaying impact on the cement setting rate. This article discusses the effect of the structural parameters of the cellulose ether on all performance aspects of cement renders.

INTRODUCTION
Cellulose ethers are well defined by their molecular weight, by the type and the degree of substitution. The effect of cellulose ethers on specific performance aspects of cement mortars has been investigated in the past. Its main effect is the water retention in mortar systems which can be adjusted by the dosage and the molecular weight of the cellulose ether [1]. The cellulose ethers preferred in dry-mix mortar application are hydroxypropyl methyl cellulose (HPMC) or hydroxyethyl methyl cellulose (HEMC). Aqueous solutions of this type of polysaccharides reversibly precipitate or form gels when heated [2]. The thermogellation temperature is controlled by the substitution of cellulose ethers. Once the cellulose ether has gelled, it looses its functionality in construction applications.

This can happen during the summer months when the mortar temperatures may exceed 40°C.

Cellulose ethers are additives which control to a large extend the rheology of a cement render. In a complex matrix of this multiphase blend they have an impact on the yield point and the shear thinning behavior [3]. The effect of cellulose ethers on the cement setting has been subject to many investigations. Adsorption of the polymers on the cement clinker phases inhibits the formation of portlandite [4]. The lower the content of methoxy groups the stonger the retardation of cement setting will be.

WATER RETENTION

The viscosity of the aqueous phase of cement render correlates well with its water retention capabilities. Increasing the dosage level or the molecular weight of cellulose ether will thicken the aqueous phase of the render and subsequently result in an increase of water retention.

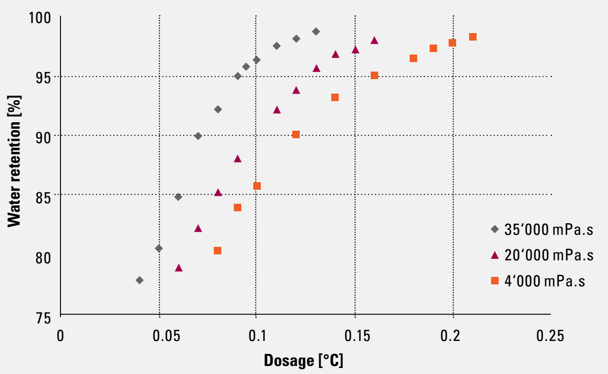

Figure 1: Water Retention of Cellulose Ethers as a Function of Dosage Level and Viscosity of a 2% Aqueous Solution

The molecular weight of the cellulose ether in figure 1 is indirectly expressed as the viscosity of a 2% solution in water measured with a Brookfield RVT Rheometer at 20 rpm. An addition level of 0.09% of the 35,000 mPa·s cellulose ether is required in order to achieve a water retention of 95% which is typically the case for a cement render. To get the same result with lower viscous grades, one has to add 0.13% of a 20,000 mPa·s cellulose ether or even 0.16% of the lowest viscous grade (4,000 mPa·s). The desire to reduce formulation cost makes the cellulose ether with the highest molecular weight the preferred additive in this formulation as the dosage level can be minimized. However, this choice may have a negative impact on the workability of the plaster as high viscous grades tend to cause stickiness.

In the application it takes only about 10 seconds, depending on the length of the spray hose, from the moment when water gets in contact with the drymix render formulation in the spray machine until it is projected onto the wall. Within this short time span, the cellulose ether has to fully solubilize in water in order to unfold its full functionality. Solubilization kinetics are depending on the grain size of the cellulose ether. The finer the product, the faster it goes into solution and maximizes its water retention capabilities.

Table 1 compares the water retention values of 15,000 mPa·s HEMC products with various contents of fine particles going through a 63 µm sieve. All three grades passed the 200 µm sieve, i.e. no coarse particles were present.

15,000 MPa·s HEMC	Fines < 63µm	40%	57%	67%
	water retention	89.9%	91.8%	91.3%

Table 1: Water Retention of a Cement Render as a Function of Fine Content (< 63µm) of the Cellulose Ether

The material with the least amount of fine powder (40% < 63µm) showed the lowest water retention. Increasing the fine content beyond 57% did not result in additional water retention. A fine content of 50% seems to be sufficient to ensure that all cellulose ether goes into solution within the short time span of spraying. The fact that some cellulose ether grains are still undissolved at the point of spraying is further confirmed by noticeable post-thickening of the render on the wall. These particles continue to dissolve during the various leveling steps after spraying and increase the consistency of the cement mortar.

Aqueous HPMC and HEMC solutions gel or precipitate when heated. The hydrophobic methyl substituents start to interact and form a reversible 3-dimensional net work, which completely redissolves upon cooling. In the gelled state the cellulose ether looses its water retention property. The gellation temperature can be controlled by the substitution level of the polymer. Methyl and to a lesser extend hydroxypropyl substituents decrease the gel point. Adding more hydroxyethyl groups would increase the gel point and can partly compensate the loss in temperature tolerance. Figure 2 illustrates the water retention values of two cement renders modified with different cellulose ethers. METHOCEL™ 267 is a HPMC with a methoxy degree of substitution (DS) of 1.75 and a molecular substitution of hydroxypropyl groups (MS) of 0.28. WALOCEL™ MKW 40,000 PP10 is a HEMC with a DS of 1.60 and a hydroxyethyl MS of 0.22.

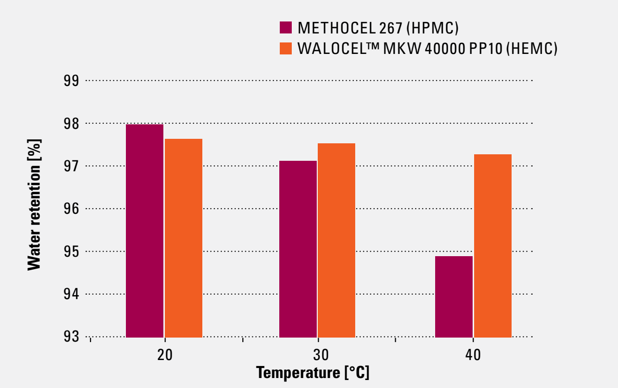

Figure 2: Water Retention of two Cellulose Ethers as a Function of Temperature

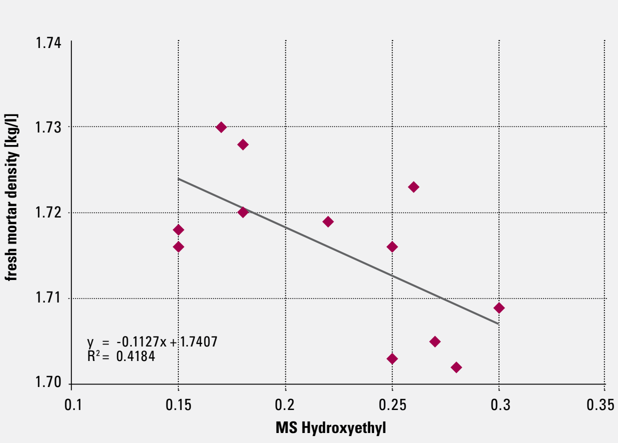

Figure 3: Plot of Fresh Mortar Density as a Function of MS Hydroxyethyl Substituents

The higher content of methyl substituents in the case of the HPMC and the presence of the less hydrophilic hydroxypropyl substituent are the reason for lower temperature tolerance compared to HEMC. Already at 40°C the HPMC looses significant water retention capability. The render will dry out too quickly before the cement can completely set and cracks will be formed. High temperature tolerance of the cellulose ether also means extended open time and good workability at the job site in warm climate conditions.

WORKABILITY AND STICKINESS

Workability and stickiness of the plaster are soft measures which are very difficult to quantify. The applicator typically gives a subjective rating. Cellulose ethers stabilize entrained air bubbles thus reducing the density of the fresh mortar. The mortar formulation itself contains anionic surfactants which act as air entrainment agents. Air bubbles are desired because they reduce the crack formation during the setting of the cement plaster. Lower mortar density is also to some extend favorable for better workability. The mort ar becomes smoother and creamier. The amount of hydroxyalkyl substitution reduces the cement mortar density as the results with a series of HEMC products demonstrate in figure 3.

High sag resistance and easy workability are often conflicting properties of a cement render. The detailed understanding of the rheological behavior is a prerequisite to resolve such conflicts. Dow has developed an oscillation rheology test method which allows to characterize complex multiphase systems like cement render and quantify yield point and shear thinning. It is very important to carefully prepare the sample and select a suitable measuring cell in order to generate reproducible data. Best results were achieved with a concentric cylinder and a vane spindle, to prevent slipping at the mortar interface (figure 4).

Figure 4: Oscillation-Rheometer for Mortar Systems

The rheological properties of the materials were measured with a stress-controlled rheometer (Physica UDS 200). The plaster is contained in a cup and deformed with a 6-vaned spindle in oscillatory shear flow at 2 Hz. The plaster was subjected to a sweep of the stress amplitude from 5 – 500 Pa. The results are reported as either a shear storage modulus (G'), a loss modulus (G"), or a loss factor (tan δ = G"/G') at yield point. Figure 5 illustrates an example of the resulting curves of this experiment. Until a shear stress of 130 Pa the storage modulus G' is higher compared to the loss modulus G". The elastic properties of the mortar dominate over the viscous component. When the shear stress is further increased, both moduli drop significantly. The storage modulus G', however, decreases even further so that beyond the yield point the loss factor G" dominates the rheological behavior and the render starts to flow. The curve of the loss factor (tan δ) shows a steep increase at this yield point.

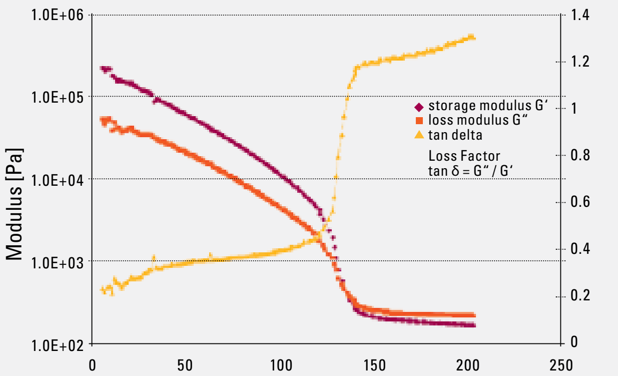

Figure 5: Plot of G', G" and tan δ as a Result of an Oscillating Amplitude Sweep of a Cement Render

The yield point determined by this method correlates very well with the sag resistance of the render on the vertical wall. At the same time, the loss factor tan δ at the yield point gives us information about the viscoelasticity of the render at this critical point. A lower loss factor at the yield point means that G' becomes bigger compared to G" and indicates that the render at the point before it starts to flow is characterized by a more pronounced elastic behavior. In fact, we could observe that cement renders with lower loss factors at the yield point show less stickiness and are easier to level out. Plotting the stickiness of the cement render against the loss factor shows a linear relationship (figure 6).

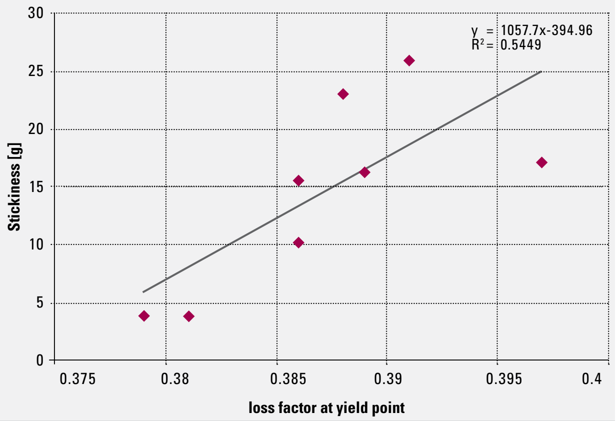

Figure 6: Plot of Loss Factor at Yield Point against Mortar Stickiness

The stickiness of the cement render has been simply determined in a lab experiment by the amount of mortar sticking to the metal cone when performing the flow table test (DIN 18550). The cellulose ethers in the above mentioned test series all had about the same viscosity level. The different rheological performance of the render can be achieved by the control of the substitution level of the cellulose ethers.

The workability of cement render in the application at the construction site is impacted by all formulation components. In our investigation we have focussed on the cellulose ether only. We could demonstrate in the lab that fresh mortar density and stickiness can be influenced by the choice of the cellulose ether. In order to find out the impact of the cellulose ether thickener in spray plaster trials we compared a number of HEMC of identical molecular weight, but different substitution levels. Our results could clearly show that the MS level of hydroxyethyl substituents plays a critical role (figure 7).

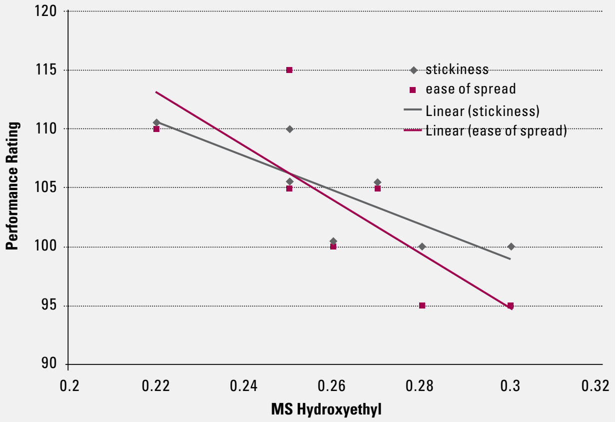

Figure 7: Plot of Plaster Performance Rating Against MS Hydroxyethyl

Our subjective rating system is based on the relative performance against a standard, which was a HEMC with a MS hydroxyethyl of 0.26. The performance of this standard was set to 100. Improvements to the standard were rated higher (105, 110,..) and lower performance was considered with lower numbers. Reducing the hydroxyethyl substitution level down to a level of 0.22 gave significant improvement over the standard in terms of easiness of spread and render stickiness. Higher levels of substitution, however, further reduced the workability. This result is in line with the rheological lab data shown in figure 6, where higher hydroxyethyl substitution causes higher loss factors at yield point and subsequently more stickiness. Reducing the MS hydroxyethyl to levels below 0.20 is not desireable, because the increasing fresh mortar density and loss of render creaminess are neutralizing the reduced stickiness. The level of methyl substitution did not have any impact on the workability rating of the cement render.

CEMENT HYDRATION

H.J. Weyer et al. [4] have monitored the cement hydration with the help of sychroton radiation in-situ in the presence of cellulose ethers. They found out that the level of methylation has a significant effect on the hydrolysis of the C2S/C3S clinker phases. They also found out that the presence of CE retards the formation of ettringite, but did so independently of the degree of substitution of the methylgroups. Pourchez et al. [5] have also studied the delay of the cement hydration with the help of conductometry measurements. They came to the same conclusion and attribute the retardation of the cement setting to the methylation level of cellulose ethers present in the formulation. In a recent presentation, Häcker and Arnold [6] could demonstrate the same effect of the methylation level on cement retardation. They based their data on heat flow calorimetric investigations of pure cement clinker phases. Figure 8 illustrates the heatflow of the cement setting reaction based on Portland cement (CEM I 42.5) water slurries (W/C ratio 0.45) with and without the addition of hydroxyethyl methyl cellulose (0.3% based on cement).

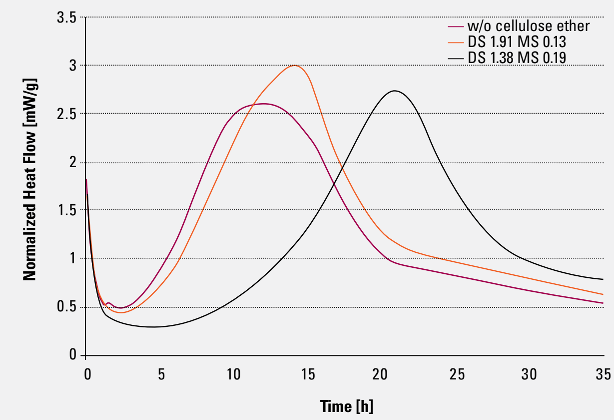

Figure 8: Heatflow Curves of Cement Setting Reaction with and without the Addition of HEMC's

As expected, the addition of cellulose ethers causes a retardation of the cement setting reaction. Without HEMC addition, the peak of the heatflow curves of the reaction exotherm can be observed after about 12 hours. Adding cellulose ether with a high DS of 1.91 shifts the peak by about 2 hours. Further delay of 7 hours is observed in the case of a lower substituted HEMC (DS 1.38). Plotting the degree of methyl substitution of various HEMC samples against the peak of the reaction exotherm (figure 9) gives a linear relationship.

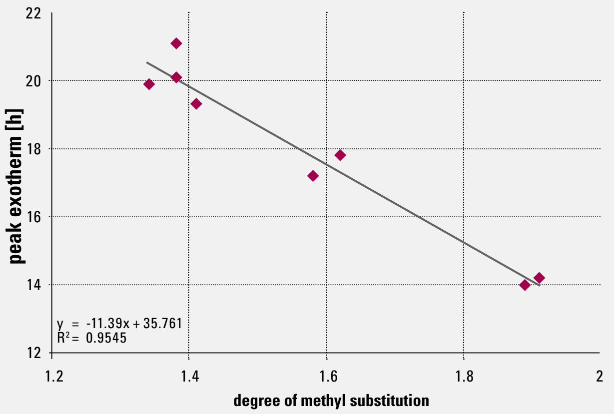

Figure 9: Linear Relationship of HEMC DS Methyl and the Peak Exotherm

SUMMARY

Cellulose ethers are used as thickeners and water retention additives in drymix mortar applications. However, beyond these main functionalities they impact many other performance parameters of cement renders as well:

– temperature tolerance
– stickiness (loss factor at yield point)
– fresh mortar density (air entrainment)
– workability (rheology)
– cement retardation

The structural properties of the cellulose ether polymers can be adjusted during the manufacturing step. It is of paramount importance to understand these structure-performance relationships in order to design products which are optimized for the specific application needs. In the case of the cellulose ethers of this study these independent structural parameters include the substitution levels of methyl and hydroxyethyl groups and the viscosity in aqueous solution. The following matrix (table 2) summarizes the effects of the cellulose ether structure on the critical performance requirements:

The level of methyl substitution also needs to be compromised. High methyl DS is favourable as the cement setting reaction is less delayed. This trend is in conflict with the negative impact of methyl DS on the temperature tolerance. We identified a methyl DS in the range of 1.60 as a good value to balance the conflicting trends.

Workability of the cement render is largely controlled by the hydroxyethyl substituents. They have a strong impact on the rheology as well as the air entrainment. The ideal cement render should be sag resistant, creamy, not sticky and very easy to spread. For this reason the MS hydroxyethyl should not be too low to allow sufficient air entrainment and reduce fresh mortar density. There should also be an upper limit for this substituent, otherwise the render becomes too sticky and workability suffers. Our investigations have shown that a range of 0.20 – 0.25 for the MS hydroxyethyl enables a good balance of properties. Dow Construction Chemicals offers a range of WALOCEL™ MKW HEMC products meeting these structural properties, which are perfectly designed for the use additives in cement render application.

	Desired direction	Viscosity	DS (Me)	MS (HE)
Water retention	↗	↗		
Temperature tolerance	↗		↘	↗
Mortar density	↘			↘
Stickiness	↘	↗		↗
Cement setting rate	↗		↗	

Table 2: Effects of Cellulose Ether Parameters on Cement Render Application

Viscosities of cellulose ethers in cement render application should not be too high as they would have a negative impact on the stickiness of the mortar. Depending on the specific render product they typically vary between 10,000 – 40,000 mPa·s (as 2% aqueous solution). Higher viscosities would potentially enable lower dosage rates at the same water retention level and subsequently lower formulation cost. However, such formulations are generally less performing and very sensitive to variations.

AUTHORS

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