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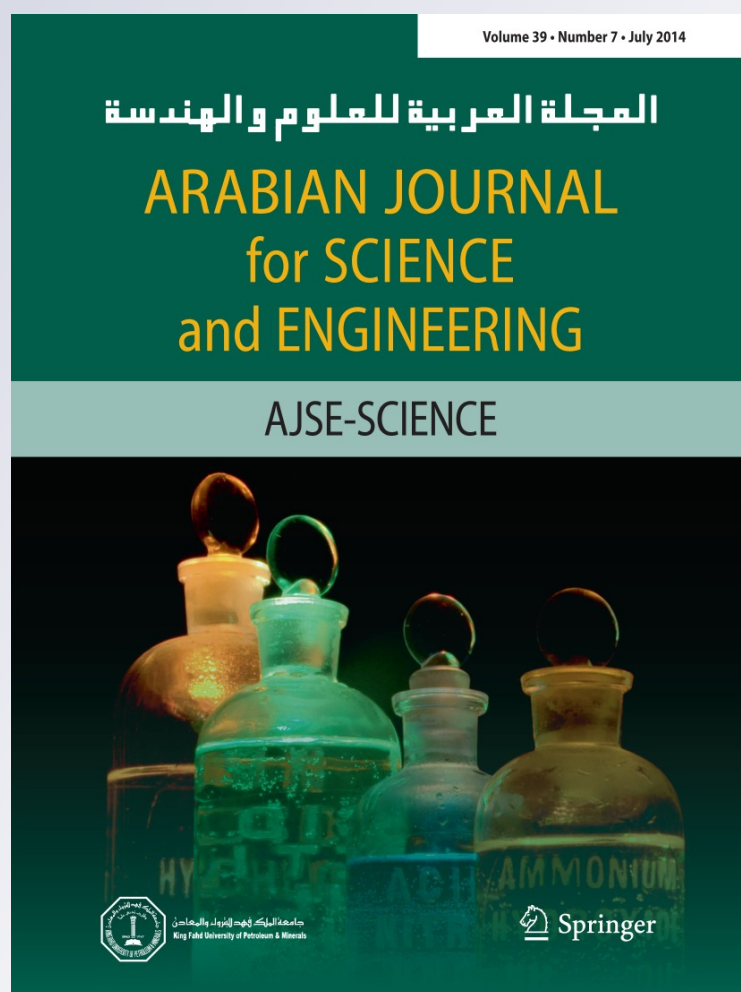
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Zeta Potential–Viscosity Relationship in Kaolinite Slurry in the Presence of Dispersants

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Abstract Kaolinite is a widely used additive for industrial applications. Hence, the properties of kaolinite affect the quality of the final materials. The fact that kaolinite is a heterogeneous mineral with dual charges has to be taken into account. In this study, the zeta potentials and the apparent viscosities of kaolinite suspensions in water in the presence of three dispersants; sodium silicate, sodium tripolyphosphate and sodium-polyacrylate at pH values of 4, 7 and 10 were determined. Diagrams of zeta potentials (ZPs) and viscosity as a function of dispersant concentration all show minima. The concentrations at which minima of these two properties appear do not coincide, except for those mineral dispersants at high pH values. The results are explained in terms of the spatial distribution of charges on basal and edge surfaces of kaolinite plates in acidic, neutral, and basic media.

الخلاصة

الكولنيت هو من المضافات المستخدمة على نطاق واسع للتطبيقات الصناعية. وبالتالي، فإن خصائص الكولنيت تؤثر في نوعية المواد النهائية. وفي الحقيقة فإن الكولنيت هو معدن غير متجانس مع شحنات مزدوجة ويجب أن تؤخذ بعين الاعتبار. وقد تم في هذه الدراسة تحديد إمكانات زيتا (ZPS) واللزوجة الظاهرية لمعلق كولنيت في الماء في وجود ثلاثة مشتتات، وسيليكات الصوديوم (SS)، وتراي بولي فوسفات الصوديوم (STPP) وبولي أكريليت الصوديوم (Na-PA) في قيم درجة حموضة من 4، 7 و 10. الرسوم البيانية لإمكانات زيتا واللزوجة كدالة للتركيز المشتت أظهرت كلها قيم دنيا. إن التراكيز التي ظهرت عندها القيم الدنيا لهاتين الخاصيتين لا تظهر تطابقا، باستثناء تلك المشتتات المعدنية في قيم درجة الحموضة العالية. وقد تم توضيح النتائج من حيث التوزيع المكاني للشحنات على أساس وحافة سطوح صفائح الكولنيت في الأوساط الحمضية والمتعادلة والقاعدية.

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1 Introduction

The physical and chemical properties of kaolinite $[(Al_2Si_2O_5(OH)_4]$ have led to its extensive use as a filler, extender, paper coater, pigment, and also as an important raw material for the ceramic, cement, refractory, catalyst, and fiber glass industries [1,2]. Layered kaolinite particles in a suspending liquid have dual heterogeneous surface charge characters. The basal planes of the kaolinite layers always exhibit negative charges throughout all pH values; this is due to a low degree of isomorphous substitution of cations with different charges within the lattice. The edge planes of the layers exhibit a pH-dependent charge character: i.e., they are either positive at low pH or negative at high pH. In spite of the
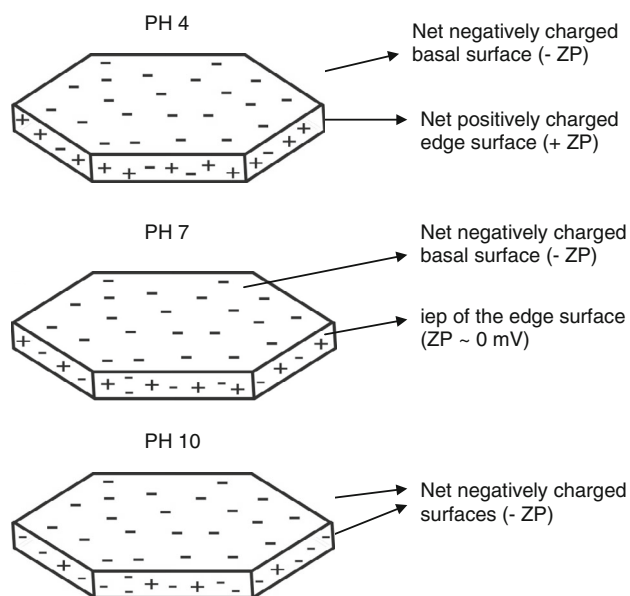
PH 4

Net negatively charged
basal surface (- ZP)

Net positively charged
edge surface (+ ZP)

PH 7

Net negatively charged
basal surface (- ZP)

iep of the edge surface
(ZP ~ 0 mV)

PH 10

Net negatively charged
surfaces (- ZP)

Fig. 1 Schematic illustration of heterogeneously charged kaolinite plates

dual-charge character, the kaolinite particles always exhibit negative charges (negative ZPs) in solution for all pH values above the isoelectric point (IEP). This is so because of the high surface area to edge area ratio. Namely, the edge surface area varies in constitution between 10–30 % of the total surface area—depending on the particle size and impurities present [3]. Kaolinite plates having high basal surface to edge surface ratios are presented in Fig. 1.

The viscosity (or, more precisely, the apparent viscosity) control of suspensions in the ceramic industry is also important for obtaining a desired final product [4–10]. The viscosity is mainly affected by the interactions between particles, as dictated by the repulsive forces (i.e., electrostatic or electrical double layer (EDL), hydration and steric forces) and the attractive Van der Walls (VDW) forces [11,12]. The ZP determines the strength of the EDL repulsive forces between particles and identifies the stability degree of colloidal suspensions when the predominant stabilization mechanism of the suspensions is electrostatic [12–14]. There are reports in the literature to the effect that a ZP increase leads to a decrease in viscosity or other rheological properties like yield (or shear yield) stress in colloidal suspensions consisting of homogeneously charged identical mineral particles [15–20]. The mathematical relationship between the ZP and the rheological property (yield stress) for such a suspension is given in [16] where the yield stress is a linear function of $(ZP)^2$. There is an inverse relation between them. That is, the yield stress increases when the absolute ZP decreases, and the yield stress (and also viscosity) reaches to its maximum value when the ZP is zero or vice versa.

Apparently well-dispersed suspensions have relatively low viscosities while agglomerations of particles result in

high viscosity values [16–18]. There are numerous studies on possible relationships between the ZP and the rheological properties of a variety of colloidal suspensions. These include suspension of clays [16,21–24], alumina [7,16,25], Ce-ZrO$_2$ [26], Si$_3$N$_4$ [17], anatase [27], zirconia [16], and cordierite [28]. The issue of the existence—or otherwise— of a relationship between viscosity and ZP has been dealt with in a number of studies [20,26,27,29]; unequivocal conclusions have not been reached. The main objective of this study is to investigate the relationship between the ZP and the viscosity data of kaolinite suspension having heterogeneous charge structure in the absence or presence of dispersants and find out what degree of correlation exists between these two quantities. We also seek to explain the underlying reasons for this relationship. So, the present work on aqueous kaolinite suspensions with and without a dispersant is one more contribution to this complex subject.

2 Experimental

2.1 Materials

The Ukrainian kaolinite was kindly supplied by the Ankara Seramik Co. of Turkey and its chemical and mineralog-

Table 1 Chemical, mineralogical, and physical properties of kaolinite

	Unit	Value
Chemical content		
SiO$_2$	%	47.59
Al$_2$O$_3$	%	35.76
CaO	%	0.19
MnO	%	0.03
Fe$_2$O$_3$	%	1.54
K$_2$O	%	0.18
Na$_2$O	%	0.03
TiO$_2$	%	1.05
SO$_3$	%	0.03
Loss of ignition	%	13.6
Mineralogical content		
Mica	%	3
Kaolinite	%	93
Quartz	%	4
Physical properties		
Specific surface area	m^2/g	20.2
Particle size distribution		
D90	μm	30.04
D50 (average)	μm	8.59
D10	μm	2.64

ical analysis and some physical properties are given in Table 1.

Also, the dispersants used and some of their properties are presented in Table 2. The other chemicals used were of analytical grade. All the dispersants and other chemical solutions and kaolinite suspensions were prepared using double-distilled water (with a specific conductivity below 2 μS/cm at its natural pH = 6.1).

2.2 Methods

Zeta potential values were obtained from electrophoresis results obtained with a zeta meter (Zeta-Meter 3.0+) as a function of each dispersant concentration (between 0.1–6 mg/g-clay) at pH values of 4, 7 and 10 at room temperature (25 ± 1 °C). 25 mg of each kaolin powder was dispersed into 50 mL of a background electrolyte solution of 1 mM NaCl. The pH was adjusted by adding 1 M HNO_3 or 1 M NaOH with a pH meter (Mettler Toledo Seven Easy) and stirred with a magnetic stirrer (Velp-Scientifica) for 15 min. Then the dispersant was added and stirred for 15 min, and finally the suspension was poured into the measuring cell.

The ZP values of kaolin particles were obtained using the Smoluchowski equation (1)

$$U = \zeta \varepsilon_0 \varepsilon / \eta \qquad (1)$$

Here U is the electrophoretic mobility of the particles in (m/s)/(volt/m), ε_0 is the permittivity of free space (8.854×10^{-12} C^2/Jm), ε is the static dielectric constant of the medium (78.5 for water at 25 °C), η is the viscosity of the medium in Poise (=0.1 kg m^{-1}s^{-1}), ζ is the ZP in V of the particles. For each ZP measurement ten particles were tracked and the averages are reported here. The ZP versus pH measurements without dispersant were performed using 1 mM NaCl.

Apparent viscosity measurements of kaolinite suspensions as a function of the pH and the dispersant concentration (between 0.1 and 6 mg/g clay) at pH values of 4, 7 and 10 were carried out, respectively, using a rotational viscometer (Brookfield DV-II+) with a spindle No. 1 at constant 100 rpm at 25 ±1 °C. The kaolin powder (100 g) was mixed with 400 g of 1 mM NaCl solution in a beaker (600 mL) and mechanically stirred at 700 rpm for 4 h. The pH of the suspension was adjusted using 1.0 M of HNO_3 or NaOH and stirred for 15 min. The viscosity readings were recorded every 5 s for 30 s.

Table 2 Chemical structures and some properties of used dispersants

Name	Abbreviation	Chemical structure	Manufacturer firm	Other properties
Sodium silicate (water glass)	SS	(Na_2SiO_3) NaO—Si—ONa ‖ O	Fluka	Na_2O: 11–12 wt%
				SiO_2: 22–24 wt%
				MW: 122 g/mol
				Density: 1.37 g/ml
				Appearance: liquid
Sodium tripoly-phosphate	STPP	($Na_5P_3O_{10}$) NaO—P—O—P—O—P—ONa	Merck	P_2O_5/Na_2O: 0.6
				Purity: 85 % (Technical grade)
				MW: 367.86 g/mol
				Appearance: powder
Sodium polyacrylate (Reotan-L as commercial name)	Na-PA	-------H_2C——CH------- C=O ONa	Lamberti	Specific gravity: 1.29–1.31 kg/l Active content: 44–46 %
				MW ≈ 4,000 g/mol
				Appearance: liquid

3 Results and Discussions

3.1 Determination of the IEP of Kaolinite

Each mineral has an isoelectric point (IEP) at a pH controlled by the mineral powder composition. By definition, at IEP there is no net charge at the surface, the total positive charges are equal to the total negative ones. H^+ and OH^- ions are potential determining ions for clay minerals as well as for all metal oxides and hydroxides [2,30]. Mineral powders typically agglomerate at IEP while going away from IEP in either pH direction results in de-agglomeration. Figure 2 presents ZP variation of the Ukrainian kaolinite as a function of pH. The ZP is predominantly negative over the entire pH range studied—from 2.5 to 11—and appears to be dominated by the properties of the negative basal faces. The IEP of Ukrainian kaolinite is at pH = 2.5. IEP values are known to be dependent on pretreatments [4,31,32].

The pH increase results in an increase in the negative charges on kaolinite due to adsorption of OH^- ions on the $\equiv$ Si–OH, $=$ Al–OH, $\equiv$ Si–OH$_2^+$, and $=$ Al–OH$_2^+$ sites or deprotonation of these groups at the edge surface of kaolinite. The decrease in pH has been reported to result in a decrease of the absolute ZP values due to adsorption of H^+ ions on the $\equiv$ Si–O$^-$, $=$ Al–O$^-$, $\equiv$ Si–OH, $=$ Al–OH groups at the edges [31,32]. Oplhen [2] has discussed three basic interaction types for layered clay minerals, such as kaolinite, depending on pH and electrolyte concentration. The first is an edge–face interaction between positively charged edges and negatively charged faces within acidic regimes below the IEP of the kaolinite edge (at pH $\approx$ 7) [16,31,33,34] which leads to the formation of a card-house structure. The second type is an edge–edge interaction, which occurs at nearly neutral pH, where the EDL repulsive force is zero but the viscosity of the suspension is lower than the viscosity resulting from the edge–face interactions. The third type is a face–face interaction, which is seen at higher electrolyte concentrations
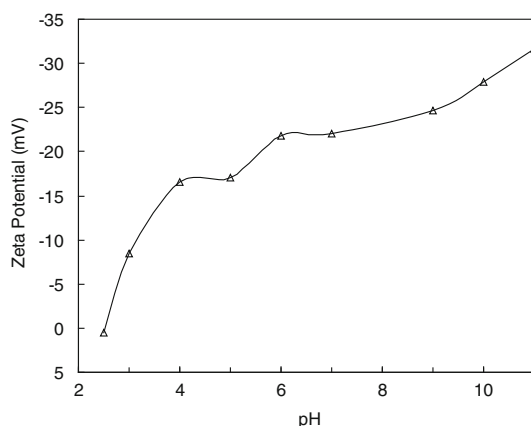
and is virtually independent of pH of the suspension. As a result, at low pHs (such as pH = 4), we have kaolinite as dual-charged particles. At increasing pH values, the kaolinite particles reach a state of charge homogeneity. For the reasons outlined above, we have chosen three pH values— namely 4, 7 and 10—to study a possible relationship between the ZP and apparent viscosity of kaolinite suspensions in the presence of a dispersant.

3.2 Zeta Potential/Viscosity and Dispersant Concentrations Curves

3.3 Sodium Silicate (SS)

Sodium silicate is an important inorganic dispersant and widely used for dispersing of clay slurries. Figure 3 shows the ZP and the viscosity variation as a function of SS concentration for moderately acidic (pH = 4) and basic (pH = 10) media.

A sharp decrease in the viscosity is shown at pH 4 due to breaking the card-house structure in a narrow concentration range which results from increasing of anionic charge on the edge. However, at pH 10, this decrease is not as sharp as at pH 4, because there is no card-house structure in the system instead the anionicity of both face and edge surfaces partially enhances the whole stability of the suspension. Similar effects appear in the presence of anionic dispersants, as reported by others previously [35,36]. In a suspension, SS undergoes hydrolysis and gives anionic silicate components such as $[Si(OH)_3]^-$, $[Si_2O_3(OH)_4]^{2-}$, and $[Si_4O_8(OH)_4]^{4-}$ [30]. The presence of such highly charged silicate species moves the ZP toward more highly negative values.

Consider a possible connection between the ZP and the viscosity vs. dispersion concentration diagrams beginning with pH = 4. The minimal value of ZP = -26 mV is seen at 0.6 mg/g clay SS concentration. The minimum of viscosity,
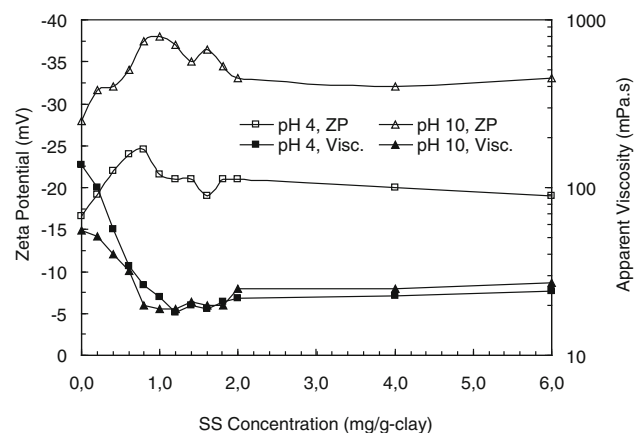

Fig. 2 The zeta potential of kaolinite as a function of pH

Fig. 3 The zeta potential and apparent viscosity of kaolinite suspension as a function of SS concentration at pH 4 and 10

equal to 19 mPa s, is seen at 1.2 mg/g clay SS concentration. However, at pH = 10, the minimum value of viscosity = 20 mPa s and the minimum of ZP, at −38 mV, appears at the same SS concentration, namely 0.8 mg/g clay. Prior to us, Duran and coworkers [19] pointed out the difficulties in relating ZP to the viscosity of suspensions. Penner and Lagaly [20] measured the dynamic mobility (DM) of kaolin and montmorillonite particles using an electroacoustic method at high-solid content. Also, they report a lack of agreement between the extreme of the DM and viscosity curves as a function of dispersant concentration.

3.3.1 Sodium Tripolyphosphate (STPP)

Sodium polyphosphates—such as sodium-trypolyphosphate $(Na_5P_3O_{10})$, sodium-hexametaphosphate $(N_6P_6O_{18})$, etc.— constitute another group of important multivalent anions used as dispersants. The action of the polyphosphate ions $(P_3O_{10}^{5-}, P_3O_{10}^{4-},$ etc.) may differ from that of polyvalent silicate anions, although both types of anions absorb electrostatically. Polyphosphate ions are also capable of forming strong covalent bonds (specific adsorption) with a number of metallic cations such as Al at the edges of clay particles, particularly at pHs below the IEP at the edges of kaolinite [2,30].

In Fig. 4, we present the respective results. For pH = 4, we see again that the minimum of viscosity = 15 mPa s at the dispersant concentration = 1.0 mg/g clay does not coincide with the minimum of ZP = −26 mV at 0.6 mg/g clay. The lowest value of ZP among all dispersants is equal to −48 mV is seen at pH = 10 for 0.4 mg/g clay dispersant concentration. Here, at the same concentration, we also see a minimum of η = 16 mPa s. The differences in behavior at pH = 4 and pH = 10 might be related to the charge density of kaolinite. Likely, kaolinite has dual charge (layer edge positive and
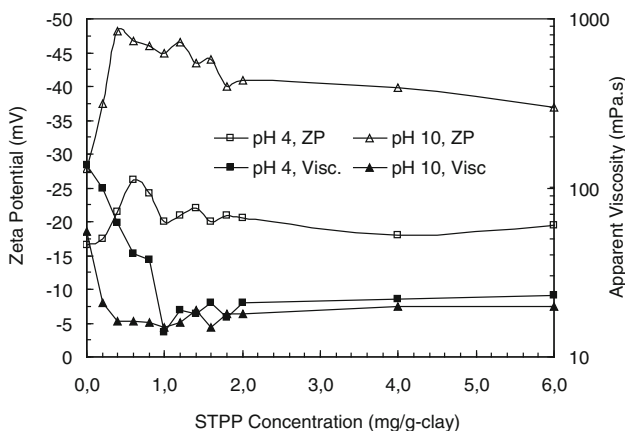
layer surface negative) at pH = 4, whereas at pH = 10 both surfaces are predominantly negative; see again Fig. 1.

3.3.2 Sodium-Polyacrylate (Na-PA)

A polymer-based anionic dispersant Na-PA is a homopolymer of sodium acrylate, widely used in ceramic slurries for viscosity and thixotropy control. Anionic polymers (with molecular weights of less than 10^3 g/mol) such as Na-PA, polymethacrylic acid, polyacrylic acid, ammonium polyacrylate provide either electrostatic or steric stabilization of colloidal suspensions [26]. Figure 5 shows ZP and viscosity changes of the suspensions depending on Na-PA concentration at pH = 4 and 10.

We see in Fig. 5 that viscosity decreases sharply at both low and high pH values, reaching minima at 0.6 and 0.2 mg/g clay for pH 4 and 10, respectively. The sharp decreases can be attributed to specific adsorption of carboxyl [–(C=O)–O⁻] groups of Na-PA with some metallic cations such as
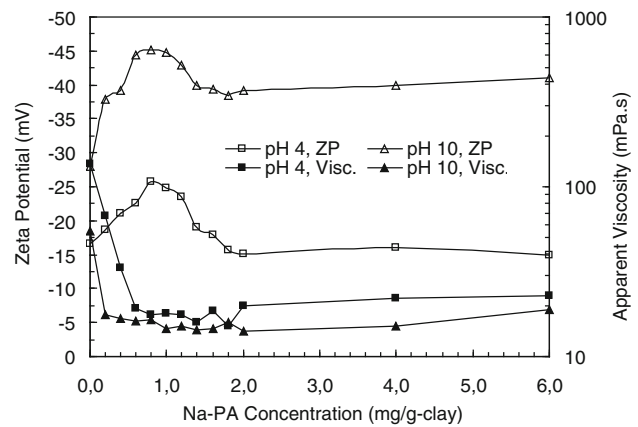

Fig. 5 The zeta potential and apparent viscosity of kaolinite suspension as a function of Na-PA concentration at pH 4 and 10
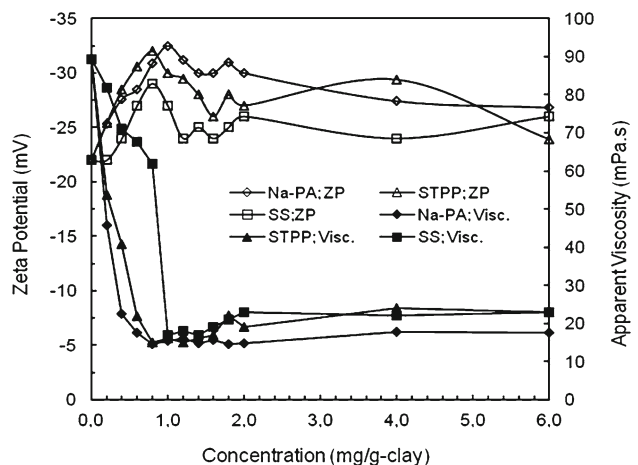

Fig. 4 The zeta potential and apparent viscosity of kaolinite suspension as a function of STPP concentration at pH 4 and 10

Fig. 6 The zeta potential and viscosity of kaolinite suspension as a function of various dispersant concentrations at pH 7

Table 3 The concentrations giving max ZP and min viscosity at different pH

Dispersant	Concentration (mg/g clay)					
	pH 4		pH 7		pH 10	
	Max ZP	Min. Visc.	Max ZP	Min. Visc.	Max ZP	Min. Visc.
Na-PA	0.8 (−26 mV)	0.6 (18 mPa s)	1 (−33)	0.8 (15)	0.6 (−44)	0.2 (16)
STPP	0.6 (−26)	1 (15)	0.8 (−32)	0.8 (15)	0.4 (−48)	0.4 (16)
SS	0.6 (−24)	1.2 (19)	0.8 (−29)	1 (17)	0.8 (−38)	0.8 (20)

Ca^{2+}, Al^{3+} existing at the edge surfaces of kaolinite (similar as in the case of STPP) [37]. Then, we have regions of nearly constant viscosity until 2 mg/g clay or so for both pH values. Afterwards, we have for pH = 4 a slight increase of viscosity which can be explained by an excess ionic strength resulting from unadsorbed polyacrylate molecules. The minima of ZP, −26 and −44 mV, for pH = 4 and 10 is seen, respectively, at 0.8 and 0.6 mg/g clay. The ZP is an indicator of electrostatic stabilization rather than the steric stabilization. Hence, maximum stability (minimum viscosity) can be obtained at low dispersant concentrations before reaching the ZP maximum. For instance, good stability (and lower viscosity) can be obtained in the presence of Na-PA for alumina suspensions [9] even at lower ZPs such as 10 mV due to the steric effect of adsorbed polymers onto the particle surface.

3.3.3 Comparison of Results for the Three Dispersants

To compare the effects of the three dispersants on the stability of kaolinite suspensions, we present the results for pH = 7 in Fig. 6. Na-PA causes a dramatic decrease in the viscosity at the lowest dispersant concentration, STPP at a slightly higher one, and SS at a still higher concentration. This may be attributed to the specific adsorption of Na-PA and STPP onto kaolinite edges and also to additional steric stabilization provided by Na-PA.

The ZP in the presence of either Na-PA or STPP is lower than that of SS. We have just seen that viscosities are lower for Na-PA and STPP than for SS. Values of dispersant concentrations at the ZP minima and viscosity minima are listed in Table 3. Among the nine concentration pairs, only in three cases do these minima coincide. Two out of these three pertain to STPP.

4 Conclusions

The following factors play a role in ZP and viscosity values of our mineral suspensions:

1. the particle shape effect: the effect due to the shape of the particles is not considered in the Smoluchowski equation. Under the same experimental conditions, the behav-ior of spherical homogenously charged particles may be different from that of charged heterogeneous layered particles—leading to variations in electrophoretic mobility in the suspension.
2. methodology: It is difficult to follow layered hexagonal kaolinite particles as compared to spherical ones by microscope during ZP measurement.
3. stabilization mechanism of dispersants: In the presence of polymer-based dispersants leading to steric stabilization, such as Na-PA, disagreement between locations of minima of ZP and viscosities is expected since ZP is related to electrostatic interactions in the system. In the presence of inorganic dispersants (SS and STPP), one expects a better connection between the minima of the two quantities—but this is not seen for SS at pH = 4.
4. pH of the suspension: as the pH increases, net surface charge on the layer edges switches from positive to negative. Thus, the charge distribution of kaolinite becomes more homogenous and a better connection between the ZP and viscosity minima is expected for inorganic dispersants.

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