Influence of Cellulose Ethers on the Consistency, Water Retention and Adhesion of Renovating Plasters

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Abstract. Cellulose ethers such as (hydroxypropyl)methyl cellulose (HPMC), (hydroxyethyl)methyl cellulose (HEMC), methyl ethyl hydroxyethyl cellulose (MEHEC) are currently used admixtures in the production of factory-made mortars. They improve such properties as water retention, workability, adhesion to the substrate, open time etc. in the cement-based mortars. The article investigates the impact of physicochemical properties (molecular weight, methods of modification, viscosity) on water retention, rheological properties and the adhesiveness to the substrates of renovation plasters. The research showed that the cellulose viscosity has a greater impact on water retention despite diversified effects of various derivatives of cellulose. The influence of viscosity of cellulose ethers is uneven. The greatest growth of retention was observed with the change of viscosity from 100 mPa·s to 15000 mPa·s. The further growth of viscosity of cellulose admixtures influenced the change of water retention with lower intensity. It was also stated that cellulose ethers improve the adhesion of renovation plasters to the substrates. Particularly beneficial results were obtained in the case of plasters consisting (hydroxypropyl)methyl cellulose (HPMC)-based admixtures.

1. Introduction

The walls of historic buildings, depending on their origin, are usually stone, brick or mixed: stone and brick walls. Lime, lime and clay mortars as well just clay were primarily used as binders [1]. Due to the long-term impact of environmental conditions: moisture, salts, temperature changes, etc., the absorption capacity of such walls varies considerably [2]. Renovation mortars are often used in their renovation [3]. Renovation plasters are a systemic solution that includes renovation rendering coat, plasters that accumulate salts (base plasters) and hydrophobic plasters. High efficiency in salt removal and drying of walls is achieved by applying a half-cover rendering coat and two layers of plasters, the first of which is made of a porous plaster absorbing salt solutions, and the other one - made of a hydrophobic porous plaster [4]. One of the important characteristics distinguishing the renovation plasters from traditional cement or cement-limestone plasters is their porosity. In most cases, these mortars are applied in thin layers, which is why they are particularly exposed to the rapid transfer of water to absorbent substrates. The time of preserving the mortar working properties is then shortened, the amount of water necessary for hydration of cement is reduced, which results in lowering the strength and adhesion properties of the hardened mortars to the substrate [5].

In order to limit this phenomenon, admixtures increasing water retention, produced on the basis of cellulose derivatives, can be added to mortars [6-9].

Cellulose derivatives are obtained through a chemical modification consisting in the etherification in hydroxyl groups present in cellulose with of organic groups. The most important simple ethers are
methylocellulose (MC), ethylcellulose (EC) and benzylcellulose (BC). Mixed ethers include ethylbutylocellulose (EBC), methylethylcellulose (MEC), Hydroxypropylmethylcellulose (HPMC), etc. Cellulose ether structures used in the studies are shown in Figure 1.

In cement and cement-lime mortars, cellulose-based admixtures have an impact on both macroscopic properties: strength, adhesion to substrates as well as consistency and change in water retention [6-9].

In fresh mortar, cellulose particles are adsorbed on the surface of aggregate or cement grains to form a monolayer. Further adsorption is prevented by the absorbing effect of the solvent [6]. Cellulose, which has not been adsorbed, forms a gel in the intergranular space. As a result of steric interactions of the cellulose chains, the cement grains and aggregates separate. The longer the cellulose chains, the greater their interaction, which in turn leads to a more stable suspension.

The influence of cellulose-based admixtures on water retention is caused by their ability to bind water [10, 11]. The mechanism of water retention by mortars with cellulose admixtures may result from the difference between the chemical potential of water in the mortar and the chemical potential of clean water. As the water evaporates from the wet mortar, the concentration of cellulose admixture increases. The presence of dissolved substance lowers the potential of water, and thus the hydrostatic pressure, which helps to retain water in the mortar. In addition, due to the presence of cellulose particles on the surface of the mortar, the diffusion space may be limited, resulting also in a decrease in water mobility and stimulating its retention in the mortar [5, 12].

Some of the important physicochemical factors of cellulose admixtures influencing the change of mortar consistency and water retention include: the type of modification, viscosity, quantity, molecular weight, granulometry, degree of substitution, etc [7, 13, 14].

Figure 1. Cellulose ether structures used in the studies: HPMC (hydroxypropyl)methyl cellulose (a), HEMC (hydroxyethyl)methyl cellulose (b), MEHEC methyl ethyl hydroxyethyl cellulose (c).
The available literature is scarce in the investigations of the influence of cellulose admixtures on the change in consistency, water retention and adhesion to substrates of renovation plasters. This paper discusses the impact of cellulose ethers, with various modifications and viscosities, on these properties.

2. Subject of the study and research methodology used
The impact of cellulose ethers of different chemical composition and viscosity, obtained on the basis of hydroxypropyl methylcellulose (HPMC), hydroxyethyl methyl cellulose (HEMC) and methylethylhydroxyethyl cellulose (MEHEC), on the change of adhesion to the substrate, consistencies and water retention properties were investigated. The research was carried using the original recipe of renovation plasters.

2.1. Tested material.
An experiment was carried out consisting in preparing dry mixes of renovation plasters containing tested cellulose admixtures in the amount of 0.3% against all dry ingredients. The admixture at the level of 0.3% was selected based on preliminary tests which had shown that this is the optimal amount of admixture that allows to clearly assess its impact on the analyzed mortar property. Properties, the method of cellulose admixtures modification and their viscosities determined in 2% solutions by Brookfield method are presented in table 1.

| Type of admixture | Brookfield viscosity 2% solutions [mPa·s] | Content of methoxy groups (OCH₃, %) | Content of hydroxyethoxy groups (OC₂H₄OH, %) | Content of hydroxypropoxyl groups (OC₃H₆OH, %) |
|------------------|------------------------------------------|-----------------------------------|----------------------------------|----------------------------------|
| HPMC1            | 6 500                                    | 24 – 30                           | –                                | 4.0 – 10                         |
| HPMC2            | 20 000                                   |                                   |                                  |                                  |
| HPMC3            | 28 000                                   |                                   |                                  |                                  |
| HPMC4            | 52 000                                   |                                   |                                  |                                  |
| HPMC5            | 67 000                                   |                                   |                                  |                                  |
| HEMC1            | 6 500                                    | 19 – 30                           | 5.0 – 8.0                        |                                  |
| HEMC2            | 14 000                                   |                                   |                                  |                                  |
| HEMC3            | 36 000                                   |                                   |                                  |                                  |
| HEMC4            | 65 000                                   |                                   |                                  |                                  |
| MEHEC1           | 6 000                                    | 18 – 28                           | 4.0 – 7.0                        |                                  |
| MEHEC2           | 15 000                                   |                                   |                                  |                                  |
| MEHEC3           | 31 000                                   |                                   |                                  |                                  |
| MEHEC4           | 60 000                                   |                                   |                                  |                                  |

The composition of the renovation mortars is shown in Table 2. The mortars were prepared in a laboratory mixer, which is described in PN-EN 196-1:2006 standard [15]. The method of samples mixing was in accordance with PN-EN 998-1:12 standard [16]. After adding water, the mortars were mixed mechanically for 30 seconds, then manually for 60 seconds using a trowel, and then for 180 seconds mechanically. The agitator speed was 160/45 rpm.

2.2. Methodology of research on the influence of cellulose admixtures on the change of renovation plasters consistency
The determination of the consistency of the fresh mortars was made using the flow table method in accordance with PN-EN 1015-3:2006 standard [17]. The consistency tests were carried out after 30 minutes from the moment of mixing dry ingredients with water. This is conventionally the time that elapses from mixing the ingredients with water until the mortar is applied in working conditions. In laboratory practice of in mortar design, it is convenient to express the effect of admixtures on the consistency with the amount of water that should be added to the dry ingredients in order to obtain the same consistency.
Table 2. The compositions of the test recipe

| Component                        | Weight fraction (%)<sup>a</sup> | Description            |
|----------------------------------|---------------------------------|------------------------|
| CEM I 42.5 Portland Cement        | 24                              | Binder I               |
| Hydrated lime                    | 7                               | Binder II              |
| Limestone powder                 | 8                               | Crushed stone I        |
| Quartz sand 0.0 ÷ 0.5 mm         | 58.11                           | Crushed stone II       |
| Cellulose admixture              | 0.3                             | Thickener              |
| Pearlite EP 150                  | 2.5                             | Lightweight aggregate  |
| Sodium α-olefin sulfonate        | 0.08                            | aerating admixture     |

<sup>a</sup> All amounts are given in relation to all dry ingredients

2.3. Methodology of research on the influence of cellulose admixtures on the ability to retain water in fresh renovation plaster

The retention of water in fresh mortar is described by the percentage of water that remains in the mortar after a brief draining of water through the filter paper [18]. In laboratory conditions, the measurement consisted of placing fresh mortar in a plastic ring with a diameter of 140 mm and a thickness of 10 mm, set on a filter paper and a plate also made of plastic. The mortar flow in the ring was covered with a plastic plate and left for 5 minutes. The amount of water adsorbed by the filter paper was adopted as the measure on the basis of which the water retention value (WRV) was calculated as a percentage determined from equation (1):

\[
WRV = 100 - W
\]

where: WRV - water retention value, W - relative loss of water in the mortar (%).

The water content in mortars was constant and amounted to 25% in relation to all dry ingredients, the w/c ratio was 1.04.

2.4. Methodology of research on the influence of cellulose admixtures on the adhesion of hardened mortars to the substrate

The adhesion of renovation plasters was determined in accordance with PN-EN 1015-12:2016-08E standard [19], as the value of critical stress during which adhesive or cohesive detachment of the layer of applied material from the substrate took place. For this purpose, the mortar was applied to a thickness of 10 mm on a concrete block. After 28 days of seasoning, 50 mm diameter discs were cut out of the plaster. After cleaning the surface of the samples, five steel discs were attached with epoxy glue at the points of incision. The critical strain was measured using the "Pull-off" device on the ProceqDyna Pull-off Tester Z-16. The consistency of the mortars was constant at the flow of 170 mm±5 mm determined by the flow table test.

3. Research results and discussion

The test results are shown in table 3.

3.1. Study of the influence of cellulose admixtures on the consistency of mortar

The results of the influence of cellulose admixtures, differing in the modification and viscosity, on the consistency change determined using the flow table method are shown in Figure 2. To quantify the
influence of the admixture on consistency, the amount of mixing water which must be added to the mortar in order to obtain a consistency of the mortar had without cellulose admixture, which was 170±5mm (determined by the flow table test).

**Table 3.** Research results showing the influence of chemical composition and viscosity of cellulose admixtures on consistency determined by the shock table method, water retention value (WRV), adhesion to concrete substrate and air content in fresh mortar.

| Type of admixture | Brookfield viscosity 2% solutions (mPa·s) | Flow (mm) at c/w=1.04 | Addition of water (%) at 170mm flow | Water retention value WRV (%) | Adhesion to the substrate (N/mm²) | Air content in fresh mortar (%) |
|-------------------|---------------------------------|-----------------|----------------------------------|-----------------------------|---------------------------------|--------------------------------|
| References threshold | –                              | 170             | 25                               | 75                          | 0.59                            | 26.5                           |
| HPMC1             | 6500                           | 144             | 26.6                             | 86                          | 0.72                            | 23.1                           |
| HPMC2             | 20000                          | 137             | 29.6                             | 96                          | 0.76                            | 26.0                           |
| HPMC3             | 28000                          | 134             | 31                               | 96.4                        | 0.79                            | 25.9                           |
| HPMC4             | 52000                          | 124             | 32.3                             | 97                          | 0.86                            | 25.8                           |
| HPMC5             | 67000                          | 112             | 33.5                             | 97.5                        | 0.85                            | 25.5                           |
| HEMC1             | 6500                           | 148             | 26.3                             | 86                          | 0.69                            | 26.4                           |
| HEMC2             | 14000                          | 137             | 28                               | 96                          | 0.71                            | 26.2                           |
| HEMC3             | 36000                          | 126             | 31.4                             | 97                          | 0.78                            | 25.8                           |
| HEMC4             | 65000                          | 115             | 33.1                             | 97                          | 0.82                            | 25.6                           |
| MEHEC1            | 6000                           | 160             | 25.9                             | 85                          | 0.64                            | 26.5                           |
| MEHEC2            | 15000                          | 150             | 27                               | 90                          | 0.68                            | 26.1                           |
| MEHEC3            | 31000                          | 146             | 28.5                             | 92                          | 0.76                            | 28.9                           |
| MEHEC4            | 60000                          | 127             | 29.8                             | 97                          | 0.72                            | 25.7                           |

**Figure 2.** Influence of the type of cellulose admixture on the amount of mixing water necessary to obtain a consistency of flow 170±5 mm, determined by the flow table method (a), the relationship between the viscosity of cellulose admixtures, determined for 2% solutions (Brookfield viscosity gauge) and the amount of mixing water (flow 170±5 mm) (b).

Analysing the results presented in figure 2a, it can be concluded that with the increase in viscosity of cellulose admixtures, the consistency of fresh mortar increases unevenly. The greatest changes in the amount of mixing water were observed when the viscosity changed from 6,000 MPa·s to 28,000 MPa·s for HPMC, from 6,500 to 36,000 MPa·s for HEMC, and in the case of MEHEC in the viscosity change from 6,000 to 31,000 MPa·s. It was also found that the degree of influence of individual admixtures on mortar consistency depends on their type of cellulose admixture. The greatest influence on the consistency was that of HPMC and HEMC-based admixtures, figure 2b.
Furthermore, the air content in fresh mortars was measured in accordance with PN-EN 1015-7:2000P standard [20]. According to the WTA instruction [21], the air content for renovation plasters should not be less than 25%. The cellulose admixtures and the increase in mortar consistency associated with their presence reduced the air content in the fresh mortar. For mortar containing no cellulose admixture, the air content was 26.5%. With increasing viscosity of admixture and a constant w/c, the air content slightly decreased, figure 3.

![Figure 3. Influence of viscosity of cellulose admixtures on the change of air content in fresh mortar at a constant w/c of 1.04](image)

### 3.2. Study of the influence of cellulose admixtures on water retention in mortar

Figure 4a. shows changes in water retention depending on the viscosity of cellulose (for 2% solutions, Brookfield viscosity gauge).

![Figure 4. Dependence of WRV water retention value on viscosity of cellulose admixtures (Brookfield 2% solution) (a), dependence of water retention on consistency of mortars determined using a flow table test (b)](image)

It can be concluded that there is a relationship between the viscosity of cellulose admixtures and the WRV water retention by the mortar, figure 4a. This effect is uneven, with the greatest intensity observed when the viscosity of cellulose admixtures changed from 6,000 mPa·s to 20,000 mPa·s in the case of HPMC, from 6,500 to 14,000 mPa·s for MEHEMC, and from 6,000 to 31,000 mPa·s. A further increase in the viscosity of cellulose admixtures had an increasingly lower effect on the change in WRV.

Analysing the obtained results, it can be stated that the HPMC- and HEMC-based admixtures had the greatest influence on the water retention of renovation mortars. For the mortar containing no
cellulose admixtures, the value of water retention was approx. 75%. For those above-mentioned admixtures with a viscosity over 15,000 mPa·s, water retention values in mortars exceeded 95%. Figure 4b. shows the changes in water retention depending on the consistency of the mortar. Analysing the graph, it can be concluded that cellulose admixtures with a comparable viscosity have a different effect on the consistency, and a different effect on WRV water retention value. Although the increase in viscosity of admixtures above 15,000 mPa·s clearly influenced the change of mortar consistency, it did not affect the water retention capacity. The increase in the viscosity of admixtures from 15,000 to 65,000 mPa·s, the resulted in WRV coefficient results being in a narrow range from 96 to 97%.

3.3. Study of the influence of cellulose admixtures on adhesion to a concrete substrate
The results of the influence of cellulose admixtures, differing in the modification method and viscosity, on the adhesion to substrates are presented in Figure 5.

![Figure 5](image)

**Figure 5.** Dependence of the adhesion of renovation plasters on the type of cellulose admixtures (a), dependence of the adhesion of renovation plasters on the viscosity of cellulose admixtures, determined for 2% solutions (Brookfield viscosity gauge) (b), at flow 170±5mm, determined by the flow table test

Analysing the results presented in Figure 5a, it can be concluded that with increasing viscosity of cellulose admixtures, adhesion to substrates increases. The influence depends on the type of cellulose admixtures. Across the entire range of admixture viscosities, the HPMC and-based admixtures had the greatest impact on adhesion. The HEMC effect was slightly lower, although, in this case, the adhesion increases with the increase of the admixture viscosity. Admixtures based on MEHEC influenced the adhesion in a very diverse way. The highest adhesion was obtained for MEHEC with a viscosity of 31,000 mPa·s. Further increase in the viscosity of this admixture caused a slight decrease in adhesion.

The standard requirements for the adhesion are mainly determined for repair mortars for concrete and tile adhesives. In the case of renovation plasters, similarly to the other plasters, a value equal to the tensile strength of the substrate is assumed to be sufficient adhesion. Based on the opinion of practitioners, sometimes, despite the fulfilment of mandatory requirements, not all renovation plasters offered by commercial manufacturers show sufficient adhesion. In particular, this applies to the use of renovation plasters on poor substrates which are quite common in the renovation of objects of historical importance. The author's technological practice shows that for renovation plasters, the adhesion should not be lower than 0.5 MPa, with the optimal value at 0.8 MPa. In the aspect of the renovation plaster under consideration, the highest adhesion was obtained for HPMC 52000.

4. Conclusion
The paper presented the influence of cellulose admixtures on consistency, water retention capacity and adhesion to substrates. As a result of the examination, it was found that due to the shaping of physical properties of renovation plasters, which were analysed in this paper, the best results can be obtained with (hydroxypropyl)methyl cellulose HPMC and (hydroxyethyl)methyl cellulose HEMC, particularly:
• With the increase in viscosity of cellulose admixtures, the consistency of fresh mortar increases unevenly. The greatest changes in consistency were observed for HPMC and HEMC when the viscosity changed, from approx. 6,000 MPa·s to approx. 30,000 MPa·s; further increase in the viscosity of cellulose admixtures influenced the mortar consistency to a lesser extent.

• There is a relationship between cellulose viscosity and water retention by the mortar. This effect is uneven, with the greatest intensity observed when the viscosity of cellulose admixtures changed from 6,000 mPa·s to 15,000 mPa·s. The influence of cellulose admixtures with higher viscosities on the change in water retention was increasingly lower.

• Adhesion of mortars containing cellulose ethers was higher than that of unmodified mortars. For the analysed renovation plaster, the highest adhesion was obtained for HPMC 52000.

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