



Effect of polyacrylamide on rheology of fresh cement pastes



Hela Bessaies-Bey^a, Robert Baumann^b, Marc Schmitz^b, Michael Radler^b, Nicolas Roussel^{a,*}

^a IFSTTAR, Laboratoire Navier, Université Paris Est, France

^b Dow Construction Chemicals, Horgen, Switzerland

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ABSTRACT

In this paper, we study the effects of polyacrylamide (PAM) on both aqueous solutions and fresh cement pastes. Our solution viscosity and hydrodynamic radii measurements in various solutions suggest that calcium ions lead to a cross-linking of anionic PAM and to the formation of PAM micro-gels. Our adsorption measurements show that the majority of these micro-gels adsorb on cement grains whereas our rheological measurements on cement pastes suggest that these micro-gels are able to adsorb simultaneously on several cement grains and bridge them increasing therefore the macroscopic yield stress of the suspension. Finally, we show that the contribution of PAM to yield stress is very sensitive to shear history, as the paste structure seems to progressively lose its ability to rebuild with each shearing or re-mixing cycle. We suggest that this feature could find its origin in the progressive flattening of the micro-gels at the surface of the cement grains.

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1. Introduction

Polyacrylamides (PAM(s)) are water-soluble polymers, the molecular structure of which can be readily tailored to provide a broad range of properties. They are therefore commonly used as thickeners or flocculants in various industries among which is the construction materials industry.

For instance, fiber cement composite production involves addition of PAM in order to tailor the flocculation properties to the casting process [1–4]. According to [5–7], PAM allows for an increase in the density of the resulting industrial product, which, in turn, is at the origin of an increase in mechanical strength. PAM is also used as an anti-washout admixture in concrete application as these molecules seem to be able to reduce bleeding and significantly increase fresh concrete cohesion when placed underwater [5,8,9]. Another example can be found in the field of mono-layer render mortar or tile adhesives, in which PAM along with cellulose ether is often added to improve sagging resistance (*i.e.* the ability of these products to stick to the supporting vertical wall and only slightly deform under their own weight) [10,11].

Compared to other chemical admixtures in the construction industry such as cellulose ethers [10–21] or poly-carboxylic ethers [22], the physical and chemical phenomena at the origin of the effects of PAM on cement paste or mortar rheology are still unclear and the use of this polymer is based on empirical data in most industrial practices. Within this frame, this work focuses on the mechanism of action of PAM in a cement suspension by combining microscopic measurement (adsorption at the surface of cement

grains and polymer conformation in various aqueous solutions) and macroscopic measurements (polymer aqueous solution viscosity and cement paste rheological properties).

We study here commercial PAM polymers with various molecular structures. We first study their viscosity enhancing effects in aqueous solutions along with their hydrodynamic radii in distilled water, calcium hydroxide solution and synthetic cement pore solution. Using Total Organic Carbon (TOC) measurements, we then measure the adsorption of these molecules at the surface of cement particles. From rheological measurements on cement pastes prepared with PAM, we discuss the changes in cement particle interaction forces in the presence of PAM. Finally, we show that the contribution of PAM to the fresh behavior is very sensitive to shear history as the paste structure seems to progressively lose its ability to rebuild with each shearing or re-mixing cycle.

2. Materials and experimental protocols

2.1. Materials

The cement used in this study is a Portland cement equivalent to ASTM Type I cement. Its chemical composition obtained through ICP-AES and ATD-ATG is given in Table 1. Its Bogue composition is also given in Table 1. The free lime was determined by extraction with hot ethylene glycol [23]. The cement maximum packing fraction was estimated to be around 60% in [24] and its Blaine specific surface is $3650 \pm 100 \text{ cm}^2/\text{g}$.

Anionic PAM(s) are hydrophilic polymers with high molecular weight (*i.e.* often more than one million Dalton). They are obtained by copolymerization of polyacrylamide with a carboxylic acid salt (*cf.*

* Corresponding author.

E-mail address: nicolas.rousseau@ifsttar.fr (N. Roussel).

Table 1

Chemical and Bogue composition of the cement studied here.

C ₃ S	C ₂ S	C ₃	C ₄ AF	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	Cl	CaO free	Ignition loss
64.00%	14.00%	2.50%	14.00%	21.04%	3.34%	4.14%	65.43%	0.83%	0.22%	0.35%	2.31%	0.02%	0.69%	1.53%

Fig. 1). Their water solubility comes from the strong solvation of their polar groups, either ionic or non-ionic. Four anionic polyacrylamides labeled M X/Y are studied in this work. X and Y correspond respectively to the molecular weight expressed in 10⁶ Da and to the degree of anionicity (in %) defined as the ratio of anionic monomers to the total number of monomers in the chain. The polymers studied here are commercial products from SNF Floerger in powder form. The monomers were polymerized in a gel form, ground and then dried. The molecular weight of the PAM molecules studied here was computed from calibrated capillary viscometry measurements. The anionic charges were measured by the reaction of a cationic colloid, a methyl glycol chitosan, which reacts stoichiometrically with the anionic charges. The excess was back titrated with potassium polyvinyl sulfate in the presence of toluidine blue. Their molecular parameters are gathered in Table 2. A polyethylene oxide (PEG) from Dow Chemical is also used in this work. It is a linear polymer, which is synthesized by ionic polymerization of ethylene oxide utilizing metal alkoxide compounds as catalysts [25,26]. For the PEG studied here, a molecular weight of 4 · 10⁶ g/mol and a hydrodynamic radius of 80 nm were obtained from rheological and dynamic light scattering measurements respectively.

2.2. Sample preparation

2.2.1. Polymer solutions

Aqueous solutions of PAM were prepared using various solvents: distilled water, calcium hydroxide solution, magnesium chloride solution and synthetic cement pore solution [27]. The polymer was dissolved in the solvent by magnetic stirring for 4 h. The solutions were prepared by adding to distilled water either 0.85 g/L Ca(OH)₂ or 1.08 g/L MgCl₂ whereas the synthetic cement pore solution was prepared by adding 1.72 g/L of CaSO₄ · 2H₂O, 6.959 g/L Na₂SO₄, 4.757 g/L K₂SO₄ and 7.12 g/L KOH. The pH levels of the calcium hydroxide solution and cement pore solution at 21 °C are respectively equal to 12.39 and 13.06. All chemicals used here were purchased from Sigma Aldrich.

2.2.2. Cement pastes

In order to focus on the influence of the presence of PAM on the rheology of cement pastes, we have chosen here a preparation protocol, which was shown to limit the consequences of any potential early chemical interaction between early hydrates and polymers [28]. The cement powder and 80% of water were first homogenized by hand and then mixed for 1 min using a Turbo-Test Rayneri VMI mixer at 840 rpm. The polymer dissolved in the 20% remaining water was added to the system 15 min after the first contact between the cement powder and water (*i.e.* we expect that most of the initial aluminates

have already nucleated). The cement paste was then mixed for 1 min. After 15 min of rest, the sample was finally mixed for 1 min before starting any measurements. In the specific case of the samples used further to study the effect of shear history on structuration, they were directly filled in the rheometer cup at the end of the 15 min resting time in order to acquire rheological data from the rheometer as early as possible through the material's history.

For all rheological measurements, cement pastes were prepared with a water to cement mass ratio (W/C) equal to 0.4. For adsorption measurements, they were prepared with a water to cement ratio (W/C) equal to 0.8 in order to facilitate the extraction by centrifugation of the interstitial fluid. Only dosage and nature of PAM varied from one sample to another. All PAM dosages are expressed in this paper as a mass percentage of water in the system. It can finally be noted that all mixing and testing protocols were carried out at 20 ± 1 °C.

2.3. Rheological measurements

2.3.1. Polymer solution viscosity measurements

The viscosity of polymer solutions was measured using a C-VOR Bohlin stress-controlled rheometer equipped with serrated parallel plates [29]. The diameter of the plates was 40 mm and the gap between the plates was 0.5 mm. The tested polymer solution was poured on the lower plate of the rheometer before the upper plate was set into position. The apparent viscosity was then measured as a function of shear rate. An increasing shear rate ramp from 1 s⁻¹ to 1000 s⁻¹ was applied during 1000 s followed by a similar decreasing ramp. As no thixotropic behavior was detected for the tested solutions in this study and, as increasing and decreasing shear rate ramps were therefore superimposed, only decreasing shear rate ramps are shown in the following. It should finally be noted that a special attention was paid to keep the temperature constant between each measurement (20 ± 0.5 °C in the present case). Finally, in order to prevent the evaporation of the solution, a water trap covered the measurement system.

2.3.2. Cement paste yield stress measurements

The cement paste yield stress measurements were carried out with a C-VOR Bohlin rheometer equipped with a spindle with Vane geometry [30]. The Vane tool diameter was 25 mm, the outer cup diameter was 50 mm and its depth was 60 mm. The cup of the rheometer was filled with the tested cement paste and the measurement sequence was started. The cement paste was first pre-sheared at 100 s⁻¹ during 90 s in order to bring it to a reference structuration state. After a resting time of 300 s, the measurement sequences started when a constant shear rate of 0.01 s⁻¹ was applied for 300 s.

Finally, the effect of the shear history on the yield stress behavior was studied by measuring the yield stress evolution of a sample submitted 14 times consecutively to the shear cycle described above

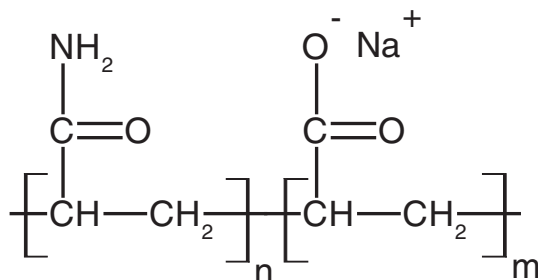


Fig. 1. Chemical structure of poly(acrylamide-co-sodium acrylate).

Table 2

Tested polyacrylamides molecular parameters.

Designation	Anionicity (%) m/(m + n)	Molar mass (10 ⁶ Da)
M8/05%	5%	8
M4/30%	30%	4
M10/30%	30%	10
M15/30%	30%	15

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