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Influence of Chemical Additives upon Both Filtration and Electroosmotic Dehydration of a Kaolin Suspension

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ABSTRACT

The filtration of a kaolin suspension and the electroosmosic dehydration of a formed cake are studied. The influence of chemical pretreatments upon these two stages is studied for various added compounds: salts, acids, coagulants, flocculants, and surfactants. The main parameters of the process, such as the cake specific resistance, the energy consumption, and the cake dryness, are compared. The added compounds influence the process parameters differently. The choice of an appropriate method of pretreatment depends on which of these process parameters is of interest to improve. However, with an appropriate pretreatment, electroosmotic dehydration allows cake dryness to be markedly increased at low energy consumption.

INTRODUCTION

Highly concentrated colloidal dispersions are generally difficult to dehydrate. Thus, obtaining a cake incorporating the smallest amount of liquid is one of the parameters determining the efficiency of filtration processes used in such different applications as chemical and mining industries and the storage of urban and industrial wastes. Many of these sectors are increasingly confronted by the problem of sludge disposal. Thus, any process allowing better dehydration at lower cost is of interest.

For suspensions such as complex melts (industrial waste), mineral dispersions (silica, titanium oxide, clay), and organic suspensions (activated sludge),

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filtration and dehydration with the application of an electric field can be remarkably effective (1). Studies have now advanced to the semi-industrial level (2, 3), but energy consumption is generally rather high. Thus, there is a need for supplementary methods to increase the efficiency of electroosmotic dehydration and in particular to decrease energy consumption.

In a preliminary study (4) we attempted to show the best conditions to dehydrate a mineral suspension when a laboratory electrofiltration cell is used. In the present work we are interested in the effect of chemical pretreatment that can increase the efficiency of the electroosmotic dehydration of a kaolin suspension. This mineral suspension was chosen because it is difficult to obtain with less than 25 to 30% moisture after filtration (4).

In an industrial filter press the filtration and dehydration (by mechanical compression of an elastic diaphragm) are carried out successively. Because the mechanical compression stage is not effective for dehydrating the studied kaolin suspension, this stage was substituted for by electroosmotic dehydration (without pressurization). This improvement of the separation process involved improvement of both the filtration and electroosmotic dehydration stages. The method we used consists of complete cake formation (ordinary filtration) followed by electroosmotic dehydration. The effectiveness of the process depends upon the presence of such chemical compounds as salts, acids, coagulants, or surfactants in the solution. Each of these compounds can be adsorbed onto the solid surfaces and thereby modify the interactions between the particles and their behavior in an electric field. Thus, some significant parameters, such as the cake porosity, the cake specific resistance, or the size of particles, are modified when chemical compounds are added (5). In consequence, the filtration and electroosmotic flow rates are also changed. The filtration flow rate, given by Darcy's law (6), is

$$q = -\frac{1}{\mu\alpha\rho_s(1-\varepsilon)}\frac{\partial P}{\partial z} \tag{1}$$

where q is a filtration flow rate ($m^3/m^2 \cdot s$); P is a pressure in the liquid phase (Pa); z is a spatial coordinate (m); μ is a viscosity ($Pa \cdot s$); ρ_s is a solid density (kg/m^3); ε is a cake porosity, and α is a cake specific resistance (m/kg) which can be deduced from the Kozeny equation (7):

$$\alpha\rho_s = \frac{36k(1-\varepsilon)}{d^2\varepsilon^3} \tag{2}$$

where k is the Kozeny constant and d is the characteristic diameter of particles (m).

Equations (1) and (2) show that the filtration flow rate, and in consequence the filtration duration, depends upon the cake structure properties (α, ε, d) which can be modified by a chemical pretreatment.

The electroosmotic flow rate is given by Ref. 8. If the electroosmosis is carried out without pressurization, the electroosmotic flow rate is given by

$$q = \frac{1}{\mu\alpha\rho_s\varepsilon}\,\sigma_s r_E i \tag{3}$$

where σ_s is an effective charge onto solid surfaces (C/m^3), r_E is a specific electric resistance (Ωm), and i is a current density (A/m^2).

Equation (3) shows that the electroosmotic flow rate depends upon both the hydrodynamic (α, ε) and electric (σ_s, r_E) cake structure properties. Therefore, Eqs. (1)–(3) show that the addition of chemical compounds can change the properties either of filtration or of the stage of electroosmotic dehydration.

The aim of this work is to study the influence of chemical pretreatment on both the filtration and the electroosmotic dehydration stages. To quantify the whole process (filtration + electroosmotic dehydration), different parameters, such as the cake specific resistance, the volume of filtrate, the energy consumption, the duration of the electric field application, and the cake dryness, are compared. A comparison between these parameters allows us to choose which pretreatment to apply to obtain an overall improvement of the process.

MATERIALS AND METHODS

Electrofiltration Cell

The cell used in this study was described in a previous publication (4). A schematic representation of the installation and its components is shown as Fig. 1. The cell is a laboratory filter-press cell (filtration area: 50 cm^2; cake thickness: 2 cm) made of two iron plates, a polypropylene frame, and two filter clothes. This cell is equipped with an electric system including a direct current generator (0–500 mA, 0–500 V) and two grid like electrodes. The electrodes system is platinum/iron. The suspension initially contained in the tank is introduced inside the cell with the help of compressed air so that a cake can be formed between the two filter clothes.

The filtrate is weighed with a precision balance ($\Delta m = 0.1$ g) linked to a computer. This allows for continuous measurement of the extracted volume, V, with time, t. The generator is also linked to the computer for continuous measurements of both the applied tension, U, and current intensity, I.

Experimental Protocol

The experiments were carried out according the following protocol. The cake was first formed under a given pressure of 2 bars. No electric field was

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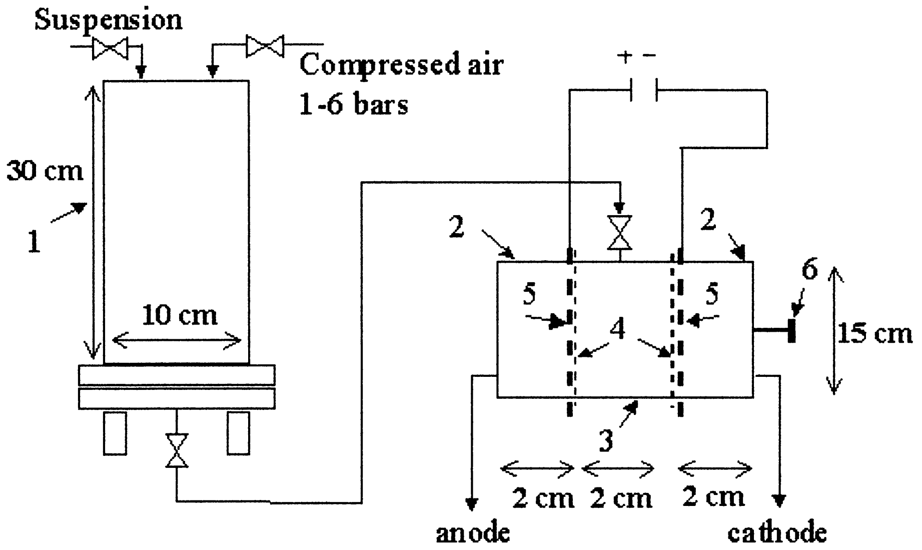

FIG. 1 Laboratory electrofiltration cell (dimensions are of the cell and tank): (1) tank, (2) iron plates, (3) polypropylene framework, (4) filter clothes, (5) electrodes, (6) locking screw.

applied through the cell until cake formation was completed. The electric field was applied only when the cake was fully formed and when the filtration flow was nil under atmospheric pressure. The electric current was then kept constant for fixed periods of time: 20 minutes at 100 mA, 10 minutes at 200 mA, and finally 100 mA once again. The voltage was increased when the amount of water contained in the cake decreased. Thus the voltage application was stopped at 250 V.

The experiments were carried out at room temperature (between 21 and 24°C). Most of them were run twice.

Chemical Compounds and Suspension

Three categories of solid chemical compounds (except liquid HCl) were dissolved in water to prepare various pretreated suspensions: 1) inorganic electrolytes [NaCl (Prolabo), $FeCl_3$ (Fluka) and HCl (Fluka)]; 2) organic flocculants [(acrylamide copolymers provided by the Degremont Co.: AS20 (nonionic), AS25 (5% anionic), AS32 (10% anionic), AS34 (20% anionic)]; and 3) nonionic surfactants [Brij 72 (HLB = 4.9) and Brij 76 (HLB = 12.4), provided by Aldrich]. Additionally, some experiments were carried out with two or more additives taken from the same or different categories.

The suspension was of the kaolin kerbriend (average size of particles = 1.2 μm, $\rho_s = 2.6$ g/cm^3). Before each experiment the dispersion was renewed. The dispersions were prepared with water (conductivity: 0.6 mS/cm). The concentration was 350 g of kaolin for each liter of water (350 g/L). The pH of the dispersion was then 7. The pH of the filtrate was still close to 7 after filtration. However, due to the electrolysis of water, once the electric field was applied the pH of the filtrate increased to 11. No significant changes in pH were observed with pretreated suspensions. Thus, in our experimental conditions (low chemicals/kaolin ratio) the electrolysis of water sets the final pH.

RESULTS

Table 1 summarizes the average results of both filtration and electroosmotic dehydration for each experiment.

Some experiments were implemented at various concentrations of additives because the pretreatment involves the determination of an optimum concentration (9, 11). Values obtained at various NaCl, AS20, or Brij concentrations confirm that if not properly adjusted, the chemical pretreatment does not improve the process efficiency. Highly concentrated solutions of flocculants (above 100 to 250 ppm) or surfactants (above 0.01 M) were not studied. In those cases the dispersion viscosity increases and filtration is very difficult. Moreover, as known from the literature (9), overdosing with flocculants can induce a deterioration in the filtration.

Table 1 shows that without any chemical pretreatment, electroosmotic dehydration leads to an increase in cake dryness. Figure 2 shows that it is possible to increase the dryness from roughly 68% up to 77%.

Moreover, using additives can improve the process efficiency. As can be seen from Table 1, almost all compounds belonging to the categories of *inorganic electrolytes* or *acids* improve the efficiency (a reduction of the filtration duration, an increase in the filtrate volume, and a decrease in the specific cake resistance). The optimum concentration has been deduced from the NaCl results and thus taken as 0.003 M. As is known from the literature (11), the addition of electrolytes raises the ionic strength in the suspension, there by reducing the effective range of electrostatic repulsion. This causes the coagulation of solid particles, which generally improves filtration performance. Table 1 illustrates the effect of some electrolytes upon the filtration stage.

Figure 3 shows plots of $t/V = f(V)$ for the filtration of a kaolin suspension with and without the addition of inorganic electrolytes. The plot labeled "kaolin" is for a pure kaolin dispersion without any pretreatment. The range of accuracy of the experiments within experimental error (deduced from three repeated experiments) is shown in Figs. 3 and 4. For reasons of legibility the range of accuracy is not shown for every value. Only the significant effects are discussed in what follows.

Figure 3 shows the best results obtained with each kind of inorganic compound added at 0.003 M. The slopes of the straight part of the curves corre-

TABLE 1

Filtration/Electroosmosis Dehydration Parameters of the Kaolin Suspension

							Cake		
	Duration of cycle (s)			Volume of filtrate (mL)				Dryness (%)	
Added compound	Filtration	Electro-osmosis	Total	Filtration	Electro-osmosis[a]	Total	Specific resistance α (Tm/kg)	After filtration	After electroosmosis
None	8,200	3,880	12,080	147	14.2 (13.2)	161.2	8.3	68.5	77
NaCl (0.001 M)	7,120	4,580	11,700	157	16.7 (13.1)	173.7	5.7	68	78.1
NaCl (0.003 M)	7,440	5,250	12,690	162	23.7 (16.3)	186.2	5.1	67	81.9
NaCl (0.1 M)	9,540	5,450	14,990	164	12.1 (8.0)	176.3	9.0	67	75.7
HCl (0.003 M)	6,750	7,800	14,550	165	22.3 (10.3)	187.7	5.7	70	83.7
$FeCl_3$ (0.003 M)	6,670	3,940	10,610	161	20.4 (18.6)	181.4	5.8	67.5	80.4
AS20 (50 ppm)	6,590	5,910	12,500	149.1	15.8 (9.6)	164.9	6.2	68	70.9
AS20 (100 ppm)	6,880	3,140	10,020	152	14.8 (17.0)	166.8	5.8	67	75.4
AS20 (250 ppm)	9,400	3,740	13,140	149.1	20.4 (19.6)	169.5	9.1	69	81.5
AS25 (50 ppm)	7,070	3,380	10,450	161.5	14.3 (15.2)	175.8	5.5	67	75.4
AS32 (50 ppm)	5,630	3,900	9,530	165	19.9 (18.4)	184.9	4.4	69	79.1
AS32 (100 ppm)	9,950	3,340	13,290	166.7	17.7 (19.1)	184.4	5.1	69	80.3
AS34 (50 ppm)	5,730	3,130	8,860	162.4	16.7 (19.2)	179.1	4.8	67	80.1
AS34 (100 ppm)	7,580	4,100	11,680	157.2	14.1 (12.4)	171.3	5.6	68	76.6
Brij 76 (0.001 M)	7,130	3,330	10,460	147.1	17.4 (18.8)	164.5	6.2	67	78
Brij 76 (0.01 M)	8,600	2,900	115,001	156.2	22.7 (28.2)	178.7	8.9	68	83.7
Brij 72 (0.01 M)	12,100	3,460	5,560	155.6	23.9 (24.9)	179.5	9.2	68	83.6
NaCl + $FeCl_3$	5,400	5,440	10,840	161	20.7 (13.7)	181.7	4.6	67	80.5
AS34 + $FeCl_3$ + HCl	5,170	4,000	9,170	153.1	7.2 (6.5)	160.3	4.0	68	72.1
Brij76 + AS34 + $FeCl_3$	9,890	3,980	13,870	131.4	23.7 (21.4)	155.1	8.3	69	85.6
Brij76 + AS34 + $FeCl_3$ + NaCl	12,060	3,880	15,940	155.4	23.3 (21.6)	178.7	7.2	70	86.1

[a] Values in parenthesis are the calculated average electroosmotic flow rates: q (mL/h) = V (mL)/t (h).

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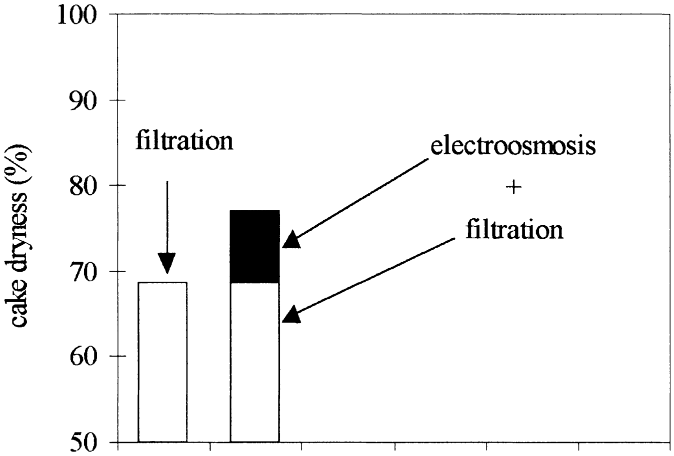

FIG. 2 Influence of filtration and electroosmotic dehydration upon cake dryness (no added chemical compounds).

spond to the values of specific cake resistance α presented in Table 1. The smaller the slope of the straight part of the curves, the lower the value of α. As can be seen from Fig. 3 and Table 1, the values of α decrease when inorganic compounds are added. For example, the addition of NaCl (0.003 M) decreases the specific cake resistance from 8.3×10^{12} to 5.1×10^{12} m/kg. The addition

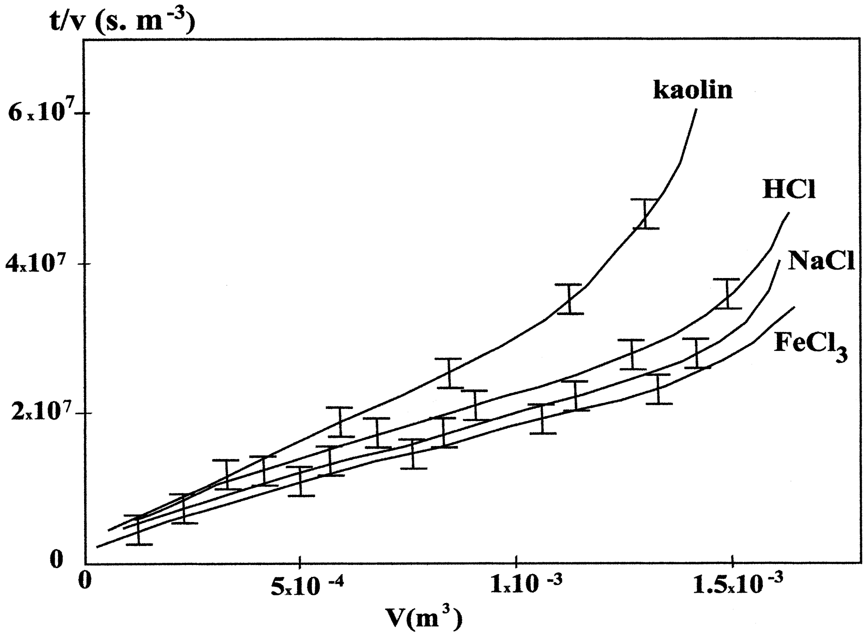

FIG. 3 Influence of inorganic compounds (0.003 M) upon cake resistance.

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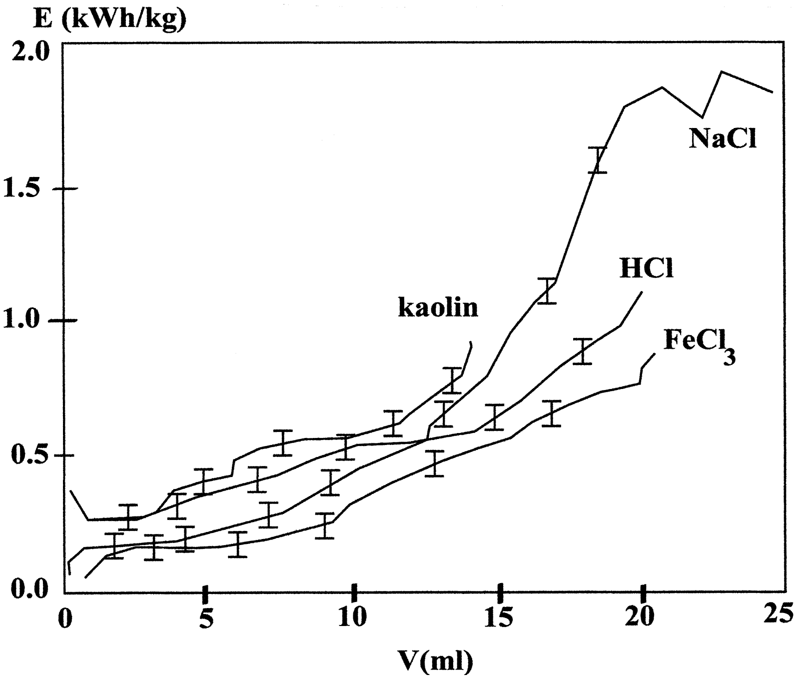

FIG. 4 Influence of inorganic compounds (0.003 M) upon energy consumption.

of HCl decreases the time of filtration (by 18%) and increases the volume of filtrate (by 11%). The addition of electrolytes also leads to an increase of the filtrate volume obtained during the electroosmotic dehydration stage. The final dryness of the cakes after both filtration and electroosmotic stages is increased (up to 83.7% for HCl), but the time of electroosmotic dehydration is somewhat longer when compared to the process without additives.

The results should also be analyzed in term of energy consumption. Figure 4 shows the energy consumption ($E = UI$) plotted versus the volume of filtrate extracted during the electroosmosic stage. As can be seen from Fig. 4, the addition of inorganic compounds increases the electroosmotic filtration volume at lower energy consumption compared with a nonconditioned kaolin suspension. Ionic compounds are therefore influential both for decreasing the time of electroosmotic dehydration duration and for lowering energy consumption.

The short electroosmotic time observed with the addition of $FeCl_3$ (Table 1) is due to a raised average electroosmotic flow rate compared to NaCl and HCl additives. The average value of the flow rate can be calculated with volumes and electroosmotic duration given in Table 1. This result can not be explained by lower cake specific resistance or lower cake porosity with the addition of $FeCl_3$. Values of these structure parameters after the filtration stage are close for the addition of various inorganic additives [except NaCl (0.1 M) which is not the optimum concentration]. Equation (3) shows that a difference in q for inorganic compounds may be due to a difference in the product $\sigma \times$

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r_E. In our protocol, electroosmotic dehydration is effected at constant electric current. In this case, E is proportional to U and to r_E. Consequently, a lower value of E involves a lower value of r_E. Figure 4 suggests that E (and consequently r_E) is lower with addition of $FeCl_3$. But Eq. (3) suggests that the product $\sigma \times r_E$ should be increased by the addition of $FeCl_3$ (because of the increased value of q). This means that the value of the effective charge (σ) should be increased by the addition of $FeCl_3$. As Fe^{3+} are multivalent complexing cations, they are more strongly bound at the kaolin/water interface than are Na^+ or H^+. This increases the value of σ and explains a shorter electroosmotic period with the addition of $FeCl_3$. It seems, therefore, that electroosmotic duration for ionic compounds depends on their effect on r_E and σ.

Most additives in the *polymer flocculants* category also improve process performance. Table 1 shows that improvement mainly concerns filtration. In fact, flocculants can markedly decrease the filtration time (up to 40%) and increase the volume of filtrate (to 11%). Therefore an interesting influence of flocculants concerns the specific resistances that can be lowered from 8.3×10^{12} to 4.4×10^{12} m/kg for AS32. This is shown in Fig. 5 which compares the best plots $t/V = f(V)$ with and without flocculants. This effect is explained by an aggregation of the solid particles induced by the addition of high molecular weight polymer flocculants. Due to our experimental conditions (small quantities of flocculants added), flocculation is based upon a bridging mechanism. In this type of flocculation, long-chain polymer molecules are adsorbed onto the particle surfaces by either electrostatic or hydrophobic bonding (11). As a consequence of the bridging, the average particle size of the aggregates

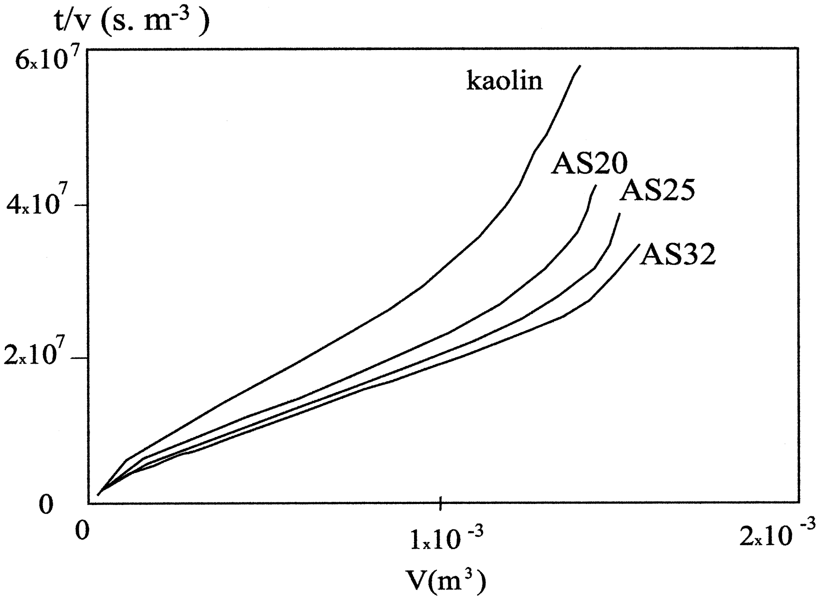

FIG. 5 Influence of flocculants (50 ppm) upon cake resistance.

increases, which causes an increase in the porosity and a better dehydration within a shorter time.

Flocculants are less efficient on the electroosmotic stage. However, they can slightly accelerate electroosmosis and decrease energy consumption Figure 6 presents the consequence of a shortened electroosmotic dehydration stage: with AS32 the energy consumption is reduced. Figure 6 also shows that the ionic degree of flocculants has an influence upon energy consumption: below roughly 8 mL of extracted filtrate, AS20 and AS25 (non or slightly ionic) are better than AS32. The situation is markedly different when more than 8 mL of filtrate is extracted. Thus the choice of a flocculant to lower energy consumption depends on the volume one wants to attain for electroosmosis dehydration. The value of the effective charge (σ) may be increased with adsorbed ionic flocculants. Thus, as for $FeCl_3$, an increased value of σ may explain the lower energy consumption due to a faster electroosmotic time through the addition of ionic flocculants.

Additives in the *surfactants* category can decrease filtration performances. Table 1 shows that long filtration time and increased cake specific resistance (Brij 72) can result from the addition of surfactants. Therefore, the determination of the optimum concentration of surfactants is also very important. This is demonstrated in Table 1 for Brij 76 at concentrations of 0.001 and 0.01 M. However, despite the low filtration increase in effectiveness (Brij 76, 0.001 M) or even some deterioration of filtration (Brij 76, 0.01 M), surfactants are most efficient on the electroosmotic stage. Among the studied compounds they are the ones which lead to the shortest electroosmotic time. For this du-

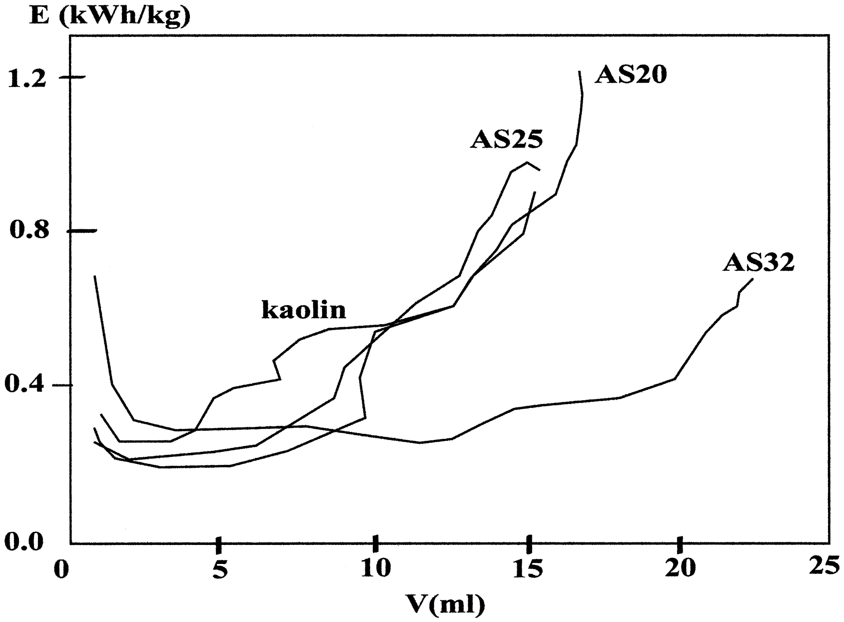

FIG. 6 Influence of flocculants (50 ppm) upon energy consumption.

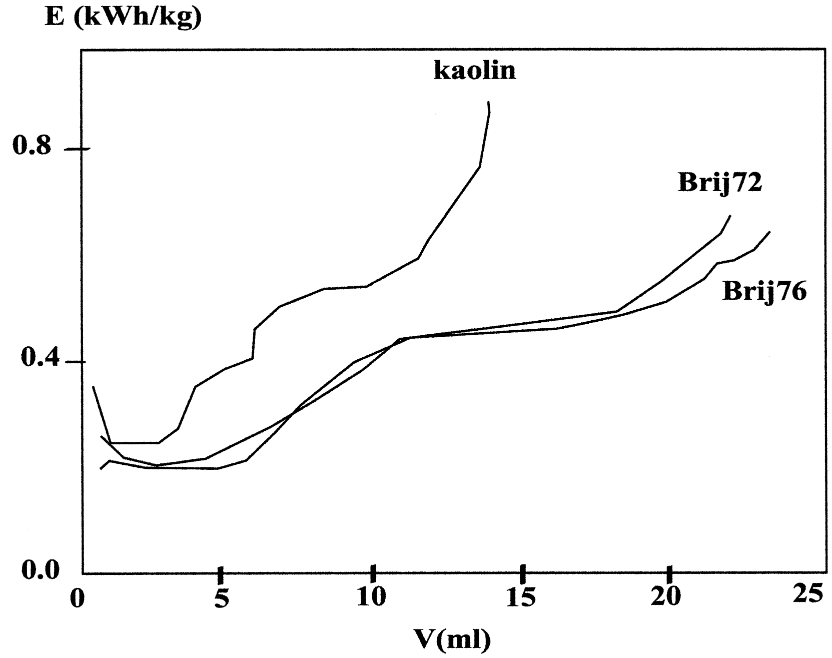

FIG. 7 Influence of surfactants (0.01 M) upon energy consumption.

ration, the energy consumption obtained is the lowest. Figure 7 shows that one obtains a 50% decrease in energy consumption in comparison to the kaolin suspension without additives. Unlike flocculants, Brij decreases the energy consumption whatever the extracted volume. This low energy consumption is due to a fast electroosmotic dehydration and a high flow rate (Table 1). As Brij are nonionics, they do not change the conductivity, the electric resistance, nor the adsorbed charge onto kaolin particles. However, adsorbed surfactants are known to markedly change the wetting properties of surfaces (10), changing them from hydrophilics to hydrophobics (a property used for mineral flotation) and enhancing the flow of water at the solid/water interface. This may induce a lower liquid viscosity at the solid/liquid interface and explain the increased flow rate obtained with surfactants during the electroosmotic stage.

Another improvement due to surfactants is the final dryness of a cake. Despite the fact that surfactants give cakes that are slightly less dried after the filtration stage, Table 1 shows that the volumes of filtrate obtained during the electroosmotic stage are roughly increased by 80%. This increase should probably be linked to the interfacial activity of the surfactants. It is known that as surface-active agents they can be adsorbed at the water/air interface and decrease the capillary pressure which takes place in porous media (10). Thus, it is possible to increase the volume of extracted filtrate without pressurization.

Finally results on the mixtures lead to the conclusion that the only parameter markedly improved is cake dryness which increases to 86.5% with AS34 + Brij76 + $FeCl_3$ + NaCl. Other results related either to specific resistance or time of filtration show that it is better to add the compounds separately rather than to mix them. The influence of each compound upon the efficiency of the process cannot be recognized when they are in a mixture.

CONCLUSIONS

The choice of a chemical pretreatment depends upon the parameters which have to be optimized during the filtration/electroosmotic dehydration process. Therefore the choice is different if one is interested in obtaining either a lower cake resistance, a low electroosmotic stage duration, a lower energetic consumption, or a high cake dryness. Moreover a given compound does not optimize all parameters of both filtration and electroosmotic dehydration stages. However, some main features can be deduced from a comparison of the influence of each category of compounds upon the most important parameters of the process: specific cake resistance, energy consumption, and final cake dryness.

Filtration. Figure 8 compares the best plots $t/V = f(V)$ obtained for each category of compounds. It is clear that the lowest specific resistance is obtained with a flocculant whereas the surfactant gives a poor result.

Electroosmotic Dehydration. Figure 9 compares results related to the decrease in energy consumption. The influence of the various compounds involved has to be divided into two parts: low or high extracted volume of filtrate (roughly above 10 mL). At a low extracted volume of filtrate, $FeCl_3$ leads to the lowest energy consumption, whereas AS34 and Brij are more efficient above 10 mL.

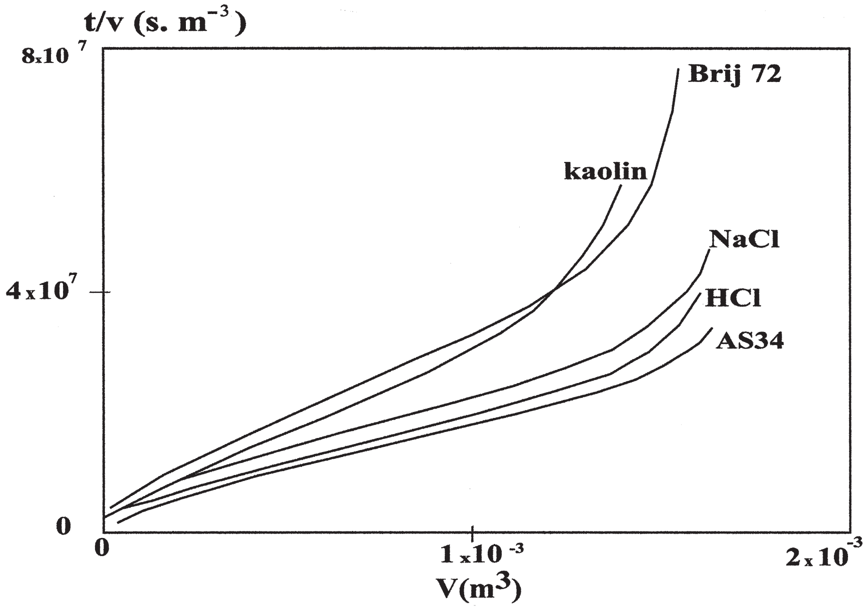

FIG. 8 Influence of various compounds on cake specific resistance.

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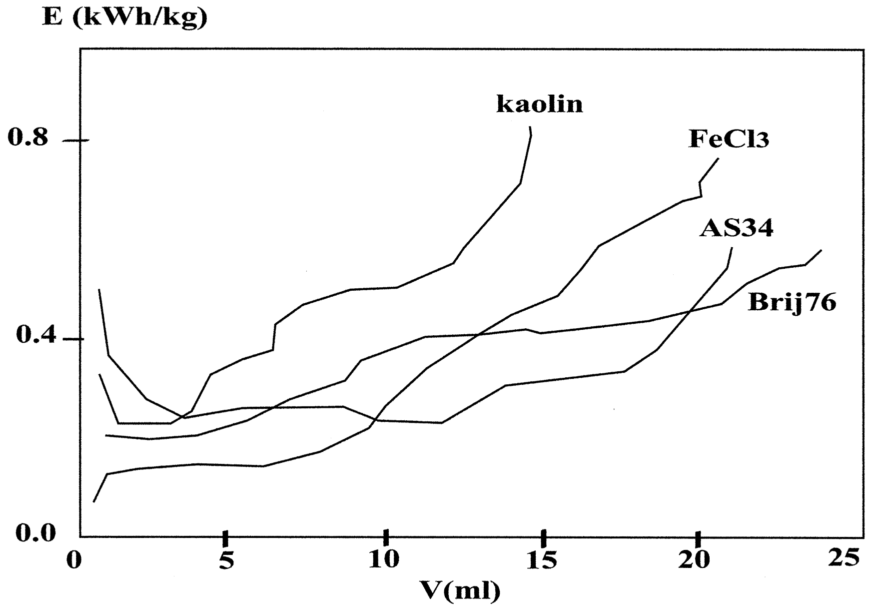

FIG. 9 Influence of various compounds on energy consumption.

Cake Dryness. Figure 10 shows the influence of various compounds on final cake dryness. As can be seen from Fig. 10, the dryness of a cake increases when the suspension is pretreated. The best results were obtained with HCl and Brij 76.

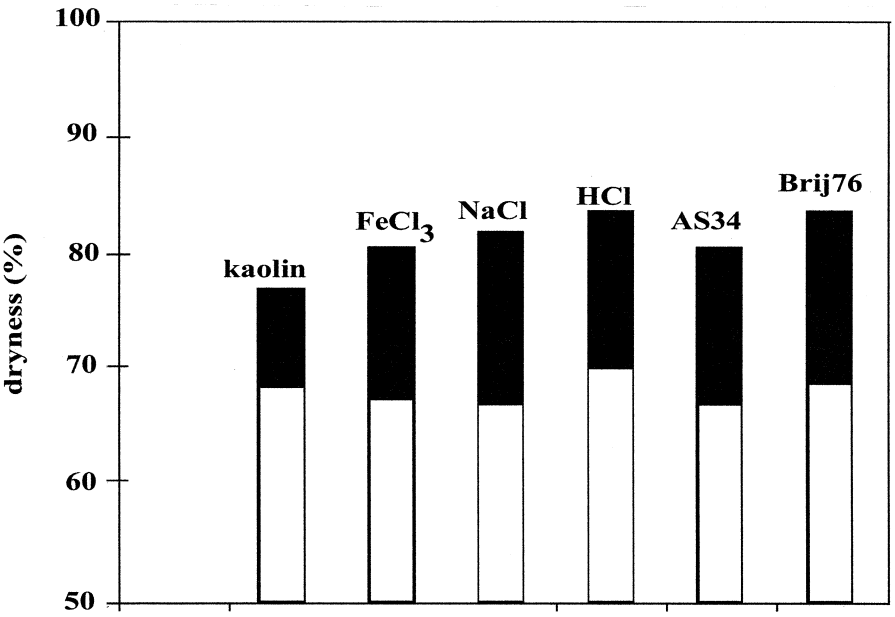

FIG. 10 Influence of various chemical pretreatments on cake dryness.

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TABLE 2
Overview of the Influence of Each Category of Compounds: (−) Negative Effect, (0) No Effect, (+) Positive Effect

	HCl	NaCl	$FeCl_3$	Flocculants	Surfactants	Mixtures
Cake resistance	+	+	+	++	0	+
Cake dryness	++	+	+	+	++	++
Energy consumption	+	+	++	+	++	+
Electroosmosis duration	−	−	−	+	+	0

Conclusions related to the other parameters are overviewed in Table 2.

In conclusion, it seems to be a general rule that the use of an appropriate chemical pretreatment of a kaolin suspension always improve both the ordinary filtration and the electroosmotic dehydration processes. The only restriction to this rule is that the electroosmotic duration can be longer when inorganic ionic compounds are added to the suspension.

NOMENCLATURE

q	filtrate flow rate ($m^3/m^2 \cdot s$)
P	pressure in liquid phase (Pa)
z	coordinate (m)
μ	viscosity (Pa·s)
ρ_s	solid density (kg/m^3)
ε	cake porosity
α	cake specific resistance (m/kg)
k	Kozeny constant
d	characteristic diameter of solid particles (m)
σ_s	effective charge onto solid surfaces (C/m^3)
r_E	specific electric resistance (Ωm)
i	current density (A/m^2)
U	tension (V)

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