

Adsorption of Poly (4-Sodium Styrene Sulfonate) on Kaolinite Clays

O. M. Sadek,¹ W. K. Mekhemer,¹ F. F. Assaad,² B. A. Mostafa¹

¹*Institute of Graduate Studies & Research, Alexandria University, 163 Horreya Avenue, P.O. Box 832, Shatby 21526, Alexandria, Egypt*

²*National Research Center, Cairo, Egypt*

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ABSTRACT: The adsorption of polymer on clay particles has great importance in many industrial applications. This work aimed to study the adsorption of anionic polymer, poly (4-sodium styrene sulfonate), on kaolinite clay surfaces. Three different Egyptian kaolinite clay samples were used. The kaolinite clays were saturated with sodium or calcium cation. It was found that the Freundlich isotherm is applicable for this polymer adsorption on kaolinite. The parameters of the Freundlich equation are very close, indicating a comparable nature for the binding behavior between the different samples of kaolinite and the adsorbed polymer.

The adsorption process was examined at different pH values (3, 6, and 9 ± 0.1) to cover the range below and above the point of zero charge of kaolinite surfaces. It was found that, below this point, the polymer adsorption increased, while above it the polymer adsorption decreased. The polymer adsorption on calcium kaolinite was higher than that of sodium kaolinite at the same pH value. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 1712–1719, 2006

Key words: adsorption; kaolinite clay; flocculation; polystyrene sulfonate

INTRODUCTION

Clay minerals are extensively used in a wide range of applications. They are a key component in the formulation of ceramic products, cement, drilling fluids, molding sand, paints, and paper, among others. An important characteristic that clay minerals are able to provide in such applications is adequate particle dispersion, which is necessary to obtain a uniform and stable system.^{1,2}

The clay minerals of the kaolinite group represent two-layer structures. The crystal units of kaolinite (1 : 1 minerals) are made of one silica (tetrahedral) layer alternating with one alumina (octahedral) layer. The two sheets of each crystal unit of kaolinite are held together by oxygen atoms, which are mutually shared by the silicon and aluminum atoms in their respective sheets. These units, in turn, are held together rather rigidly by hydrogen bonding. Consequently, the lattice is fixed and no expansion ordinarily occurs between the units when the clay is wetted. The effective surface of kaolinite is thus restricted to its outer faces. Kaolinite crystals usually are hexagonal with clean-cut edges. It has a small negative charge due to broken edges and structural imperfections. The extent of atom substitution in the kaolinite lattice is small. Therefore,

the kaolinite minerals are chemically “pure,” giving them useful properties in industrial use.^{3,4}

When colloid clay minerals are added into water, different effects on rheological and electro-kinetic properties will occur due to the interactions between particles, ions, and water molecules. The particles interact with each other through columbic and Van der Waals forces. Clay particles move randomly in the suspension by Brownian motion, causing particle-to-particle collisions. The stability of the dispersion is directly dependent on how the particles interact during the collision moments. Chemical additives (electrolytes, polymers, surface active agents, etc.) will interact with clay particles when added into a water system.

The extent of the interaction of clay particles and additives is determined by a number of parameters. These are particle size, shape, and surface charge of the clay particle. Also, clay/water ratio, additive type, and its degree of hydrolysis, pH, and temperature have great influences.^{5–8} The investigations of polymer adsorption on clays are multipurpose. The adsorption of polymer modifies the surface properties, rheology, and suspension stability of the clay, which is of great technical importance.⁷

Therefore, the objective of this work was to study the adsorption behavior of poly (4-sodium styrene sulfonate) on kaolinite clay at various pH values. Also, the effect of exchangeable cation Na⁻ and Ca on the adsorption process is investigated.

Correspondence to: O. M. Sadek.

EXPERIMENTAL

Three Egyptian kaolinite samples; Bed Selected, New Esila, (from Sinai), and Kalabsha (from Aswan), were used as adsorbent in the present study, which are designated by A, B, and C, respectively. The clay was washed, and particles sized less than $20\ \mu$ were separated.⁹ The clay surface was saturated with sodium or calcium cation.¹⁰

The polymer used in this work was anionic poly (4-sodium styrene sulfonate) having molecular weight of $70,000\ \text{g mol}^{-1}$, supplied by Sigma-Aldrich Co.

X-Ray diffraction analyses of the three different kaolinites were performed using a Shimadzu XD-3 diffractometer. The cation exchange capacity of the three kaolinite clays was measured according to the standard method.¹¹ The specific surface area of the kaolinite was determined by Permaran apparatus (Out Okumpuoy, Research Laboratory, Tapiola, Finland).

Polymer adsorption isotherms were performed in duplicated samples by equilibrating 0.5 g of Na- or Ca-kaolinite with aliquots of poly (4-sodium styrene sulfonate) solution having different concentrations, ranging from 20 to 1000 ppm. The adsorption isotherms were carried out at different pH (3, 6, and 9 ± 0.1). The systems were allowed to equilibrate for 48 h on a reciprocal shaker, adjusted to the desired temperature, $25^\circ\text{C} \pm 1$. The suspensions were then centrifuged at 2000 rpm for 15 min. The supernatants were extracted. The amount of polymer adsorbed on the clay was calculated from the change in the relative viscosity of the polymer solution before and after the adsorption process. Several workers¹²⁻¹⁵ have successfully applied the viscosity method for the determination of polymer concentration. In this method, the polymer adsorbed amount was measured twice and the average was considered. The difference between two successive determinations doesn't exceed $\pm 2\ \text{mg/g}$ clay. The obtained adsorption isotherm data were recorded in Appendix A.

RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction analysis of the used clay. It is obvious that the Egyptian clay samples are similar and composed mainly of kaolinite mineral.

The cation exchange capacity and the specific surface area of the Egyptian kaolinite clays (A, B, and C) are shown in Table I. It was observed that the magnitude of the cation exchange capacity (CEC) of samples A and B are in agreement with other types of kaolinite, such as China kaolinite¹¹ and Georgia kaolinite.¹⁶ But sample C has a high CEC value, 2 to 3 times, compared to the other kaolinites. Some of these differences could be explained by the higher specific surface area of sample C. Also, high CEC in some kaolinite is found to be due to some impurities of smectite.^{17,18}

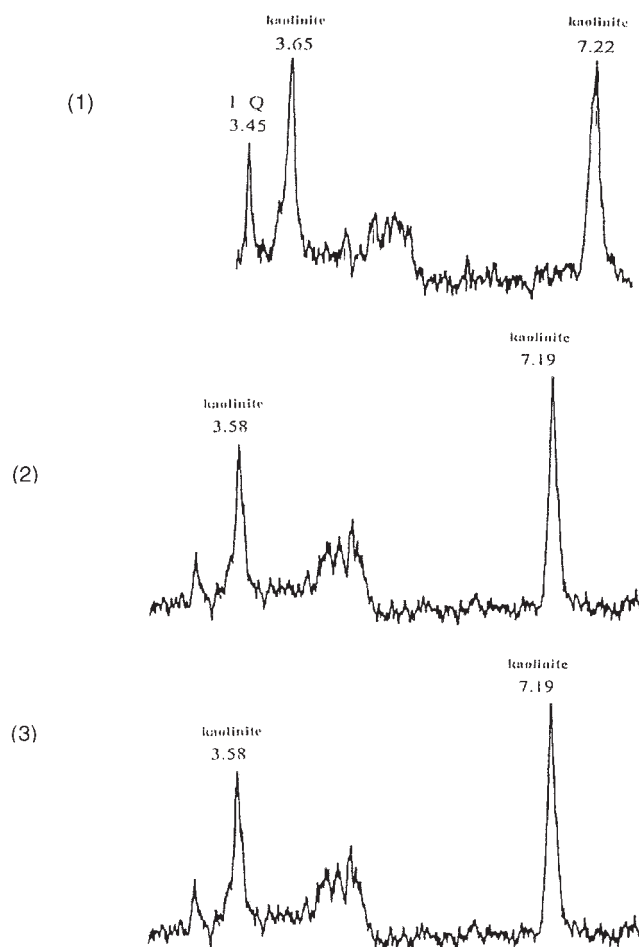


Figure 1 X-ray diffraction of the Egyptian kaolinite samples: (1) Sample A (Bed Selected), (2) Sample B (New Esila), and (3) Sample C (Kalabsha).

Adsorption isotherms

Figures 2–4 show the adsorption isotherms of poly (4-sodium styrene sulfonate) on Na- and Ca- kaolinite clays at different pH values (3, 6, and 9 ± 0.1). All the isotherms confirmed an S-shape, which indicates “co-operative adsorption,” with polymer molecules tending to be adsorbed packed in rows or clusters.¹⁹ The S-curve shows that the polymer chains are adsorbed end on the ionic group at the kaolinite surface and the polymer chains extended outwards in the solution; thus, the surface becomes rapidly more hydrophobic with increase in adsorption. The adsorbed long chain ions are vertically close-packed. The uptake of polymer by the kaolinite samples has the following order: $C > B > A$, which can be attributed to high CEC and surface area of sample C, as shown in Table I.

The previous adsorption isotherms, Figures 2–4, show that kaolinites (samples A, B, and C) saturated with divalent cation, such as Ca^{+2} , adsorb more polymer than the kaolinite saturated with monovalent cation, such as Na^{+} , because the former cation has larger

TABLE I
The Physical Properties of the Kaolinite Clay

	Sample A		Sample B		Sample C	
	Ca-	Na-	Ca-	Na-	Ca-	Na-
CEC (meq./100 gm)	7.98	7.40	12.20	11.52	20.90	21.50
Surface area (m ² /g)	10.3064		12.1621		13.5665	

hydration shells than the latter. As a result, the adsorption of flexible linear polymer onto the clay surface generally leads to desorption of numerous solvent molecules from the surface. Thus, the maximum adsorbed amount of polymer on Ca-kaolinites (samples A, B, and C) is larger than that adsorbed on Na-kaolinites. Therefore, the exchangeable cations in the clay interlayer play an indirect role in the sense that it is the size of their hydration shells that is important.²⁰ These results are in agreement with other published results, which studied the adsorption of cis/trans 1,2-dichloroethylene (CDE) vapor on smectite clay exchanged with Pb²⁺, Hg²⁺, Cd²⁺, Ca²⁺, Ag⁺, or Na⁺. They found that clay saturated with divalent cations adsorb CDE more than monovalent cations.²¹

The Freundlich empirical equation ($0.9 \leq R^2 \leq 0.95$) describes the results of polymer adsorption on kaolinite samples, indicating a multilayer adsorption process^{22,23}:

$$\log x/m = \log k + 1/n \log C$$

where x/m is the adsorbed amount of the polymer, C is the equilibrium concentration of the polymer, $1/n$ measures the nature and strength of the adsorption

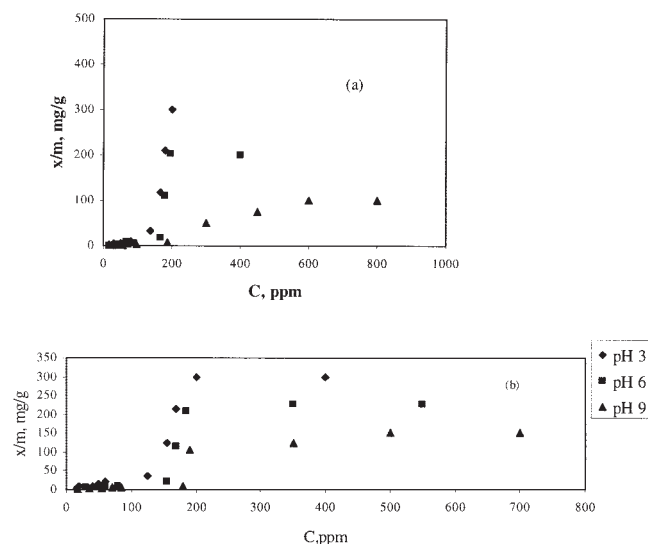


Figure 2 Adsorption isotherm of Poly (4-sodium styrene sulfonate) on Sample A: (a) Na- and (b) Ca- kaolinite, at different pH values.

process and the distribution of active sites, and k is the index for classifying the degree of adsorption. Figures 5–7 show Freundlich plots of polymer adsorption on Na- or Ca-kaolinite at different pH values. The parameters of Freundlich isotherms of polymer adsorption on Na- or Ca-kaolinite are shown in Tables II and III, respectively. Very close adsorption constant values imply a comparable nature for the binding sites independent of the origin of the studied clays. The results proved that kaolinite contains specific sites of different binding characteristics. These results are in agreement with other published results.^{24,25}

Adsorption and pH dependency

The adsorption isotherms, Figures 2–4, show that, as pH values increased, the adsorbed amount decreased.

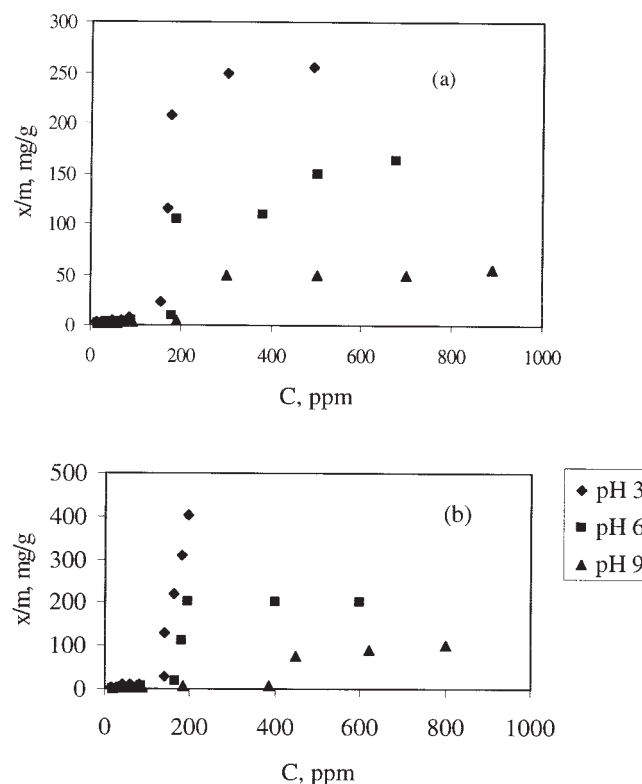


Figure 3 Adsorption isotherm of Poly (4-sodium styrene sulfonate) on Sample B: (a) Na- and (b) Ca-kaolinite, at different pH values.

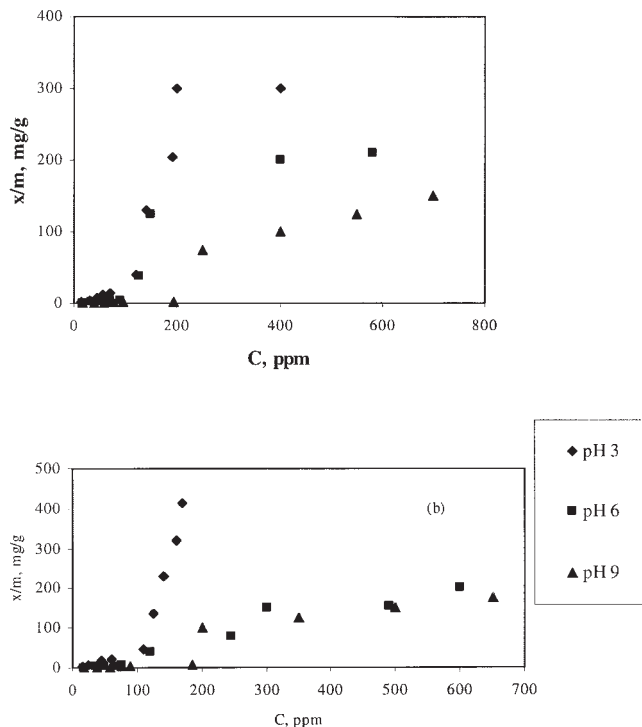


Figure 4 Adsorption isotherm of Poly (4-sodium styrene sulfonate) on Sample C: (a) Na- and (b) Ca-kaolinite, at different pH values.

This is due to the crystallographic structure of kaolinite, which corresponds to a 1 : 1 layer structure. Kaolinite particles have two different sources of charges, edges, and faces. At the faces, there are permanent charges, while edges are pH dependent charges. The

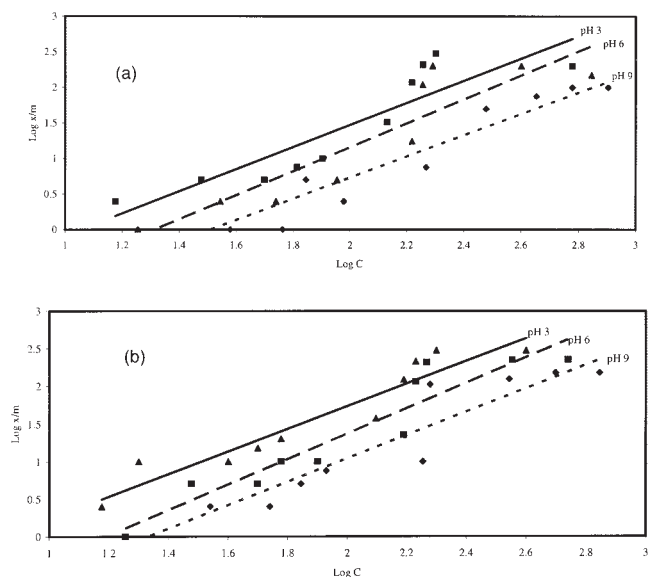


Figure 5 Freundlich isotherms for the adsorption of Poly (4-sodium styrene sulfonate) by Sample A: (a) Na- and (b) Ca-kaolinite, at different pH values.

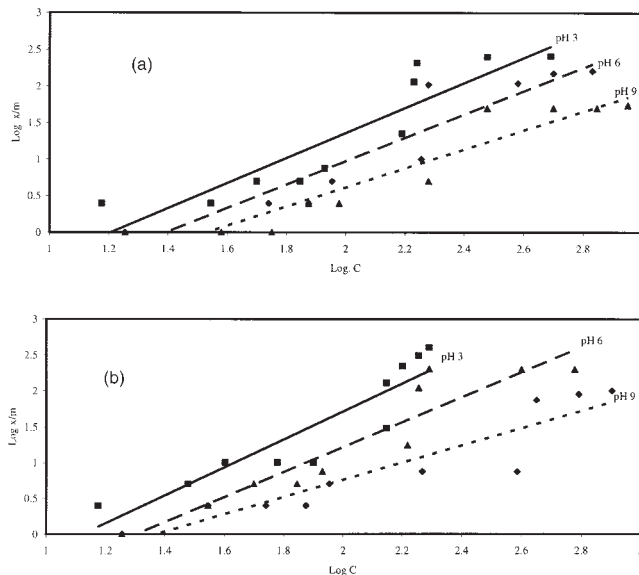


Figure 6 Freundlich isotherms for the adsorption of Poly (4-sodium styrene sulfonate) by Sample B: (a) Na- and (b) Ca-kaolinite, at different pH values.

point of zero charges (PZC) of kaolinite samples were obtained from our previous work²⁶ (Table IV). At low pH values (below PZC), the edges have positive charges and the faces exhibit negative charges. Electrostatic interaction between the polymer negative chain and positive sites of kaolinite particles occurred, leading to high adsorption capacity. Under these conditions, random coil conformations are favored. Above PZC, the edges of clays become negatively charged. Consequently, a repulsive effect takes place between

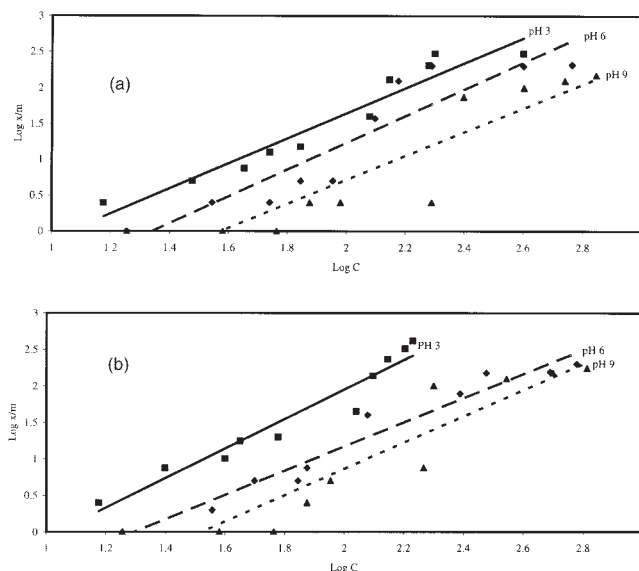


Figure 7 Freundlich isotherms for the adsorption of Poly (4-sodium styrene sulfonate) by Sample C: (a) Na- and (b) Ca-kaolinite, at different pH values.

TABLE II
Freundlich Adsorption Isotherm Constants for Na- Kaolinite at Different pH

pH	Sample A			Sample B			Sample C		
	3	6	9	3	6	9	3	6	9
l/n	1.56	1.69	1.49	1.71	1.60	1.30	1.75	1.87	1.67
K	0.0229	0.0061	0.0056	0.0086	0.0059	0.0103	0.0142	0.0031	0.0024

l/n is the intensity of adsorption; K is index for adsorption classifying.

TABLE III
Freundlich Adsorption Isotherm Constants for Ca-Kaolinite at Different pH

pH	Sample A			Sample B			Sample C		
	3	6	9	3	6	9	3	6	9
l/n	1.50	1.69	1.56	1.95	1.74	1.20	2.02	1.66	1.80
K	0.0537	0.0097	0.0085	0.0065	0.0053	0.0225	0.0081	0.0072	0.0019

l/n is the intensity of adsorption; K is index for adsorption classifying.

TABLE IV
The Point of Zero Charge (PZC) of the Kaolinite Clay

	Sample A	Sample B	Sample C
Ca-	5.60	5.60	6.00
Na-	6.08	6.24	6.12

negative sites of the clay and ionized negative polymer sulfonate. Therefore, the adsorption capacity of poly (4-sodium styrene sulfonate) on kaolinite dropped again to lower values. This indicates that the adsorbed amount of polymer onto the kaolinite is high at low pH compared to a lower adsorbed amount at high pH.^{18,27}

CONCLUSIONS

The main conclusions that could be drawn from the study of adsorption of poly (4-sodium styrene sulfonate) on kaolinite clays are as follows:

1. The adsorption isotherm of polystyrene sulfonate on kaolinite has an S-shape curve and obeys the Freundlich equation.
2. The adsorption amount of polystyrene sulfonate on kaolinite depends upon the origin of the kaolinite and pH of the medium. So, below the PZC of kaolinite, polymer adsorption quantity is higher than above PZC.
3. At constant pH value, the adsorbed amount of polymer on calcium kaolinite is higher than on the sodium form.
4. This study had a very practical application in studying the local kaolinite clay and treating it for organo-philization.

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APPENDIX

TABLE A.I
Freundlich Adsorption Isotherm Data of 0.2% Na- Bed
Selected Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	30	10	5	1.477	0.699
	60	50	10	5	1.699	0.699
	80	65	15	7.5	1.813	0.875
	100	80	20	10	1.903	1.000
	200	135	65	32.5	2.130	1.512
	400	165	235	117.5	2.217	2.070
	600	180	420	210	2.256	2.322
	800	200	600	300	2.301	2.477
	1000	600	400	200	2.778	2.300
6	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.544	0.398
	60	55	5	2.5	1.740	0.398
	80	65	15	7.5	1.813	0.875
	100	90	10	5	1.954	0.699
	200	165	35	17.5	2.217	1.243
	400	180	220	110	2.255	2.041
	600	195	405	202.5	2.290	2.306
	800	400	400	200	2.600	2.300
	1000	700	300	150	2.845	2.176
9	20	18	2	1	1.255	0.000
	40	38	2	1	1.579	0.000
	60	58	2	1	1.763	0.000
	80	70	10	5	1.845	0.699
	100	95	5	2.5	1.978	0.398
	200	185	15	7.5	2.267	0.875
	400	300	100	50	2.477	1.699
	600	450	150	75	2.653	1.875
	800	600	200	100	2.778	2.000
	1000	800	200	100	2.903	2.000

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.

TABLE A.II
Freundlich Adsorption Isotherm Data of 0.2% Ca-Bed
Selected Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	20	20	10	1.300	1.000
	60	40	20	10	1.600	1.000
	80	50	30	15	1.699	1.176
	100	60	40	20	1.778	1.300
	200	125	75	37.5	2.097	1.579
	400	155	245	122.5	2.190	2.088
	600	170	430	215	2.230	2.330
	800	200	600	300	2.300	2.477
	1000	400	600	300	2.600	2.477
6	20	18	2	1	1.255	0.000
	40	30	10	5	1.477	0.699
	60	50	10	5	1.699	0.699
	80	60	20	10	1.778	1.000
	100	80	20	10	1.900	1.000
	200	155	45	22.5	2.190	1.352
	400	170	230	115	2.230	2.060
	600	185	415	207.5	2.267	2.317
	800	350	450	225	2.544	2.352
	1000	550	450	225	2.740	2.352
9	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.540	0.398
	60	55	5	2.5	1.740	0.398
	80	70	10	5	1.845	0.699
	100	85	15	7.5	1.930	0.875
	200	180	20	10	2.255	1.000
	400	190	210	105	2.279	2.021
	600	350	250	125	2.544	2.097
	800	500	300	150	2.699	2.176
	1000	700	300	150	2.845	2.176

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.

TABLE A.III
Freundlich Adsorption Isotherm Data of 0.2% Na- New
Esila Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	35	5	2.5	1.544	0.398
	60	50	10	5	1.699	0.699
	80	70	10	5	1.845	0.699
	100	85	15	7.5	1.929	0.875
	200	155	45	22.5	2.190	1.352
	400	170	230	115	2.230	2.060
	600	175	415	207.5	2.240	2.320
	800	300	500	250	2.477	2.398
	1000	490	510	255	2.690	2.410
6	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.544	0.398
	60	55	5	2.5	1.740	0.398
	80	75	5	2.5	1.875	0.398
	100	90	10	5	1.954	0.699
	200	180	20	10	2.255	1.000
	400	190	210	105	2.279	2.021
	600	380	220	110	2.580	2.041
	800	500	300	150	2.699	2.176
	1000	675	325	162.5	2.830	2.210
9	20	18	2	1	1.256	0.000
	40	38	2	1	1.580	0.000
	60	58	2	1	1.760	0.000
	80	75	5	2.5	1.875	0.398
	100	95	5	2.5	1.978	0.398
	200	190	10	5	2.279	0.699
	400	300	100	50	2.477	1.699
	600	500	100	50	2.699	1.699
	800	700	100	50	2.845	1.699
	1000	890	110	55	2.949	1.740

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.

TABLE A.IV
Freundlich Adsorption Isotherm Data of 0.2% Ca- New
Esila Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	30	10	5	1.477	0.699
	60	40	20	10	1.602	1.000
	80	60	20	10	1.778	1.000
	100	80	20	10	1.900	1.000
	200	140	60	30	2.146	1.477
	400	140	260	130	2.146	2.114
	600	160	440	220	2.200	2.342
	800	180	620	310	2.255	2.491
	1000	195	805	402.5	2.290	2.605
6	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.544	0.398
	60	50	10	5	1.699	0.699
	80	70	10	5	1.845	0.699
	100	85	15	7.5	1.930	0.875
	200	165	35	17.5	2.217	1.243
	400	180	220	110	2.255	2.041
	600	195	405	202.5	2.290	2.306
	800	400	400	200	2.600	2.300
	1000	600	400	200	2.778	2.300
9	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.544	0.398
	60	55	5	2.5	1.740	0.398
	80	75	5	2.5	1.875	0.398
	100	90	10	5	1.954	0.699
	200	185	15	7.5	2.267	0.875
	400	385	15	7.5	2.585	0.875
	600	450	150	75	2.650	1.875
	800	620	180	90	2.792	1.954
	1000	800	200	100	2.903	2.000

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.

TABLE A.V
Freundlich Adsorption Isotherm Data of 0.2% Na-
Kalabsha Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	30	10	5	1.477	0.699
	60	45	15	7.5	1.653	0.875
	80	55	25	12.5	1.740	1.097
	100	70	30	15	1.845	1.176
	200	120	80	40	2.079	1.602
	400	140	260	130	2.146	2.114
	600	190	410	205	2.279	2.312
	800	200	600	300	2.300	2.477
	1000	400	600	300	2.600	2.477
6	20	18	2	1	1.255	0.000
	40	35	5	2.5	1.544	0.398
	60	55	5	2.5	1.740	0.398
	80	70	10	5	1.845	0.699
	100	90	10	5	1.954	0.699
	200	125	75	37.5	2.097	1.574
	400	150	250	125	2.176	2.097
	600	195	405	202.5	2.290	2.306
	800	400	400	200	2.600	2.300
	1000	580	420	210	2.763	2.320
9	20	18	2	1	1.255	0.000
	40	38	2	1	1.580	0.000
	60	58	2	1	1.763	0.000
	80	75	5	2.5	1.875	0.398
	100	95	5	2.5	1.980	0.398
	200	195	5	2.5	2.290	0.398
	400	250	150	75	2.398	1.875
	600	400	200	100	2.602	2.000
	800	550	250	125	2.740	2.097
	1000	700	300	150	2.845	2.176

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.

TABLE A.VI
Freundlich Adsorption Isotherm Data of 0.2% Ca-
Kalabsha Kaolinite at Different pH Values

pH	Co ppm	C ppm	Co-C ppm	x/m mg/g	log C	log x/m
3	20	15	5	2.5	1.176	0.398
	40	25	15	7.5	1.398	0.875
	60	40	20	10	1.600	1.000
	80	45	35	17.5	1.650	1.243
	100	60	40	20	1.778	1.300
	200	110	90	45	2.040	1.650
	400	125	275	137.5	2.097	2.138
	600	140	460	230	2.146	2.362
	800	160	640	320	2.204	2.510
	1000	170	830	415	2.230	2.618
6	20	18	2	1	1.255	0.000
	40	36	4	2	1.556	0.300
	60	50	10	5	1.699	0.699
	80	70	10	5	1.845	0.699
	100	75	15	7.5	1.875	0.875
	200	120	80	40	2.079	1.600
	400	245	155	77.5	2.389	1.889
	600	300	300	150	2.477	2.176
	800	490	310	155	2.69	2.190
	1000	600	400	200	2.778	2.300
9	20	18	2	1	1.255	0.000
	40	38	2	1	1.580	0.000
	60	58	2	1	1.763	0.000
	80	75	5	2.5	1.875	0.398
	100	90	10	5	1.954	0.699
	200	185	15	7.5	2.267	0.875
	400	200	200	100	2.300	2.000
	600	350	250	125	2.544	2.097
	800	500	300	150	2.699	2.176
	1000	650	350	175	2.813	2.240

Co, Initial concentration ppm; C, Average equilibrium concentration ppm; x/m, Adsorbed amount of polymer in mg per one gram of kaolinite clay.