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# The Zeta Potential of a Mixed Mineral Clay in the Presence of Cations

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## Abstract

In the present study, zeta potential of a mixed mineral clay (MMC) was measured in the presence of salt cations; $Na^+$, $Li^+$ and $Ca^{2+}$ and metal cations; $Zn^{2+}$, $Cu^{2+}$, and $Al^{3+}$. Results reveal that the zeta potential of the MMC, in general, becomes more positive in the presence of divalent and trivalent cations. In presence of metal ions the zeta potential of the MMC has similar trends, i.e., increase in the concentration of these ions causes an increase in the zeta potential to the positive values up to around pH 7, and then it becomes positive; and produces generally two apparent pzcs (point of zero charges). The zeta potential values of the MMC were compared with those of pure montmorillonite and pure kaolinite.

**Keywords**: Electrokinetic decontamination, mixed mineral clay, zero point of charge potential, zeta potential

## Katyonlar Varlığında Karışık Mineralli Kilin Zeta Potansiyeli

## Özet

Bu çalışmada, karışık mineralli kil (KMK) örneğinin $Na^+$, $Li^+$, $Ca^{2+}$ tuz katyonları ve $Zn^{2+}$, $Cu^{2+}$, $Al^{3+}$ metal katyonları varlığında zeta potansiyeli değerleri ölçülmüştür. Genel sonuçlar incelendiğinde, zeta potansiyeli değerlerinin iki değerlikli ve üç değerlikli katyonların varlığında daha pozitif olduğu görülmektedir. Metal iyonların varlığında KKM'nin zeta potansiyeli davranışının benzer olduğu görülmektedir. Metal iyonların konsantrasyonundaki artış, pH 7 civarına kadar zeta potansiyelini pozitif değerlere doğru olarak arttırmakta, ve pH 7 civarında zeta potansiyeli pozitif değerler almaktadır. Metal iyonları genellikle iki adet sıfır yüklü nokta (*syn)* meydana getirmiştir. KKM'nin zeta potansiyel değerleri saf montmorillonit ve saf kaolin ile karşılaştırılmıştır.

**Anahtar Kelimeler**: Elektrokinetik dekontaminasyon, karışık mineralli kil, sıfır yük noktası, zeta potansiyeli

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## 1 Introduction

In recent years, electrokinetic remediation has been developed to remove organic and inorganic compounds from contaminated soils [Shapiro & Probstein, 1993; Acar & Alshawabkeh, 1993]. The electrokinetic remediation technique involves installing trenches and/or wells to encompass the contaminated zone, inserting electrodes into the trenches and/or wells. In this technique, contaminants are transported towards either the cathode or the anode depending on their charge (cationic or anionic) and the direction of the pore water flow under the voltage gradient. Contaminates are collected at the electrodes, then extracted and subsequently treated.

Although the principle of electrokinetic decontamination is fairly simple, complications arise because physico-chemical interactions occurring at the soil-water contaminant interface. For example, a pH gradient occurs during electrokinetic remediation between anode and cathode due to electrolysis of water creates acidic pH at the anode and basic pH at the cathode. Thus, researchers [Eykholt & Daniel 1994; Kim et al. 2001; Page & Page 2002] have reported that zeta potential of fine-grained soils plays an important role in the efficiency of the electro-remediation and recommended that zeta potential of soils to be determined before electrokinetic decontamination to optimize treatment parameters. This is because the direction and rate of the electroosmotic flow is zeta potential dependent, especially in compacted clayey soils, as shown in Equation 1.

$$k_{eo} = \frac{\varepsilon\varepsilon_o \zeta}{\mu} n_e \tau \qquad (1)$$

Where $k_{eo}$ is electroosmotic permeability, which ranges between $10^{-9}$ to $10^{-8}$ $m^2V^{-1}s$, η is viscosity, $\varepsilon$ is the relative permittivity of the pore fluid, $\varepsilon_o$ is the permittivity of free space, $n_e$ is effective porosity and $\tau$ is tortuosity. As can be seen from Equation 1, the electroosmotic permeability is dependent on zeta potential [West & Stewart, 1995]. During electro-remediation processes, the surface charge of the clay is altered by both the adsorption of contaminants and by changes in pore solution pH. Such complications control the efficiency of contaminant removal [Eykholt & Daniel, 1994; Dzenitis, 1997; Kim et al. 2001; Vane & Zang, 1997]. The researchers used zeta potential to explain the observed remediation efficiency rates of cations and soils [Eykholt & Daniel, 1994; Dzenitis, 1997; Kim et al., 2001]. Although the zeta potential of fine-grained soils is negative, it is pH dependent and subsequently may show positive values. Therefore, changes in zeta potential in the presence of different conditions (pH, cations, variation in soil mineralogy, etc.) need to be known.

Although previous studies [Yukselen & Kaya, 2003; Kaya & Yukselen, 2005] reported changes in zeta potential of montmorillonite, kaolinite and quartz powder in the presence of various ions, however, there is limited data on how that of the zeta potential of the MMC changes with the salt and heavy metal cations. Thus, the purpose of this study is to determine the zeta potential of the MMC in various conditions such as cation type, pH, and ionic strength to produce much sought data for the interpretation of the results to change in zeta potential during electrokinetic decontamination applications.

## 2 Materials and Methods

Natural MMC was obtained from Çanakkale, Turkey. Cation exchange capacity (Na method – Chapman, 1965) of the MMC is 13 meq/100g. X-Ray powder diffraction pattern was obtained using a Philips diffractometer and CuK$\alpha$ radiation (Figure 1). The intensities of diffraction peaks of a mineral in a mixture are proportional to its concentration. Thus, the relative proportions of the identified minerals roughly determined using their peak intensities. According to this, X-ray diffraction analysisshowed that the test MMC material consists of 40–50 % montmorillonite, 20–30 % illite, 10–15 % kaolinite, and 3–5 % quartz minerals.

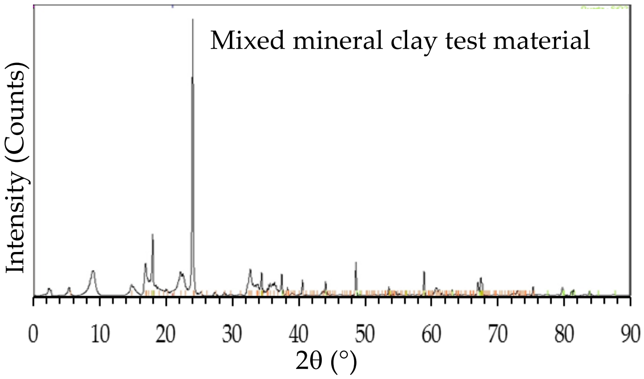

**Figure 1.** XRD pattern of the MMC test material.

Soils–clays usually contain a mixture of clay minerals. For that reason, the zeta potential of the MMC is useful for many practical purposes. Inductively coupled plasma (ICP) analysis of the MMC is given at Table 1.

**Table 1.** ICP analysis of the MMC.

| Sample # | $SiO_2$ % | $Al_2O_3$ % | $Fe_2O_3$ % | MgO % | CaO % | $Na_2O$ % | $K_2O$ % |
|---|---|---|---|---|---|---|---|
| MMC | 58.8 | 23.5 | 2.57 | 0.9 | 0.08 | 0.13 | 1.9 |

Alkali salt ions and hydrolysable metals are NaCl, LiCl, $CaCl_2.2H_2O$, $CuCl_2$, $ZnCl_2$, $AlCl_3$ all from Merck chemical.

The MMC sample was washed by ammonium acetate ($CH_3CO_2NH_4$) several times to discard different cations. The washing process was as follows: as–received sample was prepared by mixing 2 moles/L ammonium acetate for 15 minutes at a solid to liquid ratio of 1:2, diluting with distilled water to 2 to 3 % solids, allowing it to settle, and discarding the supernatant. The process of mixing distilled water, resettling and discarding the supernatant was repeated several times. Measuring the electrical conductivity in which until negligible changes occurred in the supernatant controlled the quality of the purified samples. The washed sample dried at 80 °C for over 48 hours. Dried sample was grounded and sieved from #200 mesh. The details of procedure can be found in Yukselen (2001).

In order to conduct the zeta potential measurements, 50 mg sample was transferred to a 50 ml glass per to which 50 ml of cation solution and a magnetic stirring bar were subsequently added. The pH of the solution was measured with Orion electrodes. The pH of solution was adjusted by dropwise addition of 0.1 M HCl and NaOH solutions after the clay sample addition. At the beginning of the zeta potential measurements pH profile of the MMC was evaluated as a function of stirring time at three different pHs (pH 3, pH 11 and no drop-wise addition). The mixing time was chosen as 10 minutes with respect to pH profile of the sample.

The zeta potential measurements were conducted by means of Zeta Meter 3.0+ (Zeta Meter Inc., New York), equipped with a microprocessor unit. Before and after each measurement the type-II micro-electrophoresis cell was washed with distilled water for preventing cross–contamination. After the mixing period and each zeta potential test, pH of the solution was measured. If any changes occurred in pH, the last pH reading was recorded as the pH of the solution. It was observed that change in pH before and after the each test does not exceed 0.5. This is negligible change for most of the measured values. The zeta potential of at least six particles for each test was determined and their average was recorded. The reliability of the zeta potential measurements was controlled with standard deviation of readings. Standard deviations are calculated by zeta meter from the readings. Standard deviation of each measurement was lower than 2 mV. Temperature of the room was 22.5±2.5 °C during the experiments.

## 3 Results and Discussions

### *pH Effect*

Figure 2 shows the zeta potential of the MMC as a function of pH in distilled water. As seen from Figure 2, the zeta potential of the MMC ranges from –22.6 mV at pH 3 to –41.2 mV at pH 11. As pH of the solution increases, the zeta potential becomes more negative. The observed behavior is due to disassociation of hydroxyls $(OH)^-$ on surfaces and edges of clay particles in water, which is influenced by pH [Yong & Warkentin, 1966]. The higher the pH is the greater the tendency for the $H^+$ to go into solution, and the greater the negative charge of the particle. In other words, as pH of the solution becomes more alkaline the zeta potential of the MMC becomes more negative.

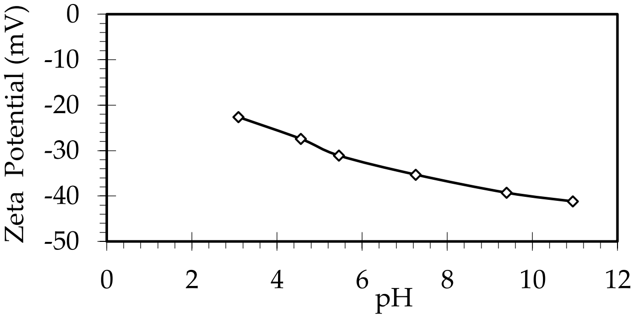

**Figure 2.** Variation of the zeta potential of the MMC in distilled water.

The MMC has negative zeta potential values within the pH range (3≤pH≤11), since there is no *pzc*. The MMC contains montmorillonite and kaolinite minerals. The surface charge of kaolinite mineral in aqueous is always negative arising, in general, directly from $Al^{3+}$ substitution for $Si^{4+}$ in the tetrahedral sheet of the mineral and charges arises from adsorption and desorption reactions in aqueous solution [Schroth & Sposito, 1997]. For montmorillonite, isomorphous substitution occurs mainly in alumina sheet, with magnesium or iron substituting for aluminum in the dioctahedral minerals [Mitchell, 1993]. Ion substitution and small contribution from broken bonds are sources of charge of montmorillonite. Thus, the observed zeta potential values for the MMC are in good agreement with what is known about zeta potential of clay minerals.

### *Effect of Salt Cations on the Zeta Potential of Mixed Mineral Clay*

Figures 3 (a), (b) and (c) show the zeta potential of the MMC in the presence of $Na^+$, $Li^+$ and $Ca2^+$ cations, respectively. The MMC has more negative zeta potential values with $1x10^{-3}$ M and $1x10^{-4}$ M NaCl with those obtained with distilled water (except pH 3 and pH 5). This means that monovalent ions at low concentration would give the most extended diffuse double layer [Yong & Warkentin, 1966; Celik et al., 1996]. However, this is not true for high ionic strengths. As can be seen from Figure 3 (a), in the presence of 1x10$^{-2}$ M NaCl solution the MMC has slightly lower negative zeta potential values with respect to other molarities (1x10$^{-3}$ and 1x10$^{-4}$). As the ionic strength increases, the zeta potential becomes less negative but never reaches zero at any concentrations. Note that only 1x10$^{-2}$ M concentration of NaCl decreases the zeta potential of the MMC over 3–5 mV. The zeta potential behavior of the MMC in the presence of Li+ cation is similar to those of Na+. Figure 3 (c) shows the zeta potential of the MMC in $CaCl_2$ solution. Divalent cation, $Ca^{2+}$, makes the zeta potential more positive at all concentrations. The $Ca^{2+}$ cations can compress the diffuse double layer of the mineral, the zeta potential changes over 20 mV; yet, it can not reverse the sign of the electrical potential, however; the MMC produces positive zeta potential values with 1x10$^{-2}$ M $CaCl_2$ around pH 3. At alkaline pH values divalent cation $Ca^{2+}$ precipitate as salt hydroxide as follows (Equation 2):

$$M^{++} + 2OH^{-} \rightarrow M(OH)_2 \downarrow \qquad (2)$$

M refers to salt cation such as $Mg^{2+}$ and $Ca^{2+}$.

Therefore, at high ion concentrations and alkaline pH values (pH>7), the measured zeta potential values are the zeta potential of the $M(OH)_2$-solid. There were some precipitates at the bottom of the cell in the presence of 1x10$^{-2}$ M CaCl2 at pH 9 and pH 11. The zeta potential of the MMC decreases as pH increases at 1x10$^{-2}$ M concentrations of $CaCl_2$ after pH 7, which is unexpected. The reason of this, at alkaline pH values, $Ca^{2+}$ ions precipitated as $Ca(OH)_2$ in the bulk solution. Therefore, the measured zeta potential is the zeta potential of precipitate covered solid. In the presence of precipitation the zeta potential of MMC becomes more positive. The *pzc* of MMC is around pH 4 in the presence of $Ca^{2+}$ ions.

It is known that $Ca^{2+}$ ions have more replacing power than ions. As a result of this, $Ca^{2+}$ ions replace with ions and make the zeta potential more positive. When the zeta potential of MMC in the presence of distilled water and 1x10$^{-4}$ M $CaCl_2$ is compared, the effect of the $Ca^{2+}$ ions on the zeta potential of MMC is seen clearly in spite of its low concentration. This is a less problem for higher concentrations of the cations (1x10$^{-2}$ M and 1x10$^{-3}$ M).

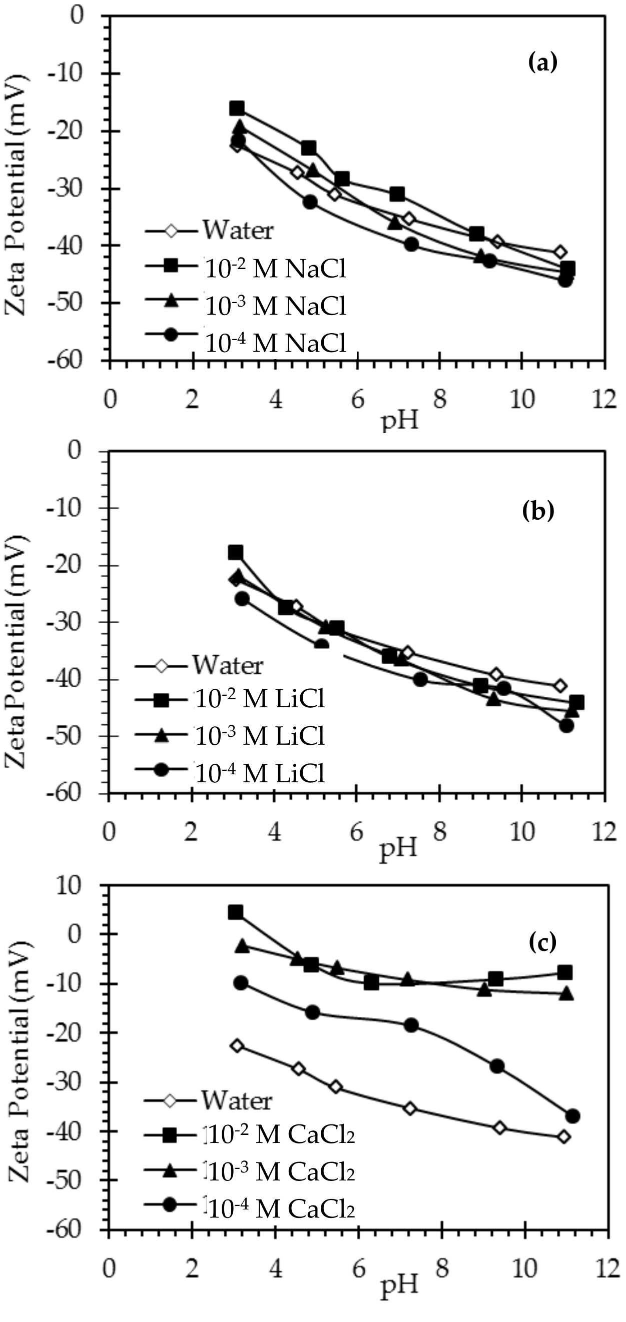

**Figure 3.** The zeta potential of MMC in the presence of NaCl (a), LiCl (b) and CaCl2 (c) solutions.

The zeta potential of MMC is compared with those of pure montmorillonite and pure kaolinite in the presence of distilled water and 1x10$^{-2}$ M $CaCl_2$ (Figure 4). The zeta potential behavior of MMC is among the pure montmorillonite and pure kaolinite in the presence of distilled water. As given in the materials and methods section, the MMC contains 40–50 % montmorillonite, 20–30 % illite, 10–15 % kaolinite. It can be said that, although its high proportion, montmorillonite cannot govern the zeta potential behavior of MMC. In the presence of $1x10^{-2}$ M $CaCl_2$, at pH 3, pH 9, and pH 11, the zeta potential of MMC becomes more positive regarding zeta potential values of montmorillonite and kaolinite. There is no data available on the zeta potential behavior of pure illite in the presence of $Ca^{2+}$ ions in this study. However, it can be said that the general behavior of MMC is not extremely different from their mineral constituents.

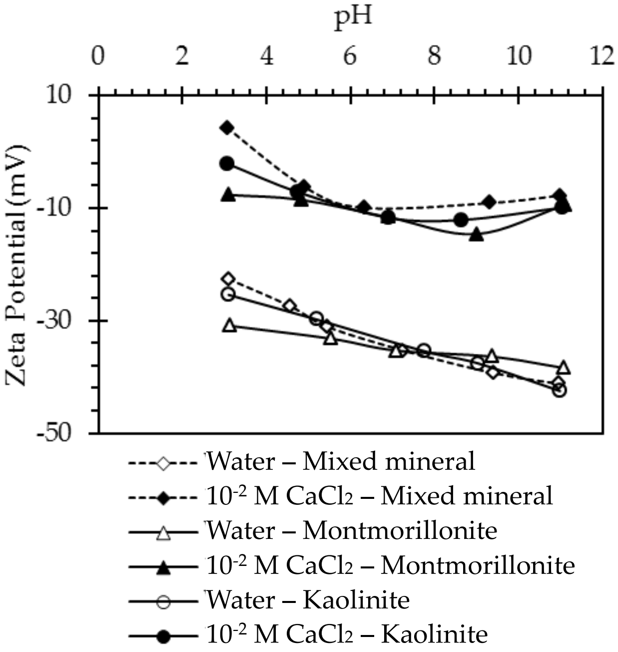

**Figure 4.** Comparison of the zeta potential of MMC with montmorillonite and kaolinite.

### *Effect of Metal Cations on the Zeta Potential of Mixed Mineral Clay*

In the presence of different concentrations ($1x10^{-2}$, $1x10^{-3}$, $1x10^{-4}$ M) of $ZnCl_2$, $CuCl_2$, $AlCl_3$ solutions the zeta potential of MMC was determined. Metal cations have strong influence on the zeta potential of MMC. The stability constant of the used $Ca^{2+}$, $Zn^{2+}$, $Cu^{2+}$, and $Al^{3+}$ cations are low enough to allow hydrolyzation. Since, they form as their hydroxides and precipitate in the bulk solution. Generally, in the presence of metal cation solutions at alkaline pHs precipitation occurs in the bulk solution as a metal hydroxide. Figure 5 presents the general shapes of the zeta potential of MMC in the presence of hydrolysable metals. As the concentration of hydrolysable metal ions increases the zeta potential becomes positive at all pH values but the effect is largest at intermediate pH, slightly above that at which precipitation would be expected in the bulk solution [West & Stewart, 1995]. Second *pzc* is attributed to the specific adsorption of the partially hydrolyzed metal ion to produce a charge reversal. At higher pH, it appeared that the adsorbed hydroxyl ion, M(OH), was converted into the metal hydroxide, which partially or completely covered the surface. At still higher pH, the zeta potential turned from positive to negative at a pH that approached the normal *pzc* of the metal hydroxide as the thickness of the hydroxide layer increased [Hunter & James, 1992]. Therefore, at high ion concentrations and high pH values, the measured zeta potential belongs to the M(OH)-solid. Under acidic conditions, the effect of hydrolysable metal ions is insufficient to cause the zeta potential to reverse [West & Stewart, 1995]. In this study, at low pH values (< pH 7) the zeta potential is less negative, and generally around pH 7 it has positive peak values; then rapidly becomes more negative as pH increases.

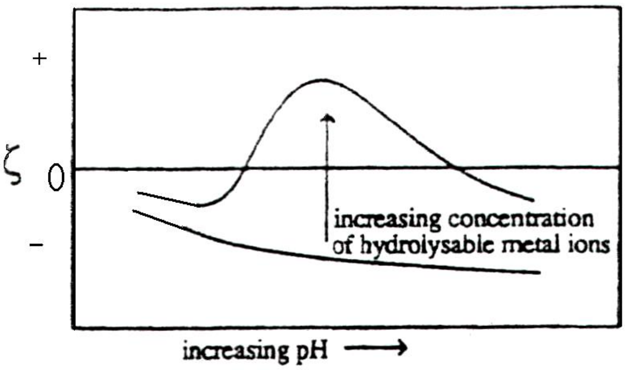

**Figure 5.** Schematic illustration of the zeta potential behavior of the MMC in the presence and absence of hydrolysable metal ions (adapted from West & Stewart 1995).

Figures 6 shows the zeta potential of the MMC in the presence of $Zn^{2+}$ cations. The MMC has two *pzcs* at near pH 3.5 and pH 7 in the presence of $10^{-2}$ M $ZnCl_2$. Hunter & James (1992) also observed this trend for kaolinite treated with $Cu^{2+}$, $Co^{2+}$, and $Cd^{2+}$ ions. They reported that under alkaline conditions, the zeta potential behavior tends towards that of the solid metal hydroxide. Dillard & Koppleman (1982) showed that $Co^{2+}$ ions are covalently bonded to the clay under acidic conditions, whereas under alkaline conditions the clay is coated with a layer of electrostatically bound cobalt hydroxide. This explains why the zeta potential values at high pHs tend towards those of the metal hydroxide covered solid. This observation becomes more dominant with increase in the ion concentration. Similar to $Co^{2+}$ ion, $Zn^{2+}$, $Cu^{2+}$ and $Al^{3+}$ ions cover the solid surface as metal hydroxide. In the presence of $1x10^{-3}$ and $1x10^{-4}$ M $ZnCl_2$ zeta potential values are negative cannot reach to positive values.

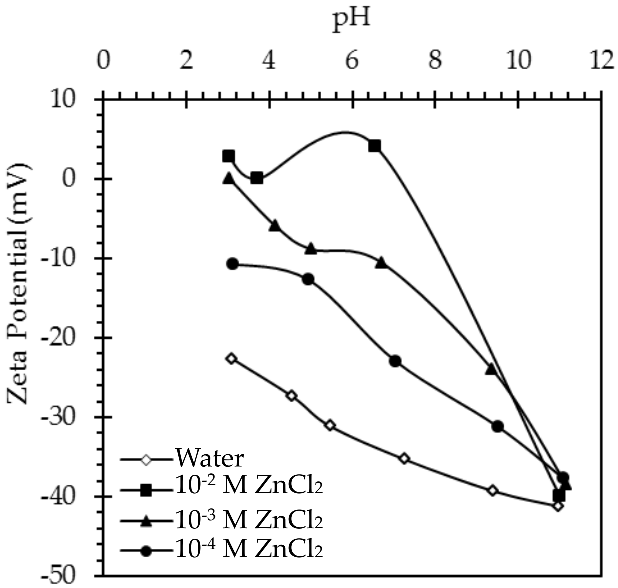

**Figure 6.** The zeta potential of MMC in the presence of $ZnCl_2$ solution.

The MMC has only one *pzc* at pH 10 at $1x10^{-2}$ M concentration and three pzcs at $1x10^{-3}$ M concentration around pH 3, pH 5 and pH 8 with $Cu^{2+}$ ions (Figure 7a). Divalent $Cu^{2+}$ ions can compress markedly the diffuse double layer of MMC (from –30 mV to +35 mV at pH 8). The effect of $Cu^{2+}$ ions is largest at intermediate pH. $Al^{3+}$ cation increases the zeta potential up to +40.mV. Even $1x10^{-4}$ M concentration of $Al^{3+}$ can compress diffuse double layer, so the zeta potential reaches positive values. The cation valence effect can be seen clearly when the zeta potential values are compared in the presence of $1x10^{-4}$ M $CuCl_2$ and $1x10^{-4}$ M $AlCl_3$. The $Al(OH)_3$ precipitate is positively charged at pH values up to between 8–10, due to the adsorption of positively charged $AlOH^{2+}$ and $Al(OH)^{2+}$ species, and negatively charged above these pH values owing to the those of $Al(OH)_4^-$ species. The MMC has different *pzcs* values at all concentrations of $AlCl_3$, between pH 7.5–10 (Figure 7b).

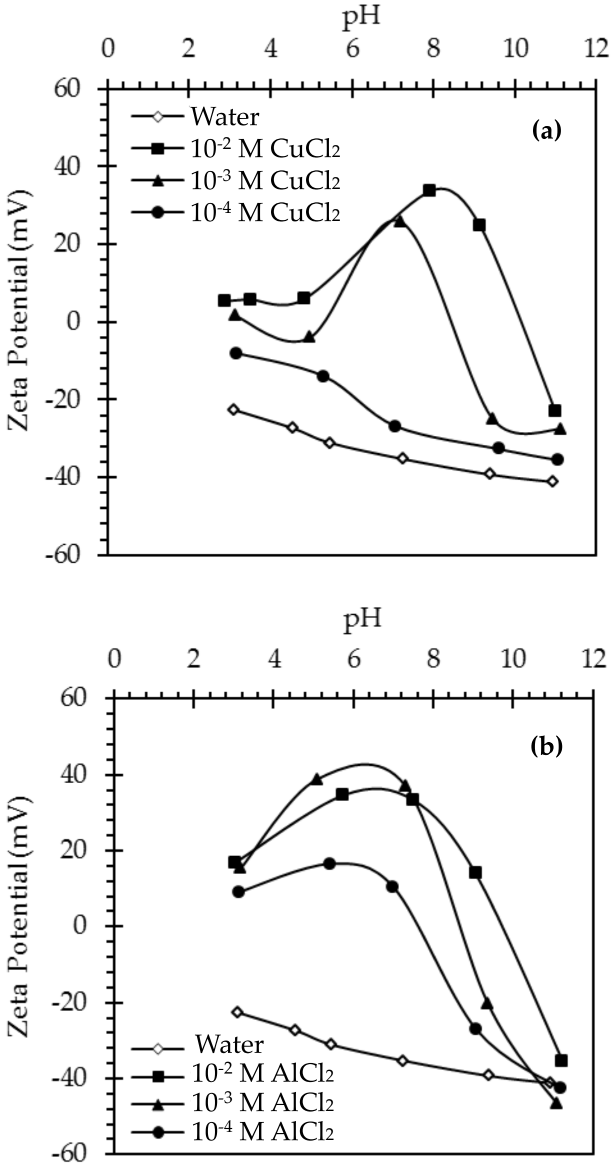

**Figure 7.** The zeta potential of MMC in the presence of (a) $CuCl_2$ and (b) $AlCl_3$ solutions.

Figure 8 shows the cation valence effect on the zeta potential. As can be seen, as cationic valence increases, the zeta potential becomes more positive. However, there are some zeta potential differences between the divalent cations, for example $Ca^{2+}$ and $Cu^{2+}$. This is possibly due to the hydrated ionic radius difference between the cations. Hydrated ion size has influence on the diffuse double layer system. The larger the ion is the thicker the layer and makes the zeta potential more negative.

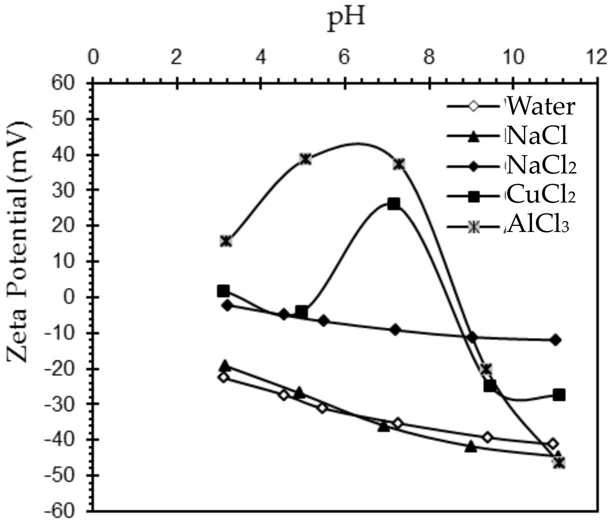

**Figure 8.** Cation valence effect on the zeta potential of MMC ($10^{-2}$ M concentrations of cations).

### *Importance of Results in Terms of Electrokinetic Decontamination*

As stated above, the efficiency of electrokinetic decontamination is controlled, among other parameters, by the zeta potential of soil-pore water and ions as described in Equation 1. It is also noted that magnitude and sign of the zeta potential changes with ionic strength and pH of the system. The results shown here confirm that, indeed, the zeta potential of the MMC mineral changes in the presence of earth and alkali ions. The changes in zeta potential depend on the ion type and ionic strength. The results further indicate that heavy metals such as Zn2+, Cu2+, and $Al^{3+}$ precipitate as metal hydroxide at alkaline pHs (pH>9) and at high ionic strength, which should be expected to happen around a cathode at which high pH and high ion concentration occurs. Precipitation of these heavy metal ions as metal hydroxide around the cathode, of course, is not desirable since it decreases the efficiency of the remediation process. The results suggest that precipitation of metal oxides can be prevented via controlling the pH around the cathode by making it more acidic solutions. In other words, zeta potential of soil and the targeted of cations with surfactant agent to be determined in the laboratory before electrokinetic remediation application for optimizing the parameters treatment set up.

## 4 Conclusions

The zeta potential of MMC was investigated in the presence of different cations and surfactants. General behavior of MMC is not different from its mineral constituents. As pH increases, the zeta potential of MMC becomes more negative in the presence of distilled water. In the presence of distilled water, within the pH range (pH 3–11 ) MMC has negative zeta potential values, since there is no pzc. The zeta potential of MMC with hydrolysable metal cations present similar trends: as the concentration of hydrolysable metal ions increases, the zeta potential becomes more positive at all pH values, the effect is the largest at intermediate pH, slightly above that at which precipitation of metal hydroxide would be expected in the bulk solution. The zeta potential of MMC becomes more positive as pH increases until pH 7; it has peak positive values around pH 7, and then rapidly becomes more negative under alkaline conditions, where metal hydroxides precipitate. MMC has two *pzcs* with some type of metal cations.

The practical implications of contaminated soils should be determined before conducting any remediation method, as mentioned before researchers have obtained that zeta potential is one of the factor that affect the contaminant removal rate.

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