

Static and Dynamic Filtration Properties of Aqueous Suspensions of Clays and Electrolytes

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J. Durrieu, J. F. Argillier, E. Rosenberg, Y. Li. Static and Dynamic Filtration Properties of Aqueous Suspensions of Clays and Electrolytes. Revue de l'Institut Français du Pétrole, 1997, 52 (2), pp.207-218. 10.2516/ogst:1997020 . hal-02079093

HAL Id: hal-02079093
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STATIC AND DYNAMIC FILTRATION PROPERTIES OF AQUEOUS SUSPENSIONS OF CLAYS AND ELECTROLYTES

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FILTRATIONS STATIQUES ET DYNAMIQUES DE SUSPENSIONS AQUEUSES D'ARGILES ET D'ÉLECTROLYTES

Les propriétés de filtration de suspensions colloïdales d'argile en présence d'électrolytes (NaCl, $CaCl_2$ et KCl) dans l'eau ont été étudiées en conditions statique et dynamique. Les expériences de filtration, combinées à l'observation des cakes formés par cryomicroscopie à balayage et microscopie électronique à transmission, mettent en évidence l'importance de la taille et la forme des particules d'argiles, ainsi que leur mode d'association en suspension, sur la texture du cake, sa perméabilité et ses propriétés de relaxation. Ces paramètres dépendent fortement de la nature de l'électrolyte.

Le mode de dépot en dynamique est lié à l'état d'agrégation de la suspension au départ, sa polydispersité et la sensibilité des agrégats au cisaillement. Dans tous les cas, le processus de croissance du cake est ralenti et on obtient des volumes de filtrat plus élevés qu'en statique. Le cisaillement a deux effets: d'une part, dissocier les agrégats lâches présents en suspension et d'autre part, exercer un tri granulométrique des particules dans le cas d'une suspension polydispersée. À fort gradient de cisaillement on obtient rapidement un cake fin d'épaisseur constante. L'épaisseur de ce cake dépend de la fraction de petites particules présentes en suspension, ou susceptibles d'être formées par dissociation d'agrégats lâches sous l'action du cisaillement. La perméabilité du cake en dynamique, comme en statique, est conditionnée par la taille et la forme des particules qui le constituent.

STATIC AND DYNAMIC FILTRATION PROPERTIES OF AQUEOUS SUSPENSIONS OF CLAYS AND ELECTROLYTES

Filtration properties of aqueous colloidal suspensions of clays in presence of electrolytes (NaCl, KCl, $CaCl_2$) have been studied in static and dynamic conditions. Filtration experiments combined with cake observation by cryo-SEM and TEM show the influence of the associating mode of clay particles in suspension on the texture, the permeability and the relaxation properties of the cake. These parameters are very much dependent on the nature of the electrolyte added.

In dynamic conditions the construction of the cake is related to the state of aggregation of the clay suspension, its polydispersity and the sensitivity of the aggregates to shear rates. In all cases the build up of the cake is slowed down and dynamic filtrate volumes

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are larger than static filtrate volumes. The shear rate has two effects: first to dissociate loose aggregates in the suspension and second to exert a granulometric sorting of the particles in the case of a polydisperse suspension. At high shear rate a cake of constant thickness is quickly obtained. The thickness of this cake depends on the fraction of small particles present within the initial suspension or that are formed by dissociation of loose aggregates under dynamic conditions. In dynamic like in static the permeability is controlled by the size and shape of particles that constitute the cake.

FILTRACIONES ESTÁTICAS Y DINÁMICAS
DE SUSPENSIONES ACUOSAS DE ARCILLAS Y
0DE ELECTROLITOS

Se han estudiado, en condiciones estática y dinámica, las propiedades de filtración de suspensiones coloidales de arcilla en presencia de electrolitos (NaCl, $CaCl_2$ y KCl) en el agua. Las experiencias de filtración, combinadas con la observación de los cakes formados por criomicroscopia de exploración y miscroscopia electrónica de transmisión, hacen resaltar la importancia de las dimensiones y la forma de las partículas de las arcillas, así como su modo de combinación en suspensión, sobre la textura del cake, su permeabilidad y sus propiedades de relajación. Estos parametros dependen muy ampliamente de la naturaleza del electrolito.

El modo de aglutinación en fase dinámica guarda relación con el estado de agregación de la suspensión en su punto de partida, su polidispersidad y la sensibilidad de los agregados al cizallamiento. En todos los casos, el proceso de crecimiento del cake se ve moderado y se obtienen volumenes de filtrado más elevados que en fase estática. El cizallamiento presenta dos efectos: en primer lugar, disociar los agregados sueltos presentes en suspensión y, en segundo lugar ejercer una clasificación granulométrica de las particulas en el caso de una suspensión polidispersa. Operando con un gradiente elevado de cizallamiento se obtiene rápidamente un cake fino de espesor constante. El espesor de este cake depende de la fracción de las pequeñas partículas presentes en suspensión, o susceptibles de formarse por disociación de agregados sueltos, bajo la acción del esfuerzo de cizallamiento. La permeabilidad del cake en fase dinámica, así como en fase estática depende de las dimensiones y la forma de las partículas componentes.

INTRODUCTION

When drilling a well, it is of major importance to control the loss of fluid through the wellbore into the porous formation to improve the wellbore stability and decrease risks of formation damage when reaching the reservoir. Static or cross flow filtration has been the subject of several laboratory studies [1-10]. It has been shown that for dynamic filtration, a steady state situation could be reached characterized by constant filtration flow rates and constant thickness of the filter cake. It is not clear whether this equilibrium thickness results from the erosion of the cake or from particle deposition stopping due to the shearing action of the fluid. Many authors have observed that equilibrium dynamic filtration rates are difficult, sometimes impossible to re-establish after a period of static filtration, leading to conclusion that once formed, a cake is difficult to abrase. This gives the preference to the second mechanism according to which the shearing action sweeps away solid particles from approaching the outer surface of the cake. At one point particle deposition becomes thus impossible when the drag force exerted on arrival of particles at the surface via filtration transport becomes lower that the tangential hydrodynamic shear force that tends to keep them circulating. According to this hypothesis, colloidal state of the suspension will play a major role in cake buildup and should have a significant effect on the overall filtration process.

Our objective in this study was to obtain from an experimental work using different shear conditions and different states of agregation of an aqueous suspension more insight of dynamic filtration mechanisms. In particular we tried to establish relationships between cake texture obtained by filtration of the suspension and its macroscopic properties, mainly filtration properties. A parallel is made with static filtration experiments for which this kind of correlation has already been reported [11-13].

The base fluid studied is a colloidal suspension of bentonite. Bentonite is characterized by a plate-like shape and small size of the particles. The basic unit of the clay is a layer of 1nm thickness and the clay crystal is defined as a parallel stacking of these layers. The colloidal state of the suspension is then characterized by the size and shape of these elementary crystals (this parameter can be described by transmission electron microscope TEM observations) and by the association mode of these crystals at higher scale (which can be

described by low temperature scanning electron microscope LTSEM). Degree of aggregation of clay platelets is controlled by the electrochemical nature of the suspending medium, in particular the type of electrolytes added. In this study we studied the influence of addition of salts (monovalent and divalent electrolytes) on filtration properties and cakes textures obtained in static and dynamic conditions.

1 EXPERIMENTAL

1.1 Colloidal clay suspension as a model drilling fluid

A classical component of water-based muds is montmorillonite clay. The ability of bentonites to enhance viscosity and control filtration arises from their swelling properties. Montmorillonites are clays where surface charges are mainly due to octahedral substitutions of Mg by Al. This overall negative charge is compensated by exchangeable mono or divalent cations located in the interlamellar spaces. In this study we used a Wyoming clay "Greenbond" from *CKS Inc.*, that is a seventy percent sodium, thirty percent calcium smectite. The cationic exchange capacity CEC of the clay is about 0.56 meq/g. A concentration of 30 g/l of bentonite in suspension was used. Different electrolytes have been studied: monovalent salt NaCl, KCl and a divalent salt $CaCl_2$. Fluids were prepared by mixing the clay in distilled water at double concentration and letting the clay particles to swell for 24 hours. Electrolytes (typically 0.5 mol/l) were added after the preswelling of the clay.

1.2 Filtration experiments

Two types of filtration experiments have been performed: API static and dynamic filtrations on paper filters. API filtrations were performed on a Baroid filter press under a pressure drop of 7 bar on paper filters. Dynamic filtration experiments have been performed using a dynamic filter press with a cone/plane geometry. Rotation of the cone provides a shear rate that is uniform across the surface of filtration. This equipment is illustrated in Figure 1 and has been described elsewhere [8]. Filtration experiments were performed with a typical pressure drop of 7 bar and shear rates at the surface ranging from 50 to 400 s^{-1}.

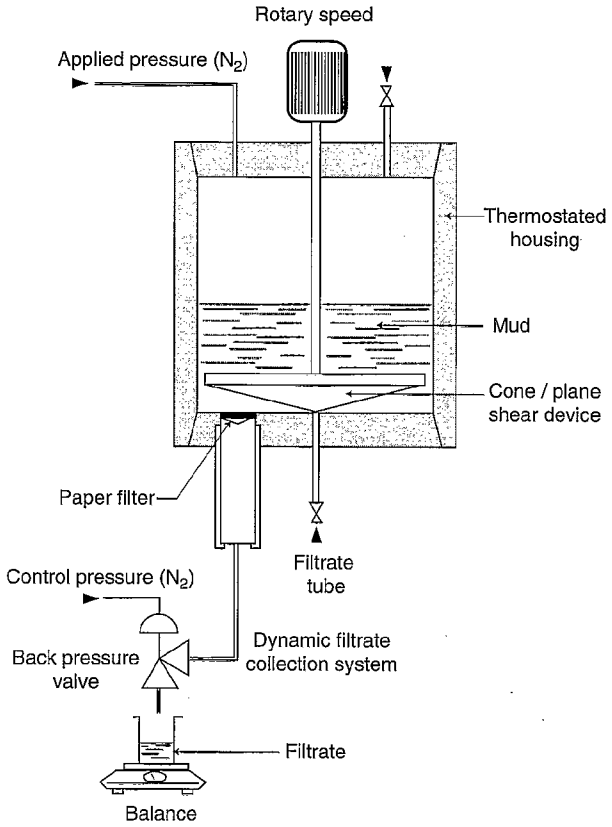


Figure 1

Cone plane-geometry dynamic filtration cell.

1.3 Cryo-SEM cake observations

When hydrated samples are subjected to drying, the capillary forces created at the air/water meniscus may exceed 100 bar in small pores leading to considerable shrinkage and distortion of the original structure. These effects can be overcome by special preparation methods like critical point drying, exchange of water by other solvants, or cryofixation. In the latter method, the ideal goal is the vitrification of water or microcrystalline ice formation that does not disturb the structures. Several sophisticated methods have been developped to reach this goal on pure water or biological samples but they apply to very small droplets or thin sandwiches (<50 microns thick) which are not realistic options for the study of cakes. A compromise has to be made between the necessity of extracting a

small piece compatible with the freezing process and avoid damage associated with sampling process. Fortunately, freezing of cake is not freezing of pure water: cakes are concentrated suspensions and contain a great quantity of salts which increases the osmotic potential and therefore changes the freezing conditions by decreasing the .ice nucleation temperature. Quenching methods appear then to be the most appropriate. This fast freezing reduces ice crystal damage without completly avoiding it. Conditions have been defined to get reproducible results and avoid radial distorsions that are characteristic of insufficient freezing velocity. Cryoscanning electron microscopy observations of filter cakes have been performed according to the previously described procedure [11].

2 RESULTS AND DISCUSSIONS

2.1 Comparison of static and dynamic filtrations (system bentonite + NaCl)

Let us first describe the results obtained with the system composed of bentonite 30 g/l and NaCl 0.5 mol/l. On Figure 2 are represented the evolution of the filtrate volume as a function of square root of time for different shear rates, from static to 400 s^{-1}.

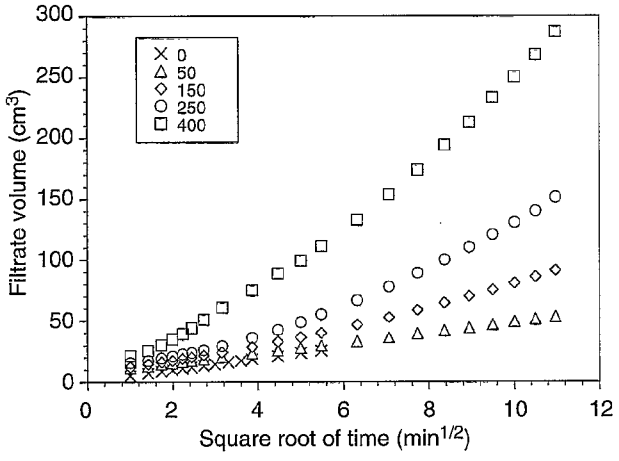


Figure 2

Influence of shear rates on filtration properties for a suspension of bentonite GB + NaCl. Filtrate volume as a function of square root of time.

We observe as expected an increase of the filtrate volume (and consequently of the flow rate) when increasing the shear rate. On static conditions or at very low shear rates, the filtrate volume is linear with $t^{1/2}$, whereas at larger shear rates after a short time the regime gets linear with time. Qualitatively it is possible in dynamic conditions to distinguish 3 filtration regimes:

- a spurt regime that corresponds to the filtrate volume that appears almost instantaneously. This volume is the volume necessary for building a continuous film of cake of low permeability at the surface or within the paper filter. This spurt was not observed in static conditions on paper filters;
- an intermediary regime where the cake thickens and the filtrate volume increases linearly with square root of time. This regime corresponds to a quasi-static regime;
- a filtration regime that appears to be linear with time. This regime is observed for long times and at high shear rates and corresponds to the filtration through a cake of constant thickness.

The laws describing the filtration process can be easily described for the two limiting cases.

2.1.1 Static filtration

Assuming that no particle are able to go through the filter and defining b as the cake specific volume, i.e. the volume of cake deposited by unit of filtrate volume:

$$b = \frac{V\text{cake}}{V} = \frac{eA}{V}$$

Darcy's law can be written as:

$$Q = \frac{dV}{dt} = k\frac{A^2\Delta P}{\eta bV}$$

where k is the cake permeability and η the filtrate viscosity.

Assuming that the cake permeability and the cake specific volume are constant throughout the filtration (this point is in agreement with cake observation by cryo-SEM and has been discussed elsewhere [11-13]), it is easy to see that in static condition the filtrate volume (V) as well as the thickness of the cake (e) increases linearly with square root of time.

$$V = \left(\frac{2\Delta PA^2}{\eta}\frac{k}{b}t\right)^{1/2} \quad \text{and} \quad e = \left(\frac{2\Delta P}{\eta}kbt\right)^{1/2}$$

From these equations it comes that the filtration velocity (U) decreases continuously with time, inversely proportional to square root of time:

$$U = \frac{Q}{A} = \frac{1}{A}\frac{dV}{dt} = \frac{1}{2}\left(\frac{2\Delta P A^2}{\eta}\frac{k}{b}\right)t^{-1/2}$$

2.1.2 Dynamic filtration: limiting case where a cake of constant thickness is obtained

We suppose here that for given shear conditions the cake reaches a constant thickness $e = e^*$. We can then define a critical filtration velocity U^* which is simply given by Darcy's law:

$$U^* = k\frac{\Delta P}{\eta e^*}$$

In this regime the filtrate volume varies linearly with time and the filtration velocity is constant. Results obtained with GB + NaCl confirm the obtention of this regime (Fig. 3) and we observe that the critical filtration velocity U^* increases with shear rates.

2.1.3 Modelisation

During the dynamic filtration process, as the filtrate passes through the filter, solid particles constituting the suspension are either deposited on the cake and make it thicker or are swept away by tangential hydrodynamic shear forces. In order to describe the filtration mechanism, we assume that the particles are isolated from each other in the suspension. Then a particle is submitted to two main forces:

- a normal drag force F_y, caused by the filtrate flow through the filter, proportional to the radius of the particle;
- a tangentiel hydrodynamic force F_x, proportional to the square radius of the particule and to the shear rate. This force is constant during the whole filtration.

Using Stokes law these two forces can be written as [2]:

- $F_y = \alpha\, 6\pi\eta\, a\, U_y$
- $F_x = \alpha\, 6\pi\eta\, a\, U_x = \alpha\, 6\pi\eta a^2\gamma$, as $U_x = a\gamma$

where α is a shape factor.

At any time during the filtration, the particle of radius a will be deposited only if $F_x < f\, F_y$, where f is a friction coefficient.

As U_y and thus also F_y decreases during the early stages of the filtration due to the thickening of the cake the filtration of a monodisperse suspension will stop when $F_x = f\, F_y$, that is:

$$a\gamma = f\, U_y^*$$

It appears that the critical filtration velocity should vary linearly with γ. This relationship between shear rate and filtration velocity is illustrated on Figure 4 for shear rates larger than 50 s^{-1}. For 50 s^{-1} low shear rate, the filtration properties of the cake are very near to the static ones and the equilibrium flux is not reached after 2 hours of filtration.

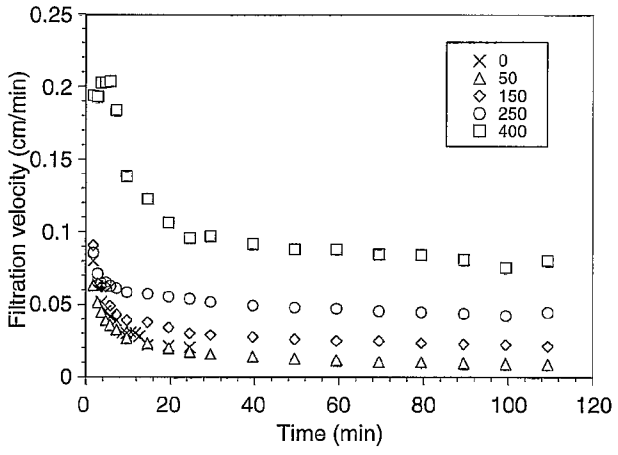


Figure 3

Influence of shear rates on filtration properties (system GB + NaCl). Evolution of the filtration velocity with time for different shear rates.

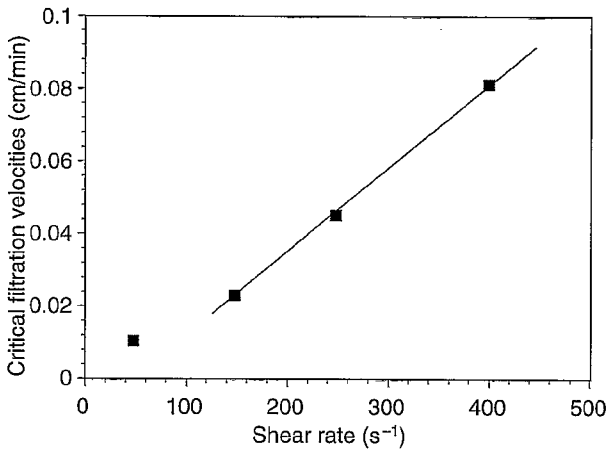


Figure 4

Influence of shear rate on filtration velocities obtained after 2 hours of filtration.

2.2 Influence of the added electrolyte nature

We have studied the influence of addition of electrolytes to the bentonite suspension. Two monovalent salts (NaCl and KCl) and a divalent salt ($CaCl_2$) have been studied.

2.2.1 Static filtration

Filtration measurements

Filtration curves are reported on Figure 5. They show that the filtrate volume increases in the order GB + NaCl < GB + $CaCl_2$ < GB + KCl

Cakes textures

In previous experiments [11] and [12], we have compared the influence of two electrolytes NaCl et $CaCl_2$ and found good correlations between cakes textures observed at the micrometer scale by cryo-SEM and filtration properties of the mudcake. A strong aggregation within the cake was observed in presence $CaCl_2$ which drove us to the conclusion that the permeability was related to the aggregation mode of particles at this scale. The complementary work on KCl suspensions done here lead us to the conclusion that this parameter is not the only one governing cake permeability. As seen on Plate 1, the cake GB + KCl is characterized by a very similar texture to that of GB + NaCl, though its permeability is found much higher than that of the GB + NaCl cake (66.2 μDarcy for GB + KCl instead of 13.5 μD for GB + NaCl). TEM observations were therefore undertaken in order to describe cake texture at the nanometer scale. Observations were made on thin sections of the freeze-dried cakes after embedding in resin. The results reported on Plate 2 show that the arrangement of crystals within the pore walls of the cake are very different from each other. In GB + NaCl cake, crystals are found as 2D, flat and flexible platelets partially overlapping each other, thus enabling the construction of a continuous impermeable film, whereas GB + KCl cake is composed of an imperfect stacking of rigid and isotropic "brick-like" crystals. The arrangement of crystals within the pore walls seems to play a major role in cake properties. In order to give an other evidence of the imperfect stacking of crystals in the case of GB + KCl, we have measured the relaxation properties of the cake by submitting it to a filtration of water using a cycle of different differential pressures, namely 2 bar, 7 bar and return to 2 bar. We defined a relaxation coefficient as the ratio of the permeability measured at 2 bar after the cake has been submitted to a higher pressure (7 bar) over the first permeability measured at 2 bar. The relaxation coefficient varies in the order NaCl < $CaCl_2$ < KCl as shown on table 1, which illustrates a high capacity of GB + NaCl and GB + $CaCl_2$ cakes to recover their initial properties after being compressed and the high sensitivity to pressure of the cake GB + KCl. The deformability of crystals and their ability to overlap each other seems to be an elementary condition to form a resistant and impermeable cake.

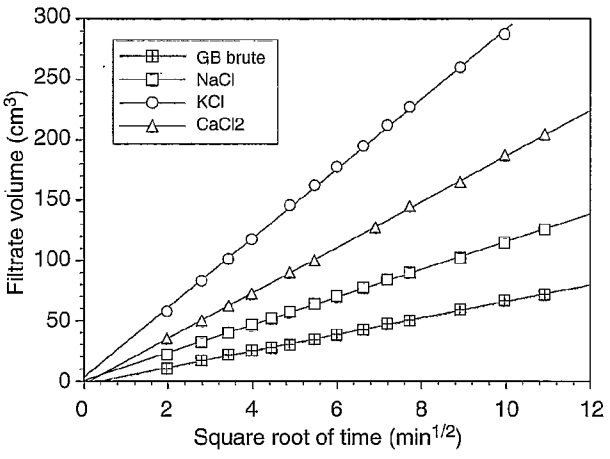
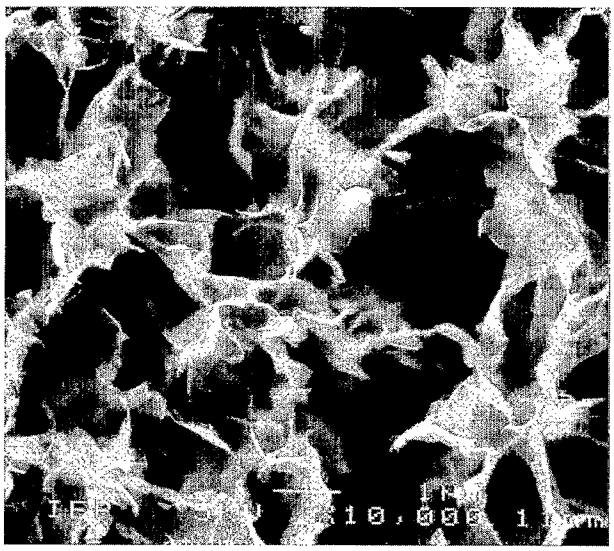


Figure 5

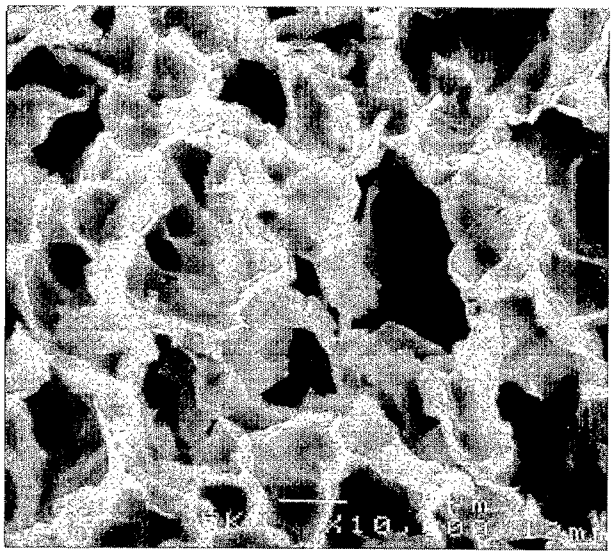
Filtration properties of different systems (bentonite GB + electrolytes 0.5 mol/l).

TABLE 1

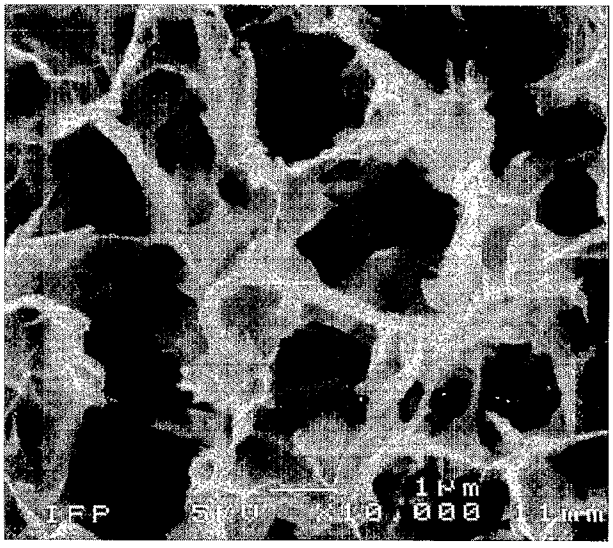
Electrolyte (0.5 mol/l)	Relaxation coefficient (%)
None	85
NaCl	51
$CaCl_2$	64
KCl	21



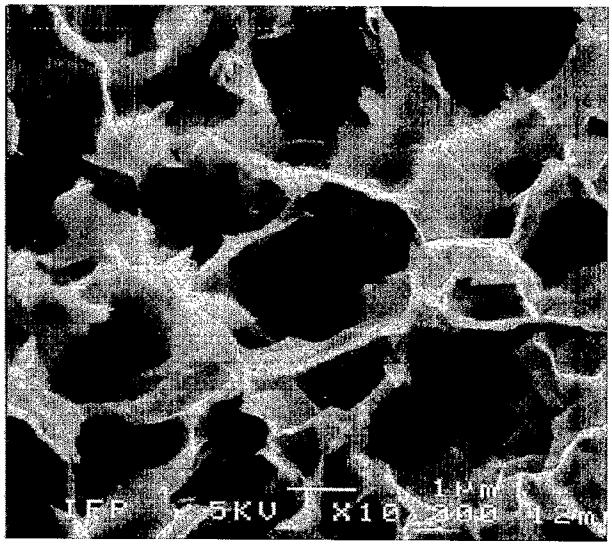
GB



GB + $CaCl_2$



GB + NaCl



GB + KCl

Plate 1

Influence of electrolyte on cake texture. Static filtration Cryo Scanning Electron Micrographs (white scale bar = 1 micron).

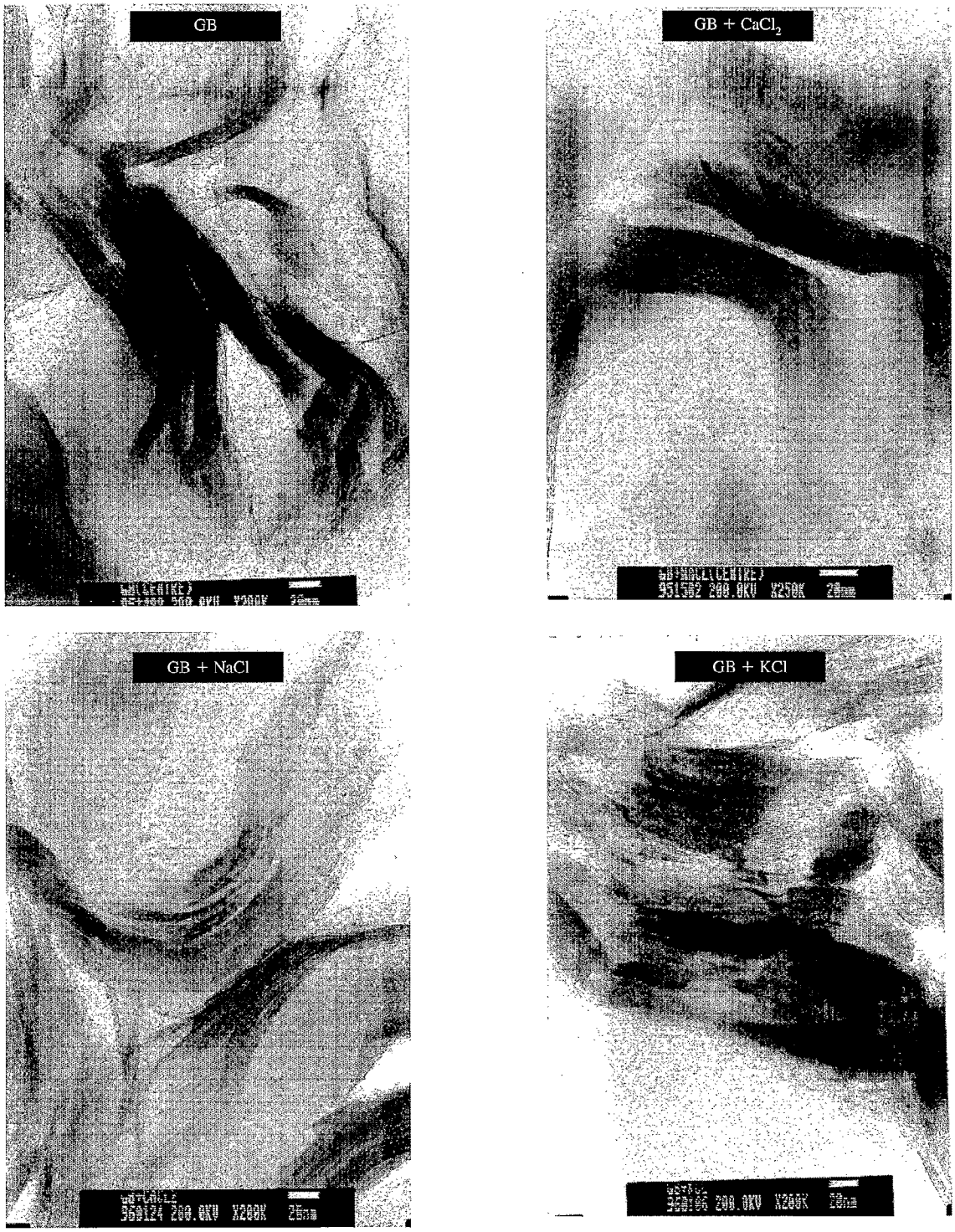


Plate 2

Influence of electrolyte on clay texture. Static filtration Transmission Electron Micrographs (white scale bar = 20 nm).

2.2.2 Dynamic experiments

Filtration measurements

Filtration properties at 400 s^{-1} expressed as the evolution of filtration velocities as a function of time are presented for the 4 systems on Figure 6. If we compare the two systems GB + KCl and GB + $CaCl_2$ we observe that equilibrium velocity is not reached after two hours filtration for both systems. Velocity decreases rapidly in the very first minutes of filtration and then decreases very slowly with time during the whole filtration. When compared to static results, it can be noted that filtration properties of the two cakes are not in the same order, as GB + $CaCl_2$ gives higher filtrate volumes in dynamic and GB + KCl in static. In order to explain this behavior, we have measured the thickness of the cakes by cryo-SEM and calculated their permeabilities. Note that cake thicknesses have been measured after opening of the pressurized cell and cake relaxation. As a consequence the measured thickness could be a bit larger than the in situ thickness and the permeabilty would then be a little overestimated. Measurements and calculations for the 4 systems are reported in Table 2. It appears that static and dynamic cakes have very similar permeabilities for a given suspension, except for GB + $CaCl_2$ which exhibits a singular behavior: the permeability of the dynamic cake is much lower that the static one, and the large filtrate volumes obtained are due to the very low thickness of the mud cake.

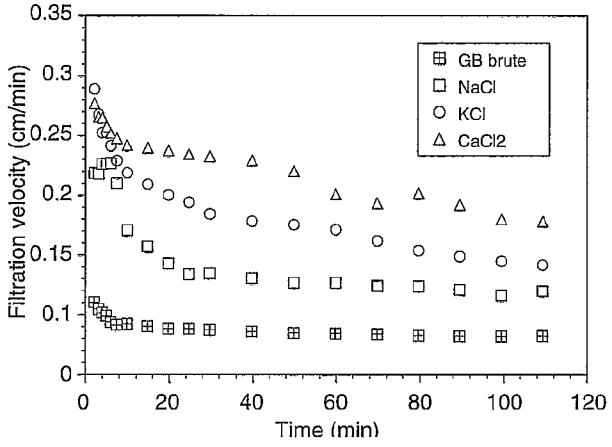


Figure 6

Evolution of filtration velocities with time for different systems (bentonite GB + electrolytes 0.5 mol/l).

TABLE 2

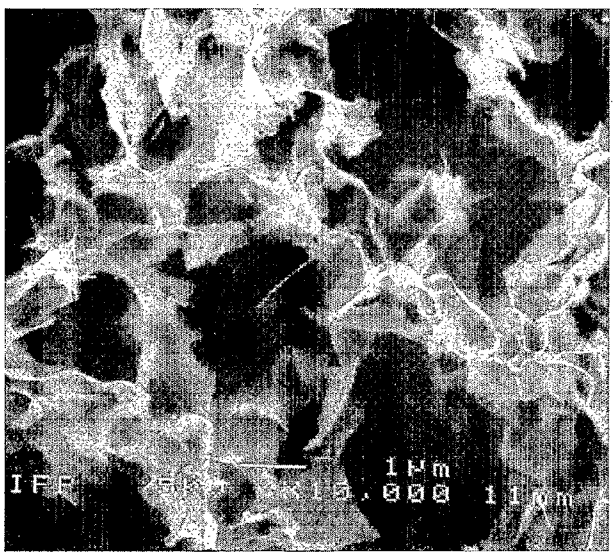
Systems	Cake thickness (mm) 30" static	Cake thickness (mm) 120" dynamic	Static K (μD)	Dynamic K (μD) (400 s^{-1})
GB	4	0.5	4.5	4.4
GB + NaCl	7	0.8	13.5	15.5
GB + KCl	16	1.5	66.0	39.0
GB + $CaCl_2$	11	0.2	44.0	7.5

Textural study of dynamic cakes

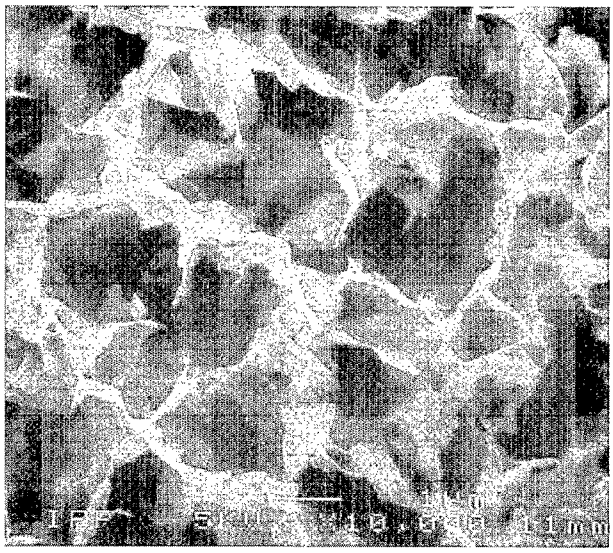
Textural study of dynamic cakes shows an increasing homogeneity and a decreasing pore size of the bentonite network. Plate 3 illustrates the effect of shear rate on a GB suspension: shear rate induces the dissociation of loose aggregates in the suspension leading to shorter pore walls and smaller cells but the structure of the pore walls remains the same. This has no effect on the permeability of the cake GB as shown on Table 2. Similar results are obtained in the case of GB + NaCl and GB + KCl. For GB + $CaCl_2$ the texture of static and dynamic cakes are very different (Plate 4): for the cake obtained in dynamic conditions, cells are smaller and the pore walls are no more made of large and flocculated aggregates. The structure of the pore walls is very similar to the one of GB + NaCl obtained in the same conditions.TEM results brings some more details about the arrangement of crystals in the pore walls: in the dynamic GB + $CaCl_2$ cake, pore walls are made of the association of very small and thin crystals comparable to the bentonite crystals observed in the absence of electrolyte.

Comparison GB + KCl and GB + CaCl: discussion

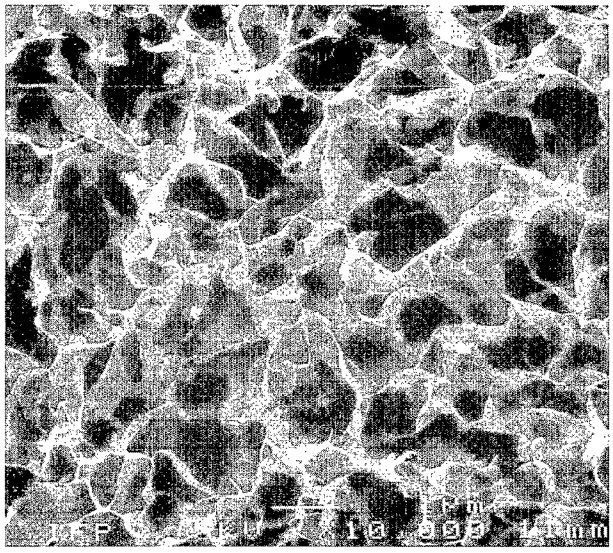
Due to divalent cations the suspension GB + $CaCl_2$ is mainly composed of large flocculated particles. During dynamic filtration, very quickly large particles are not able to deposit because of hydrodynamic shear forces. Only the smallest particles are able to deposit, which represents a very small fraction of the population. As a consequence the rate of deposition of these small particles is very low. After a few minutes of filtration, the cake has already reached its equilibrium thickness. The cake remains very thin. The permeability of the cake is low compared to static cake because it is only constituted of small particles. When compared



Static



50 s^{-1}



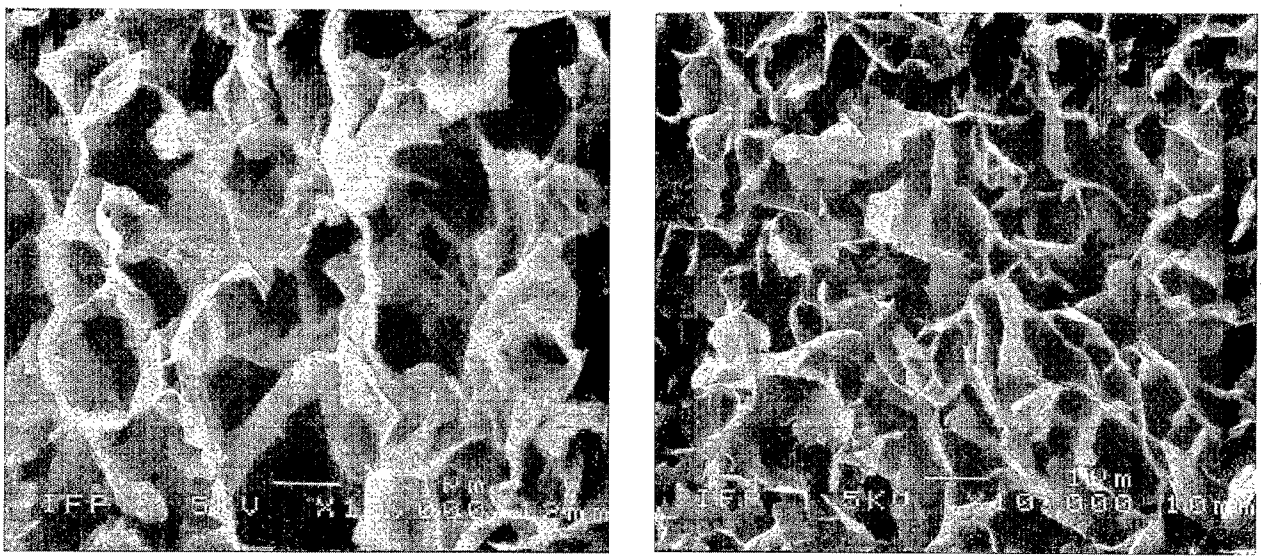
400 s^{-1}

Plate 3

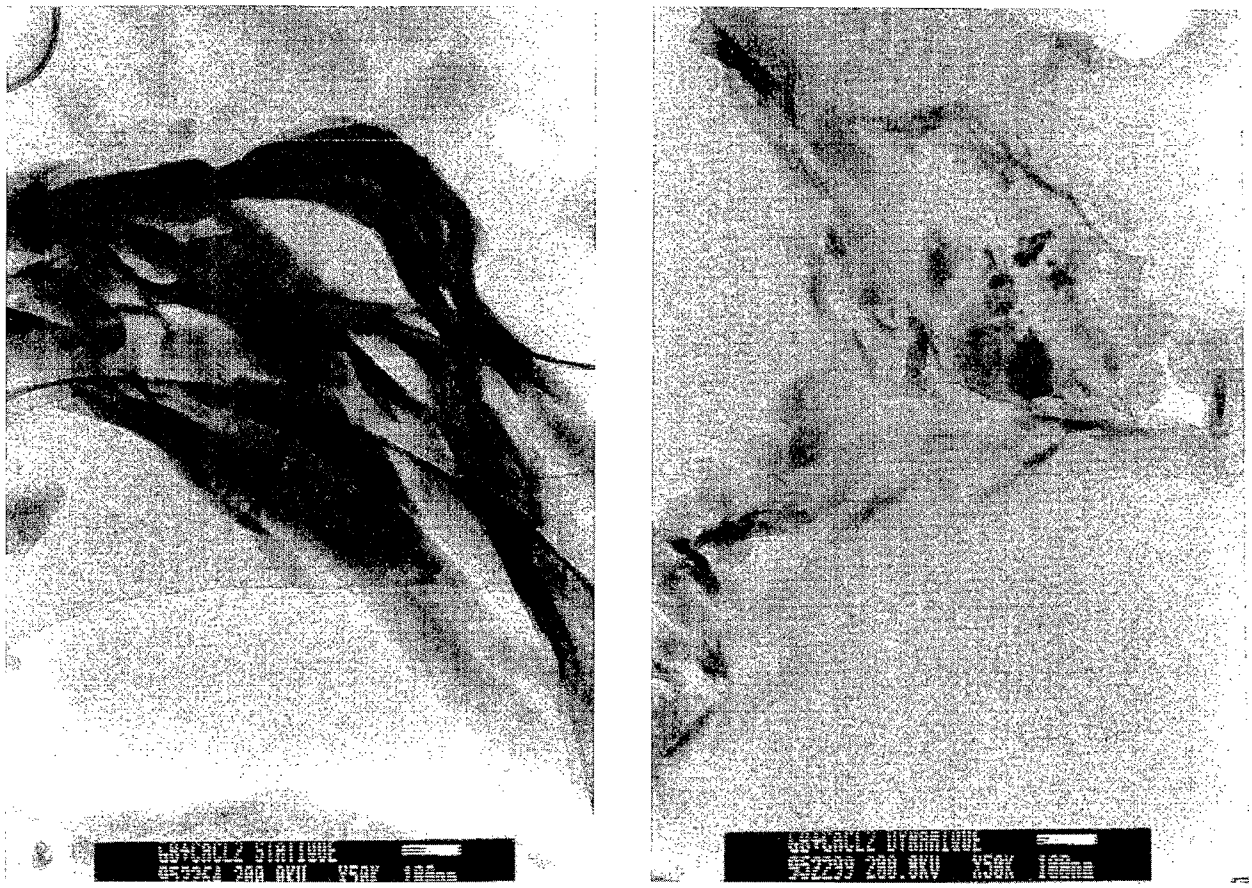
Influence of the shear rate on the cake texture. Greenbond cake.

to GB and GB + NaCl cakes one can note parallel to the very similar cake structures very close values of permeabilities. As filtration properties depend on both characteritics, rate of particle deposition and cake permeability, filtration properties remain poor with $CaCl_2$ because of the very small thickness of the cake.

Within the system GB + KCl the fraction of small particles is large. The rate of deposition is high and the cake is growing continuously. But the permeability of the cake is high in dynamic like in static because of the rigid morphology of the particles and as a consequence filtration properties are poor.



SEM micrographs (scale bar = 1 micron)



TEM micrographs (scale bar = 100 nm)

Plate 4

Influence of the shear rate on the granulometric fraction deposited. Left: GB + $CaCl_2$ static. Right: GB + $CaCl_2$ dynamic (400 s^{-1}).

For the two systems GB + $CaCl_2$ and GB + KCl dynamic filtration properties are poor but for different reasons: low rate of particle deposition for GB + $CaCl_2$ and large cake permeability for GB + KCl.

CONCLUSIONS

We have studied static and dynamic filtration properties of bentonite suspensions. Different states of aggregation have been obtained by using different electrolytes. TEM and cryo-SEM have been used as imaging technics for characterizing cake textures. Influence of the colloidal state of the clay suspensions can be summarized as follows:

- In static conditions, the rate of deposition is constant throughout the filtration and the permeability of the cake is mainly a function of the morphology of crystals or particles constituting the pore walls: overlapping of crystals that leads to the formation of impermeable pore walls is easier in the case of anisotropic, flexible and deformable particles.
- In dynamic conditions the rate of deposition is a function of the fraction of small particles that are initially in the suspension or formed after dissociation of loose aggregates by shear rates. The second action of shear rates is to exert a granulometric sorting of particles that are able to deposit. Permeability of the cake is controled by the morphology of particles that have been able to deposit.

ACKNOWLEDGMENT

Contribution of A. Audibert (*IFP*), H. Vandamme (*CRMD-Orléans*) and C. Allain (*FAST-Orsay*) to this study is gratefully acknowledged. The authors also wish to thank Ph. Dascotte, F. Lasquellec, M.C. Lynch (*IFP*) and J. Montes (*EAP*) for their help in this work.

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Final manuscript received in January 1997