



HAL
open science

Surface pre-coating of talc particles by carboxyl methyl cellulose adsorption: Study of adsorption and consequences on surface properties and settling rate

Patrice Bacchin, Jean-Pierre Bonino, François Martin, Marylène Combacau, Patrick Barthes, Sabine Petit, Jocelyne Ferret

► To cite this version:

Patrice Bacchin, Jean-Pierre Bonino, François Martin, Marylène Combacau, Patrick Barthes, et al.. Surface pre-coating of talc particles by carboxyl methyl cellulose adsorption: Study of adsorption and consequences on surface properties and settling rate. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 2006, 272 (3), pp.211-219. 10.1016/j.colsurfa.2005.07.026 . hal-00201138

HAL Id: hal-00201138

<https://hal.science/hal-00201138>

Submitted on 24 Dec 2007

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Surface pre-coating of talc particles by carboxyl methyl cellulose adsorption : study of adsorption and consequences on surface properties and settling rate

P. Bacchin¹, J-P. Bonino², F. Martin^{3,4}, M. Combacau⁵, P. Barthes^{1,2}, S. Petit⁵, J. Ferret⁶

¹ Laboratoire de Génie Chimique - UMR CNRS 5503 - Université Paul Sabatier, 118 route de Narbonne, 31062 Toulouse, France

² CIRIMAT - UMR CNRS 5085 – Université Paul Sabatier, 118 route de Narbonne, 31062 Toulouse, France

³ Laboratoire de Mécanisme de Transfert en Géologie - UMR CNRS 5563 –, 31062 Toulouse cedex, France

⁴ Hydr'asa - UMR CNRS 6532 – Université de Limoges – 87060 Limoges Cedex, France

⁵ Hydr'asa - UMR CNRS 6532 – Université de Poitiers – 86022 Poitiers Cedex, France

⁵ Talc de Luzenac Europe - S. A., BP 1162, 31036 Toulouse Cedex, France

* Corresponding author. bacchin@chimie.ups-tlse.fr, Fax number : (33) 5 61 55 61 39

Abstract

This paper investigates the adsorption of different sized carboxyl methyl cellulose (CMC) onto talc particles (adsorption isotherm, adsorption reversibility), its consequences on the particles properties (electrophoretic mobility and surface wetting) and its effect on their dispersion (settling coefficient). Throughout the paper, the properties of talc particles dispersed in CMC solution are compared to CMC pre-coated talc particles: talc particles dried in a solution of CMC before their redispersion. The adsorption of CMC onto talc was quantified at around 0.4 mg of CMC per m² of talc and was seen to be irreversible on washing the talc particles with distilled water. When characterising talc surface properties, it was found that CMC adsorption leads to an increase of the negative surface charge and to an increase of the wettability. The settling velocity of CMC pre-coated talc particles in water can be around 50 % lower than that of the initial talc particles. The pre-coating of talc particles by CMC is then assumed to increase their stabilization : particle aggregation is hindered by adsorbed CMC layer inducing electrosteric repulsion between the talc particles. The technique of talc pre-coating with CMC makes the talc dispersion easier and could open interesting perspectives in engineering processes using talc dispersions.

1. Introduction

Talc is widely used in numerous applications in the paper, paint and plastics industries. Furthermore, the morphology of talc in sheets confers it interesting lubricant properties which could also be used to develop new low friction composite materials [1,2]. However, in these different processes, it is often necessary to have a good dispersion of small talc particles. Because of its hydrophobic properties (low surface surface energy), it is difficult to disperse talc in water and without formation of foam or aggregate [3].

In mineral science, Carboxyl Methyl Cellulose CMC (long chain polysaccharide) is a well known polymer depressant for talc particles [4,5] i.e. it reduces the floatability of talc. Morris et al. [4] proposes that CMC adsorbs both to the edge and the face of talc particles. The adsorption could occur in a flat conformation via hydrophobic interactions leading the hydrophilic hydroxyl and carboxyl groups of CMC to extend out from the talc surface. Such a conformation could then substantially improve the hydrophilic character of talc particles leading to a better dispersion in water [4]. Floatability studies show that the molecular weight of CMC does not have a decisive effect on talc floatability (Shortridge et al. 2000 [5]).

However, the use of CMC in solution to help dispersion of talc is not always possible. For a process requiring the use of a complex solution with talc (as for example electrolytes mixtures for electrochemical codeposition), the adding of significant quantities of CMC can disturb solution equilibrium and then alter process efficiency. Furthermore, the adsorption of CMC to the talc can be reduced because of competitive adsorption of other components of the solution on the talc surface. In order to overcome these limitations, we report in this paper the development of a technique for pre-coating talc with CMC to aid dispersion.

The aim of this study was to quantify CMC adsorption on talc particles and to investigate the effect of adsorption on the surface properties of talc and its settling properties when dispersed. Three CMC with different degree of substitution and molecular weights were tested.

2. Materials and methods

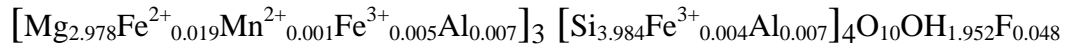
2.1. *Materials*

Talc mineral

- Mineralogical background information

Talc is a 2:1 layer clay (phyllosilicate family) which links two tetrahedral sheets with one octahedral sheet in their structure (Fig. 1). Talc represents the trioctahedral magnesium

end member with the formula: $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$. Studies with various spectroscopic methods show complex and low amounts of cation substitutions in both tetrahedral and octahedral sites [6] with conservation of the electro-neutrality of the structure. For example, the structural formula obtained with standard Trimouns talc (commercial product of Talc Luzenac Europe), on the basis of 11 oxygen atoms, is :

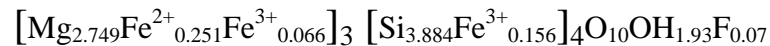


In the tetrahedral sheet, a charge deficit appears : 0.011. In the octahedral sheet, there is a charge excess of 0.012. However, it appears clearly that a deficiency in tetrahedral charges, due to substitutions of tetravalent cations Si by trivalent cations Fe and Al, are compensated by an excess of octahedral charges generated by the replacement of the divalent cation Mg by trivalent cations Fe and Al [6]. All pure talc samples from all over the world show complex structural formulae, but with electro-neutrality of the talc-cell.

Talc samples

Two talcs were used in this study.

(i) talc I (labelled Brazil) (used in section 4.3) comes from a mineralogy collection (Toulouse, France), and is a pure talc [7] composed of very well defined centimetric plates (photography by SEM observation in fig. 2). The structural formula, according to chemical analyses and various spectroscopic methods [8] is:



(ii) talc II used in this paper (excepted in section 4.3) was supplied by Luzenac Europe (France). It is a pure talc (no chlorite or other mineral phases). The chemical formulae is very close to that of standard Trimouns talc. Particles have a specific surface area determined by N_2 adsorption with the BET method at $16.7 \pm 0.5 \text{ m}^2.\text{g}^{-1}$. The average diameter when diluted in osmosed water was measured by photon correlation spectroscopy (Zetasizer 4, Malvern Instrument) at 1.3 micrometers.

Carboxyl methyl cellulose (CMC)

Three different types of carboxyl methyl celluloses (referenced by Sigma Aldrich as 21901, 21900 and 21903) were added to the talc dispersions. The properties of CMC molecules (schema of chemical structure in figure 3) are summarized in table 1. CMC molecules present a relatively high average number of glucose hydroxyl groups substituted by $-\text{CH}_2\text{COO}^- \text{-Na}^+$ per glucose monomer (degree of substitution) indicating strong surface charge. Furthermore, these CMC present different molecular sizes leading to important changes in viscosity. The

water used to prepare the CMC solution was osmosed water without any buffering then typically displaying pH between 5.5 and 6.5 and conductivity less than 20 micro-siemens/cm.

2.2. Methods

Talc dispersion

Talc particles (100 g) were progressively dispersed in 1000 ml of osmosed water (or CMC solution) by small additions of water. The dispersion was then homogenized with an UltraTurrax T50 (IKA, Staufen, Germany) at 5200 rpm for 20 minutes. This apparatus generates important stress yield within the small space between the rotating pestle and the tube breaking any clusters of particles resulting from aggregation.

Adsorption isotherms

Adsorption isotherms were determined by a classical method based on analysis of equilibrium CMC concentration in solution after a contact with talc particles. Experiments were conducted by dispersing talc at 100 g/l in 1000 ml of CMC solution with an initial concentration of between 0.5 g/l and 3 g/l. After a contact time with a continuous stirring of 24 hours, the dispersion was centrifuged. Initial concentration and residual concentration were determined by COT analysis (TOC 5050A, Shimadzu). The quantity of CMC adsorbed was determined by mass balance.

Zeta potential and size measurements

Zeta potential and particle size were measured with a Zetasizer 4 (Malvern Instruments, Malvern, UK). Samples were diluted (1:1000 in weight) in pure water buffered with 0.001 M in KCl. Mobility measurements were performed at 25°C from pH 3 to pH 12 by adding small quantities of HCl and NaOH with a titrator. Zeta potential was calculated using the Smoluchowski equation. For a given pH, three experiments were conducted in order to check the repeatability of the analysis.. Size was determined by dynamic light scattering at 90° with same dilution and same apparatus.

ATR MIR analysis

ATR FT-MIR (Attenuated Total Reflectance on Fourier Transform Middle InfraRed) analysis was performed on talc particles with a Nicolet 510 FTIR spectrometer (Hydr'Asa Laboratory, Poitiers University). In the ATR technique, the IR beam samples a surface by internal reflection of the light ; the IR light penetrates the molecules placed at the surface via

the evanescent field. The MIR approach can enhance the ATR technique by bouncing the light off the surface several times. This method can then be sensitive to the vibrational modes of molecules at the surface.

Settling experiments

Sedimentation was studied with a Turbiscan MA (Formulacion, Toulouse, France). This apparatus determines variations of sample turbidity along the sample height at different times. The stability and the homogeneity of the dispersion can thus be estimated and the settling velocity is quantified from the movement with time of the sedimentation front. The glass tube containing the sample has an internal diameter of 12 mm and a height of 140 mm. The turbidity is measured by an optical head (infrared light) recording the transmitted and back-scattered light intensities and the sample is scanned every 40 μm over its whole height. This allows clear identification of the sedimentation front (between the settling particles and the supernatant). Its position is defined in this study as the position where the back-scattered intensity is 50% of the total front height. The settling velocity, determined as the rate of variation of front position with time, can be defined as a mean settling velocity : 50 % of the particles having a settling velocity below this value and 50 % above.

Talc wetting

Wetting experiments were performed on a large sheet of talc from Brazil (collection of mineralogy, Toulouse, France), in order to determine the effect of CMC on the wettability of talc particles. With the large surface of talc, we were able to deposit a drop of water on the surface or an air bubble under the immersed surface. The latter technique was chosen to avoid drying the talc surface after contact with the CMC solution. The contact angle between the bubble and the surface was measured with a Digidrop Fast (GBX, Romans sur Isère, France). The contact angle is defined as the angle made by the liquid/solid line and the liquid/air line. The contact angle obtained with the classical sessile drop technique, θ , is then determined as $180 - \theta'$, where θ' is the angle of the bubble as it could be determined for a drop a water on a surface.

Pre-coating of talc with CMC

A procedure was developed in this work to have dry talc particles pre-coated with CMC. A first dispersion of talc was prepared with 3g/l of CMC. The dispersion was then centrifuged

for 1 hour at 4000 rpm. The centrifugation pellet was then dispersed in osmosed water and centrifugated again to wash off the excess CMC. The CMC concentration in the supernatant was analyzed to quantify the reversibility of CMC adsorption. This is discussed later in the paper. This last operation was repeated once. The resulting pellet is then dried and ground manually. These talc particles are referred to as CMC pre-coated talc.

3. Study of CMC adsorption on talc

3.1. CMC adsorption on talc particles

Adsorption isotherms for three different CMC on talc particles are presented in figure 4 where adsorbed CMC amount (in mg) per particle surface area (in m²) is plotted versus CMC concentration at equilibrium. The adsorption isotherm shows classical monolayer adsorption behaviour with a plateau. Plateau coverage was reached for a concentration of free CMC in solution of around 1 g.L⁻¹ and corresponds to around 0.4 mg.m⁻². This value is below that determined by Morris et al. [4] (0.6 mg.m⁻²) but higher than that of Steenberg et al. [9] 0.22 mg.m⁻². These differences could be attributed to the physico-chemical properties of talc particles, the CMC molecule used (size and degree of substitution) and characteristics of the solution (pH and ionic strength). The adsorption seems greater when the size of the CMC molecules is low. Steric exclusion between polymer chains may occur leading to slightly greater adsorption with small CMC. On another hand, the high degree of substitution of CMC 21903 does not lead to an increase in adsorption which could mean that adsorption is not controlled by electrostatic effects. This tends to support the adsorption mechanism controlled by hydrophobic interactions as proposed by Morris et al. [4].

The fraction of talc area occupied per adsorbed CMC molecule, σ , can be estimated from the following relationship :

$$\sigma = \frac{m_a}{M_w} N_A S_{CMC} \quad [1]$$

Where m_a is the amount of CMC adsorbed per surface area of talc (figure 4), N_A is Avogrado's number and S_{cmc} is the estimated area occupied by a CMC molecule. The area of a glucose unit of the CMC can be estimated at 0.47 nm² [4]. For a molecule of 100 000 g/mol, i.e. 455 monomer units, the area occupied by a CMC molecule is around 212 nm² if adsorption is assumed to occur in an extended, linear flat conformation. The area occupied on the talc surface is reported in table 4 from the calculation for different molecular weight of CMC at an equilibrium concentration of 1 g/l. When assuming that adsorption occurs in flat conformation, the adsorbed molecules represent around 60% of the total surface area.

SEM FEG micrographes were taken on talc particles without CMC and after a coating with CMC (respectively in fig. 5a and 5b). Comparing them reveals a difference in topography of the talc layers. On the untreated talc surface, successions of planes and steps occur, characteristic of piling up of talc layers, whereas on the surface of particles treated by CMC, the pattern is diffuse unlike that caused by the presence of steps. The covering "veil" seems to result from the irreversible adsorption of CMC on the greater part of the talc surface. These observations were confirmed by Attenuated Total Reflectance on the Middle InfraRed region (ATR MIR) of talc particles with and without pre-coating. The observations were made on the well extended (a, b) plane (Figure 2) and the InfraRed beam was perpendicular to this plane and parallel to the OH-direction (Figure 1), here accounting for the absence of OH stretching vibrations in the $4000 - 3600 \text{ cm}^{-1}$ region (not presented in this article). Figure 6 shows that CMC pre-coating leads to the detection of vibrational bands characteristic of CMC molecules. These bands mask the Si-O bands which are detected on naked talc particles. Adsorption of CMC to the surface of talc particles is then confirmed by these analysis.

3.2. Analysis of adsorption reversibility

After CMC adsorption, talc particles were washed twice with osmosed water as detailed in the methods section relative to the pre-coating procedure. Table 2 gives the loss in adsorbed CMC after one and two washing.

Around 75-80 % of CMC (21900 and 21901) adsorbed on particles was still present after two washings. Only 35-40 % of CMC 21903 remained on the talc after two washing. The larger size of CMC 21903 could lead to greater reversibility of adsorption by washing as the polymer chain can be stressed by the hydrodynamic forces exerted by centrifugation.

Then, after the two washing steps, the centrifugation pellets were dried and reground to be dispersed again in water. When analysing the water where dispersion of CMC pre-coated talc particles was made it was found by total organic carbon TOC analysis that the concentration of CMC was equivalent to 5-10 % of the total amount adsorbed remaining after two washings (table 3). The reversibility of adsorption is again greater with long-chain CMC.

To conclude, CMC adsorbs irreversibly for a great part to the talc surface. Pre-coating of talc with CMC is possible leading to talc powder with CMC adsorbed on the surface.

4. Effect of adsorption on properties of talc particles

4.1. Zeta potential measurements

Zeta potential measurement of talc particles were performed in the absence or in the presence of the CMC polymer at 1, 2 and 3 g/l within the pH range 3 to 11. Results for the 3 CMC are plotted in figure 7 for the same concentration of 3 g/l and compared to the zeta potential for bare talc particles in water. The evolution of zeta potential with pH is classical with a slightly positive value at low pH and an increasingly negative value as the pH rises. The isoelectric point of talc is around 3. This value is higher than that found by Morris et al. [4] (pH=2). For a neutral pH around 8, the zeta potential of bare particles is around - 35 mV whereas with CMC (21900 and 21901) it reaches a value of - 42 mV and - 49 mV for CMC 21903. It can be noted that the zeta potential is directly correlated to the degree of substitution of the CMC (table 1): the same results being observed for CMC with same substitution degree (0.78 and 0.79) whereas larger negative values were observed with the CMC with the highest degree of substitution (0.92). Experiments with lower CMC concentrations (1 and 2 g/l) showed exactly same trends. For high pH a cross-over in zeta potential versus pH can be observed for CMC 21900 and 21901. This cross-over has already been observed and explained by Morris et al. [4]. Adsorption leads to a shift of shear rate plane away from the particle surface leading to a smaller apparent zeta potential. Particles coated with negative macromolecules can then exhibit zeta potentials slightly below the ones determined for bare particles. Furthermore, zeta potentials were determined on CMC pre coated talc particles (figure 8). The zeta potential is always more negative when talc is precoated with CMC. The negative charge is greater with CMC 21900 and 21903 (- 48 mV at neutral pH). These results confirm that the CMC adsorbed after precoating the talc remains.

4.2. CMC adsorption and apparent particle size

Particle size was determined by dynamic light scattering. Figure 9 presents the mean hydrodynamic diameter of talc as a function of CMC solution concentration. These measurements show an increase of particle size when CMC is adsorbed on the particle surface being greater for CMC having a higher molecular weight. For the larger CMC an increase of 1 μm of the initial particle diameter of 1.5 μm is observed on adding CMC. This can not be attributed to the adsorption of extended molecule on the surface (the macromolecular chain can not increase the apparent hydrodynamic size to this extent). As it will be discussed later, the increase of particle size could result of the formation of aggregate between particles because of bridging flocculation.

4.3. CMC adsorption and talc wetting phenomena

CMC is a macromolecule with carboxyl groups and hence relatively hydrophilic chain. In contrast, talc is hydrophobic. Wetting experiments were performed to study talc wetting. The experiments were carried out on a large sheet of Brazilian talc by a bubble captured under the immersed talc surface. Figure 10 shows the evolution of the bubble contact angle, θ' , for different CMC in solution and for a pre-coated talc surface. It can be noted that talc is found to be hydrophobic when immersed in water: the bubble contact angle is equivalent to a contact angle with a sessile drop technique of 92° . This value is close to that found by Shrader and Yariv [3] between $81-88^\circ$. In the presence of CMC, the bubble contact angle increased meaning that talc loses its hydrophobicity when immersed in a CMC solution. These variations cannot be explained only by the change in surface tension of the liquid. To prove this, measurement of wettability were performed on a talc surface after CMC contact and washing (pre-coated talc) and talc still exhibited a hydrophilic character. On the another hand, experiments were carried out on capillary elevation in a tube containing talc particles with a Wilhelmy balance method (Balance 3S, GBX, France). These complementary experiments showed same trend; no water rising in the initial talc powder because of its hydrophobicity and a rise of water in pre-coated talc particles at a significant rate meaning that talc particles are then hydrophilic.

5. Dispersion and settling of talc particles with CMC

First, CMC facilitates dispersion of talc in aqueous solution. We noted that the use of CMC reduces the formation of foam and leads to equivalent dispersion with less energy. This can be explained by the effect of CMC on the hydrophobicity of the talc surface. Wettability experiments have shown that the talc surface becomes hydrophilic in the presence of CMC because of CMC adsorption on talc particles. With a hydrophobic surface, air can be trapped between talc particles leading to foam whereas in the presence of CMC the dispersion of particle in aqueous solution is improved. Recent experiments confirmed the presence of air nano-bubbles between sheets of talc leading to floatation “air entrapment in the porous structure of talc are possible causes for the poor dispersability of micronised talc” [10]. By reducing the hydrophobicity of talc surface with CMC (see section 4.3), air entrapment can be limited then leading to a better dispersability of particles.

Settling properties of talc suspensions were determined for talc particles in the absence and presence of CMC and for CMC pre-coated talc particles. Figure 11 presents the variation of settling velocity with the CMC concentration for various CMC molecules.

Settling of bare talc particles occurs at around 1.5 mm/h. A plateau with lower settling velocity was found for CMC concentrations above 1 g/L. For these conditions, the settling velocity decreased as the molecular weight of CMC increased. Table 5 presents the particle diameter calculated for various CMC concentrations from settling experiments. Calculations were made with the classical settling law by accounting for the change in supernatant viscosity (experimentally determined by a co-axial rheometer) and a talc density of 2.8. For bare particles, the settling corresponds to particles of around 2 μm in diameter. For talc particles in CMC solution with a concentration of over 1 g/L, the settling is fully linked to CMC viscosity : the apparent size calculated from settling law by using the experimental supernatant viscosity is almost constant in presence of CMC (Table 5) . However, these calculated talc particle size is two times less than the one obtained for bare particles.. So, dispersion of talc particles is better in the presence of CMC at concentrations over 1 g/L. This suggests the existence of electrosteric repulsion between particles because of the brush-like talc surface induced by charged CMC adsorption which is fully linked to an increase in negative zeta potential (see above). On the other hand, for concentrations less than 1 g/L CMC 21903 (highest molecular weight) a higher settling rate was measured than with bare particles. This can be attributed to flocculation between particles because of dilution of polymer adsorbed to the talc surface and leading to the formation of bridges between particles (bridging flocculation). For higher polymer concentrations the phenomenon of stabilisation by electrosteric repulsion occurs leading to a decrease of settling velocity.

Settling velocity was determined for CMC pre-coated talc particles dispersed in water. The results are presented in table 6. It shows that the settling velocity is slightly lower than that obtained with bare particles which can not here be attributed to changes in viscosity but to a stabilization of particles after pre-coating. Furthermore, it was experimentally found that dispersion of pre-coated particles was very easy requiring less energy than with bare particles.

6. Conclusions

Determination of adsorption isotherms has shown that the three CMC tested adsorb on the talc surface. This adsorption on talc particles was confirmed by Attenuated Total Reflectance on the Middle InfraRed region (ATR MIR) and SEM FEG observations. Washing of talc particles leads to a weak desorption of CMC meaning that CMC adsorption is mainly irreversible (except for the CMC with the highest molecular weight). Furthermore, pre-coating the talc with CMC is possible leading to talc powder with CMC irreversibly adsorbed on the surface.

Dispersion and settling studies of talc have shown that the presence of CMC drastically improves the dispersion of talc particles by avoiding foam and aggregate formation. This effect can be explained by wettability experiments showing that CMC adsorption confers more hydrophilic properties to talc. Furthermore, when dispersed in CMC solution at concentrations over 1 g/l (in 100 g/l talc suspension), the settling velocity is reduced mainly because of the high viscosity of the CMC solution. But results also show that presence of CMC induces a complementary decrease in settling velocity which could be due to the better dispersion of the talc in the solution induced by electrosteric repulsion between talc surfaces where CMC is adsorbed. This is confirmed by measurement of zeta potentials of talc where adsorption leads to a higher negative zeta potential. This positive effect of CMC is also observed when CMC pre-coated talc particles are dispersed in water.

For future applications, it has been shown that it is possible to pre-treat talc particles with CMC to improve their dispersion in aqueous solution. This method [2] could be very interesting for the dispersion of talc in complex aqueous solutions where the presence of CMC could change the properties of the solution or where competitive adsorption could occur at the talc surface.

7. Acknowledgements

We have benefited over the years from results obtained by Reda El Berrichi and William Vautrin and from very helpful discussions with Stephane Vaillant and Jean-Luc Trompette. We are grateful to the CNRS for supporting this study through the projet “Traitements de surface et dépôts de couches minces” and to Luzenac Europe (Richard Baëza and Sandrine Péjo-Soucaille) for providing the talc material, infrared data and for research discussions.

8. References

- [1] J-P. Bonino, S. Vaillant., F. Martin, P. Bacchin, J. Alexis, “Composite Coatings with Talc Particles”, Vth International Colloquium on Chromium Plating – Saint Etienne, France, May 24-27 2004.
- [2] Matériau composite utilisable comme revêtement lubrifiant, Patent FR 2848219 (2004).
- [3] M.E. Schrader, S. Yariv, Wettability of clay minerals, J. Colloid Interf. Sci., 136 (1990), 85-94.

- [4] G.E. Morris, D. Fornasiero, J. Ralston, Polymer depressants at the talc-water interface: adsorption isotherm, microflotation and electrokinetic studies, *Int. J. Miner. Process* 67 (1-4) (2002) 211-227.
- [5] P.G. Shortridge, P.J. Harris, D.J. Bradshaw, The effect of chemical composition and molecular weight of polysaccharide depressants on the notation of talc, *Int. J. Miner. Process* 59 (3) (2000) 215-224.
- [6] F. Martin, P. Micoud, L. Delmotte, C. Maréchal, R. Le Dred, Ph. de Parseval, A. Mari, J.P. Fortuné, S. Salvi, D. Béziat, O. Grauby, J. Ferret, The structural formula of talc from the Trimouns deposit, Pyrénées, France, *Canadian Mineralogist* 37 (4) (1999) 975-984.
- [7] E. Ferrage, F. Martin, A. Boudet, S. Petit, F. Fourty, F. Jouffret, P. Micoud, Ph. de Parseval, S. Salvi, C. Bourgerette, J. Ferret, Y. Saint-Gérard, S. Buratto, J.P. Fortuné, Talc as nucleating agent of polypropylene: morphology induced by lamellar particle addition and interface mineral-matrix modelization, *Journal of Material Science* 37 (2002) 1561-1573.
- [8] S. Petit, F. Martin, A. Wiewiora, Ph. de Parseval, A. Decarreau, Crystal-chemistry of talcs: a Near Infrared (NIR) spectroscopy study, *American Mineralogist* 89 (2004) 319-326.
- [9] E. Steenberg, P.J. Harris, Adsorption of carboxymethyl cellulose, guar gum, and starch onto talc, sulphides, oxides and salt-type minerals, *South Afr. J. Chem.* 37 (1984) 85.
- [10] M. Zbik, R.St.C. Smart, Dispersion of kaolinite in aqueous solution: nanomorphology and nano-bubble entrapment, *Miner. Eng.* 15 (2002) 277-286.

Table 1 : Main properties of the CMCs used for dispersion of talc

CMC type	Degree of Substitution	Molecular weight
21900	0.79	100-150 000
21901	0.78	50 000
21903	0.92	250 000

Table 2 : Reversibility of CMC adsorption on talc particles after one or two washings (centrifugation and dispersion in water) by analysis of CMC in the supernatant. Calculations based on initial quantity adsorbed.

CMC type	Loss in % (of initial adsorbed amount) after one washing	Loss in % (of initial adsorbed amount) after two washings
21900	16	22
21901	18	23
21903	47	63

Table 3 : Reversibility of CMC adsorption onto talc particles after centrifugation, drying of the pellet and dispersion in water after regrinding. Calculation based on quantity adsorbed after two washings.

CMC type	Loss in % (of amount adsorbed after 2 washings) after drying and regrinding of the talc
21900	7
21901	6
21903	22

Table 4 : Calculation of the fraction of talc surface covered by CMC when assuming macromolecular adsorption in a flat conformation.

CMC	Mw (g/mol)	Molecule area (nm ²)	Amount of CMC adsorbed on talc (mg/m ²)	Fraction of talc surface covered by CMC
21901	50000	107	0.46	0.59
21900	125000	267	0.42	0.54
21903	250000	535	0.39	0.50

Table 5 : Talc particle diameter deduced from settling experiments for talc dispersion in the absence and presence of various CMC. Calculations used a classical settling law corrected with the supernatant viscosity.

	No CMC added	CMC 21900		CMC 21901		CMC 21903	
		2 g/L	3 g/L	2 g/L	3 g/L	2 g/L	3 g/L
Settling velocity (mm/h)	1.5	0.21	0.16	0.50	0.46	0.10	0.08
Supernatant viscosity (Pa.s)	0.001	0.0018	0.0021	0.0008	0.0011	0.0038	0.0048
Particle diameter (μm)	2.04	1.02	0.97	1.05	1.19	1.03	1.03

Table 6 : Settling velocity of talc particles in the absence of CMC and for CMC pre-coated talc particles.

	No CMC added	Pre-coated talc particles with		
		CMC 21900	CMC 21901	CMC 21903
Settling velocity (mm/h)	1.5	1.58	1.04	0.69

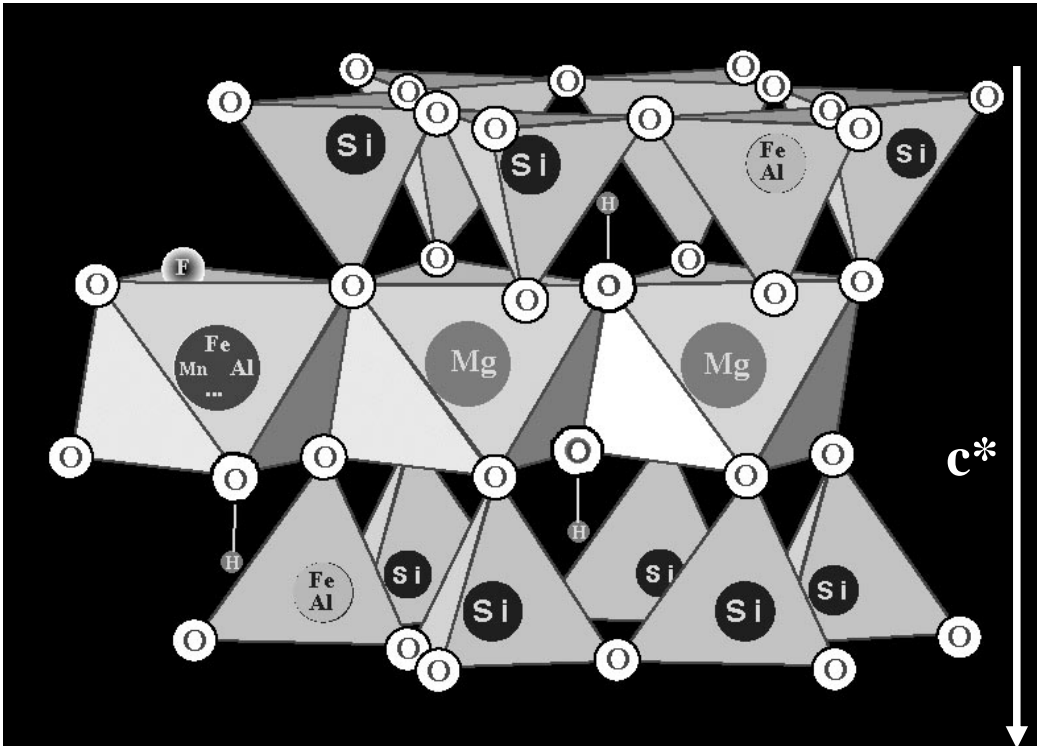


Fig. 1: Talc structure along the c^* -axis : two Si inverted tetrahedral sheets (with few Fe^{3+} and Al substitutions) sandwich one Mg octahedral sheet (with few Fe^{2+} , Al, Mn, ... substitutions)

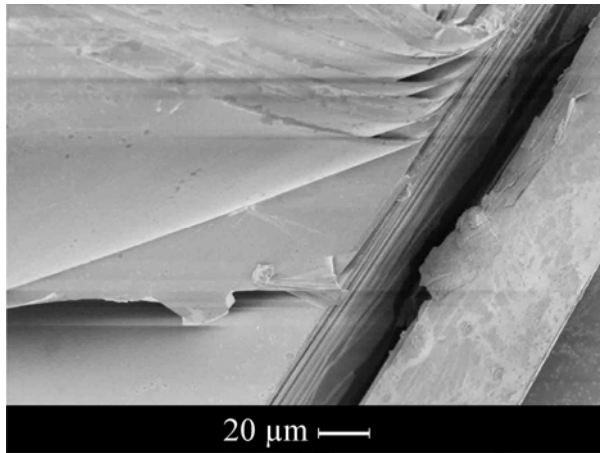


Fig. 2: SEM (Scanning Electronic Microscopy), using a LEO 435 VP, in the Ecole Nationale Supérieure de Chimie de Toulouse; observation of talc from Brazil constituted by very long, perfectly stacked flakes

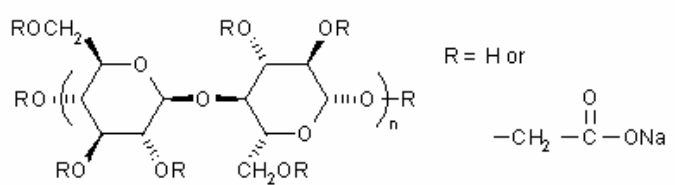


Figure 3 : Chemical structure of CMC

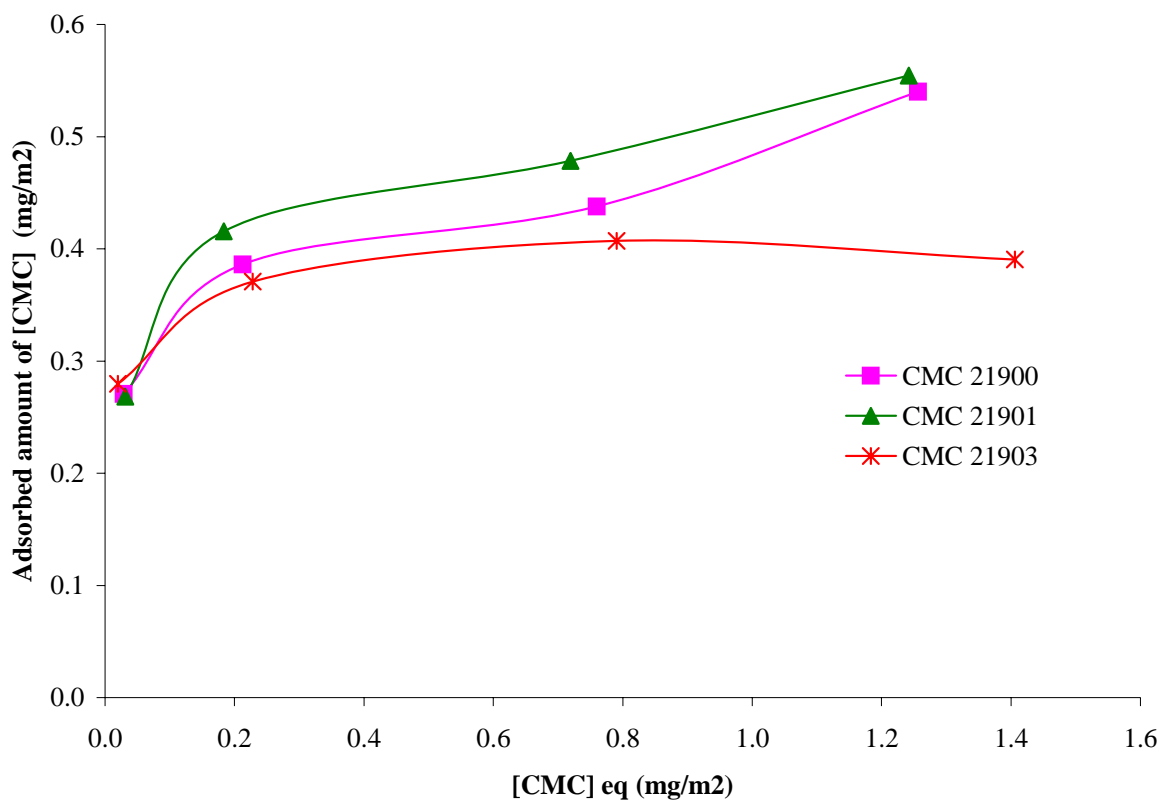


Figure 4 : Adsorption isotherm of CMC on talc at 25 °C. Talc concentration of 100 g/l.

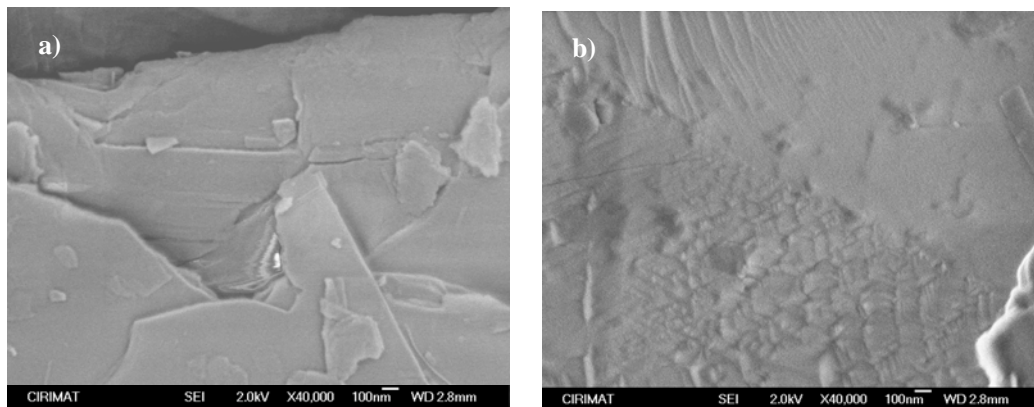


Figure 5 : SEM FEG photography's of talc particles before a) and after CMC pre-coating b).
Surface aspect of talc is modified after pre-coating with CMC.

Mis en forme : Anglais
(Royaume-Uni)

Mis en forme : Anglais
(Royaume-Uni)

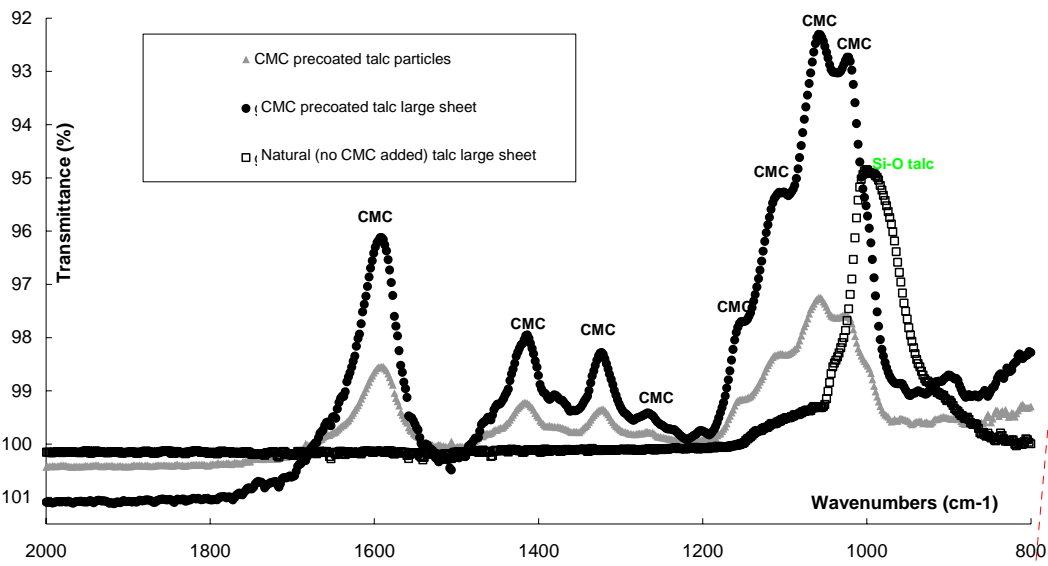


Figure 6 : ATR MIR analysis of talc particles without added CMC – white squares – of talc particles precoated with CMC – grey triangles – and of a large talc sheet precoated with CMC – black circles -. IR Vibrations bands of CMC are detected on precoated talc. These bands cover the Si-O band of the talc.

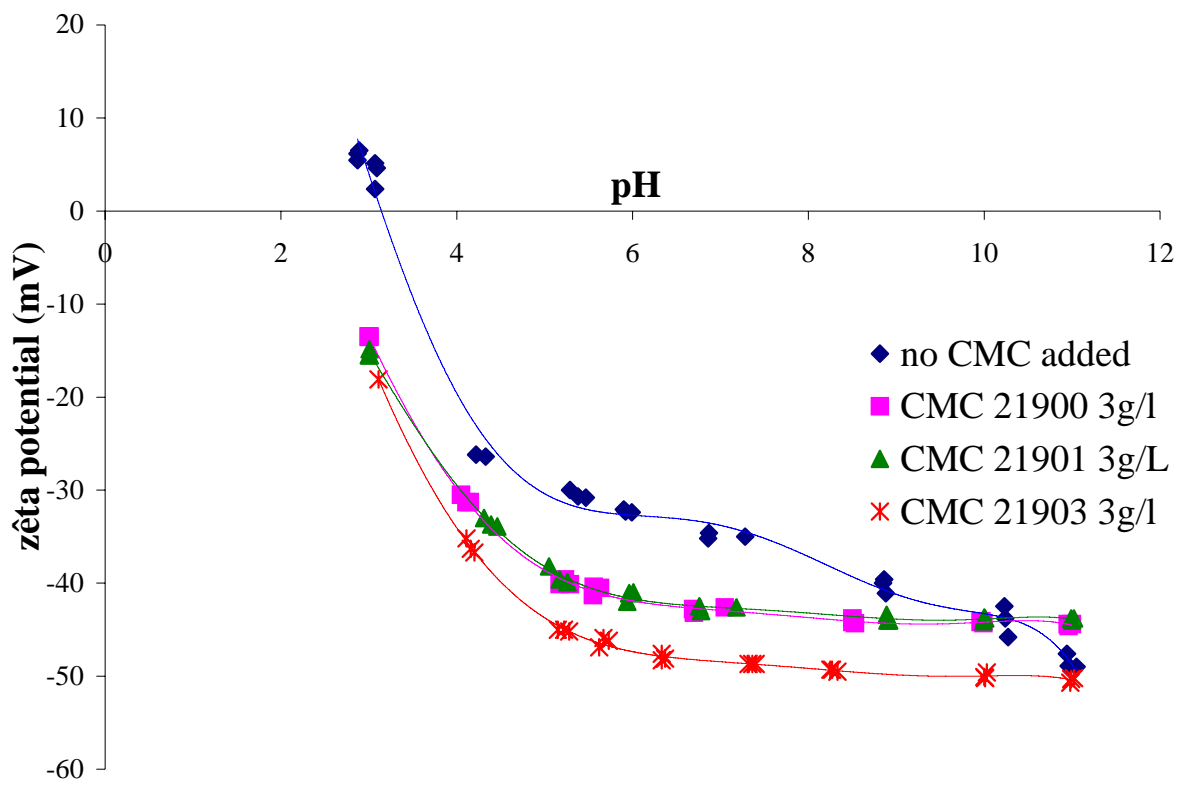


Figure 7 : Zeta potential of talc as a function of pH in absence or in presence of CMC.

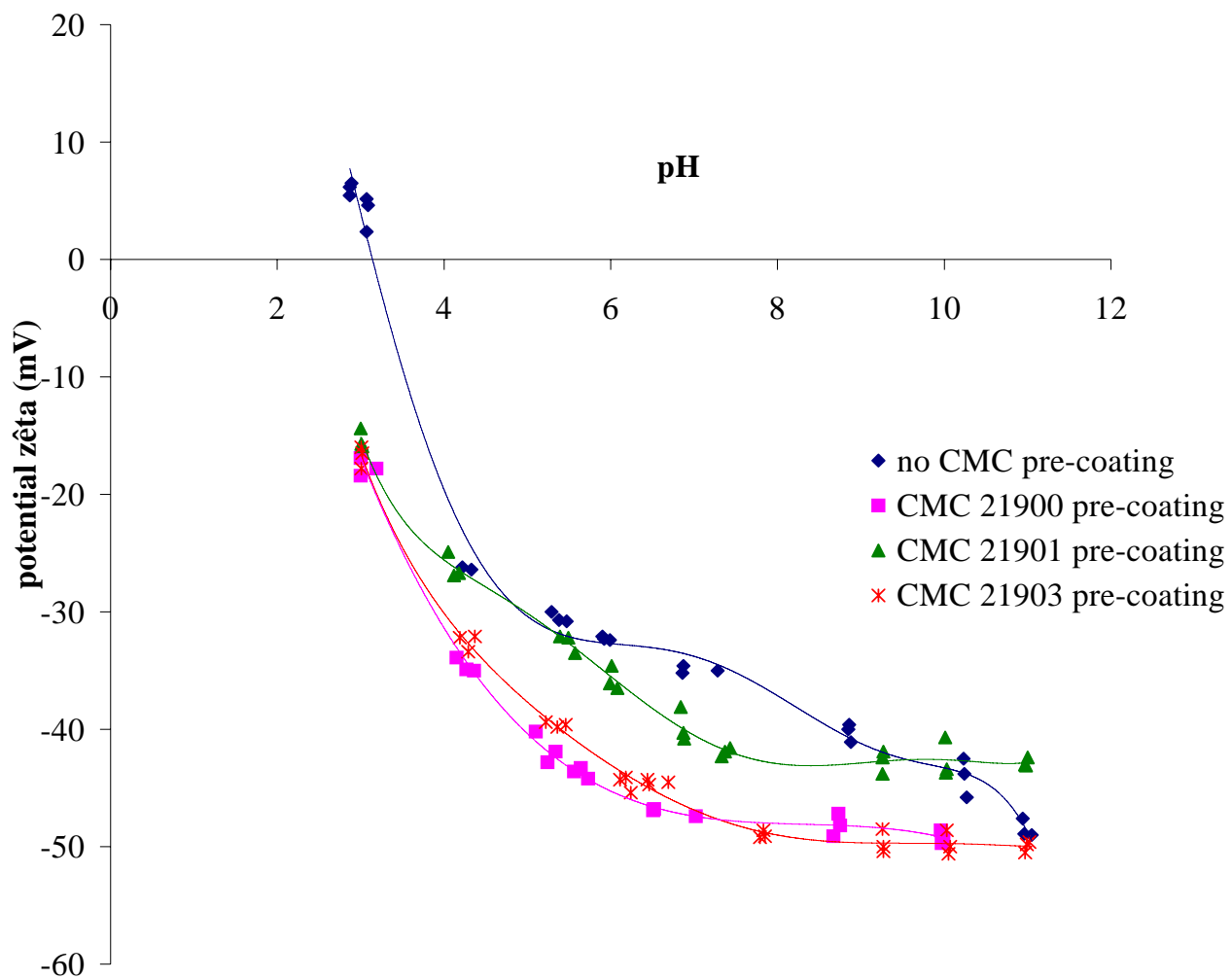


Figure 8 : Zeta potential of CMC pre-coated talc particles as a function of pH.

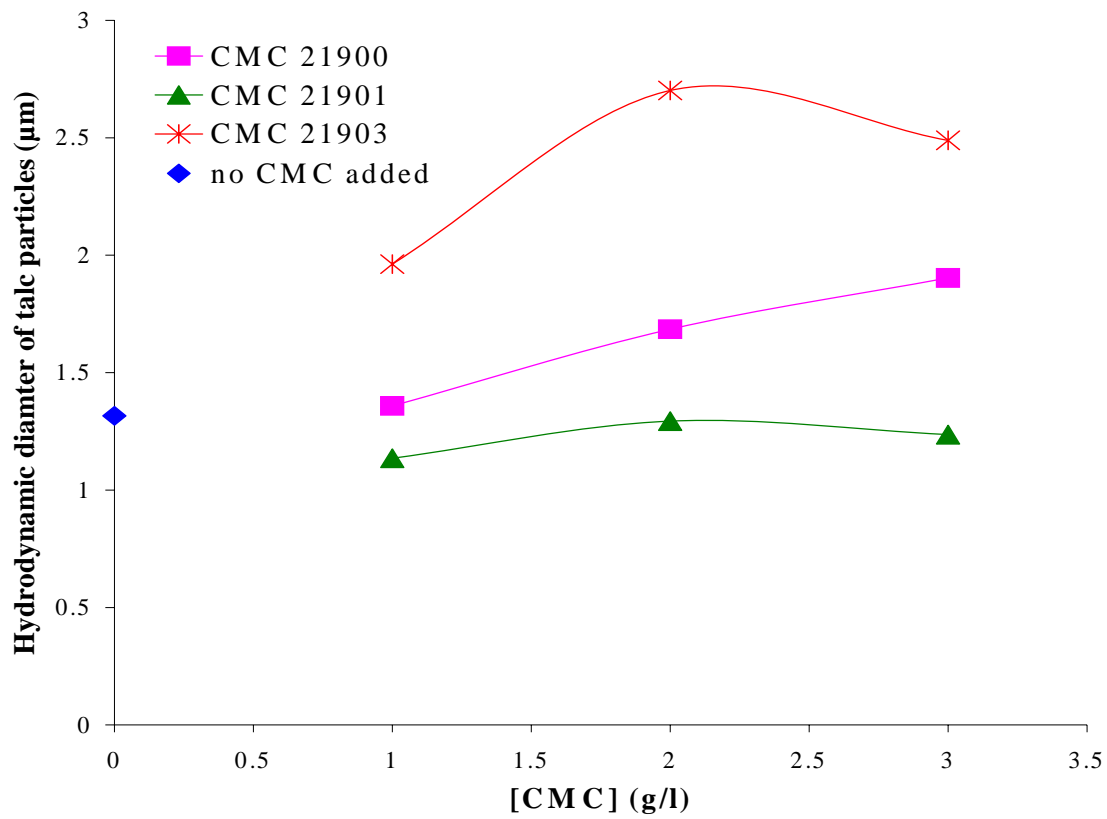


Figure 9 : Mean particle diameter of talc particles in absence or in presence of CMC.

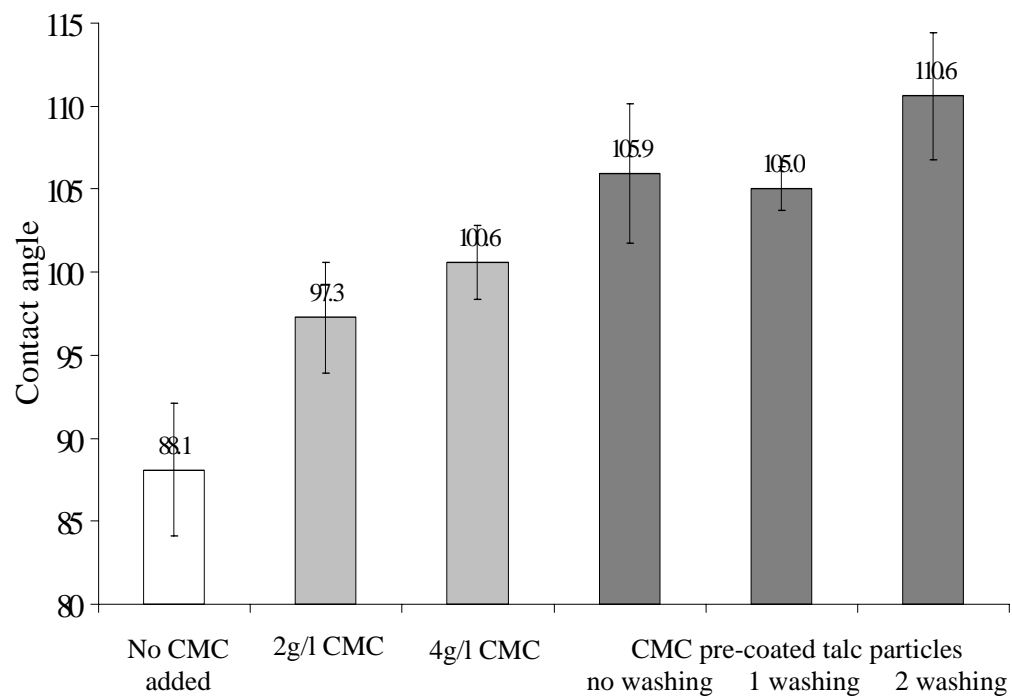


Figure 10 : Contact angle on a talc sheet in absence of CMC, in presence of CMC and for CMC pre-coated talc particles (CMC 21900).

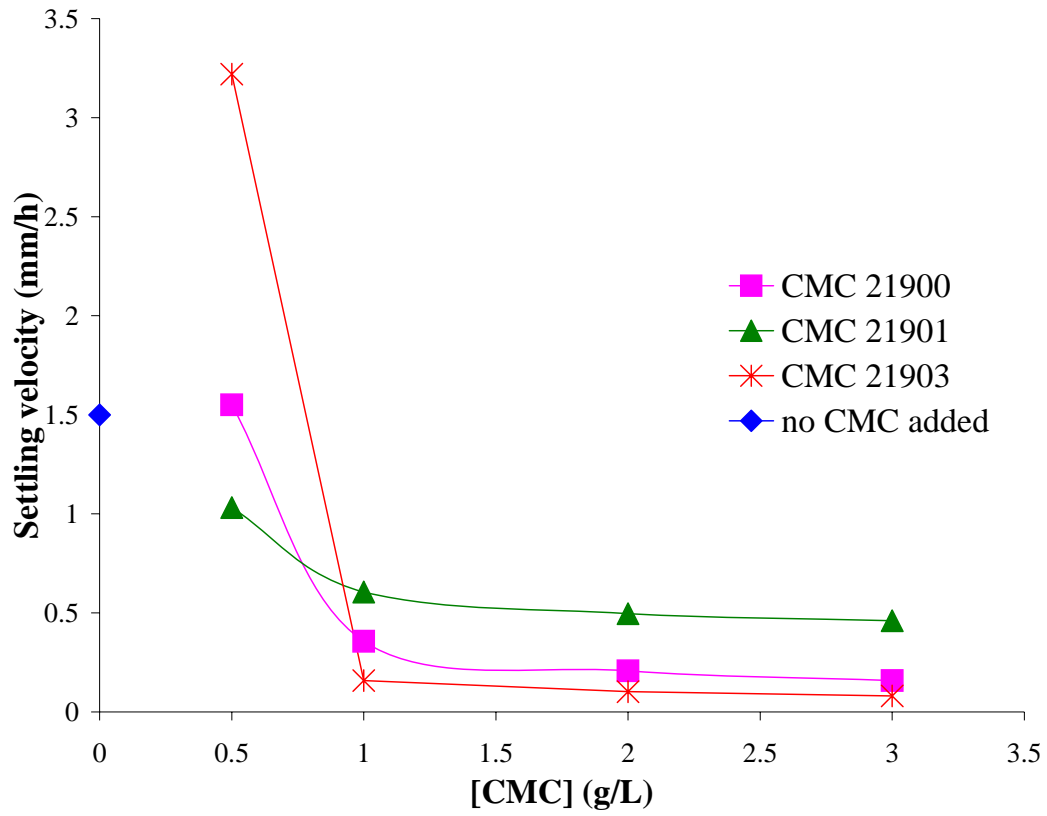


Figure 11 : Settling velocity of talc particles as a function of CMC concentration.