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Evaluation of various cellulose ethers performance in ceramic tile adhesive mortars

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ABSTRACT

Eleven cellulose ethers (CE) were selected and tested in a cement based ceramic tile adhesive (CTA) formulation designed to highlight the effect of the CE on the end-use values of the mortars. Results showed that the end-use values, either in the fresh or hardened state, are strongly influenced by the latex powder/CE choice. This is due to the competitive adsorption between these two components; in the fresh state, it affects the CE concentration in the pore solution and hence the viscosity of the mortar, setting time, and skinning. In the hardened state, when CE films do not prevent evaporation at the tile–mortar interface, adhesion in hot curing conditions is lowered compared to dry tensile adhesion values. Test results showed that CTA formulations should be thought of in terms of the CE/latex powder couple, since interactions between this couple and the cement, strongly influence end-use values of the mortar.

1. Introduction

Adhesive mortars are widely used to fix ceramic tiles as decorative finishes or for fixing polystyrene sheets onto building walls as part of external insulation systems; this is because their bond strength and flexural properties increase the cladding's lifetime. To achieve the required adhesive properties, polymer dispersions are added to the mortar formulation. Liquid polymer dispersions can be spray-dried and used as a powder component in dry-mix mortar formulations. When mixed with water, the powder redisperses and forms films in the cement matrix, which further enhance the mechanical properties of cementitious mortars.

Cellulose ethers are commonly introduced into industrial mortar formulations in order to obtain some of the required properties of the adhesive mortar; from the fresh paste to the hardened material [1]. These cellulose derivatives are molecules which improve the water retention and workability of the fresh material by a process of air entrapment, as noted in Fig. 1. They also improve the adhesion to substrates [2]. Among the wide variety of existing cellulose ethers, four types are commonly used in mortar manufacturing: methyl cellulose (MC), hydroxypropylmethyl cellulose (HPMC), hydroxyethylmethyl cellulose (HEMC) and hydroxyethyl cellulose (HEC) (Fig. 1). The chemical nature of HEC is determined mainly by two parameters i.e. the molecular weight (Mw) and the hydroxyethyl content (% OC_2H_4OH). Conversely, HPMC and HEMC are characterised by three structural parameters i.e. the molecular weight, the methoxyl content (% OCH_3) the hydroxypropyl (in HPMC, % OC_3H_6OH) or the hydroxyethyl content (in HEMC, % OC_2H_4OH).

However, one negative aspect of the use of these macromolecules in mortar formulation is the delay in cement hydration [3–5]. Pourchez et al. highlighted various delays in cement hydration induced by cellulose ether (from 10 min up to several hours) [6,7]. This delay seemed to depend mainly on the chemical structure of the molecule and in particular, on the degree of substitution. It was shown that the adsorption of the polymers onto the cement clinker phases inhibits the formation of portlandite [8]. It was assumed that the CE strongly effects hydrated calcium silicate precipitation, leading to a decrease in the amount of initial hydrated calcium silicate nuclei, delaying the formation of a continuous hydrated calcium silicate shell around the tricalcium silicate grain, and finally delaying the formation of a thicker and more permeable hydrated calcium silicate layer. When a CE is added to a cementitious matrix, a gradual reduction of tricalcium aluminate dissolution rate is observed; this is also associated with ettringite and calcium hydroaluminate precipitation. Hydroxyethyl cellulose induces a stronger adsorption on calcium hydroaluminates and a longer tricalcium aluminate hydration delay than does hydroxypropylmethyl cellulose [9]. The lower the content of methoxyl groups, the larger will be the retardation of cement setting time.

Abbreviations: CE, cellulose ether; MC, methyl cellulose; HPMC, hydroxypropylmethyl cellulose; HEMC, hydroxyethylmethyl cellulose; HEC, hydroxyethyl cellulose; RDP, latex redispersible powder; CTA, ceramic tile adhesive

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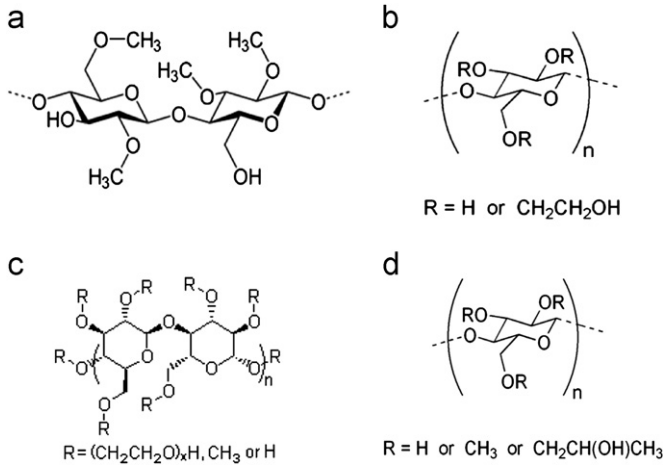

Fig. 1. Structure of cellulose ethers: (a) MC, (b) HEC, (c) HEMC, (d) HPMC.

Table 1
Mix design for ceramic tile adhesive.

Component	CTA mix design
CEM I 52,5 N cement (mass % of dry mixture)	36
Silica sand (mass % of dry mixture)	58.36
Calcium carbonate (mass % of dry mixture)	2
Cellulose ether (mass % of dry mixture)	0.34
Calcium formate (mass % of dry mixture)	0.3
RDP (mass % of dry mixture)	3
Water/powder (mass %)	25

More generally, the properties of the CE depend on both the molecular weight of the polymer and on the degree of etherification. The mechanical properties of the ethyl cellulose are reported to be influenced more by the former, whereas other physical properties such as solubility and water absorption are influenced more by the latter [10]. Such information is not generally provided as it is considered to be confidential by CE manufacturers; however, it has been demonstrated that the average molecular weight of the polymer is linked to its solution viscosity, when it is dissolved in water at a set concentration and reference temperature [11].

The present study aims at evaluating the effect of CE type on the fresh paste parameters of adhesive mortars for ceramic tile, formulated with a latex redispersible powder (RDP). Such data are necessary to highlight some of the mechanisms affecting workability of RDP mortars over time and which will affect their use on construction sites.

2. Materials and mixture proportioning

A typical C2TE ceramic tile adhesive formulation was used to investigate the effect of various CEs on RDP mortars (Table 1). C2TE refers to the fresh and hardened adhesive properties as defined in EN 12004 and ceramic tile adhesive refers to a fine inorganic aggregate Portland cement based mortar modified with polymer material (redispersible powder) capable of adhering two other materials together by means of surface attachment.

In this investigation, a redispersible powder based on a modified vinyl acetate copolymer was used. This polymer had neither carboxylic nor hydroxyl groups on its surface, minimising any possible interactions between the RDP and the CE. Polyvinyl alcohol was added at the end of the production process as a drying aid to assure the redispersibility of the powder.

Eleven sets of CEs were selected: 2 MC-based, 6 HEMC-based, and 3 composed of a mixtures of HEMC and HPMC. The viscosities of a 2% solution of each ranged from 5000 to 44500 mPa.s. Data on the cellulose ether are summarised in Table 2. The viscosities of the CE solutions were taken from the technical data sheets of the manufacturer. All viscosity measurements of the polymer solutions (concentration at 2% by mass) were done using a Brookfield viscometer, with an RV type spindle turning at 20 rpm and at a set temperature of 23 °C.

Table 2
Cellulose ether employed in this investigation according to their type and use.

CE Type	Code	viscosity of the 2% CE solution (mPa s)
MC	MC1	5500
	MC2	13,500
HEMC	HEMC1	5000
	HEMC2	14,000
	HEMC3	25,000
	HEMC4	25,000
	HEMC5	30,000
	HEMC6	44,500
Mixed HEMC/ HPMC	Mixed1	6000
	Mixed2	15,000
	Mixed3	17,000

Table 3
Characteristics of the CEM I 52.5 N cement.

Chemical characteristics (%, in mass)		Physical characteristics	
SiO_2	20.0	specific gravity (g/cm^3)	3.1
Al_2O_3	5.1	Blaine specific area (cm^2/g)	4200
Fe_2O_3	3.3	Compressive strength (Mpa)	
CaO	63.9	2 days	24
MgO	0.8	7 days	37
SO_3	3.1	28 days	62
K_2O	0.73	Vicat setting time (min)	
Na_2O	0.32	Initial	160
S^-	0.03	final	240
Cl^-	0.06		
CO_2	1.1		
Free CaO	1.8		
Na_2O eq	0.8		
L.O.I	2.0		
Clinker	97.0		
other	3		

The cement used was a CEM I 52.5 N complying with the EN 196-1 standard, with a Blaine fineness of 4200 cm^2/g and composed mainly of clinker. Physical and chemical characteristics of the cement are summarised in Table 3.

3. Test methods

As mortar properties can be affected by the mixing procedure and equipment and in order to have comparable results, the same mixer and the same mixing sequence were used for each set of mortars. The RDP and cellulose ether were incorporated into the cement prior to mixing. The mortars were prepared in batches of 2 l using a helicoidal mixer rotating 62 rpm. The mixing consisted of adding the water into the mixer and then introducing the powders (binder, redispersible powder, cellulose ether and sand) gradually over 30 s with the mixer off. The mortar was then mixed during 30 s at a speed of 62 rpm. After a rest period of 30 s, the mixing was resumed for an additional 90 s at 62 rpm. Prior to sampling, the mixture was left at rest for 5 min before a final 15 s homogenisation mixing. The raw materials were conditioned to assure the targeted test temperature of the mixed mortars. In order to avoid heat loss or