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Effect of sepiolite on the flocculation of suspensions of fibre-reinforced cement

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ABSTRACT

Sepiolite is used to increase thixotropy of cement slurries for easier processing, to prevent sagging and to provide a better final quality in the manufacture of fibre-reinforced cement products. However, the effect of sepiolite on flocculation and its interactions with the components of fibre cement are yet unknown. The aim of this research is to study the effects of sepiolite on the flocculation of different fibre-reinforced cement slurries induced by anionic polyacrylamides (A-PAMs). Flocculation and floc properties were studied by monitoring the chord size distribution in real time employing a focused beam reflectance measurement (FBRM) probe. The results show that sepiolite increases floc size and floc stability in fibre-cement suspensions. Sepiolite competes with fibres and clay for A-PAMs adsorption and its interaction with A-PAM improves flocculation of mineral particles.

1. Introduction

Since the prohibition to use asbestos fibres to manufacture fibre-reinforced cement, cellulose and poly(vinilalchohol) (PVA) have been commonly used as fibre-reinforcement in the Hatschek process.

The manufacture of fibre-reinforced cement composite sheets in the Hatschek process consists of the formation of a multilayer fibre-cement sheet by dewatering the fibre-cement slurry using a cylindrical sieve, a felt and an accumulation roll. First, a soft porous wet film of fibre cement is formed on the surface of the cylindrical sieve, which rotates in contact with a dilute water-based slurry of fibres, Portland cement and mineral materials. Some water is removed by vacuum through the sieve. Then, the solid wet film is transferred to a felt in which more water is removed by vacuum and pressure dewatering. The resulting layer is transferred to an accumulator roll by further dewatering at high pressure. The dewatering of the accumulated films in contact with each other binds the layers together forming a solid sheet. When the number of wrapped films on the accumulation roll is sufficient to form a fibre-cement sheet with the required thickness, the stack of films is removed, corrugated and carried to the curing section [1,2].

Therefore, fibres must interact with the mineral particles in the fibre-cement suspension to make possible the manufacture of a composite sheet with the required properties. The interaction among particles and asbestos fibres was strong due to the inorganic nature, morphology, density and surface properties of asbestos. However, the interaction of cellulose and PVA fibres with mineral particles is weak due to the organic nature and the low density of these fibres. Consequently, the use of a flocculant is usually required to retain mineral particles in the composite sheet [1–4], being anionic polyacrylamides (A-PAMs) the most frequently used agent [4–7].

However, phenolic groups from cellulose fibres extractives and carboxylic groups of flocculants can affect notably the hardening of cement and, therefore, the mechanical properties of the fibre-reinforced product [5,6,8]. To overcome the effect of cellulose fibres on cement hardening two different partial solutions have been developed: (1) to replace part of the cellulose fibres by PVA fibres, which are more expensive than cellulose but do not affect cement hardening (air curing), and (2) to speed up the cement hardening in an autoclave, by increasing temperature and pressure, without requiring the use of synthetic fibres (autoclave curing).

Furthermore, it is necessary to optimize both the flocculant properties and its dosage to minimize its effect on cement hardening.

Flocculants are not the only additives used in the manufacture of asbestos free fibre cement; minerals as microsilica, clay, aluminium oxide or sepiolite are used with different aims. Sepiolite is used to improve rheological properties and plasticity of fibre-cement suspensions, what reduces the irregularity of the sheet surface and avoids the separation of layers when the sheet is corrugated [1,2,8]. Controlling the viscosity of the fibre-cement slurry is essential to improve productivity of the Hatschek machine since the friction of the cylindrical sieve with the slurry increases with the viscosity of the latter [2]. Its effect on the viscosity of the slurry is due to the morphology of this mineral. Sepiolite particles, with a length of 2–10 μm, exhibit a porous micro-fibrous morphology and tend to aggregate in bundles. When they are dispersed in water, they form networks that improve rheology of the suspension and contribute to the homogeneous distribution of particles in the system [9]. This improves the process and the product quality. Significant improvements on mechanical properties (compressive and

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Table 1
Composition of studied fibre-cement mixes. Dry weights.

Raw materials	M1 (g)	M2 (g)
Cellulose	0.64	1.92
ASTM II cement	18.2	8.8
Microsilica	0.8	–
PVA	0.36	–
Silica	–	7.8
Al_2O_3	–	0.8
Clay	–	0.8

Table 3
Single component suspensions: weight of dry solids suspended in 400 mL.

Raw materials	Weight (g)
Cellulose	0.64
PVA	0.36
ASTM II Cement	18.2
Microsilica	0.4
Silica	7.8
Al_2O_3	0.8
Clay	0.1

bending strengths) of cement have been also observed when sepiolite is used as additive [10,11]. However, no studies have been published about the effect of the use of sepiolite on the flocculation process during fibre-cement manufacture.

The aim of this paper is to study the effect of the use of sepiolite on flocculation of fibre-cement suspensions induced by A-PAMs and to assess its effect on the interaction between A-PAM and the different compounds found in the fibre-cement suspensions. Since there are great differences in composition and behaviour between the fibre-cement slurries employed in air curing and those used in autoclave curing processes, the present research studies a fibre-cement suspension representing each process.

2. Materials

Two different fibre-reinforced cement slurries (M1 and M2) were used. Their composition is summarized in Table 1. Cellulose fibres are refined unbleached pine Kraft pulp (35 °SR).

M1 is similar to the fibre-cement formulations used in the manufacture of air cured fibre cement.

M2 is a mixture used to produce fibre cement cured in autoclave.

The flocculation of each fibre-cement mixture was studied in the absence of sepiolite and with a sepiolite content of 1.25%. Two sepiolites, PANGEL® HV and Cimsil FRC were studied; both were supplied by TOLSA S.A. The surface of sepiolite FRC has a higher anionic charge density than the surface of sepiolite HV.

Molecular weight and charge density of the flocculant determine the flocculation process. Therefore, three commercial A-PAMs having different values of the mentioned characteristics were used as flocculants in the Hatschek process. Their characteristics are summarized in Table 2.

Flocculants were dissolved in distilled water to prepare solutions of A-PAM with a concentration of 1.5 g/L.

To determine the interaction between sepiolite and A-PAM with each component of the fibre-cement mixtures, a series of suspensions of each component was prepared using water saturated in $Ca(OH)_2$. Table 3 summarizes the solids concentration of these suspensions.

3. Flocculation trials

Flocculation and floc properties of the different suspensions were studied by using a focused beam reflectance measurement (FBRM) device, M500L, manufactured by Mettler Toledo, Seattle, WA, USA.

Table 2
Characteristic of the flocculants used.

Floculant	Molecular weight (g/mol)	Charge density (%)
A-PAM1	Medium (10^6)	8.7%
A-PAM2	High ($7.5 \cdot 10^6$)	6.2%
A-PAM3	High ($7.4 \cdot 10^6$)	13.4%

3.1. FBRM technique description

The FBRM probe measures in real time the chord length distribution of particles in the suspension, whose evolution enables us to monitor flocculation, deflocculation and reflocculation processes. A laser beam is projected through the probe and focused on the focal point by means of a rotating lens. The focal point describes a circular path on a 20 µm plain outside the surface of the probe, immersed into the suspension, at high rotation speed (2000 rpm). Every time a particle in suspension crosses the circular line circumscribed by the focal point, light is reflected from the surface of the particle and it reaches the detector. A computer calculates the chord length of the particle on the basis of the reflectance time and focal point speed. Each measurement provides a particle chord length distribution representative of the size and shape of the population of particles in the suspension, the mean of which (mean chord size) is selected to study flocculation, deflocculation and reflocculation processes [3,12,13].

3.2. Flocculation trials with fibre-cement suspensions

Trials were carried out with 400 mL of fibre-cement suspension, prepared with water saturated in $Ca(OH)_2$. 0.25 g of sepiolite was added after 6 min of stirring at 800 rpm. 4 min after sepiolite addition, the stirring intensity was reduced to 400 rpm. 100 ppm of A-PAM was added 5 min after that speed change, and the evolution of flocs was studied at 400 rpm for 4 min. Then, the stirring intensity was increased to 800 rpm during 2 min to break down the formed flocs and, finally, reduced again to 400 rpm to facilitate the reflocculation of the particles. Fig. 1 shows the sequence of additions and mixing rate changes.

A set of trials were carried out adding the sepiolite 15 s after the A-PAM addition instead of adding it at the beginning of the trial.

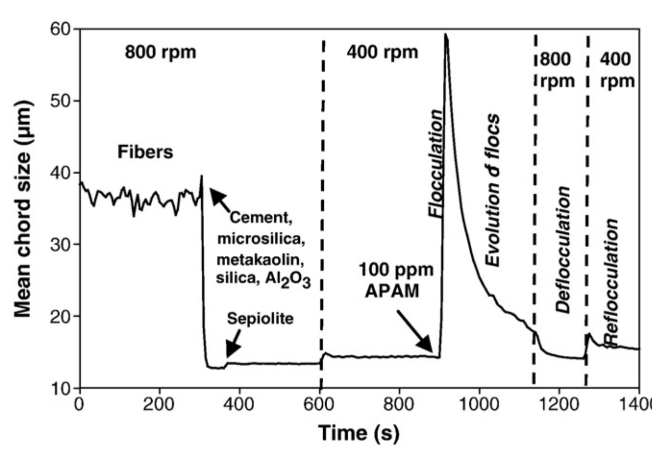

Fig. 1. Procedure to study effect of sepiolite on flocculation.

3.3. Flocculation trials with single components suspensions

Flocculation trials were carried out with the suspension of each isolated component of the fibre-cement mixture, to characterize the interaction of A-PAM with each of them as well as the effect of sepiolite on this interaction.

In these trials the mass of each raw material used to prepare 400 mL of suspension in water saturated in $Ca(OH)_2$ (Table 3) was the same as the mass of that raw material in the fibre-cement mixture. Initial stirring rate was set to 800 rpm to disperse the particles, 0.25 g of sepiolite was added after 7 min of stirring. 4 min after sepiolite addition, stirring rate was reduced to 400 rpm. A dosage of 100 ppm of A-PAM was added 4 min after stirring rate reduction. Floc evolution, deflocculation and reflocculation processes were studied following the procedure used with fibre-cement suspensions.

4. Results

4.1. Effect of sepiolite on fibre-cement flocculation and floc properties

Figs. 2 and 3 show the evolution of the mean chord size during flocculation, deflocculation and reflocculation of fibre-cement mixtures with the three A-PAMs. When A-PAM was added to any of the fibre-cement suspension, the mean chord size increased indicating the aggregation of particles (molecules of A-PAM were dissolved before its addition and they were not detected by the FBRM as shown in Fig. 4). After reaching a maximum, the value of the mean chord size decreased because of the erosion and breakage of flocs under the hydrodynamic conditions at 400 rpm. Large flocs are more sensitive to hydrodynamic forces and, thus, they broke first. This stage between the reaching of the maximum mean chord size and the increase of stirring rate is called

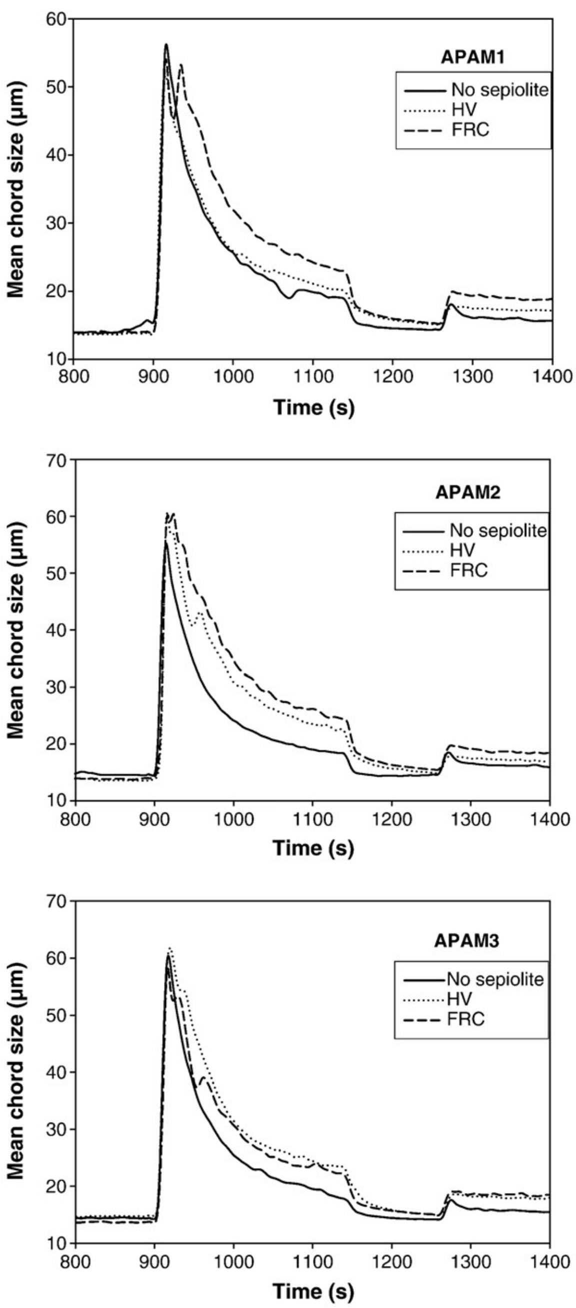

Fig. 2. Effect of sepiolite on the flocculation of M1 induced by100 ppm of different A-PAMs.

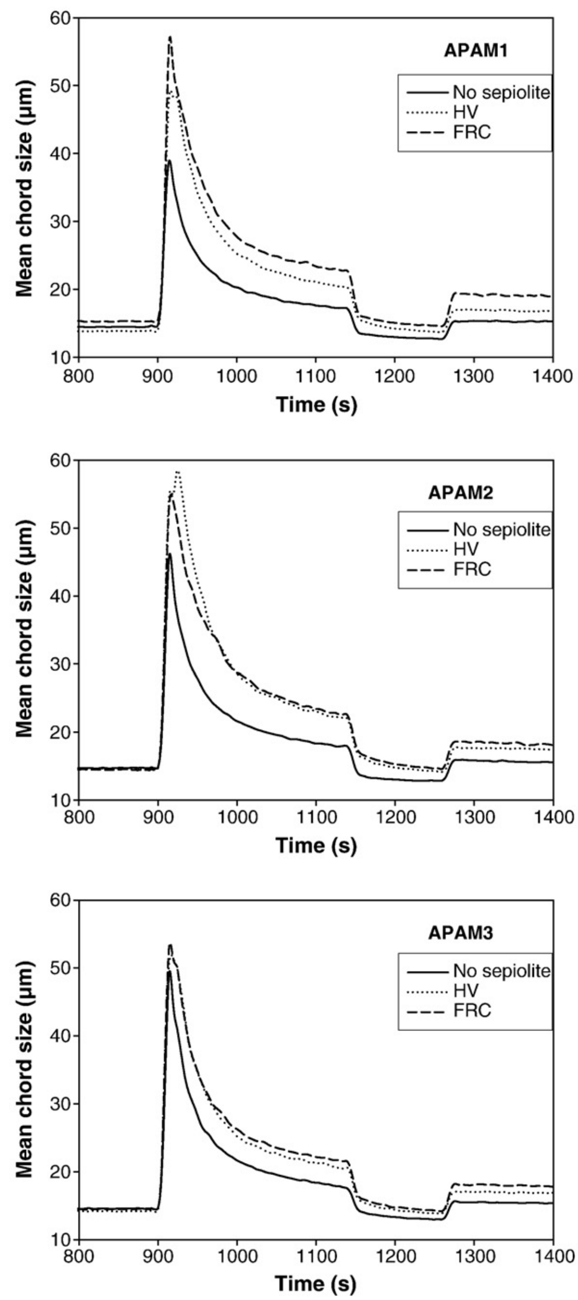

Fig. 3. Effect of sepiolite on the flocculation of M2 induced by100 ppm of different A-PAMs.

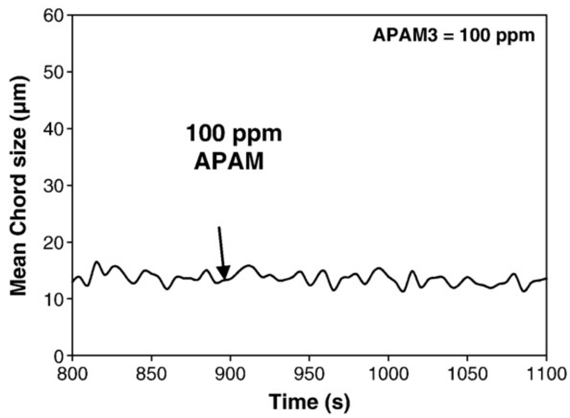

Fig. 4. Evolution of the mean chord size when 100 ppm of A-PAM was added to 400 mL of water saturated in $Ca(OH)_2$, without particles or fibres.

"evolution of flocs" to distinguish it from the deflocculation at 800 rpm [6]. When stirring intensity was increased, the mean chord size decreased, indicating that the remaining flocs were broken. Reflocculation of the system was shown by the increase of the mean chord size when the stirring intensity was decreased back to the initial value.

4.1.1. Effect of fibre-cement formulation and flocculant properties

The interaction of A-PAMs with fibre-cement slurries depends on the composition of the latter: The smallest flocs were obtained when A-PAMs were added to M2. Similar floc sizes were reached after evolution of flocs with the three A-PAMs when they were added to M1, but differences were notable when A-PAMs were added to M2. In this latter case, the highest floc sizes were reached with the A-PAMs of higher molecular weight (A-PAM2 and A-PAM3). Charge density of A-PAM3 is higher than that of A-PAM2 and flocs induced by A-PAM3 in the M2 suspension were the largest. Therefore, the floc size increased with molecular weight and charge density of the A-PAM. Fig. 5 shows that M2 is the mixture with the highest content of small particles as the distribution of chord lengths is displaced towards the lowest sizes with respect to M1. This is due to its high content of clay, which is formed by very small particles. This, along with the high content of cellulose fibres and the low content of cement particles affected the flocculation efficiency of A-PAM, as shown below. As a consequence, differences in flocculation ability of A-PAM were manifested easily with this mixture.

4.1.2. Effect of sepiolite on flocculation and floc properties

The use of sepiolite increased the mean chord size obtained after the evolution of the flocs, 240 s after the addition of A-PAM (Table 4) and after the reflocculation process; in many cases, it also increased

Table 4
Values of mean chord size alter the evolution of flocs.

	A-PAM1 (μm)	A-PAM2 (μm)	A-PAM3 (μm)
M1-No sepiolite	19	18	18
M1-HV	21	22	24
M1-FCR	23	24	22
M2-No sepiolite	17	18	18
M2-HV	20	22	21
M2-FCR	23	23	22

the maximum mean chord size reached just after the addition of the polymer. When A-PAM1 was used, the effect of sepiolite on flocculation increased slightly with the charge density of its surface, as shown by the higher mean chord size value reached after the evolution of flocs and after the reflocculation process.

To analyze the causes of the effects of sepiolite on fibre-cement flocculation, several trials were carried out adding the sepiolite 15 s after the flocculant to M2. The results show that the maximum mean chord size reached just after flocculation did not increase because sepiolite was added after or just in that moment; but the mean chord size after the evolution process was similar to the one reached when sepiolite was added before A-PAM (Fig. 6).

4.2. Effect of sepiolite on the interaction of A-PAM with single component of fibre-cement suspensions

Interaction between A-PAM and each component of the fibre-cement mixtures was studied to facilitate the interpretation of the results obtained with the fibre-cement mixtures.

Figs. 7 to 13 show the evolution of the mean chord size or the square weighted mean chord size during flocculation, deflocculation and reflocculation of each fibre-cement component induced by 100 ppm of A-PAM3 and the effect of the presence of sepiolite on these processes.

4.2.1. Interaction of fibres with A-PAM

Figs. 7 and 8 show that the addition of A-PAM 3 to cellulose or PVA fibres did not induce their flocculation. This could indicate that A-PAM3 did not interact with fibres, which are anionic too, or that its interaction did not induce flocculation. To clarify this, some trials were carried out by adding sepiolite 15 s after A-PAM addition instead of adding it before. When sepiolite was added to the suspension before adding A-PAM, the mean chord size decreased due to the small size of the particles of sepiolite. Addition of A-PAM3 to the suspension of fibres in the presence of sepiolite induced a fast formation generation of flocs formed by sepiolite particles. These flocs were broken down by

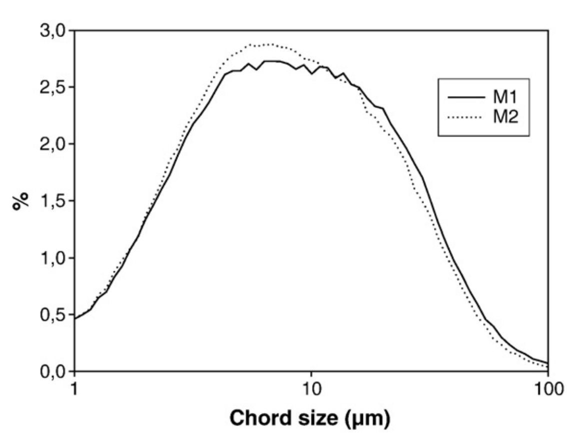

Fig. 5. Chord lengths distribution of the fibre-cement mixtures.

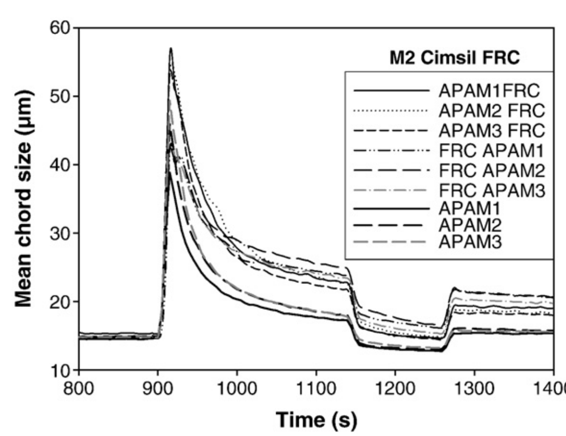

Fig. 6. Effect of the addition order of the sepiolite and A-PAM on flocculation and floc properties. Series named according to the addition order.

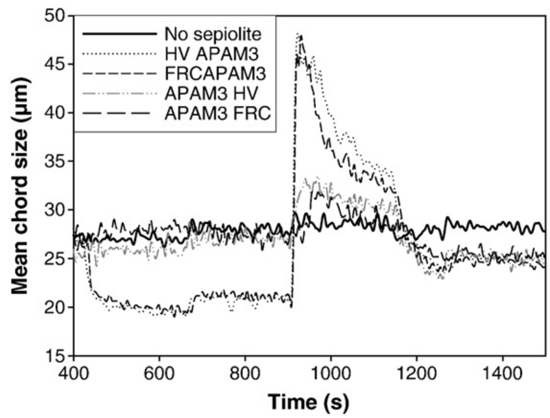

Fig. 7. Effect of sepiolite on cellulose flocculation.

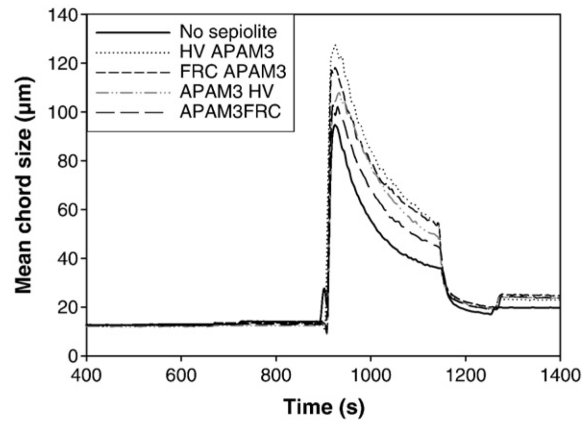

Fig. 9. Effect of sepiolite on ASTM II Cement flocculation.

increasing the stirring rate to 800 rpm and their reflocculation ability was very low. The addition of sepiolite 15 s after the A-PAM3 did not induced the formation of flocs (in the case of PVA suspension) or they were small (in case of cellulose suspension). This indicates that A-PAM not only interacted with sepiolite, but it also adsorbed onto fibres and these fibres competed with sepiolite in the adsorption process. When A-PAM 3 was added before sepiolite, many polymer chains adsorbed onto fibres but they did not induce flocculation.

The mean chord size reached when A-PAM was added to the suspension of PVA with sepiolite was lower than that reached in the case of cellulose suspension and the addition of sepiolite 15 s after adding A-PAM to the PVA suspension did not induce flocculation as mentioned above. These two facts indicate that the interaction of A-PAM with PVA was stronger than the interaction of A-PAM with cellulose. This causes the adsorption of A-PAM on PVA with flat conformation that avoids flocculation of these fibres. When sepiolite was added after A-PAM, flocculant had been already adsorbed on PVA fibres and was not enough A-PAM free chains in solution to interact with sepiolite and to induce its flocculation. In the case of cellulose, there was some A-PAM available to induce flocculation when sepiolite was added. The reason for this is that, at pH around 12, interaction between the acrylic group of A-PAM3 and cellulose ionized carboxylic groups requires the presence of cations (Ca^{++}), but the interaction between PVA and A-PAM can be driven by hydrogen bonds between the acrylic groups of A-PAM and hydroxyl groups of PVA.

4.2.2. Interaction of cement with A-PAM

The addition of A-PAM3 to the cement suspension induced the formation of flocs with a high mean chord size, higher than the values obtained when A-PAM was added to the fibre-cement mixtures. The presence of sepiolite increased the floc size obtained and no differences between the effects of both sepiolites were observed (Fig. 9).

4.2.3. Interaction of microsilica with A-PAM

The addition of A-PAM3 to the microsilica suspension (Fig. 10) induced the slow formation of small but stable flocs; most of them were not broken down when stirring rate increased to 800 rpm. Sepiolite increased the mean chord size reached and the flocculation rate, but the flocs formed were broken down by hydrodynamic forces at 800 rpm and their reflocculation ability was negligible. In this case, the effect of Pangel HV on floc size was higher than the effect of Cimsil FRC.

4.2.4. Interaction of silica with A-PAM

Fig. 11 shows the evolution of the square weighted mean chord size of silica suspension in the presence of A-PAM. Mean chord size is not only a function of the size of the particles, but it also depends on their shape. Therefore, when the shape of flocs becomes too much irregular, mean chord size can decrease. To study this case, square weighted mean chord size was required. The addition of A-PAM3 to the silica suspension induced the rapid flocculation of particles to form large flocs. Sepiolite increased floc size, but not the floc strength at 800 rpm. No significant differences between the effects of both sepiolites were observed.

4.2.5. Interaction of aluminium oxide and clay with A-PAM

The addition of A-PAM to aluminium oxide or clay induced the formation of very small but stable flocs (Figs. 12 and 13). The addition of sepiolite to these suspensions increased the mean chord size reached after flocculation induced by A-PAM and, in the case of clay, the mean chord size after the reflocculation process. Results obtained

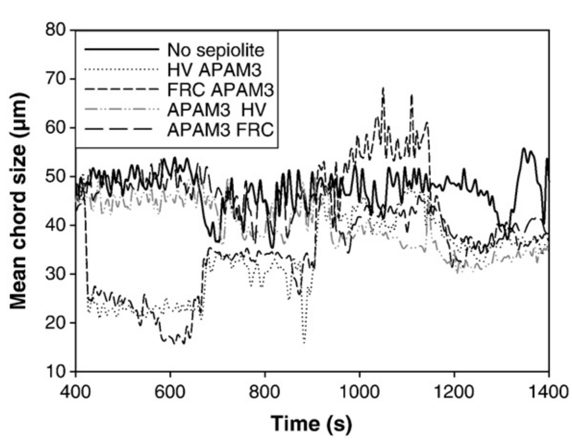

Fig. 8. Effect of sepiolite on PVA flocculation.

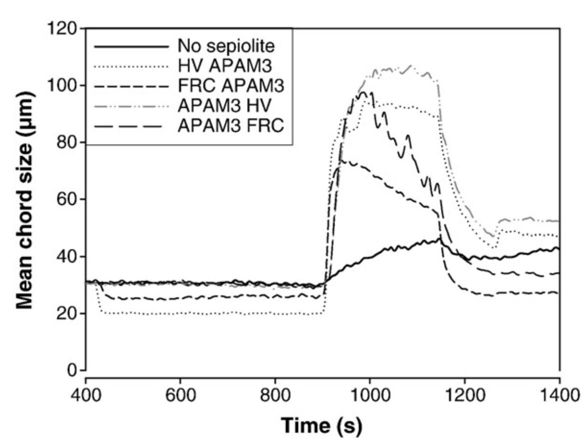

Fig. 10. Effect of sepiolite on microsilica flocculation.

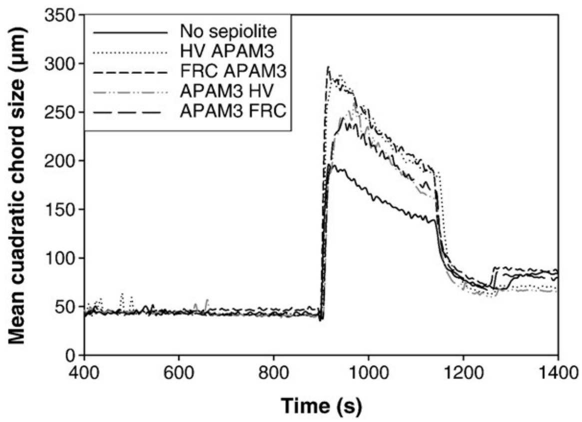

Fig. 11. Effect of sepiolite on silica flocculation.

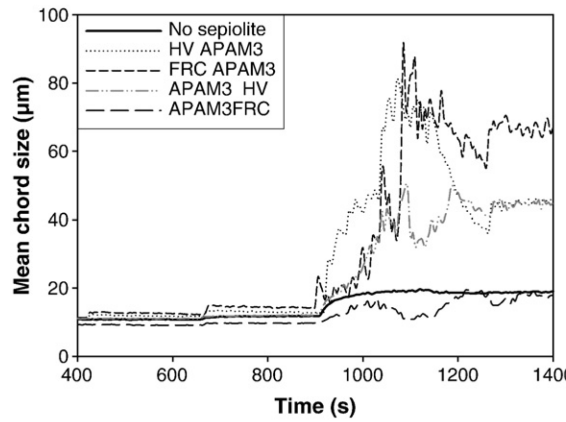

Fig. 13. Effect of sepiolite on clay flocculation.

when sepiolite was added 15 s after A-PAM show that sepiolite did not compete with aluminium oxide for A-PAM adsorption but it competed with clay.

5. Discussion

5.1. Effect of fibre-cement composition and flocculant characteristics on flocculation

In the absence of sepiolite, flocculation induced by A-PAMs depends on the composition of the fibre-cement mixture, being the smallest flocs the ones obtained from M2. This fibre-cement mixture had the highest content of fibres and the lowest content of cement. Cellulose fibres competed with the other particles in adsorption of A-PAM and this adsorption did not induce their flocculation (Fig. 7) because they were aggregated by mechanical flocculation due to their high length and concentration [14,15]. Furthermore, M2 contained the highest percentage of clay and aluminium oxide, small particles whose interaction with A-PAM led to a very little increase in the mean chord size of the suspension (Figs. 12 and 13). These two facts explain the lower mean chord size reached after adding the polymer to M2 and the higher effect of the A-PAM characteristics on flocculation compared with the fibre-cement mixture M1. The interaction between A-PAM and anionic mineral particles in fibre-cement suspension was improved by the presence of Ca^{++}. The cations supplied by the $Ca(OH)_2$ were in the medium from the beginning, but most of Ca^{++} cations were produced in the cement hardening process. Since the percentage of cement in M2 was the lowest, the production of Ca^{++} was lower than that in M1. This may explain the weaker interaction between A-PAMs and mineral particles in M2 mixture.

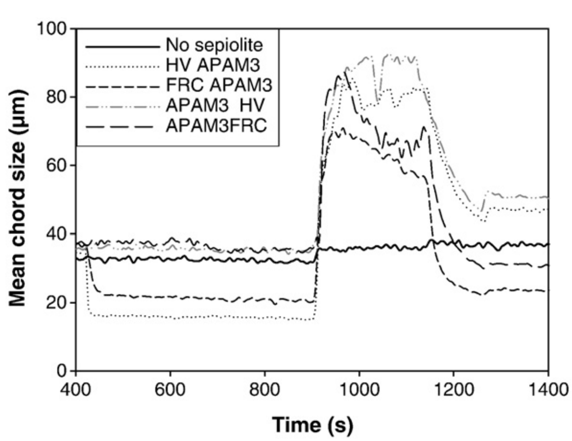

Fig. 12. Effect of sepiolite on Al_2O_3 flocculation.

Flocculation of particles in M2 depends on the conformation of A-PAMs chains. A-PAM3 had high molecular weight and the highest charge density, thus the conformation of A-PAM3 chains was longer and more extended than that of A-PAM1 and A-PAM2, and their bridging ability was higher than the one of A-PAM1 and A-PAM2.

5.2. Effect of sepiolite on the flocculation mechanism

The addition of sepiolite to fibre-cement slurry before its flocculation reduced the amount of A-PAM that interacted only with cellulose, PVA and clay, because sepiolite competes with them successfully in A-PAM adsorption. This fact increases the flocculation efficiency of A-PAM. However, this is not the only mechanism that can explain the effect of sepiolite on flocculation, because:

1. The addition of sepiolite to the M2 after A-PAM caused similar increase in the value of the mean chord size reached after the evolution of flocs with respect to the one reached by adding it before the flocculant (Fig. 6 and Table 4). Therefore, sepiolite interacted with A-PAM and was able to increase the stability of flocs at 400 rpm (increasing the mean chord size value reached after the evolution of flocs) without being affected by the addition order.
2. The flocculation of cement is also improved by the addition of sepiolite (Fig. 9) and, in this case, it was not due to a competition for A-PAM, because the effect of A-PAM3 and sepiolite addition order on the mean chord size were low and the flocs formed by cement particles when A-PAM was added were larger than the flocs formed by isolated sepiolite in the presence of A-PAM (Fig. 14). Moreover, flocculation of silica was also intensified by the presence of sepiolite

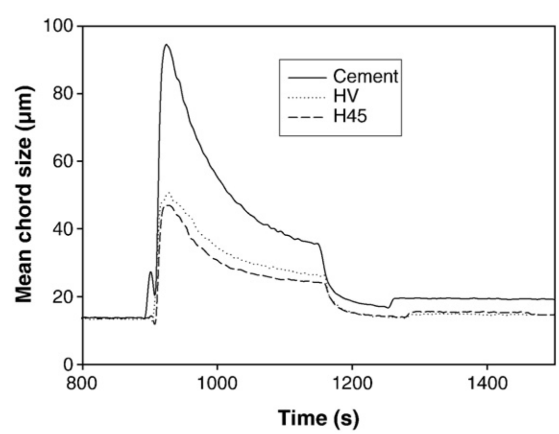

Fig. 14. Flocculation of cement suspension and sepiolite suspension induced by A-PAM3. Sepiolite suspensions: 0.25 g of sepiolite in 400 mL of water saturated in $Ca(OH)_2$.

or by its addition after the A-PAM3. This indicates that sepiolite improved the flocculation of cement or silica induced by A-PAM3 through its interaction with the flocculant and particles, which led to the formation of large flocs containing sepiolite and cement or silica particles, and increased the stability of flocs (mean chord size after evolution of flocs increased in the presence of sepiolite).

Since cement and silica were the principal components of the fibre-cement mixtures, the interaction of sepiolite with A-PAM and these particles increased the size and stability of flocs formed from the fibre-cement suspensions.

The improvement of floc properties by sepiolite will also favour retention of additives, which explains the product quality improvement observed.

6. Conclusions

Flocculation and floc properties induced by A-PAMs depended on fibre-cement composition. The lowest floc size was formed from the mixture containing the highest percentage of cellulose and clay and it increased with the molecular weight and charge density of the studied A-PAMs.

The use of sepiolite in the fibre-cement formulation increased the size and stability of flocs induced by A-PAMs. Floc stability increased slightly with the charge density of sepiolite when A-PAM1 was added to M1 or M2, but no clear effect of the charge density of sepiolite on floc size was observed in the other cases.

There are at least two mechanisms explaining the effect of sepiolite on fibre-cement flocculation induced by A-PAM: (1) sepiolite competes advantageously with fibres and clay for A-PAM adsorption, reducing the amount of A-PAM chains that adsorb onto fibres and clay without inducing the formation of large flocs; and (2) sepiolite interacts with A-PAM and mineral particles improving their aggregation and increasing their floc size and stability. This could lead to an increase in the retention of mineral particles during fibre-cement formation.

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