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from fine particle filter cakes*

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# ELECTRICALLY ENHANCED WASHING OF IONIC SPECIES FROM FINE PARTICLE FILTER CAKES

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## ABSTRACT

A technique to enhance the removal of solutes and unwanted liquors from fine particle filter cakes by the application of an electric field during washing has been developed. For aqueous feeds of titania (rutile), the effects of DC electric field strength as well as cake and suspension properties such as pH and ionic strength were investigated experimentally.

Cake washing experiments showed how the rate of removal of cations ( $\text{Na}^+$  and  $\text{Al}^{3+}$ ) and wash liquors could be increased substantially at raised electric field when the downstream electrode worked as the cathode and the suspension forming the cake was at a pH well removed from its iso-electric point (IEP). In some circumstances, cations remaining from the initial production process of rutile could be removed from the particle surfaces by the electric field. For anions ( $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$  and  $\text{NO}_3^-$ ) under the same experimental conditions, the removal rate also varied with the electric field but in a manner generally contrary to that observed for the cations. When the polarity of the electrodes was reversed reduced effects were observed, however, a modest increase in the removal rate of anions was detected at longer wash times.

With suspensions adjusted to their IEP, filter cakes formed with more open structures and larger values of porosity. For cake washing under these circumstances an increase in the cation removal rate was noted, however, liquor flow rates tended to reduce when the electric field strength was increased and cake porosity decreased with time during the course of washing.

## KEYWORDS

Electric field; Cake washing; Electroosmosis; Ion removal; Particle surface cleaning

## INTRODUCTION

Displacement washing is used in industry to separate solutes from the voids in a porous medium by the application of fresh wash liquor or solvent that is miscible with the retained mother liquor. Soluble impurities are flushed out of the voids to leave them filled by the purer liquid [1].

For fine ( $<5\ \mu\text{m}$ ) and porous particles, such as pigments, nano-sized products and catalysts, inherent properties cause extended cake formation times and the low permeability cakes generally exhibit slow cake washing and deliquoring rates. In the extreme, the operating cycle of an industrial filter can be over 24 hours per batch and excessively large volumes of wash liquor may be consumed. In some processes the latter leads to high regeneration costs before the wash liquor can be re-used. The traditional filter for separating these solids has been the filter press and it is still the most widely used, but it suffers from the problems associated with long operating cycle times when processing very fine particles or solids that tend to form a gel-like structure. With increasing demands on product purity and environmental acceptability of waste materials, accompanied by gradual reduction in the quality of raw materials, there is also a need for better washing devices and the improvement of existing ones.

When dispersed in an aqueous medium, most particulate substances acquire a surface electric charge that may arise from ion dissociation, adsorption and/or dissolution. The charge tends to produce an ordering of the surrounding solution. In particular, ions of opposite charge are attracted towards the surface to form an electrical double layer (EDL) comprising the charged surface and a neutralising excess of counter ions that may span several micrometers. The degree of charge is frequently inferred through a measured zeta-potential and its presence has led many researchers to consider how particle surface charge can be utilised to improve the separation of solid/liquid mixtures. The processes investigated include electrofiltration, where a DC electric field is applied during deadend cake formation [e.g. 2-4] and the reduction of membrane fouling during crossflow filtration [e.g. 5-10].

There has been considerable recent interest in using electric fields to enhance filter cake post treatment processes. Most work has concentrated on electrodeliquoring where an electric field is applied across a cake to aid the pressure driven removal of the interstitial liquid and/or contaminants [11-21]; the principles underlying the technique are similar to those used to remove moisture and heavy metal ions from soils [22-29]. Increases in deliquoring rates for particulate filter cakes of up to several hundred percent have now led some researchers towards examining electrowashing, a technique where a DC electric field is applied during an otherwise conventional displacement washing process to promote the removal of solute(s) or prior to electrodeliquoring a cake is washed to alter its conductivity [30-32].

In electrowashing, a porous material such as a filter cake is saturated with electrolyte and placed in an external electric field,  $E$ . The potential gradient sets the ions in motion to create a current that passes through the material – in the context of Figure 1 a cation flow is created in the downward direction. In the absence of an applied pressure gradient, the velocity gradient and the associated shear stresses only arise to balance the pressure forces on the liquid. Thus the fluid velocity in a pore rises from zero at the plane of shear (the effective location of the solid-liquid interface) to a limiting value  $u_e$ . Only a small fraction of the total liquid volume lies within the EDL layer, thus the interstitial seepage velocity is approximated by  $u_e$ . The process is termed electroosmosis and the velocity,  $u_e$ , is the electroosmotic velocity. In addition, if a macroscopic pressure gradient  $\Delta p$  is applied across the porous medium to induce a further fluid flow, the Darcy seepage velocity,  $u$  – in Figure 1 this hydraulic flow is again in the downward direction.

When both the potential gradient ( $E$ ) and the hydraulic pressure gradient ( $dp/dx$ ) are applied to a filter cake, the superficial velocity  $u_o$  is induced such that:

$$u_o = u + u_e = \frac{k}{\mu} \frac{dp}{dx} + \frac{\zeta D \epsilon}{\mu} E \quad (1)$$

where  $k$  is the cake permeability,  $\zeta$  the zeta potential of the particles,  $D$  the liquid dielectric constant,  $\mu$  the viscosity of the liquid and  $\epsilon$  the porosity of the filter cake. The liquid flows due to the electric and pressure fields both induce solute removal from the cake interstices, with the motion induced by the electric field moving otherwise inaccessible ions toward the bulk flow.

The experimental data presented in this paper quantify the effects of operational parameters on liquor and solute removal rates and attempt to differentiate between the individual enhancements of mass transfer and electroosmotic flow. The experimental apparatus is described along with the characterisation procedures employed. The influences of ionic concentration, wash liquor flow rate, electric field strength and surface potential of the particles forming the cakes are shown.

## EXPERIMENTAL

An automated experimental apparatus that facilitated downward constant pressure filtration and displacement cake washing is shown schematically in Figure 2. The arrangement, made from stainless steel, comprised suspension and wash liquor feed reservoirs and a means for delivering compressed air to the filter at a controlled rate to maintain a constant pressure. Liquid from downstream of the filter test cell (area 120 cm<sup>2</sup>) was either directed to a PC interfaced electronic balance or, during washing, collected in 1 of 20 sample bottles mounted on a rotary indexing table. A PC was used to record the mass of liquids discharged from the filter and the filtration/washing pressures and also to control the air pressure on the feed side of the filter cell. Iridium coated titanium mesh was chosen as a reasonably inert material for both the upper and lower electrodes. The upper electrode was positioned such that it touched the top of the cake surface when formation was complete. The lower electrode was just below the underside of the porous metal support on which the 0.2 µm rated Gelman Versapor filter medium rested. In all experiments the electrode spacing was maintained at 13 mm to accommodate the thickness of the cake, membrane and support. A DC power supply (SM 35-45) manufactured by Delta Elektronika BV was connected to the electrodes inside the filter cell and operated in the constant voltage mode. In most experiments the voltage was applied such that the anode was above the filter medium/cake. For one sequence of experiments the electrode polarity was reversed to position the anode just below the filter medium.

An 8.6% v/v suspension of titania (TiO<sub>2</sub> rutile: density 4260 kg m<sup>-3</sup>, mean particle size ~0.3 µm) in a solution of 0.001M or 0.01M NaNO<sub>3</sub> was initially prepared by adding a known amount of the dry powder to an aqueous NaNO<sub>3</sub> solution. The resultant mixture was homogenised using an Ultra-Turrax T25 homogeniser (Janke & Kunkel, IKA Labortechnik) with a 25 mm dispersing tool at 8000 rpm for approximately 20 mins. The pH of all feed suspensions was adjusted to the required value with either nitric acid or sodium hydroxide. A stock salt solution was made by adding NaNO<sub>3</sub> into deionised (DI) water supplied from a Millipore MilliRX20 water purification system; the Na<sup>+</sup> and NO<sub>3</sub><sup>-</sup> were used as the tracer ions to track the progress of the displacement washing phase. Zeta-potentials for rutile were measured with a Malvern Instruments ZetaSizer and the variation of zeta-potential with pH is shown in Figure 3. Measurements were carried out at two different ionic strengths over the pH range 3.5 to 9, where the zeta-potential decreased with increasing pH. For a 0.001M solution the IEP occurred at pH 5.2 whereas for 0.01M solution a pH of 5.8 corresponded to the IEP.

Prior to each experiment the pipework throughout the apparatus was rinsed with DI water to ensure that it was free of any particle/solute residue from earlier experiments. Suspension at the required pH and ionic strength and DI water were added into the suspension vessel and washing liquid vessel respectively. A filter cake of the required 10 mm thickness was formed in the conventional manner by constant pressure filtration in the absence of an electric field. Cake formation was followed by a displacement washing phase using DI water either with or without the application of an electric field. In a typical sequence of experiments the applied voltage was varied over the range 0 to 30 V. During the course of cake washing, both the hydraulic washing pressure and the voltage remained constant and up to 20 wash liquor samples were collected and subsequently analysed for ion concentration. The cations were measured using atomic absorption spectroscopy (Varian SpectrAA-200) and the anions using liquid chromatography (Dionex Chromatography). Ions initially present on the surface of the rutile particles were identified using X-ray photoelectron spectroscopy (XPS). The moisture content and porosity of a filter cake were determined using a gravimetric analysis either before or after the washing phase depending on the nature of an experiment.

## RESULTS

The experiments were initially designed to observe the effects of the electric field, ionic strength, pH (zeta-potential) and electrode polarity on cake washing in terms of washing liquor flow rates and concentrations of Na<sup>+</sup> and NO<sub>3</sub><sup>-</sup> ions. In the light of experimental results, the same protocols

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were subsequently extended to include analyses for  $\text{Al}^{3+}$ ,  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$  ions. Four sets of experiments were carried out and the conditions are listed in Table 1. Initial experiments were performed to ensure reproducibility of the experimental data. For example, at pH 7.8 and 0.001M  $\text{NaNO}_3$  the specific cake resistance between repeat tests was  $3.13 \times 10^{12} \pm 1 \times 10^{10}$  and the mass of wet to dry cake ratio was  $1.24 \pm 0.01$ .

### Effects of Electric Field Strength

A typical way to describe cake washing is through a washing curve comprising a plot of normalised instantaneous solute concentration ( $C/C_0$ ) in the wash effluent versus wash time ( $t$ ). Figures 4 and 5 show how cation ( $\text{Na}^+$ ) and anion ( $\text{NO}_3^-$ ) concentrations in the wash liquor varied with wash time and applied voltage. In all cases the filtration pressure ( $\Delta p_f$ ) and washing pressure ( $\Delta p_w$ ) were 400 kPa and the initial suspensions were prepared in 0.001M  $\text{NaNO}_3$  and adjusted to pH 7.8. For these conditions, the suspensions exhibited a zeta-potential well removed from their IEP ( $\zeta \approx -47$  mV). During cake washing without an electric field, the instantaneous  $\text{Na}^+$  ion concentration in the wash liquors decreased gradually in the initial period up to  $\sim 600$  s and then reduced more rapidly with a tendency toward a limiting value. When an electric field was applied the measured and normalised  $\text{Na}^+$  ion concentrations initially increased with time and reached the maximum values of 1.02 ( $\Delta V = 10$  V), 1.18 ( $\Delta V = 20$  V) and 1.24 ( $\Delta V = 30$  V), implying an accelerated removal of sodium ions from the cake with increasing electric voltage. A mass balance on the sodium ions washed from the cake suggested that some removal of  $\text{Na}^+$  ions from the particle surfaces may also have occurred; additional reslurry washing experiments without an electric field showed that there were indeed  $\text{Na}^+$  ions on the particle surfaces that were removable by washing. It is also evident that following the initial period where significant differences in washing performance could be observed, the wash curves became quite similar. In general, an electric field improved cake washing most effectively during the initial stages of washing and when the electric field strength was greater.

At longer washing times the  $\text{Na}^+$  ion concentration is higher when an electric field is applied than is the case when there is no applied potential. The implication from the 'tails' of the curves shown on Figure 4 is that there is a slow mechanism removing ions from the filter cake [1] and that more ions are removed when an electric field is applied. This latter point is reinforced when the area under the curves is calculated, showing more  $\text{Na}^+$  removal at higher applied voltages. These observations are consistent with the reslurry washing experiments that showed  $\text{Na}^+$  ion desorption from the particle surfaces.

Regarding the  $\text{NO}_3^-$  anions, Figure 5 shows how the washing curve changed form as the applied voltage was raised. The plot shows how the number of anions removed in the wash liquid decreased with an increasing electric field, which was a consequence of the chosen electrode polarity. When the bottom electrode (downstream of the filter medium) is the cathode, the anions within the cake are subjected to an additional upward velocity. The direction of motion of the anions opposes both the electroosmotic flow and the flow induced by the hydraulic pressure gradient. The force causing counter-flow of the anions becomes more dominant as the applied voltage is increased.

The typical effect of an electric field on wash liquor flow rate is shown in Figure 6. In all cases the flow rates tended to reduce during electrowashing with the greatest reduction being observed toward the start of washing. For the case of no electric field, there was a relatively small reduction of the flow rate. In the presence of an electric field a more severe reduction of flow rate was observed due to a combination of a lowering cake porosity and a decaying contribution from electroosmosis as the ions originally present in the cake were replaced by the DI wash water. Superimposition of the electroosmotic flow on the hydraulically induced flow typically caused a 25% increase in wash liquor flow rate at longer times; greater increases were observed during the initial stages of washing and with larger applied voltages. It is interesting to note that the application of a small field causes a substantial increase in flow above the zero field case, but

further increases in the field do not lead to proportionate increases in the wash liquor flow rate. Such behaviour is in qualitative accordance with the Smoluchowski equation [32] which infers that electroosmotic flow is proportional to current density. Figure 7 gives an example of how the measured electric power/current reduced (whilst the applied voltage remained constant) during the course of washing.

To determine if the ions initially present on the surfaces of the rutile particles could be removed by electrowashing, samples of the dry powder were analysed using XPS. The majority of the ions remaining from the manufacturing process and attached to the surface were found to be  $\text{Na}^+$  and  $\text{Al}^{3+}$  cations as well as  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$  anions. Concentrations of  $\text{Al}^{3+}$  ions in the wash liquor samples were determined using atomic absorption spectroscopy and some typical data are presented in Figure 8. In the absence of an electric field, the  $\text{Al}^{3+}$  ions were progressively removed only after the majority of the potentially mobile  $\text{Na}^+$  ions from the particle surfaces and those from the  $\text{NaNO}_3$  tracer salt had left the cake. The application of a 20 V electric field, however, had a significant effect on the kinetics of cation removal. For  $\text{Al}^{3+}$  ions, there was a relatively rapid removal from the start of washing and the concentration reached a maximum value before falling, presumably because the majority of the mobile  $\text{Al}^{3+}$  ions had been removed. In the presence of the electric field it was possible to simultaneously remove both  $\text{Al}^{3+}$  and  $\text{Na}^+$  ions; the removal kinetics of the latter are more clearly observed on Figure 4.

The  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$  ions, along with the added  $\text{NO}_3^-$  ions, discharging in the wash liquor were detected using liquid chromatography. Examples of the anion concentration variations with time are plotted in Figure 9 for both the no field case and an applied potential of 20 V. The results indicate how nitrate ions were marginally more difficult to remove in the wash liquors and more marked differences were observed with the application of the electric field. As noted earlier, the chosen electrode polarity tended to induce forces on the anions that opposed the bulk flow of liquid in the cake. Results suggested that nitrate ions were influenced most by the electric field, although all data showed a reduced washing performance with regard to anion removal when the electrode polarity was such that the anode was above the filter cake.

### Effects of Ionic Strength

A sequence of washing experiments was also performed with the concentration of  $\text{NaNO}_3$  raised to 0.01M. All other experimental conditions remained the same with filtration and washing pressures maintained at 400 kPa, 10 mm thick cakes and pH 7.8. Results showed the same general trends as those recorded in Figures 4-6 for 0.001M  $\text{NaNO}_3$  with similar forms of variation for both cation and anion concentration with wash time. A slightly increased maximum dimensionless  $\text{Na}^+$  concentration was noted when an electric field was applied (e.g. at 30 V and 0.01M,  $(C/C_0)_{\text{max}}$  equalled 1.29 in comparison to 1.24 for the 0.001M case). For a raised  $\text{Na}^+$  ion concentration in the wash liquor there was generally a reduction in the  $\text{NO}_3^-$  ion concentration indicating a reduced washing performance with respect to anion removal.

Wash liquor flow rates were generally higher at 0.01M  $\text{NaNO}_3$ . The cake porosities were almost identical at each molar concentration, suggesting that there was an increased contribution from electroosmosis. As more salt is added to the initial suspension the double layer shrinks, tending to reduce the zeta potential of the rutile particles as shown in Figure 3. The current density arising from the application of an electric field is closely related to the ionic strength of the suspension filtered and the applied voltage (see also Figure 7). As expected, at the higher ionic strength a larger current and power were recorded, particularly toward the beginning of washing where the ion concentration was at its highest (a maximum power consumption of ~30 W was observed for  $\Delta V = 30$  V). In the cases where an electric field was applied, the current decayed to a low value within a short wash period and this decay was more rapid at raised field strengths.

### Effects of pH (Zeta-potential)

To assess the impact of zeta-potential on electrowashing, filter cakes were also formed from suspensions close to their IEP (0.001M NaNO<sub>3</sub>; pH 5.2) and subsequently washed both with and without applied electric fields. Due to higher filter cake porosities it was necessary to reduce both filtration and washing pressures to 100 kPa to facilitate the experimental measurements. The data shown in Figures 10 and 11 suggest that both anions and cations behave in a generally similar manner to that noted for electrowashing at pH 7.8. However, Figure 12 shows how the flow rate of wash liquor was dramatically reduced with applied voltage and elapsed wash time and was attributable to rearrangements of cake structure induced by the electric field and the flow due to the hydraulic pressure. When 30 V was applied across a cake, washing stopped soon after the start and no further ions were removed at the 100 kPa washing pressure.

The variation of cake porosity with wash time was determined by performing two series of otherwise identical experiments where either no field was present or a 20 V potential was applied between the electrodes. Within each series, an experiment was stopped at a set time and cake samples taken to gravimetrically determine a porosity value corresponding to a given position on the wash curve (see Figure 13). For the case of no electric field the largest change in cake porosity was noted during the first few minutes of washing after which there was essentially no further change. However, with an applied voltage of 20 V the average cake porosity was seen to fall from an initial value of 0.57 to a near constant value of 0.47 after a wash time of ~1000 s. At all times following the start of washing the cake porosity with no electric field exceeded that measured at an identical time with an applied field of 20 V.

### Effects of Electrode Polarity

A few experiments were performed with the electrode polarity reversed such that the cathode was the electrode in contact with the top of the cake during washing. In this case the filtration pressure was 400 kPa and the cakes were formed from suspensions initially made up in 0.001M NaNO<sub>3</sub> at pH 7.8. Here, the cations in the EDL and the interstitial liquids of the cake were subjected to a force in the opposite direction to the bulk liquid flow. For the given experimental conditions, an applied voltage of 15 V caused the liquid flow from the filter cell to cease as the electroosmotic flow counteracted the hydraulic liquid flow due to the applied hydraulic pressure.

Over the 0-10 V range investigated, the electric field had a negligible effect on the cation (Na<sup>+</sup>) removal rate, presumably because the Na<sup>+</sup> ion velocity towards the cathode was significantly less than the liquid velocity towards the anode when the reversed direction of the electroosmotic flow was used. Figure 14 shows that for an applied voltage of 5 V the NO<sub>3</sub><sup>-</sup> ion concentrations were always lower throughout washing compared with the no electric field case. Here, it seems that the electric field is not strong enough to influence the nitrate ion mobility in the desired manner. When the applied electric voltage was increased to 10 V the solute concentrations at longer times were above those recorded for identical washing times and no field indicating an improved washing performance with regard to anions.

## DISCUSSION

The experimental data presented in Figures 4-14 show that electric fields are able to enhance the removal of solute during a displacement washing process and also removes ions previously adhered to particle surfaces. Although the processes occurring at the microscopic level are likely to be complex, it is clear that an electric field induces additional ion movements and in many cases these are sufficient to move ions into the bulk wash liquid flow and thence out of the cake. For Na<sup>+</sup> and NO<sub>3</sub><sup>-</sup> ions these movements are represented in Figure 15 where the schematics reflect the general experimental observations.

When the electrode polarity is arranged such that the positive electrode is in contact with the top of the cake then the generated field has a tendency to induce cation (Na<sup>+</sup>) movement in the direction

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of the bulk liquid flow. Simultaneous with this, the induced anion ( $\text{NO}_3^-$ ) velocity opposes both the bulk liquid and cation motion. For the  $\text{Na}^+/\text{NO}_3^-$  ion combination, the anion mobility is ~42% higher than the cation mobility (see Table 2) and the net result is that from the onset of washing there is a hold-up of both anions and cations within the cake. Because of the bulk liquid flow, however, both the anions and cations tend to translate toward the bottom of the cake and the translation is likely to become more prominent as washing proceeds and the influence of the electric field is reduced. From the data in Table 2, calculations indicate that for an applied voltage of 30 V and typical flow conditions towards the start of an experiment, the ion and bulk liquid velocities are similar and in the region of  $120 \mu\text{m s}^{-1}$ . A combination of phenomena account for the observed behaviour with electric fields. On Figure 15, point (a) represents the place where the maximum cation concentration is leaving the cake. The concentration here is higher than the concentration at zero washing time due to the time lag that results from the cation hold-up. The initial reduction in anion concentration is a consequence of the induced upward anion velocity and fewer anions emerge from the cake during the initial stages of washing. When points (a) and (b) are reached, however, the electric field has less effect because of the reduced ion concentration in the cake and, particularly for the cations, there is a tendency for the washing curves to return to those recorded without an electric field.

The effects of the imposed electric field on washing become more difficult to interpret when a cake is filtered from suspension close to its IEP. Here, washing proceeds in conjunction with a continually changing cake porosity, particularly when the electric field is applied. Whilst both cation and anion removal rates can still be influenced by the electric field, the changing ion composition surrounding each particle simultaneously brings about a change of cake structure and thus a lowering of liquor flow rate. Table 1 shows that rutile cake formed at the IEP pH of 5.2 exhibits significantly higher porosity than cakes formed at pH 7.8 under otherwise identical conditions. When the DI wash water flows through the cake the average pH of the liquid phase within the cake is likely to increase as the wash displaces and subsequently mixes with the initial cake liquors. For rutile, an increase in pH from pH 5.2 will promote some spatial particle rearrangement within the cake and bring about the recorded reduction in porosity.

With a reversed electrode polarity the removal rates of both cations and anions became similar to those observed for no applied electric field. As the anion velocity is significantly higher than the cation velocity and now in the same direction as the bulk liquid flow, the induced cation velocity will tend to have a much reduced effect on the measured cation concentrations at the cake exit. Likewise, the counter flow to the anion flow is relatively small and the anions move toward the cake exit at a constant, but marginally reduced, rate and thus the measured concentrations are similar to those observed with no electric field.

The permeability of the cake when no electric field is applied ( $E = 0$ ) can be calculated from the first term in equation (1). At the end of washing, the permeabilities of cakes that started with their pores filled by 0.001M, 0.01M and 1M  $\text{NaNO}_3$  solutions at pH 7.8 were  $1.5 \times 10^{-18}$ ,  $1.6 \times 10^{-18}$  and  $4.2 \times 10^{-18} \text{ m}^2$ . The reproducibility of these data was  $\pm 0.1 \times 10^{-18} \text{ m}^2$ . Reversal of the electrode polarity when 10 V was applied caused a permeability reduction of 25% even though the cake porosity was the same in each experiment, reflecting the effect of the osmotic flow direction being changed to oppose the pressure gradient.

Cakes that were formed close to the IEP had a higher porosity throughout washing than those formed when the magnitude of the zeta potential was greater, and their porosity changed during the washing (Figure 13). At 100 kPa, the permeabilities of cakes when using 0 and 20 V were  $8.7 \times 10^{-18}$  and  $1.8 \times 10^{-18} \text{ m}^2$  respectively. The microscopic processes that take place during the washing of these cakes is complex and involves:

- an increase of the solution pH in the pores from 5.2 to about 8, due principally to the removal of  $\text{NaNO}_3$  and  $\text{HNO}_3$  as well as (potentially) the presence of electrolysis products;
- a change of the zeta potential from about  $-7 \text{ mV}$  to about  $-40 \text{ mV}$ ;

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- c. a deaggregation of the particles; and
- d. a reduction of the cake porosity.

Modelling of this situation is necessary in order to understand the effects of the roles of each mechanism further, and it will also be necessary to investigate the fluid mechanics of flow of electrolytes in nanopores.

Equation (1) is applicable only when the porosity is uniform through the thickness of the cake and when it is invariant with time. Although the former may be approximated, the results from this work show that the latter assumption is violated. The porosity reductions shown in Figure 13 represent significant changes to the flow rate when the applied pressure is constant. For example, the final porosities for the 0 and 20 V data are 0.533 and 0.471 respectively. Hence, using the Carman-Kozeny equation, if the differences in flow rates between the two sets of data were due to cake structural effects alone the flow rate at 0 V would be 1.86 times the flow rate at 20 V. Inspection of Figure 12 shows that this flow rate ratio is actually 4.83.

The electroosmotic contribution to the flow rate can be calculated from the second term in equation (1). On Figure 6, where there is no variation of porosity during washing, the increase in total flow due to electroosmosis is only evident at up to 10 V (increasing the potential above 10 V did not increase the flow rate further). For the experiments shown on Figure 12, where the porosity decreases during washing, according to equation (1) the electroosmotic flow rates at 10 and 20 V are 0.027 and 0.054 cm s<sup>-1</sup> respectively. These data also suggest that increasing the potential to 20 V does not have the expected effect of increasing the flow rate, as the calculated flow is rather greater than the actual measured value. The observed competing effects of flow reduction by structural changes of the cake and flow increase by electroosmosis further support the need for modelling the process. It is also insufficient to just measure filtrate conductivity to track the electrowashing process. Previous work [32] has reported only a marginal effect of an electric field on washing when this is done and separate determinations of anion(s) and cation(s) must be performed to accurately determine the magnitude of the effects occurring.

## CONCLUSIONS

In this experimental study, the effects of imposing an electric field on an otherwise conventional displacement washing have been investigated. From the data presented it is concluded that the removal rates of solute and wash liquor from a cake can be enhanced substantially by an electric field when the downstream electrode acts as a cathode and the particles initially carry a negative surface charge. Both the ionic strength and pH of the suspension from which a cake is filtered play an important role in determining the magnitude of the enhancements due to an applied field. In general, for a given value of cake thickness and hydraulic pressure difference, a raised electric field strength leads to greater cation removal and enhanced liquor flow rates, the latter being particularly noticeable when the ionic concentration is higher. When the pH is adjusted to bring a suspension closer to its IEP, an applied electric field is less able to improve the liquor removal rate due to progressively decreasing cake porosity throughout the washing process, however, it is still possible to enhance the cation removal rate.

The applied electric field can also alter the removal kinetics of ions adhering to particle surfaces, particularly when the valency of the potentially mobile ion is higher and the ion radius is smaller. Although such results have yet to be fully investigated, the consumption of electric power in all electrowashing experiments was found to be low and this potentially makes the wider application of the technology attractive.

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## NOMENCLATURE

$C$	instantaneous wash liquor concentration ( $\text{kg m}^{-3}$ )
$C_0$	concentration of wash liquor at the start of washing ( $\text{kg m}^{-3}$ )
$D$	dielectric constant ( $\text{C}^2 \text{J}^{-1} \text{m}^{-1}$ )
$E$	electric field gradient ( $\text{V m}^{-1}$ )
$h$	cake thickness (m)
$k$	permeability ( $\text{m}^2$ )
$\Delta p$	applied hydraulic pressure (Pa)
$t$	washing time (s)
$u$	liquid velocity due to a hydraulic pressure gradient ( $\text{m s}^{-1}$ )
$u_e$	electroosmotic velocity ( $\text{m s}^{-1}$ )
$u_o$	superficial velocity of the liquid ( $\text{m s}^{-1}$ )
$x$	distance into filter cake (m)
$\Delta V$	applied voltage potential difference (V)

### Greek symbols

$\varepsilon$	cake porosity (-)
$\mu$	liquid viscosity (Pa s)
$\zeta$	zeta potential (V)

### Subscripts

$an$	related to anion
$cat$	related to cation
$f$	denotes filtration phase
$w$	denotes washing phase

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## FIGURE AND TABLES

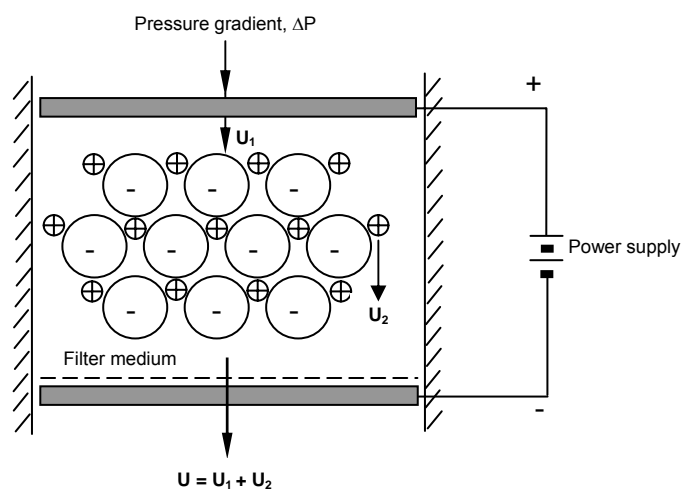


Figure 1: Effects of applying both electric field and hydraulic force across a filter cake.

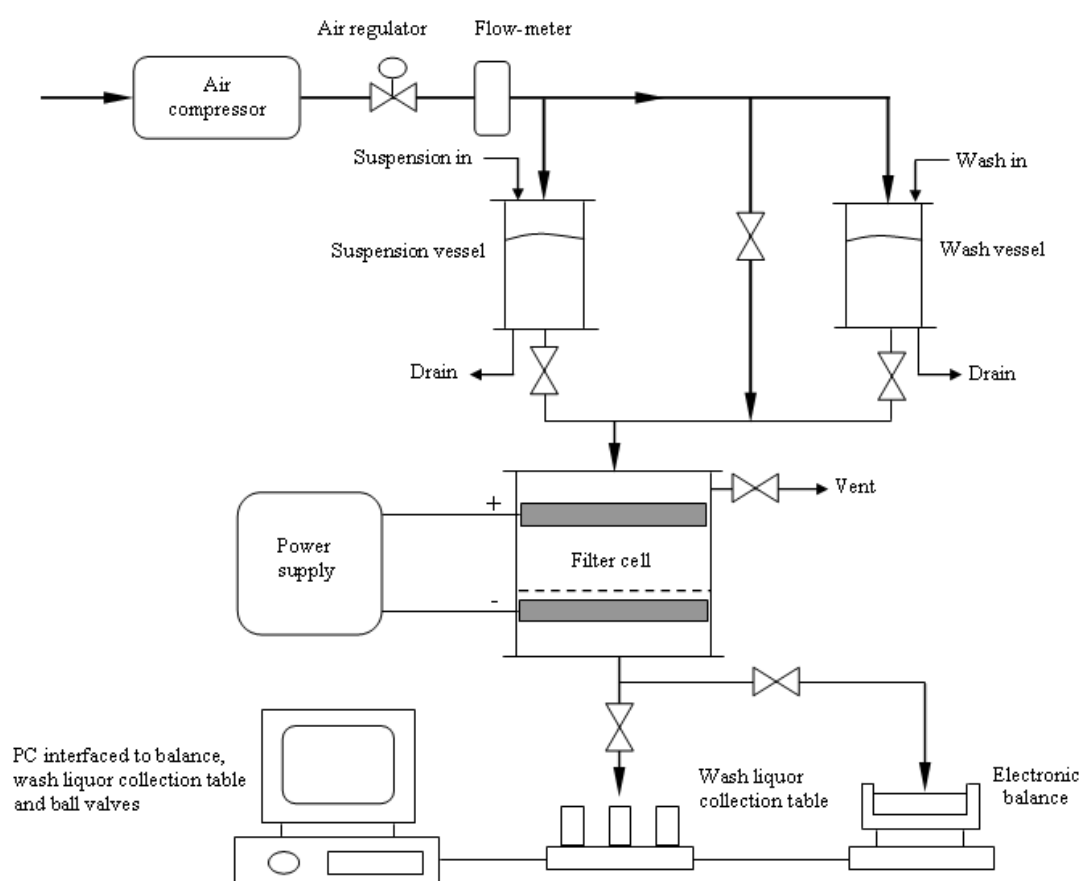


Figure 2: Schematic diagram of the experimental apparatus with the electrode polarity shown in the normal configuration.

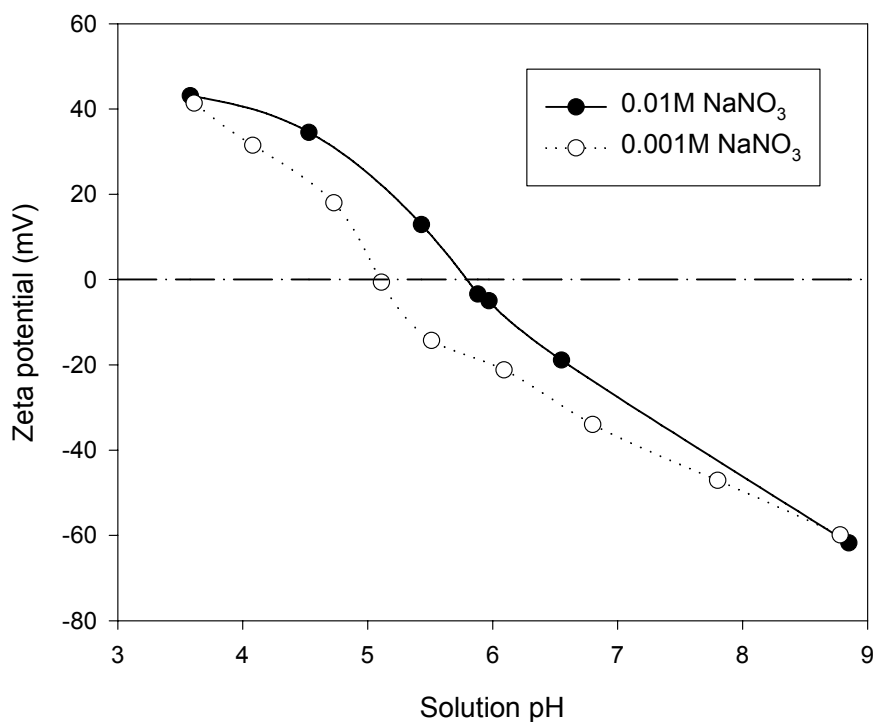


Figure 3: Zeta-potential of TiO<sub>2</sub> (rutile) in aqueous solutions, pH adjusted with either HNO<sub>3</sub> or NaOH.

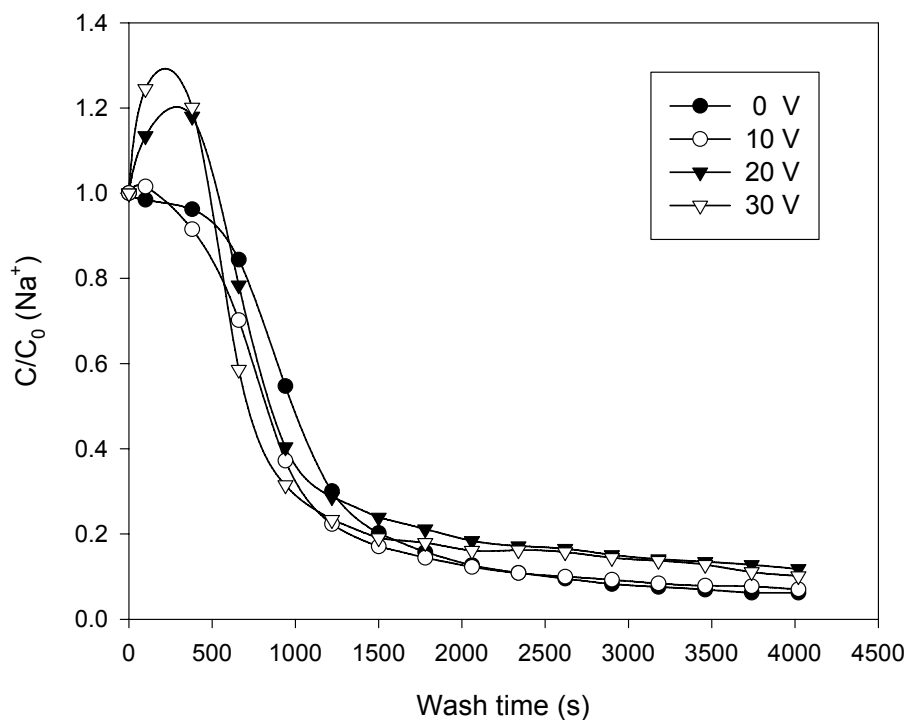


Figure 4: Variation of dimensionless Na<sup>+</sup> instantaneous concentration ( $C/C_0$  (Na<sup>+</sup>)) in the wash liquor with time ( $t$ ) and applied voltage.  $h = 10$  mm; initial pH 7.8; NaNO<sub>3</sub> concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

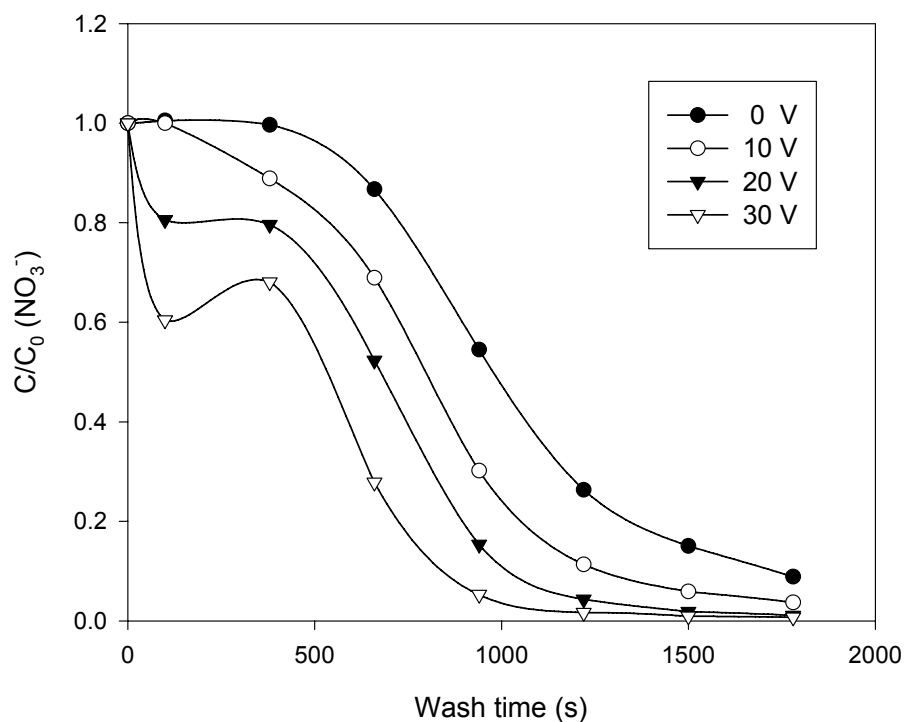


Figure 5: Variation of dimensionless  $\text{NO}_3^-$  concentration ( $C/C_0(\text{NO}_3^-)$ ) in the wash liquor with time ( $t$ ) and applied voltage.  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

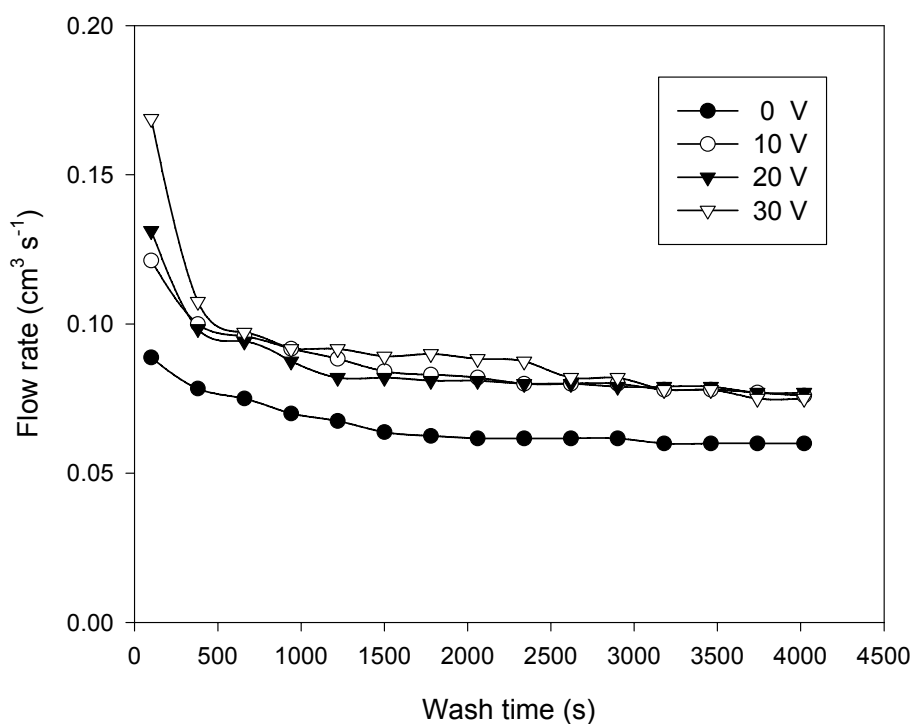


Figure 6: Variation of wash liquid flow rate with washing time and applied voltage.  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

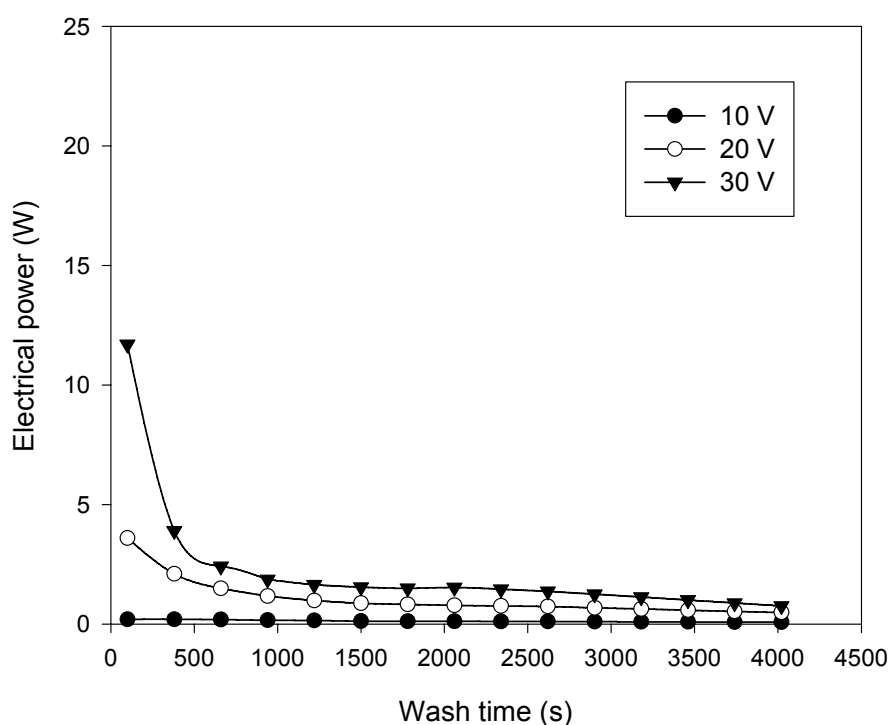


Figure 7: Variation of electrical power consumption with wash time and applied voltage.  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

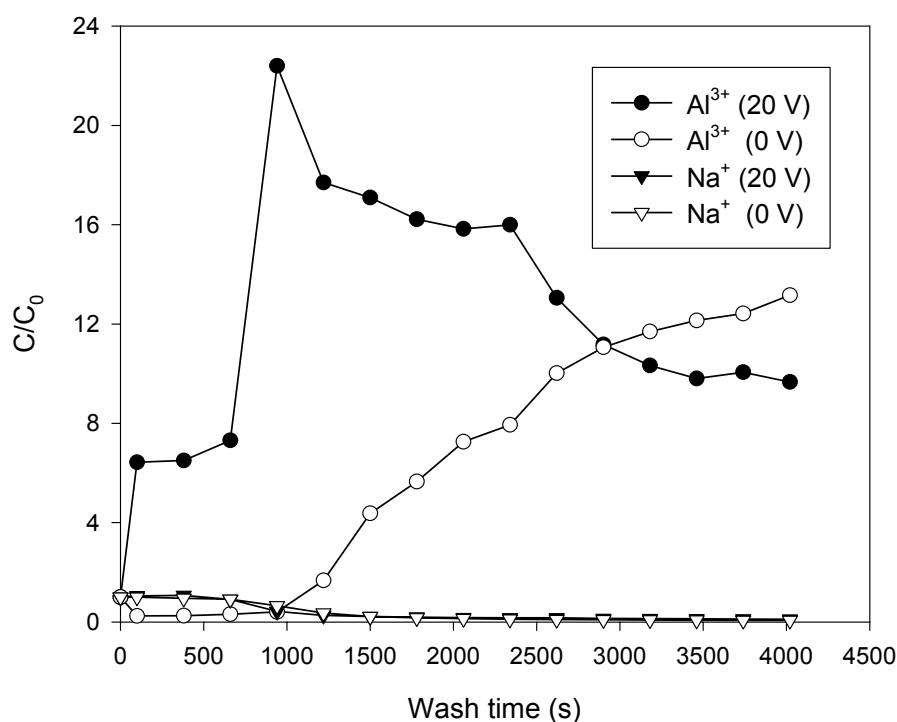


Figure 8: Variation of dimensionless cation concentration in the wash liquor with time and applied voltage.  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

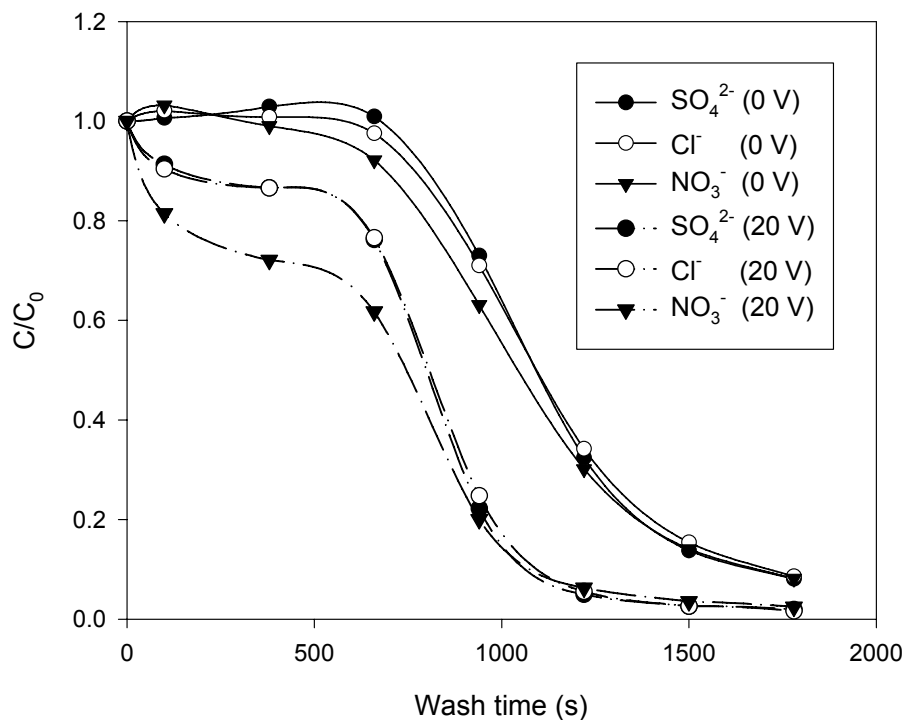


Figure 9: Variation of dimensionless anion concentration in the wash liquor with time and applied voltage.  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 400$  kPa.

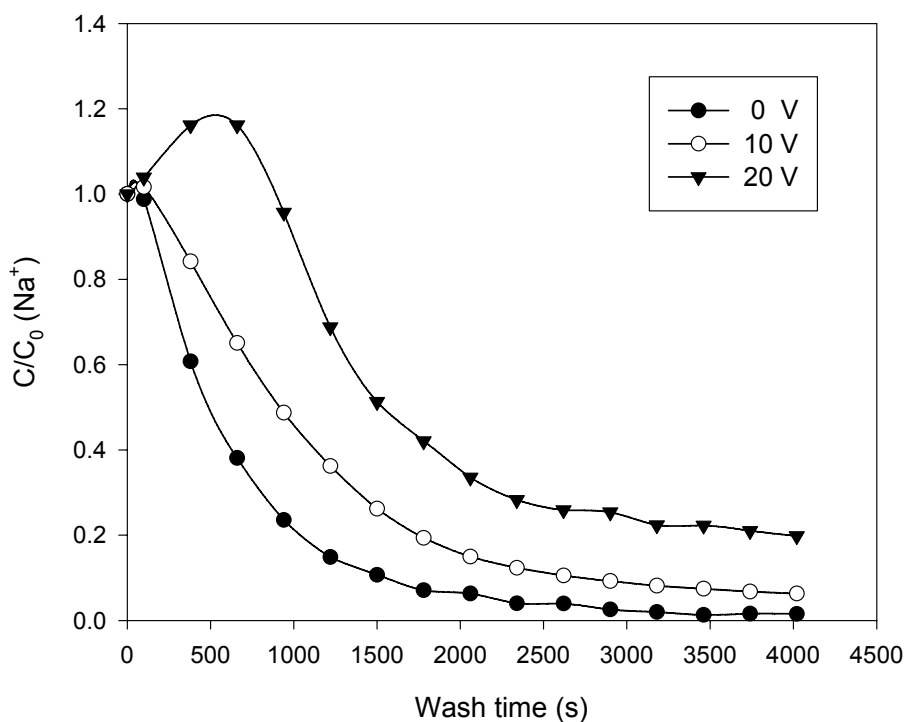


Figure 10: Variation of dimensionless  $\text{Na}^+$  concentration ( $C/C_0 (\text{Na}^+)$ ) in the wash liquor with time ( $t$ ) and applied voltage.  $h = 10$  mm; initial pH 5.2;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 100$  kPa.

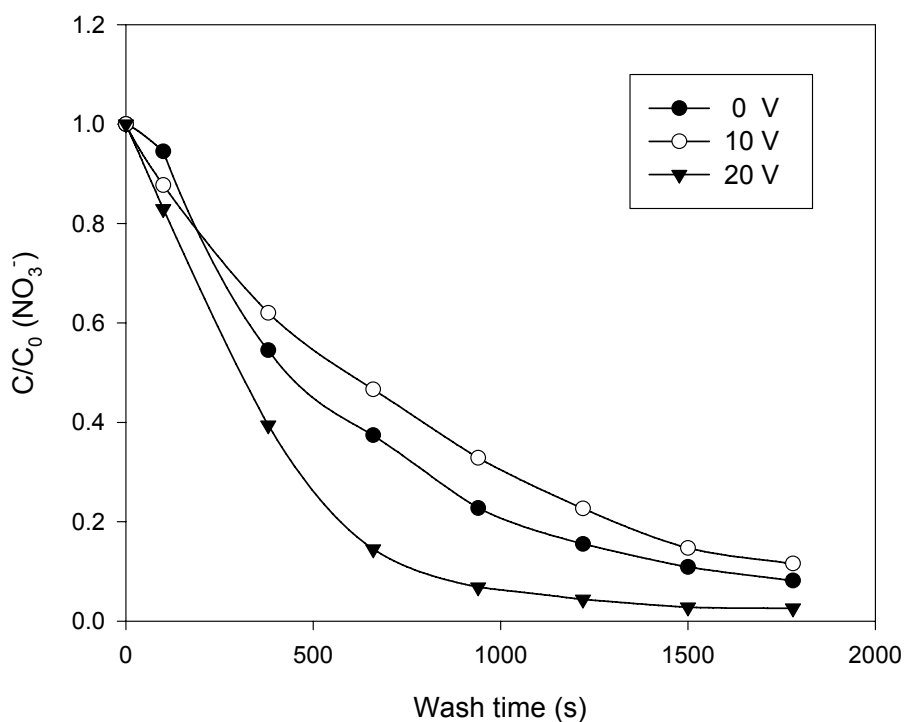


Figure 11: Variation of dimensionless  $\text{NO}_3^-$  concentration ( $C/C_0 (\text{NO}_3^-)$ ) in the wash liquor with time ( $t$ ) and applied voltage.  $h = 10$  mm; initial pH 5.2;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 100$  kPa.

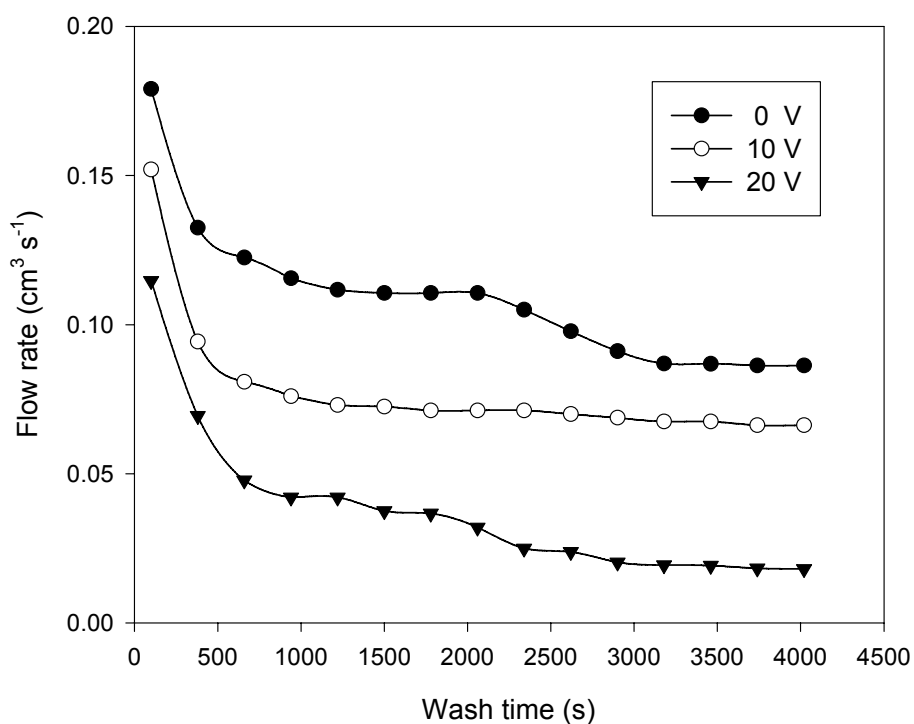


Figure 12: Variation of wash liquid flow rate with washing time and applied voltage.  $h = 10$  mm; initial pH 5.2;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 100$  kPa.

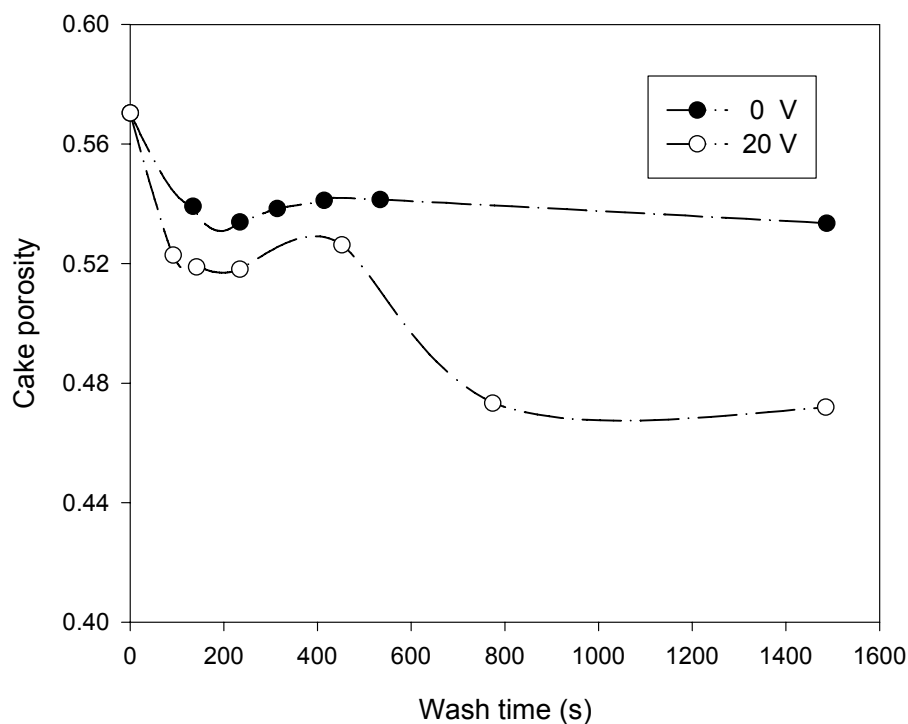


Figure 13: Variation of cake porosity with wash time.  $h = 10$  mm; initial pH 5.2;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_w = 100$  kPa.

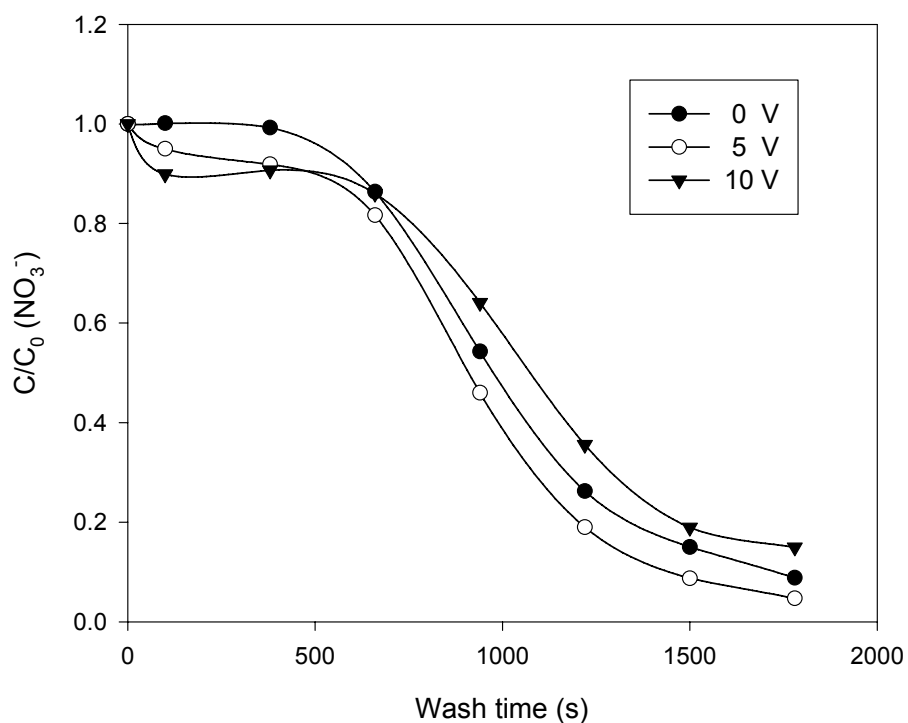


Figure 14: Variation of dimensionless  $\text{NO}_3^-$  concentration ( $C/C_0(\text{NO}_3^-)$ ) in the wash liquor with time ( $t$ ) and applied voltage (reversed electrode polarity).  $h = 10$  mm; initial pH 7.8;  $\text{NaNO}_3$  concentration = 0.001 M;  $\Delta p_f = \Delta p_r = 400$  kPa.

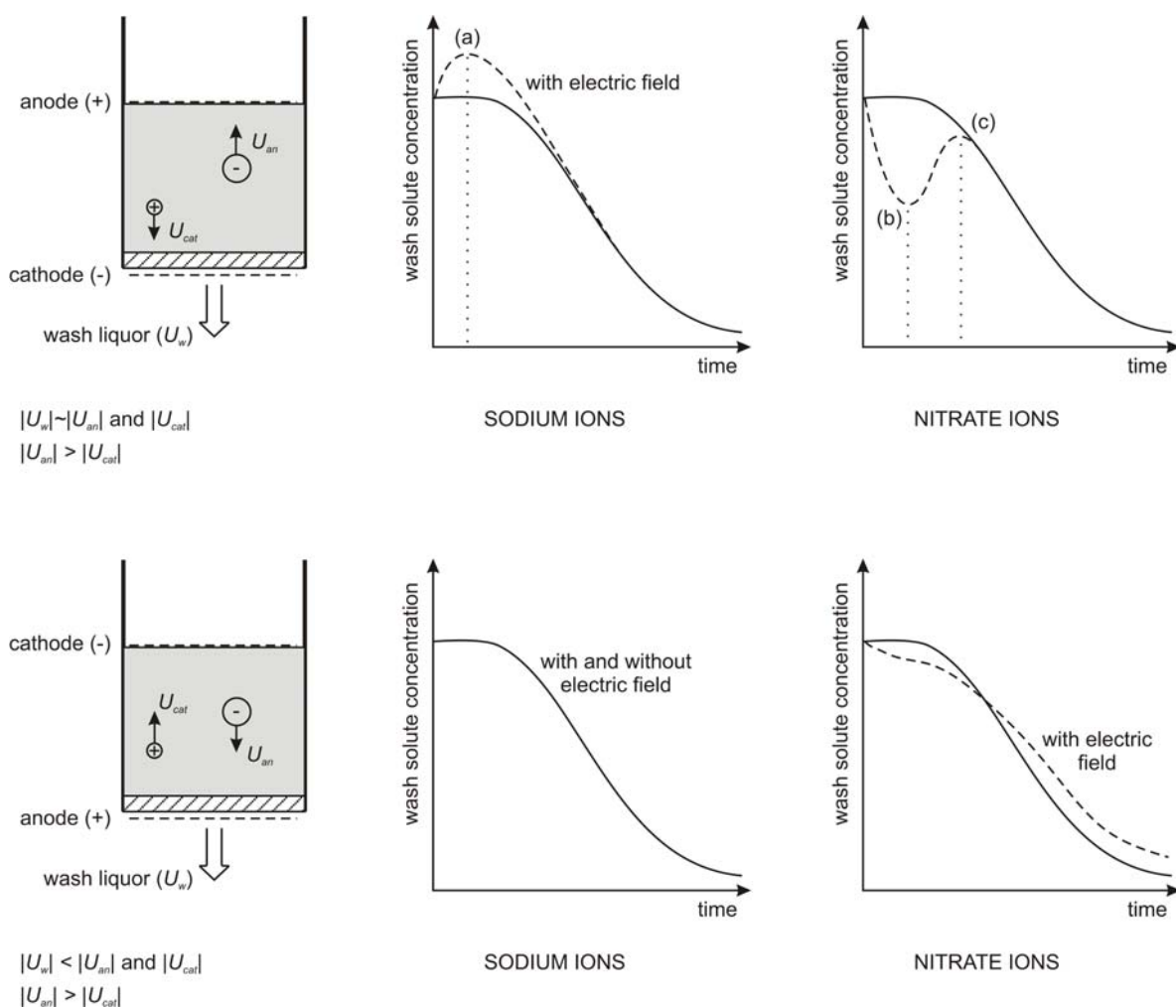


Figure 15: Schematic representations of washing performance both with and without an electric field.

Sequence number	Suspension of rutile used for cake formation	Typical cake porosity at the start of washing	Washing pressure (kPa)	Applied voltage (V)
1	0.001M NaNO <sub>3</sub> , pH 7.8	50.0%	400	0, 10, 20, 30
2	0.01M NaNO <sub>3</sub> , pH 7.8	50.2%	400	0, 10, 20, 30
3	0.001M NaNO <sub>3</sub> , pH 5.2	60.3%	100	0, 10, 20
4*	0.001M NaNO <sub>3</sub> , pH 7.8	50.0%	400	0, 5, 10

\*as sequence 1 but with reverse polarity electrodes.

Table 1: Conditions used in the washing experiment sequences.

Ion	Valency	Ion mobility in H <sub>2</sub> O at 25°C (m <sup>2</sup> s <sup>-1</sup> V <sup>-1</sup> )	Crystal ion radius (Å)
Na	1+	5.19x10 <sup>-8</sup>	0.97
Al	3+		0.51
Cl	1-	7.91x10 <sup>-8</sup>	1.81
N			1.71
S			1.84
NO <sub>3</sub>	1-	7.40x10 <sup>-8</sup>	
SO <sub>4</sub>	2-	8.29x10 <sup>-8</sup>	

Table 2: Properties of elements and molecules present in the electrowashing experiments.<sup>33</sup>