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Effect of High Molecular weight Cellulose Ethers on the Resistance in Shearing of Fresh Mortars

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Abstract: This paper presents an experimental study on the effect of three types of water-soluble high molecular weight polymers on the resistance in shear of cement mortars in fresh state. The role of molecular weight of cellulose ethers in modifying the yield strength of mortars in shear have also been investigated. To determine the rheological properties the Vane-Cylinder test was used. Hershel Bulkley model has been used to determine the rheological parameters, including yield stress. It is found that at low shear rates, all the mortar mixes behaved as a shear-thinning fluid. However at high shear rates, we observed a difference between the mixes corresponding to the different cellulose ethers. The investigation of the influence of molecular weight on the properties of fresh mortars has shown a similar observation to the reported research in literature. The yield stress of the mortar decreases with the increase of molecular weight. This decrease is not significant at low molecular weights, and becomes much more significant at high molecular weights.

Keywords: Mortar; High molecular weight, Yield strength; Cellulose.

INTRODUCTION

The term 'cellulose ether" refers to a wide range of commercial products and differs in term of substituent, substitution level, molecular weight (viscosity), and particle size. The most widespread cellulose ethers used in dry mortars as admixtures are methyl cellulose (MC), methyl-hydroxyethyl cellulose (MHEC) and methyl-hydroxypropyl cellulose (MHPC) [1].

According to their properties, cellulose ethers are used in various industrial fields, including food industry, pharmaceutical industry, in paints and adhesives, etc. They significantly modify the properties of materials even if they are introduced in small amounts (0.02-0.7 % [1]).

In mortar, cellulose can be added before or during the mixing as thickening and water retaining agents. However, the effect of cellulose ethers on the mortar in fresh state was not fully studied [2]. For example, there are few studies on the effect of methyl-hydroxyethyl cellulose (MHEC) on the resistance of fresh mortar in tension and in shear.

Cellulose ethers such as hydroxyethyl methyl cellulose (HEMC) is a common admixture in factory made mortars for various applications including cement spray plasters, tile adhesives, etc. The influence of HEMCs have been published by many researchers in the case of various application fields, such as biological macromolecules [3-5], carbohydrate polymers [6-8], etc. However, there are few published studies concerning the influence of HEMCs on the fresh state properties of cementitious materials including cement grouts [9, 10], cement-based mortars [11].

Patural *et al.*, [9] had investigated the influence of cellulose ether on the properties of mortars in fresh state, in which the molecular weight of polymers is rather low (90-410 kDa). The effect of high molecular weight cellulose ether hasn't been studied. Thus, it is interesting to deal with high molecular weight cellulose ether in order to complement the effect of molecular weight of cellulose ether on the properties of fresh mortars.

The influence of high molecular weight cellulose ether on the properties of fresh mortars had been investigated, which indicated an important role of molecular weight of cellulose ether on controlling the adhesion force, the cohesion force and the interface adherence [2]. In this paper, the influence of three types of HEMCs on the yield strength of fresh mortar in tension and in shear will be examined.

MATERIALS AND EXPERIMENTAL METHODS

Mix-design

The binder comprises a Portland cement (CEM I 52.5 N CE CP2 NF from Teil-France) and a hydraulic lime (NHL 3.5Z). In order to minimize phase separation, the standard sand CEN EN 196-1 ISO 679 has been used. In this study, the effect of three types of high molecular weight cellulose ethers have been investigated. Typical characteristics of HEMCs are introduced in Table-1.

Table-1: Typical physical characteristic of three types of HEMCs

Properties	Type A	Type B	Type C
Form	Powder	Powder	Powder
Solubility	Water soluble	Water soluble	Water soluble
Viscosity(1), mPA.s	20000	30000	70000
pH (2% solution)	Neutral	Neutral	Neutral
Molecular weight	600.000	680.000	1.000.000

(1) solution in water, Haake Rotovisko RV 100, shear rate 2.55 s^{-1}*, 20°C*

A certain dosage rate of a commercial air-entraining agent, NANSA LSS, is used to guarantee moderate rheological properties within the resolution range of the rheometer.

The weight proportion of each constituent of the mortar is represented in Table-2.

Table-2: Mortar formulation

Constituent	Cement	Lime	Sand	Air entraining	HEMC	Water
% wt. of dry mixture	15	5	80	0.01	0.19-0.31	19

The polymer content in the mortar formulation is varied according to the following proportions: Ce =[0.19; 0.21; 0.23; 0.25; 0.27; 0.29; 0.31] % by weight. The water dosage rate is fixed to 19% by weight for all the investigated samples. The mortar composition corresponds actually to a basic version of commercially-available render mortar [2].

Vane – Cylinder test

For characterizing the rheological properties of the fresh mortars, the rheometer AR2000ex is equipped with 4-blade vane geometry. Vane geometry is appropriate for high yield stress fluids such as dense granular suspensions, including mortars [11], as slippage can be avoided and the material can be sheared in volume.

The yield stress is measured with the vane-cylinder geometry in stress controlled mode in which a "ramp" of steps of increasing stress levels is applied to the vane immersed in the material, and the shear rate is measured as a function of applied stress. The yield stress is determined from the critical stress at which the material starts to flow.

Depending on each specific experiment, test will be performed at least three times to determine the best possible experimental procedure. In the first run, the interval between two successive steps must be chosen large enough to reduce the duration of the test. The yield stress is determined, but with a low precision. For latter runs, the measuring points must be increased around the determined yield point. That would help to determine a high accuracy yield stress of the materials.

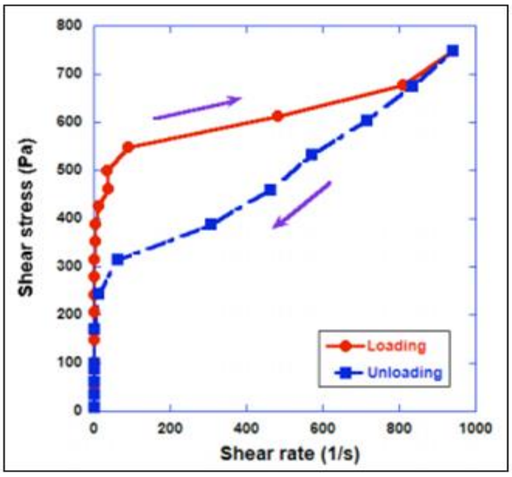


Fig-1: Typical flow curves of mortar with the addition of 0.29% of polymer

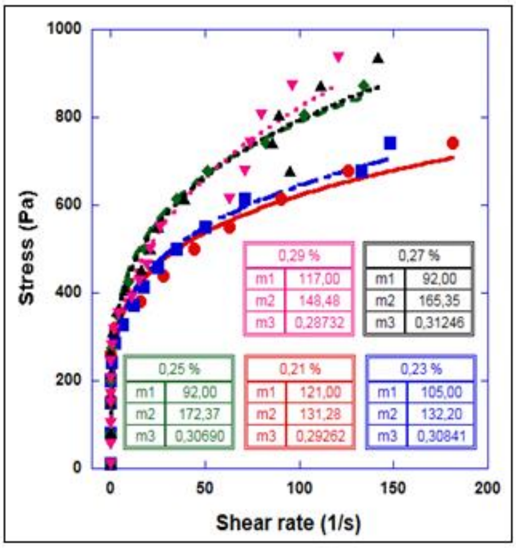


Fig-2: Perform the best fit of flow curves to Herschel-Bulkley models

A typical curve obtained in the rheology test is presented in Figure 1. The yield stress is determined by the critical stress at which we observe the transition from solid state to liquid state of the material. However, in actual experiments, almost all cases, the transition from solid to liquid state is occurred gradually and is hard to detect. Therefore, it is difficult to determine the exact value of the yield stress. So, different models have been developed in order to determine the value of the yield stress as well as other rheological parameters by fitting the flow curves' data with the model's equation. In this study, the most general models for concentrated suspensions, Herschel-Bulkley's, which is characterized by the following equation, has been used:

$$\tau = \tau_0 + K.\dot{\gamma}^n$$

The consistency coefficient K, the fluidity index n, and the yield stress τ_0 are three parameters characterize Herschel-Buckley fluids. In some cases the use of Herschel-Bulkley model leads to non-physical values of the yield stress (negative), this parameter is then determined by the applied stress at which we obtained a finite shear rate (0.01 s^{-1}). Figure 2 shows the best fits of flow curves to Herschel-Bulkley models in the variation content of A, in which m1=yield stress.

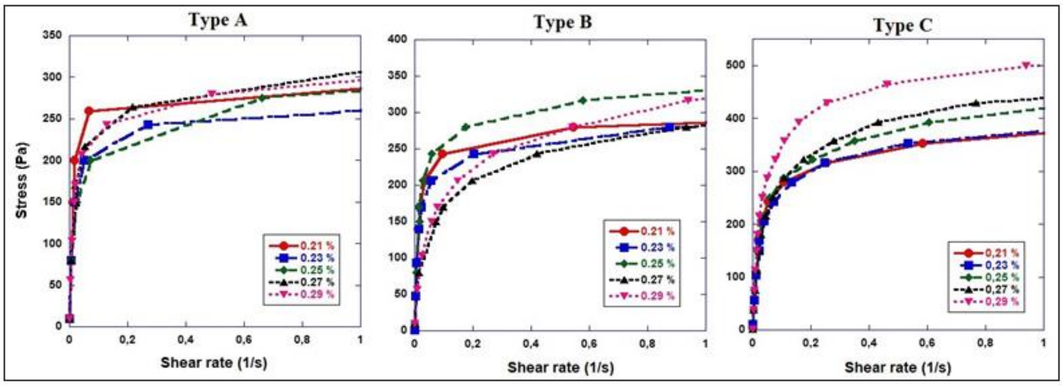
EXPERIMENTAL RESULTS

Effect on the rheological properties

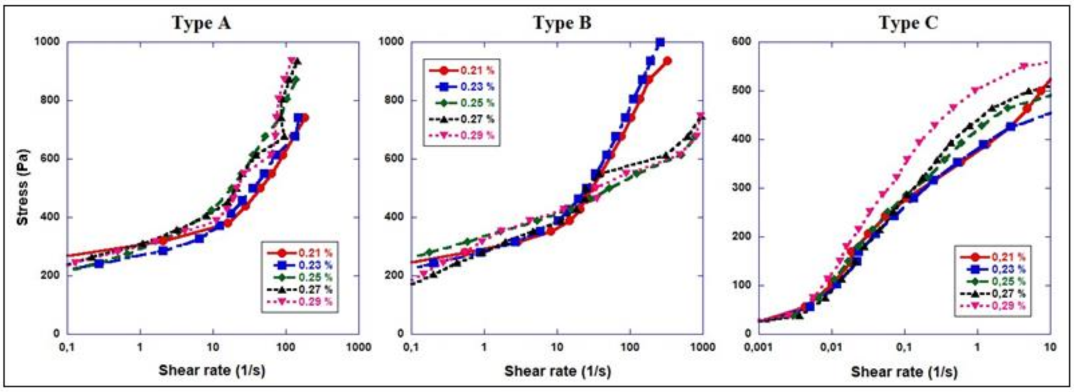
A comparison of the loading curves corresponding to different polymer contents is presented in Figure 3, both in linear and in logarithmic scale. A qualitative similarity of the rheological behaviour with the increasing of polymer content has been observed. The flow curves indicate that the mortar pastes behave as a shear thinning fluid with a yield stress.

Considering the evolution of the applied stresses as a function of recorded shear rate at some given stresses and for different polymer contents of B, we can see that: At certain stress, for instance 600 Pa, the recorded shear rates are about 60 s-1 for 0.21%, and 500 s-1 for 0.25% and 0.29%. This indicates that for certain given applied stresses, the recorded shear rates increases with the increase of polymer content. This observation is inverse to that in case for mortars with polymer A. The crossover of the flow curves indicates that the evolution of the apparent viscosity (stress divided by shear rate) versus polymer content is dependent of the shear-rate interval considered. This may be attributed to the different antagonistic effects of the polymer.

In case of C, the mortar rheological behavior is close to that of a Bingham fluid. The mortars are shear thinning at lower polymer content. However if we zoom in the flow curves around low shear rates (see figure 3b) we can observe that the mortar behave rather as Herschel-Bulkley shear-thinning fluids for all the dosages rates. This change in the rheological behavior of mortar pastes at low and high shear rates is represented by the evolution of the rheological parameters, including yield stress, which will be discussed in the following.



a) Linear plot



b) Logarithm plot

Fig-3: Flow curves comparisons with the variation of polymer contents

Evolution of the yield stress in shear

The yield stress in shear are determined by performing the best fits of the loading curves with the Herschel-Bulkley model. Figure 4 represents the evolutions of the yield stress as a function of polymer content, in case of different polymer type.

It can be seen that an optimum is observed in the evolution of the yield stress with the variation of polymer content. The yield stress reaches a minimum for a content of 0.25%. The observation of such a minimum has already reported by several authors concerning other types of mortars [9, 10]. This has been attributed to the air-entraining effects of cellulosic ether polymers [10]. In fresh state, the air bubbles in the mortar may lead to an increase of the resistance to flow initiation due to capillary forces. However, these bubbles along with the lubrication effects of the polymer would decrease the resistance to flow initiation due to decrease of granular contacts. These effects have opposing impacts. The interplay between them would lead to the appearance of minimum value in the resistance to flow initiation.

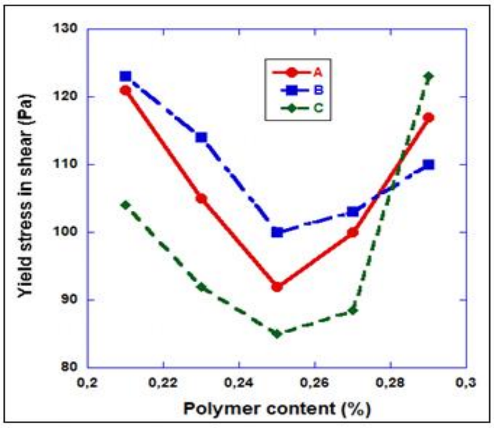


Fig-4: Evolutions of the yield stress as a function of polymer content, in case of different polymer types

Influence of the molecular weight Mw on the yield stress

The effect of molecular weight on the yield stress of the mortar is highlighted in Figure-5. It can be seen that we observe an evolution with an optimum for a concentration of 0.25 % independently of the molecular weight. As discussed in the previous sections, several authors have reported the presence of such a minimum and this has often been attributed to the air-entraining effects of cellulose ether polymers. There is no direct correlation between the depth of the minimum and the molecular weight.

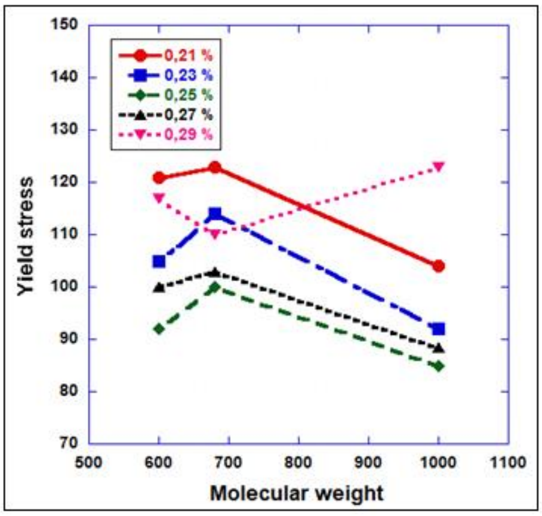


Fig-5: Evolution of yield stress in shear for the variation of molecular weight

The figure-5 shows the dependency of the yield stress on the molecular weight. Increasing the molecular weight first leads to a slightly increase of the yield stress to reach a maximum value, followed by decrease of the yield stress. These trends are observed for all the polymer concentrations expect the highest one (0.29%). For this dosage, the maximum transforms into a minimum.

CONCLUSIONS

The evolution of the resistance of mortar in shearing has been investigated by varying the content of three types of hydroxyethyl methyl cellulose denominated A, B and C. These polymers differ from each other mainly in their molecular weights.

At low shear rates, all the mortar mixes behaved as a shear-thinning fluid. However at high shear rates, we observed a difference between the mixes corresponding to the different cellulose ethers. In case of A, the mortar pastes behave as shear-thinning fluids for all investigated concentrations. In case of B, the rheological behavior of mortar is shear thinning at low concentrations, while it behaves as Bingham fluids at high contents. In case of C, the mortars behaved much like Bingham fluids through the entire shear-rate interval investigated.

The investigation of the influence of molecular weight on the properties of fresh mortars has shown a similar observation to the reported research in literature. The yield stress of the mortar decreases with the increase of molecular weight. This decrease is not significant at low molecular weights, and becomes much more significant at high molecular weights.

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