

## EFFECT OF COAGULANTS AND FLOCCULANTS ON DEWATERING OF KAOLIN SUSPENSIONS

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### ABSTRACT

Kaolin tailings are commonly generated in the mineral industry. They are invariably negatively charged and consequently tend to form stable dispersions with poor flocculation characteristics. Coagulation–flocculation treatments are suitable methods for removing colloidal particles from wastewater. In this study, the effects of mono/multivalent ions (coagulants) and polymers (flocculants) on the sedimentation and electrokinetic behaviours of kaolin have been investigated. In experimental studies,  $Al_2(SO_4)_3$ ,  $FeCl_3$ ,  $MgCl_2$ ,  $CaCl_2$ ,  $NaCl$  were used as coagulant, while as flocculant anionic (A150), cationic (C521) and nonionic (N100) polymers were used. Isoelectric point of kaolin was determined as pH 4.2. The effectiveness of coagulants increased with the increase in the ionicity degree of the metal ions. Among the coagulants,  $FeCl_3$  provided the highest efficiency (91%). Low sedimentation velocities (6.3-12.2 mm/min) were obtained with coagulants. The highest sedimentation efficiency (94%) was achieved with anionic flocculants and the same sedimentation velocity (58 mm/min) was reached with all flocculants.

**Keywords:** Kaolin, dewatering, polymer, metal salts, zeta potential

## KAOLEN SÜSPANSİYONLARININ SUSUZLAŞTIRILMASINDA KOAGÜLANT VE FLOKÜLANTLARIN ETKİSİ

### ÖZ

Kaolin atıkları mineral endüstrisinde yaygın olarak üretilir. Negatif yüklü olan bu atıklar zayıf flokülasyon özelliklerine sahip kararlı dağılımlar oluşturmaya eğilimlidirler. Koagülasyon-flokülasyon işlemleri, koloidal parçacıkları atık sularından uzaklaştırmak için uygun yöntemlerdir. Bu çalışmada, mono/multivalent iyonların (koagülant) ve polimerlerin (flokülant) kaolenin çökeltme ve elektrokinetik davranışlarına etkisi araştırılmıştır. Deneysel çalışmalarda, koagülant olarak  $Al_2(SO_4)_3$ ,  $FeCl_3$ ,  $MgCl_2$ ,  $CaCl_2$ ,  $NaCl$  ve flokülant olarak, anyonik (A150), katyonik (C521) ve noniyonik (N100) polimerler kullanılmıştır. Kaolenin sıfır yük noktası pH 4.2 olarak belirlenmiştir. Koagülantların etkinliği, metal iyonlarının iyoniklik derecesinin artması ile artmıştır. Koagülantlar arasında  $FeCl_3$  en yüksek verimi (%91) sağlamıştır. Koagülantlar ile düşük sedimentasyon hızları (6.3-12.2 mm/dk) elde edilmiştir. En yüksek sedimentasyon verimliliği (%94) anyonik flokülant ile sağlanmış ve tüm flokülantlar ile yaklaşık olarak aynı çökeltme hızına (58 mm/dk) ulaşılmıştır.

**Anahtar Kelimeler:** Kaolen, susuzlaştırma, polimer, metal tuzları, zeta potansiyeli

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## 1. INTRODUCTION

Being a clay mineral, kaolin has the unit cell formula of  $\text{Al}_2\text{Si}_2(\text{O}_5)(\text{OH})_4$ . There are two different basal cleavage faces in kaolin. One of these includes tetrahedral siloxane ( $-\text{Si}-\text{O}-\text{Si}-$ ) while the other contains octahedral alumina ( $\text{Al}_2\text{O}_3$ ) sheets. Demonstrating an inert siloxane structure, the basal face is loaded with a permanent negative charge; the reason of this charge development is the isomorphous substitution of  $\text{Si}^{4+}$  by  $\text{Al}^{3+}$  groups. At the edge of the crystal, the octahedral alumina and tetrahedral silica sheets are disrupted and broken bonds exposing aluminol ( $\text{Al}-\text{OH}$ ) and silanol ( $\text{Si}-\text{OH}$ ) groups occur. Due to the protonation and deprotonation in the surface hydroxyl groups, depending on pH, charge development may occur in the face of the edge [1-3].

Mineral tailings generally contain clay minerals like kaolin. They may cause dewatering problems which may include high yield stress, high flocculant demand, low settling rates and poor supernatant clarity due to their colloidal size, anisotropic form and basal faces with permanent charge [4-6]. In industrial applications, usually filtration is used to accomplish dewatering. Yet, pre-aggregation of the colloidal particles in the suspension is required to ensure an acceptable dewatering efficiency [7]. Inclusion of multivalent cations (coagulation) and/or polymers (flocculation) may be used to accomplish this. Salts of multivalent cations viably lessening the surface charge of the particles are called coagulants, on the other hand, high molecular weight water-soluble polymers extensive enough to connect the particles are referred to as flocculants [8]. Polymers may be anionic, cationic or nonionic. They mostly have structures consisting of linear chains. It can be easily synthesized, up to very high molecular weights. Despite being an efficient flocculant, the activity of nonionic homopolymer (100% acrylamide) can be increased through copolymerisation with different monomers. By co-polymerizing acrylamide with a cationic monomer, cationic polyacrylamide (PAM) may be formed. It is possible to produce anionic PAM by copolymerization of acrylamide with acrylic acid. The amide groups of PAM can be hydrolysed to give carboxylic acid groups which can ionize to give anionic sites along the chain. To achieve charges at varying densities, the level of hydrolysis may be regulated. For flocculation, the molecular weight and charge density of polymers can be defined as the most significant characteristics [9]. Flocculation may involve different destabilization mechanisms including polymer bridging, charge neutralization, formation of polymer-particle surface complex or combination of these mechanisms, based on the characteristics of polymers and structure of tailings [10, 11]. It has been seen that there are two mechanisms, namely, charge neutralization and sweeping mechanism during coagulation. Inorganic salts of multivalent metals are more economical than polymers, yet, their application is constrained due to low sedimentation performance [12].

Among the main factors determining the effectiveness of coagulation-flocculation processes are coagulant/flocculant type/dosage. As a result, these need to be optimized to increase the treatment effectiveness in the processes. Additionally, understanding the interaction between kaolin and coagulant/flocculant is important to optimize sedimentation performance. The zeta potential,  $\zeta$ , is an important electrokinetic properties of clay minerals. Chemical and physical events such as ion exchange, adsorption, swelling, colloidal stability, and flow behaviour are controlled by the electric charges of clay surfaces [13, 14]. It is common knowledge that addition of oppositely charged coagulants and flocculants to a colloidal dispersion usually leads to a significant decrease in the zeta potential and to a change in the sign of the particle charge, which is accompanied by destabilization of the system. As a result, beneficial information on the colloid destabilization mechanisms is achieved thanks to the data on zeta potential [9]. Toward this aim, in this study, the effects of multivalent ions and polymers on the settling velocity, supernatant turbidity, zeta potential and suspension pH were investigated for kaolin suspensions.

## 2. MATERIAL AND METHODS

### 2.1. Materials

Kaolin sample of the Bilecik (Turkey) region, obtained from MATEL Raw Material Industry & Trade Inc. Co. was utilized in this study. Chemical analysis, particle size and general properties of the sample are given in Table 1. It is containing kaolinite at least 66%. Particle size analysis of kaolin powder was carried out using Sympatec Helos H1735 particle size analyser and it was found that 75% of it was less than 17  $\mu\text{m}$ .  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{FeCl}_3$ ,  $\text{MgCl}_2$ ,  $\text{CaCl}_2$  and  $\text{NaCl}$  were used as coagulants in the coagulation tests. Polyacrylamide based flocculants were used in the flocculation tests. Anionic (A-150), cationic (C-521) and nonionic (N-100) Superfloc flocculants, obtained from the American Cyanamid Company (Cytec). The molecular weights of A-150 and N-100 polymers are in the range of  $5-15 \times 10^6$  and that of C-521 is  $10 \times 10^3-0.5 \times 10^6$ . Coagulants and flocculants used in the experiments were prepared as 5% and 0.01% solutions, respectively. Stock solutions of the coagulants and flocculants were prepared with distilled water and diluted in required rates in the experiments [15].

*EFFECT OF COAGULANTS AND FLOCCULANTS ON DEWATERING OF KAOLIN SUSPENSIONS*

**Table 1.** Chemical analysis, particle size and general properties of the kaolin

Chemical Analysis (%)		Particle Size (µm)		General Properties	
SiO <sub>2</sub>	53.0	d <sub>10</sub>	1.28	Volumetric Weight(g/L)	1675
Al <sub>2</sub> O <sub>3</sub>	30.6	d <sub>25</sub>	2.57	Casting Concentration(%)	65
Fe <sub>2</sub> O <sub>3</sub>	1.9	d <sub>50</sub>	5.91	Drying Shrinkage(%)	5
CaO	0.5	d <sub>75</sub>	16.96	Total Shrinkage(%)	12
Na <sub>2</sub> O	0.2	d <sub>95</sub>	50.72	Dry Strength(kg/cm <sup>2</sup> )	55
MgO	0.6	d <sub>97</sub>	59.50	Water Absorption(%)	0.25
K <sub>2</sub> O	2.3			Whiteness	66
TiO <sub>2</sub>	1.3			Mineral Composition (%)	Kaolinite-70 Free Quartz-11 Illite-4 Others-5
LOI	9.6				

\*These values were taken from the manufacturer

## 2.2. Methods

Experiments were carried out in 500 mL beakers at mechanical stirrer with a mixing speed control. Suspensions were prepared by dispersing 2.5 wt% kaolin. In experiments, Firstly kaolin–water suspensions were pre-conditioned for 15 min at 850 rpm in order to obtain a well-dispersed suspension. Then, the speed was reducing to 200 rpm and the coagulant/ flocculant was added into the suspension. After the suspension was stirred at 3 minutes and allowed to settle for 30 min. After that, a 10 ml supernatant sample was pipetted off and transferred to a glass cuvette to determine the turbidity. Turbidity of kaolin suspensions was measured in nephelometric turbidity units (NTU) using a turbidity meter (Velp Scientifica turbidimeter). The conductivity and zeta potential analysis of the samples was also performed using a Zeta Plus apparatus from Brookhaven. pH was measured using Jenco-6230 pH meter. The turbidity removal was calculated according to Eq. (1) [6, 16]:

$$\text{Turbidity Removal (\%)} = \frac{\text{Initial Turbidity} - \text{Supernatant Turbidity}}{\text{Initial Turbidity}} \times 100 \quad (1)$$

## 3. RESULTS AND DISCUSSION

### 3.1. pH Profiles of Kaolin

Natural pH of prepared kaolin suspensions was 7.86. The pH profiles of kaolin suspension (2.5 wt.%) as a function of time is presented in Figure 1 for an initial pH of natural, acidic (pH 3) and basic medium (pH 11). When the initial pH of kaolin suspension is adjusted to 3, the suspension pH increased to 5.1 in 300 min. The reason for this can be attributed to the adsorption of H<sup>+</sup> ions on to the negatively charged surface of kaolin and exchange with H<sup>+</sup> ions of cations (Si<sup>4+</sup> and Al<sup>3+</sup>) in the tetrahedral and octahedral layers of the mineral. When it is adjusted to pH 11, it was remaining almost constant (it was coming down to 10.6 in 300 min).

### 3.2. Effect of pH on the Zeta Potential and Isoelectric Point of Kaolin

The IEPs of kaolin particles determine the charges carried by these particles at various pH. Tetrahedral silica sheets in kaolin particles are known to carry permanent pH independent negative charges resulting from the isomorphous substitution of Si<sup>4+</sup> by Al<sup>3+</sup> groups. Gupta and Miller [18] explained that these tetrahedral silica sheets carry an IEP at pH < 4. The IEP of the Si-OH and Al-OH edge sites is between pH 5.0–7.0 [17] and that of octahedral alumina sheets is between pH 6.0–8.0 [18]. According to these IEPs, at high pH, the number of positively charged sites on kaolin surfaces should be low to exist in negligible quantities [19]. The zeta potential of kaolin as a function of pH are given in Figure 2. IEP value of kaolin was determined as 4.2. This is within the typically reported range for kaolin IEP pH 2.8 to 4.2 [20, 21]. The zeta potential of the particles became more negative with the increase of suspension pH and finally decreased to approximately -22.88 mV at a pH 11. At natural pH of kaolin suspension (pH 7.86), zeta potential value was determined approximately -21.74 mV. It can be said that the kaolin suspensions had a relatively high stability owing to the high negative surface charge of the particles in natural pH.

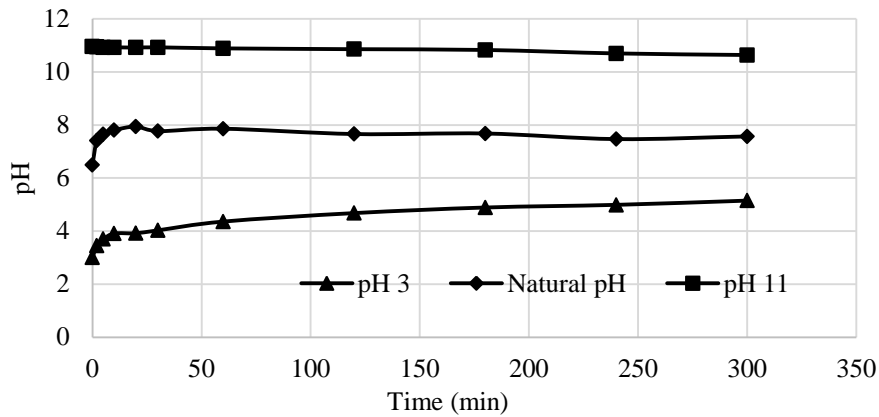


Figure 1. pH profiles of kaolin at an initial pH of natural, acidic and basic medium

### 3.3. Natural Sedimentation of Kaolin Suspension

The kaolin suspension leaved to settle naturally for 180 min. Figure 3 represents the variation of turbidity and interface height with settling time to evaluate the natural stability of the kaolin suspension. After 150 min, sedimentation velocity of suspension decreased. The final turbidity value of the suspension slowly decreased with increasing sedimentation time, and at the end of 180 minutes it was 63.4 NTU. At the end of this period, the turbidity removal efficiency is 78.94%. Kaolin particles are negatively charged in the natural pH of suspension (pH 7.8), causing their particles to remain in suspension without aggregating for long periods of time. These data show that natural sedimentation of kaolin suspension is not sufficient and that treatment is necessary.

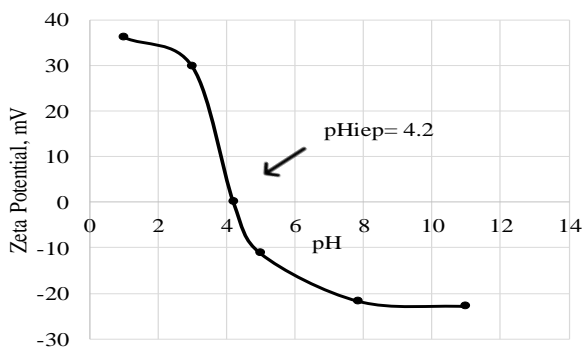


Figure 2. The effect of pH on the zeta potential of kaolin

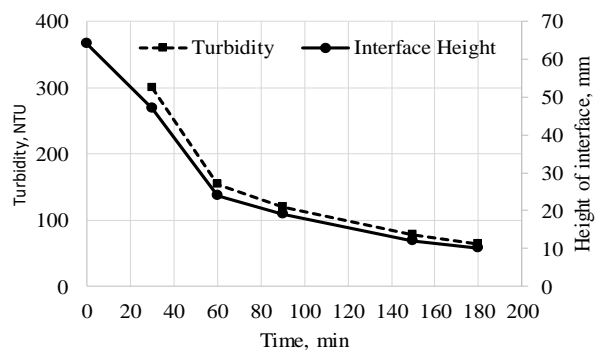


Figure 3. Variation of supernatant turbidity and interface height with settling time

### 3.4. Effect of Coagulants and Flocculants on Sedimentation Velocity and Turbidity of Kaolin Suspension

At the end of the 30 minutes natural settling period, zeta potential, turbidity, settling velocity, conductivity and final pH values of kaolin suspension were determined as; -25.74 mV; 301 NTU; 0.30 mm/min; 806  $\mu$ S/cm; 7.86, respectively. The sedimentation tests were performed in the presence of various coagulants and flocculants at different concentrations at the natural pH of kaolin suspension (7.86). Figure 4 reveals the effect of coagulant type and its concentration on the turbidity (a) and settling velocity (b) of kaolin suspension at the end of the 30 minutes settling period.

For all studied coagulants, concentrations reaching the lowest turbidity values and the turbidity values (efficiency of sedimentation) reached at these concentrations are as follows:

- $\text{FeCl}_3$  250 mg/L- 26.90 NTU (91%)
- $\text{Al}_2(\text{SO}_4)_3$  1000 mg/L- 29.10 NTU (90%)
- $\text{MgCl}_2$  500 mg/L- 72.30 NTU (76%)
- $\text{CaCl}_2$  2000 mg/L- 108 NTU (64%)
- $\text{NaCl}$  125 mg/L- 225 NTU (25%)

EFFECT OF COAGULANTS AND FLOCCULANTS ON DEWATERING OF KAOLIN SUSPENSIONS

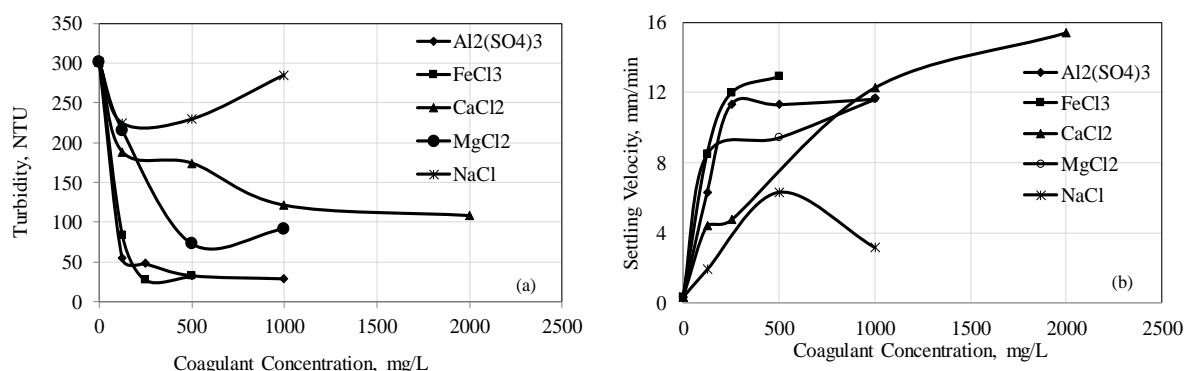


Figure 4. Effects of mono/multivalent metal salts concentrations on the turbidity (a) and settling velocity (b) of kaolin

The turbidity of suspension decreased with higher ionic value of metal salts. In terms of efficiency, trivalent coagulants showed approximately the same performance, whereas univalent coagulant coagulated the suspension with the worst performance (Figure 4a). Coagulant concentrations that reached the highest settling velocity are given below.

- CaCl<sub>2</sub> 2000 mg/L- 15.40 mm/min
- FeCl<sub>3</sub> 500 mg/L- 12.89 mm/min
- MgCl<sub>2</sub> 1000 mg/L- 11.63 mm/min
- Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> 250 mg/L- 11.32 mm/min
- NaCl 500 mg/L- 6.29 mm/min

When sedimentation velocity and turbidity values are compared, it is seen that the best sedimentation velocity (15.40 mm/min) was achieved with CaCl<sub>2</sub>, which coagulated the suspension with 64% efficiency. The best mean sedimentation velocity calculated for FeCl<sub>3</sub>, which coagulated the suspension with 91% efficiency, was 12.89 mm/min. As known, the coagulation/flocculation processes are performed to increase the sedimentation rate of the suspended particles in the suspension and remove turbidity of water. Decreasing turbidity of the water along with sedimentation of the particles is an expected result. However, a rapid sedimentation may result in a higher supernatant turbidity. Still, a slower sedimentation velocity will result in a more stable sedimentation so supernatant turbidity may also be lower. In other words, the conditions where the sedimentation velocity is optimum may not produce the same result for turbidity also. Some studies in the literature are in good agreement with this [22].

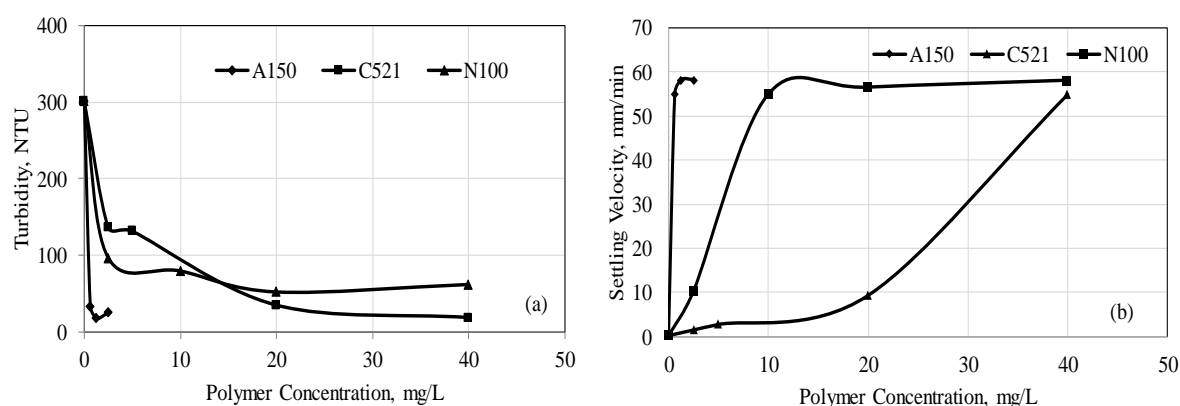


Figure 5. Effects of polymer concentrations on the turbidity (a) and settling velocity (b) of kaolin

The anionic (A150) and cationic (C521) polymers flocculated the suspension at about the same performance (94%). However anionic polymer has provided the same efficiency at a lower concentration compared to cationic polymer. In some studies, it has been shown that high molecular weight anionic polymers can be used in the sedimentation of negative charged clay [4, 23, 24]. In this case, the polymer bridging mechanism is primary importance. The turbidity of the kaolin suspension decreased with increasing concentration of polymers. A increasing trend was observed above a particular concentration for anionic polymer (Figure 5a). While high

V. ÖNEN, M. GÖÇER, H.A. TANER

polymer concentrations may restabilize the suspension, insufficient amount of polymer may result in insignificant flocculation. For all studied flocculants, concentrations reaching the lowest turbidity values and the turbidity values (efficiency of sedimentation) reached at these concentrations are as follows:

- Anionic polymer (A150) 1,25 mg/L - 17,10 NTU (94.3%)
- Cationic polymer (C521) 40 mg/L - 18,22 NTU (93.9%)
- Nonionic polymer (N100) 20 mg/L - 51,50 NTU(82.8%)

All flocculants reached approximately the same settling velocity (58 mm/min). However, this velocity was achieved at a quite low concentration with anionic flocculant (Figure 5b). In recent years, in some researches, high settling velocity (1–10 m/h) have been achieved for kaolin tailings with the aid of polymer [25].

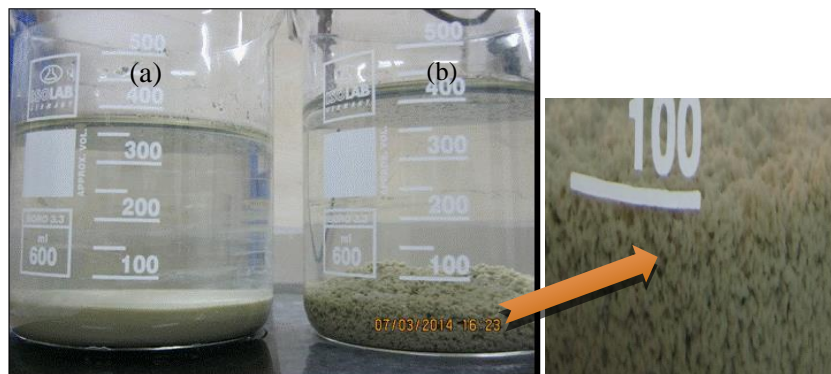
Understanding the interaction between kaolin and polymer is important to optimize flocculant performance. Several studies have been performed, examining the adsorption of polymer on to kaolin [26, 27]. All authors have concluded that the adsorption of polymer occurs primarily onto the edge surface of kaolin (i.e., on the broken bonds of aluminol (Al–OH) and silanol (Si–OH) groups) via hydrogen bonding. These electrostatic attractions between the positively charged polymer and the negatively charged kaolin promotes the adsorption mechanism [4]. Polymer bridging is the dominant bonding mechanism between anionic - nonionic polymer molecule and a kaolin particle [28]. A nonionic PAM molecule can be adsorbed onto both face and edge sites of kaolin particles. However, the most likely particle association is face-to-face (FF) in the presence of nonionic PAM due to a much larger proportion of face area compared to edge area on the kaolin particle [29].

Efficiency-cost analyses, which are essential for engineering studies, were taken into account while optimum concentration values were being determined. For example, the optimum concentration for  $Al_2(SO_4)_3$  was determined to be 125 mg/L, at which the efficiency was 81.9%, when the coagulant dosage was increased to 1000 mg/L, efficiency was 90.3%. As can be seen, when the concentration was increased by 8 times, the efficiency increased by only 8.4%. Therefore, the optimum concentration was determined to be 125 mg/L. The findings obtained during the 30-minute sedimentation period for optimum concentrations determined in the experimental studies on sedimentation of kaolin suspension are summarized in Table 2.

**Table 2.** Summary findings of sedimentation of kaolin suspension

	$Al_2(SO_4)_3$	$FeCl_3$	$MgCl_2$	$CaCl_2$	$NaCl$	A150	C521	N100
Opt. concentration (mg/L)	125	250	500	1000	125	<b>1.25</b>	20	20
Turbidity (NTU)	54.20	26.90	72.30	121	225	<b>17.1</b>	34.4	51.5
Zeta potential (mV)	-23.35	-17.36	-16.86	9.69	23,07	-26.79	-22.16	-19.7
Efficiency (%)	81.99	91.06	75.98	59.8	25.25	<b>94.32</b>	88.57	82.89
Conductivity ( $\mu S/cm$ )	936	962	1287	1147	1201	752	750	755
pH	7.22	6.40	7.50	8.50	7.50	7.1	8.2	8
Settling Velocity (mm/min)	6.29	11.94	9.43	12.26	1.88	<b>58.18</b>	9.43	56.61

Larger floc sizes were obtained with polymer than coagulants due to the higher molecular weight (Photograph 1). In general, polymers provide the better and the faster sedimentation due to large floc size but in the filtration, the coagulants tend to be more effective. In coagulation, the flocs are small and the resulting filter cake is a uniform porous structure, which allows rapid dewatering. Polymers have some disadvantages, such as, high cost, high volume of filter cake which is not easily dewaterable.



**Photograph 1.** Agglomerats in coagulation (a) and flocculation (b)

EFFECT OF COAGULANTS AND FLOCCULANTS ON DEWATERING OF KAOLIN SUSPENSIONS

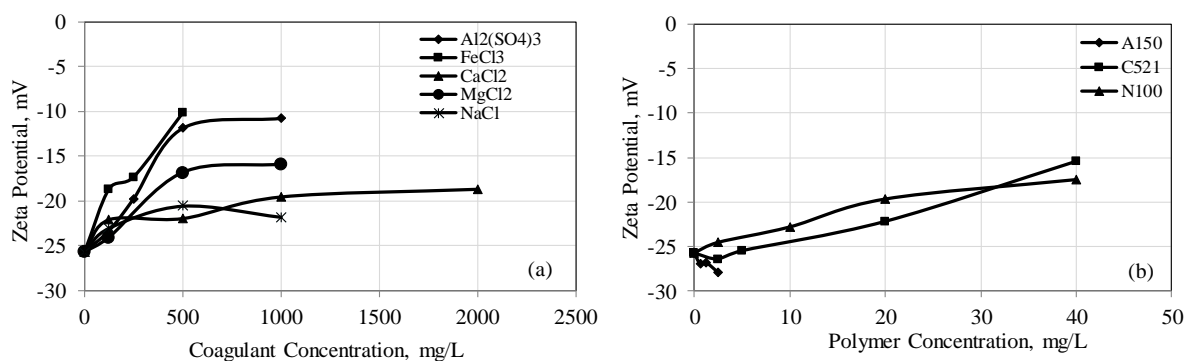


Figure 6. The variation of the zeta potential of kaolin with coagulant (a) and polymer (b) concentrations

3.5. Effect of Coagulants and Flocculants on Zeta Potential of Kaolin Suspension

The zeta potential of kaolin particles is determined using electrophoretic mobility in the coagulants and flocculants as functions of concentration.

As can be seen, the zeta potential of kaolin particles approaches zero with the increases of coagulant concentration. For example, the zeta potential of kaolin was -25.74 mV in naturally, which increases to -10.2 mV when 500 mg/L of FeCl<sub>3</sub> was added (Figure 6a). Previous studies have shown that di- and tri-valent cations bring the zeta potential value of clays closer to zero more than monovalent cations [30, 31]. The results of the current study are similar to those obtained in previous studies, and the highest zeta potential values were usually obtained with Al<sup>3+</sup> and Fe<sup>3+</sup> salts in mineral suspensions. It was also found that the coagulation behaviour of the kaolin suspension in the presence of coagulants was in good agreement with the electrokinetic properties.

In flocculation, zeta potential value increased with increasing concentration for cationic and nonionic polymer (it was approaching zero). For example, when 40 mg/L cationic polymer was added in the kaolin suspension, it was -15.36 mV. However, the zeta potential of kaolin became even more negative with increasing concentration of anionic polymer. when 2.5 mg/L anionic flocculant was added in the suspension, it was -27.86 mV (Figure 6b). Zeta potential measurements indicated that a polymer bridging mechanism played a more significant role in flocculation of kaolin suspensions with anionic flocculant.

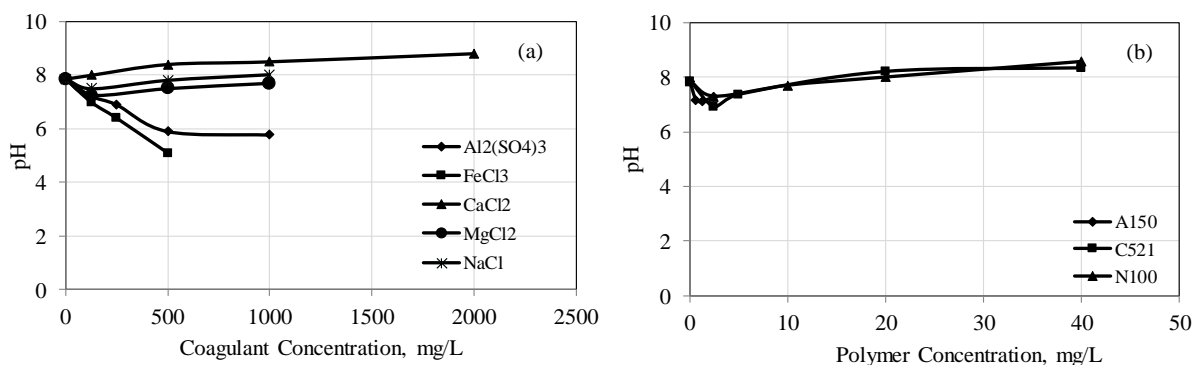


Figure 7. The variation of the suspension pH with coagulant (a) and polymer (b) concentrations

The pH value of natural kaolin suspension is 7.86. Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and FeCl<sub>3</sub> are acidic so the suspension pH decreased by increasing coagulant concentration. CaCl<sub>2</sub> is basic so it increases the suspension pH. When 500 mg/L FeCl<sub>3</sub> is added to kaolin suspension, pH becomes 5.1 and when the same concentration of CaCl<sub>2</sub> is added, pH becomes 8.4 (Figure 7a). Significant ups and downs, which was the case for coagulants, were not observed in flocculation experiments. The pH change range for kaolin suspensions is 6.90-8.57 in flocculation (Figure 7b).

4. CONCLUSIONS

- Kaolin suspensions have relatively high suspension stability due to high negative surface charge. 78.9% sedimentation efficiency could be provided at the settling time of 180 minute in natural sedimentation.

V. ÖNEN, M. GÖÇER, H.A. TANER

- In coagulation, the best performance (91%) was obtained with FeCl<sub>3</sub> (250 mg/L). At this concentration, the settling velocity is 11.94 mm/min. The effectiveness of coagulants increased with increasing ionic value of metal salts.
- Polymers showed better sedimentation efficiency than coagulants due to the higher molecular weight. The anionic and cationic polymers flocculated the suspension at about the same performance (94%-58 mm/min). However anionic flocculant has provided the same efficiency at a lower concentration.
- In coagulation, trivalent cations have a greater influence on the zeta potential of the samples. In flocculation, zeta potential value increased with increasing concentration for cationic and nonionic polymer (it was approaching zero). However, the zeta potential of kaolin decreased with increasing anionic polymer concentration. This indicates that, the main mechanism is the polymer bridging mechanism in flocculation of kaolin suspensions with anionic flocculant.

## ACKNOWLEDGEMENT

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*EFFECT OF COAGULANTS AND FLOCCULANTS ON DEWATERING OF KAOLIN SUSPENSIONS*

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