FLOCCULATION AND COAGULATION OF Ca- AND Mg-SATURATED MONTMORILLONITE IN THE PRESENCE OF A NEUTRAL POLYSACCHARIDE

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Abstract—The objective of this study was to observe flocculation of montmorillonite in the presence of a glucose polymer (dextran) and to observe the effect of saturating cation and coagulant addition on the flocculation process. Flocculation of montmorillonite was dependent on polymer molecular weight, polymer/clay ratio (w/w), nature of exchangeable cation, and ionic strength of the suspension to which the polymer was added. The T500 dextran (molecular weight = 5×10^5) caused enhanced sedimentation of Ca-montmorillonite suspension at a polymer/clay ratio of ≤ 0.01. Increasing the polymer concentration above this level stabilized the suspension such that sedimentation was less than or equal to that of the control. The T2000 dextran (molecular weight = 2×10^6) caused a similar increase in the sedimentation of Ca-montmorillonite at polymer/clay ratios of <0.1. The ability of the T2000 polymer to cause flocculation at greater polymer/clay ratios as compared to the T500 polymer was attributed to the lower osmotic pressure between clay particles for equal concentrations of the two polymers. Flocculation of Ca-montmorillonite by dextran was enhanced when the clay had initially been coagulated by the addition of salt. Reduction of the diffuse double layer upon addition of salt permitted the polymer to extend beyond the electrostatic barrier of the clays. Dextran was not able to flocculate Mg-montmorillonite suspensions with or without the presence of coagulant. The displacement of water molecules at the clay surface rather than within the hydration shell of the more highly polarizing Mg cations by polymer segments resulted in a greater polymer collapse on the clay surface leaving fewer and shorter polymer loops and tails available for contacting adjacent clay particles.

Key Words—Coagulation, Dextran, Exchangeable cation, Flocculation, Montmorillonite.

INTRODUCTION

The presence of polymeric biomolecules such as polysaccharides in soil is believed to facilitate clay flocculation and the formation of stable soil aggregates (Tisdall and Oades 1982, Chenu *et al* 1987, Fuller and Goh 1992). A significant relationship between carbohydrate content of soil aggregates and the amount of ultrasonic energy required to disperse clay from the aggregates has been demonstrated (Fuller and Goh 1992, Fuller *et al* 1995). More energy was required to disperse clay from soil aggregates possessing the greatest concentration of carbohydrate and exchangeable Ca. Thus, it can be hypothesized that the presence of polysaccharides and Ca contribute to the stabilization of clays within the aggregate structure as the interaction between organic polymers and exchangeable cations is fundamental to flocculation of clay (Parfitt and Greenland 1970a, 1970b).

Destabilization of clay suspensions occurs via coagulation and/or flocculation. The terms "coagulation" and "flocculation" have often been used interchangeably in colloid chemistry. Coagulation has been defined as particle aggregation induced by electrolyte addition while flocculation is defined as aggregation resulting from the linking or bridging of several particles by a polymer chain (LaMer 1966, Theng 1979). A coagulant acts to reduce the thickness of the diffuse double layers of adjacent clay particles thus reducing the interparticle distance; whereas, a flocculant is able to span the interparticle distance. These two processes may act individually or in concert to affect the sedimentation of clays and therefore are fundamental to the initial stages of aggregation.

The adsorption of dextran polymers by montmorillonite has been well established (Clapp *et al* 1968, Parfitt and Greenland 1970a, 1970b, Olness and Clapp 1975, Chenu *et al* 1987). The adsorption isotherms are generally of the high-affinity type. These polymers interact strongly with montmorillonite surfaces possibly through the adsorption of many polymer segments or "trains". The adsorption is essentially irreversible since the likelihood is small that all adsorbed trains will be desorbed at the same time (Theng 1982). Moreover, if adsorption is related to the probability of polymer segments contacting the clay surface, it is reasonable to expect larger polymers to be adsorbed to a greater extent than lower molecular weight polymers. Maximum

adsorption of dextran by Na-montmorillonite has been found to vary from 445 mg g^{-1} (Olness and Clapp 1975) to as low as 25 mg g^{-1} (Chenu *et al* 1987).

Dextran adsorption is also influenced by the nature of exchangeable cation on the clay. Clapp and Emerson (1972) suggest that initial adsorption of polysaccharides could occur by hydroxyl groups displacing water molecules surrounding exchangeable cations as has been shown for the adsorption of alcohols (Dowdy and Mortland 1967). The free energy of adsorption for polyethylene glycol was shown to arise primarily from an increase in translational entropy associated with the desorption of water molecules and is less negative with an increase in the polarizing power of the cation (Parfitt and Greenland 1970a).

The initial slope of dextran adsorption isotherms decreased as more strongly polarizing cations (e.g., Ca^{2+} and Al^{3+}) were present on the exchange complex (Parfitt and Greenland 1970b) since displacement of water molecules around more strongly polarizing cations will be more difficult. Therefore, the ability of the polymer to compete with water molecules for adsorption sites will determine the capacity for polymer adsorption on the clay. Thus, large molecular weight polymers are adsorbed to a greater extent than small molecular weight polymers since the former are better able to compete for adsorption sites.

The objective of this study was to examine the effect of polymer molecular weight, polymer/clay ratio, saturating cation, and presence of a coagulant on the flocculation of montmorillonite by a flexible random coil polymer such as dextran.

MATERIALS AND METHODS

Crook County Na-montmorillonite from Wyoming (SWy-1) was obtained from the Source Clays Repository of the Clay Minerals Society, Columbia, Missouri. A bulk sample was dispersed using a laboratory homogenizer. The clay fraction was fractionated by repeated sedimentation and decantation. The clays were saturated with Ca^{2+} or Mg^{2+} by addition of 0.6 M $CaCl_2$ or 0.6 M $MgCl_2$. This was repeated five times to ensure complete saturation of the clays. Excess salt was removed by washing with deionized water until no positive test for chloride could be observed using 0.01 M $AgNO_3$.

Two dextran polymers were obtained from Pharmacia (Sweden). These polymers had a molecular weight of approximately 500,000 (T500) and 2×10^6 (T2000).

The sedimentation was observed in clear 15-ml centrifuge tubes. Sufficient clay was obtained from the stock suspensions and added to the centrifuge tubes to ensure a 2% clay concentration in a 10-ml final volume. A 2% clay suspension was used because preliminary investigations showed that the boundary between the clear supernatant and clay was not distinct for clay suspensions with a concentration <2%. The pH of the stock solution was lowered to 7.0 with dilute HCl. The dextran polymers were solubilized in deionized water so that an appropriate volume of the solution would deliver pre-determined amounts of polymer. Polymer/clay ratios of 0.001, 0.002, 0.01, 0.02, 0.1, and 0.2 (w/w) were prepared by mixing the clay suspension with the appropriate volume of dextran solution. A control was also prepared with no polymer. The final volume was made to 10 mL and the tubes were gently shaken for 1 h to ensure thorough mixing of clay and polymer. The tubes were then set aside on a laboratory bench to allow sedimentation to begin. The temperature of the room remained at 22°C ± 1°C.

A traveling microscope, fitted with a Vernier scale was used to measure the depth of the sedimentation; the distance from the top of the clear supernatant to the top of the sedimenting clay was measured. Measurements were taken every 24 hours. The depth of sedimentation was used to infer the extent of flocculation of the suspension. It was assumed that the larger flocs would sediment more quickly than non-flocculated clay particles.

The effect of coagulation on the flocculation of montmorillonite by the T500 dextran was studied by adding 0.01 M $CaCl_2$ or 0.01 M $MgCl_2$ to the clay-dextran complexes. This was accomplished in one of two manners. In the first case, the clay-dextran mixtures were prepared as described above. However, following the one-hour shaking time, the salt was added to the mixture and the tubes were shaken again for one hour. Thus the coagulant was added after the flocculant. In the second case, the salt was added to the clay suspension prior to addition of the polymer. In this case, the coagulant was added before the flocculant.

RESULTS AND DISCUSSION

The sedimentation of montmorillonite was influenced by the presence of the T500 dextran polymer (Figure 1). Ca-saturated montmorillonite sedimented at a faster rate than the control when the polymer/clay ratio was ≤0.01. Each curve in Figure 1 reached a plateau at polymer/clay ratios of 0.01 or less indicating that at a ratio of 0.001, dextran was able to cause flocculation to the same extent as a dextran at a ratio of 0.01. Beyond a ratio of 0.01, additional polymer no longer enhanced the sedimentation of the clay. Within the first 72 hour of sedimentation at a polymer/clay ratio of 0.02, there was only a slight positive effect on the depth of sedimentation relative to the control. However, after 96 hours at a polymer/clay ratio of 0.02, the polymer more greatly enhanced sedimentation of clay relative to the control. Within the first 72 hours of sedimentation, the polymer at ratios of 0.1 and 0.2 resulted in similar sedimentation depths as the control.

There was essentially no flocculation of the clay when Mg^{2+} was the exchangeable cation (Figure 1). Recently, Heil and Sposito (1993) have shown that Ca was sig-
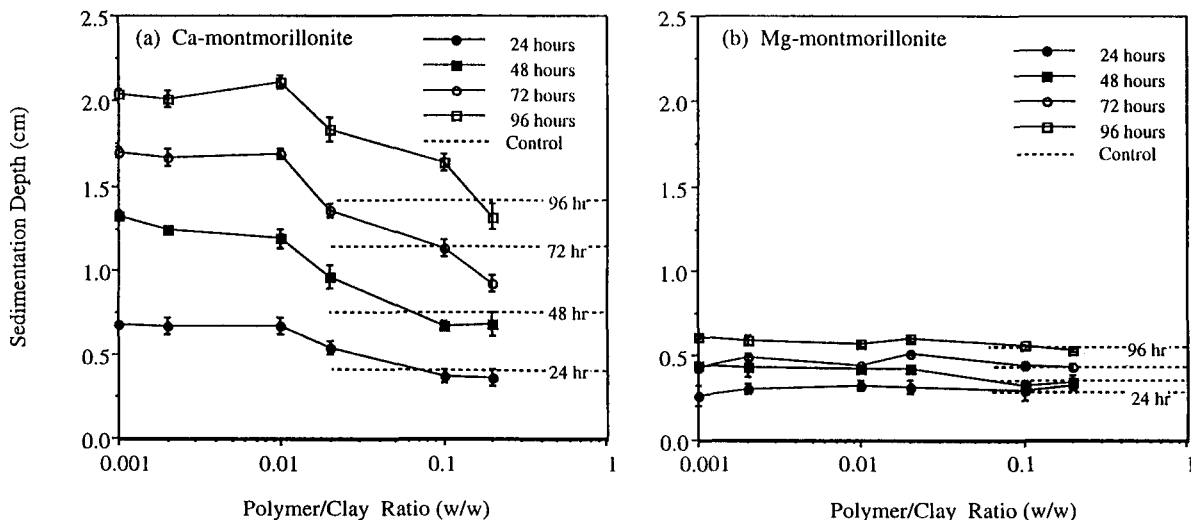
Figure 1.   Sedimentation of (a) Ca-saturated montmorillonite; and (b) Mg-saturated montmorillonite, in the presence of dextran (T500) at various polymer/clay ratios (w/w).

nificantly more effective than Mg in causing flocculation of illitic soils. These results for Mg-saturated montmorillonite contrast strongly with those for the Ca-montmorillonite, indicating that the polymer-clay interaction was affected by the exchangeable cation. The reason for this difference may be two-fold. Firstly, the affinity of the polymer for the clay surface was reduced by the presence of the greater polarizing Mg cations on the clay thereby making the displacement of water from the hydration shell of the exchangeable cation more difficult (Parfitt and Greenland 1970a). Secondly, it is also possible that dextran adsorption on the Mg-saturated clay resulted in greater collapse of the polymer on the surface. Thus, there would be shorter and fewer loops and tails extending away from the surface and flocculation would be inhibited. Greater polymer collapse at the clay surface can be expected where the polymer successfully competes with adsorbed water on the clay surface rather than within the hydration shell of the exchangeable cations.

It is also possible that little adsorption of dextran by the Mg clay had occurred. If this is so then flocculation or interparticle bridging would be limited. Since the amount of dextran adsorbed at each polymer/clay ratio was not measured, the lack of dextran adsorption by Mg clays cannot be eliminated as an explanation. However, since dextran has been shown to be adsorbed by both Ca and Na clays (Parfitt and Greenland 1970b, Olness and Clapp 1975, Chenu et al 1987), it is reasonable to expect dextran adsorption to occur on Mg clays.

The addition of the T2000 dextran polymer to Ca-saturated montmorillonite also resulted in enhanced sedimentation of the clay (Figure 2). The same tendency toward increased sedimentation relative to the

control at polymer/clay ratios of 0.01 or less occurred with the T2000 polymer. However, in contrast to the T500 dextran, an increase in sedimentation was still observed at a polymer/clay ratio of 0.1. The depth of sedimentation was much less for a polymer/clay ratio of 0.2 and was less than or equal to that of the control.

The T2000 dextran did not cause flocculation of the Mg-saturated clay. The presence of Mg on the exchange sites inhibited the flocculation of this clay by T2000 dextran as it did for the smaller T500 dextran.

The observed sedimentation in the presence of the T500 dextran was typical of colloid-polymer interactions. Polymers have been used for industrial purposes as either flocculants ("stickers") or stabilizers ("bumpers"). The factors that determine whether a polymer acts as a bumper or a sticker is the concentration of polymer relative to that of the colloidal particles. If the polymer concentration is too high, the colloidal particles become saturated with the polymer and an osmotic pressure builds up between colloidal particles (Lafuma et al 1991). In this way the colloidal particles are stabilized and the polymer acts as a bumper keeping the particles in a dispersed state. Repulsive interactions between interacting polymer loops and tails also contribute to stabilization of the clay suspension and is known as steric stabilization (Theng 1979). At a lower polymer concentration, the colloidal particles are not saturated and the buildup of osmotic pressure is insufficient to stabilize the suspension. If polymer loops and tails extend beyond the electrostatic barrier between charged colloidal particles, they act as "stickers" as flocculation is possible (Lafuma et al 1991). The distance over which the electrostatic barrier extends for clay particles depends on the thickness of the diffuse double layer which in turn depends on the saturating
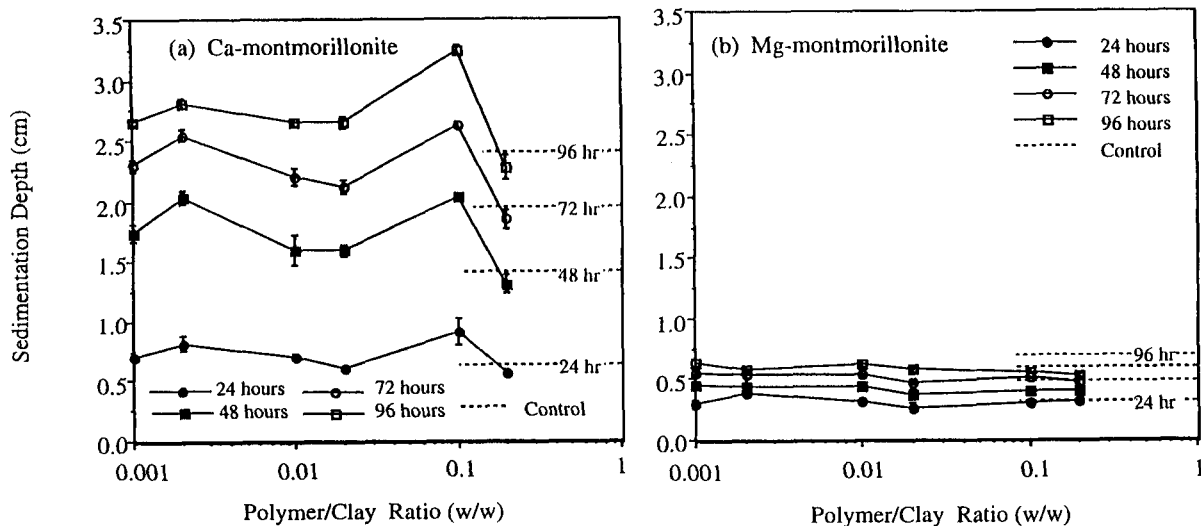
Figure 2.   Sedimentation of (a) Ca-saturated montmorillonite; and (b) Mg-saturated montmorillonite, in the presence of dextran (T2000) at various polymer/clay ratios (w/w).

cations. Therefore, polymer size and the length of loops and tails as well as thickness of the diffuse double layer will determine if flocculation occurs at a given polymer concentration.

Within the first 96 h, the presence of the T500 dextran at polymer/clay ratios of 0.1 and 0.2 resulted in similar or less sedimentation than the Ca-saturated control (Figure 1). If all the polymer was adsorbed, it would correspond to an adsorbed dextran concentration of 100 and 200 mg g^{-1} clay, respectively. This is the maximum dextran adsorbed by Ca-saturated Upton montmorillonite documented by Parfitt and Greenland (1970b). If the Ca-saturated montmorillonite is saturated with dextran at these concentrations, then the absence of flocculation relative to the control would be expected according to the reasoning of Lafuma *et al* (1991) as discussed previously. Within the first 96 h, the T500/clay ratio at which flocculation occurred was 0.01 or less (w/w). At these concentrations there were at least ten times less polymer present than at the adsorption maximum of dextran on Ca-saturated Upton montmorillonite as found by Parfitt and Greenland (1970b). It was clear that low polymer/clay ratios enhanced the depth of sedimentation of the Ca-saturated montmorillonite. Rennie *et al* (1954) were able to show an increase in aggregation with the addition of only 0.02% (w/w) of a polysaccharide produced by *Agrobacterium radiobacter*. In fact, most microbial polysaccharides cause an increase in aggregation at concentrations of only 0.02 to 0.2% (w/w) (Clapp *et al* 1962, Martin and Richards 1969, Martin 1971). Relative to the control, enhanced sedimentation in this study occurred at polymer concentrations of 0.1 to 1% (w/w). The slightly higher polymer concentrations that induced sedimentation may be attributed to the ad-

dition of polymer directly to a clay suspension whereas in the work cited previously, polymer was added directly to soil.

The T500 polymer must have been adsorbed in such a configuration that loops and tails extended far enough out from the surface of clay particles to overcome the electrostatic barrier between the particles. Complete collapse of positively charged polymers are believed to occur upon adsorption (Theng 1982). However, uncharged polymers, such as dextran, are adsorbed with only part (30 to 40%) of the polymer chain attached to the surface (Theng 1982). Therefore, the molecular weight of the polymer will determine the distance the loops and tails extend from the surface of the clay particle.

The T2000 and T500 dextran had similar effects on sedimentation at polymer/clay ratios of 0.01 and 0.002. However, after 96 hours of sedimentation, the T2000 polymer at a polymer/clay ratio 0.1 caused the greatest sedimentation of the Ca-montmorillonite. This is in contrast to the T500 dextran system where sedimentation depth was similar to the control at the ratio of 0.1. The greater flocculation in the presence of T2000 may be the result of the formation of a three-dimensional floc network as observed by Chenu *et al* (1987) for scleroglucan. The larger dextran polymer promoted the combination of smaller units into larger units by enhanced flocculation. This is largely attributed to lower osmotic pressure for the higher molecular weight dextran at the same dextran/clay ratio. The molecular weight of the T2000 polymer is four times that of the T500 polymer. Therefore, the molar concentration for the T2000 polymer will be one-quarter that of the T500 polymer for an equivalent mass of polymer in a given volume of solvent. The osmotic pressure developed by
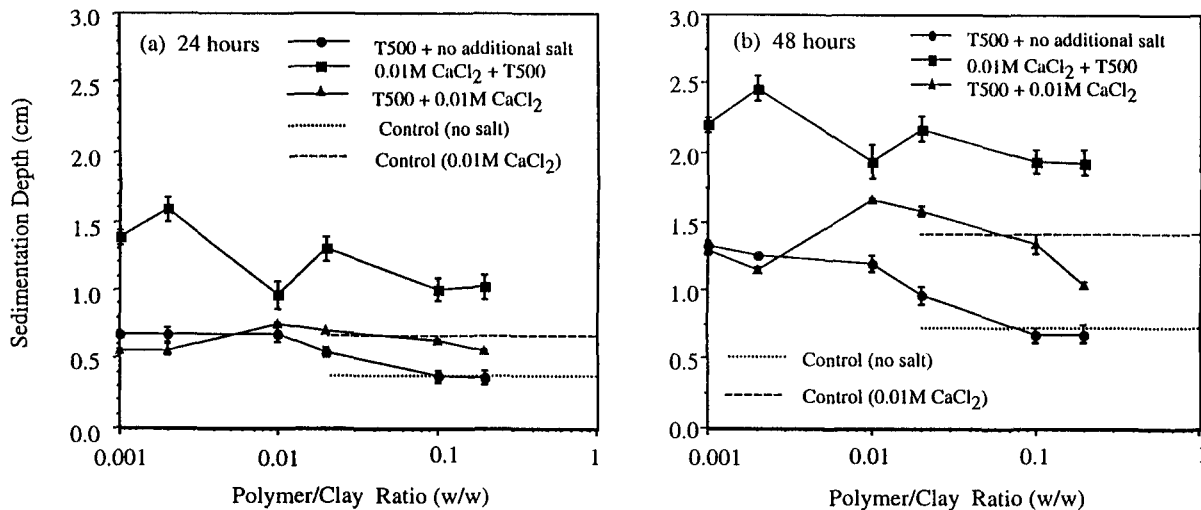
Figure 3.   The effect of the addition sequence of 0.01 M CaCl$_2$ and T500 dextran polymer on the sedimentation of Ca-saturated montmorillonite.

the presence of the T2000 polymer will be less than that of the T500 polymer since osmotic pressure is directly related to the molar concentration of the solute. Consequently, repulsion of clay particles due to osmotic pressure would be decreased.

The reason for the difference in sedimentation behavior between the Ca and Mg-saturated montmorillonite is not known with certainty. If polymer adsorption occurs via water displacement around exchangeable cations then the affinity of dextran polymers for the more strongly polarizing (higher charge density) Mg cation would be less. The more negative hydration enthalpy of Mg relative to that of Ca would make it more difficult for the polymer to displace hydration water around the Mg cation. As a result, the polymer may displace water adsorbed to the clay surface instead. A greater collapse of the polymer on the clay surface may then occur as more segment-surface contacts would be possible. This then would reduce the number of loops and tails that are available to extend beyond the electrostatic barrier around the clay.

The order in which 0.01 M CaCl$_2$ and T500 dextran were added to the Ca-saturated clay suspensions affected the sedimentation behavior of the clay (Figure 3). The case where no additional salt was added is indicated by the closed circles and is the same data found in Figure 1. When salt was added to the clay suspension after addition of dextran, the polymer no longer enhanced sedimentation (closed triangles) after 24 hours. After 48 hours, it appeared that the polymer actually inhibited sedimentation of the clays relative to the control (dashed line) by either disrupting or preventing the association of polymer with clay, which was evident where no additional salt was added (closed circles).

By comparison, the addition of the salt to the clay

suspension prior to the addition of polymer molecules resulted in enhanced sedimentation at all polymer concentrations (closed squares, Figure 3). Addition of salt would result in a greater shielding of colloid charge resulting in coagulation of the clays and hence the clays would approach each other more closely. Adding a flocculant, such as T500 dextran, now results in enhanced flocculation of the coagulated clays permitting the polymer to bridge clay particles. Even the higher concentrations of polymer resulted in flocculation of the clay suspension. Therefore, when coagulation precedes the introduction of a polymer, the probability will increase that polymer loops and tails will be present that are long enough to contact adjacent clay particles.

It has already been shown that the T500 dextran polymer did not cause flocculation of Mg-saturated montmorillonite (Figure 1). Addition of 0.01M MgCl$_2$ to the clay-dextran mixture did not enhance the sedimentation of the clay over that of the control (closed triangles, Figure 4). After 48 hours, the presence of polymer at all concentrations caused the sedimentation depth to decrease relative to the control. The inhibition of sedimentation by the polymer suggests that the polymer stabilized the suspension.

When salt was added prior to the polymer, there was essentially no increase in the depth of sedimentation except at the polymer/clay ratio of 0.001. Coagulation of the clays did not enhance the sedimentation of the Mg-saturated clay. The collapse of the polymer on the clay with few loops and tails extending beyond the electrostatic barrier of adjacent clay particles was invoked to explain the absence of flocculation in the Mg-saturated clay system. Addition of salt decreases the electrostatic barrier and the distance between clay particles is reduced. The polymer loops and tails should
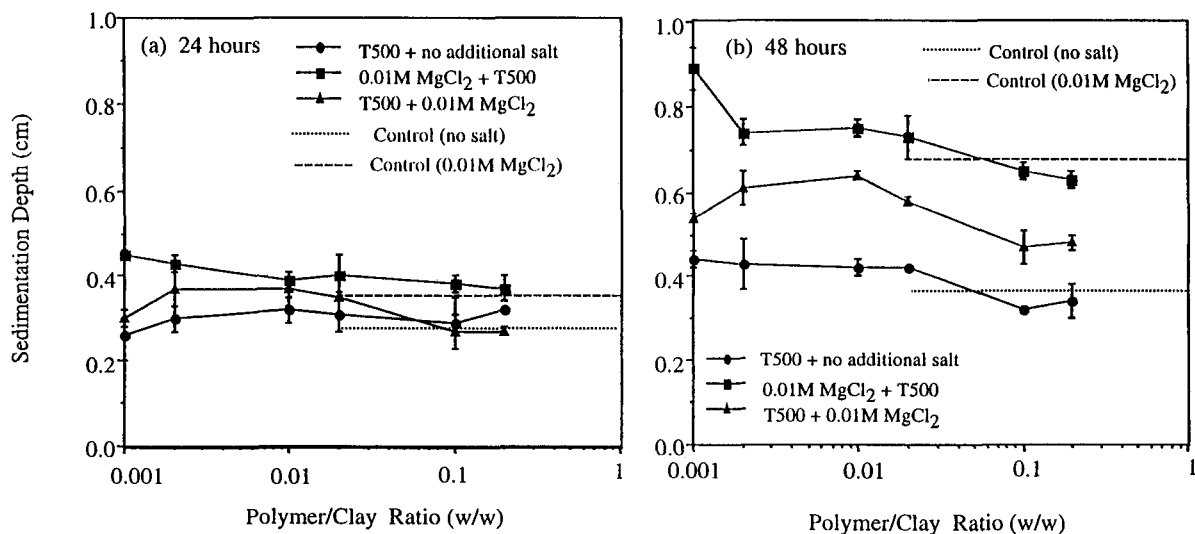
Figure 4.   The effect of the addition sequence of 0.01 M MgCl$_2$ and T500 dextran polymer on the sedimentation of Ca-saturated montmorillonite.

therefore be able to interact with adjacent clay particles. The absence of the marked sedimentation enhancement, as observed for the Ca-saturated clays for all polymer concentrations, may indicate that the adsorption of the polymer by Mg-saturated clay results in polymer collapse. A collapsed polymer with shorter and fewer loops and tails would not be able to bridge the coagulated clays.

The absence of a marked alteration of clay fabric upon adsorption of dextran (molecular weight of 2 × 10^6) by Ca-montmorillonite has recently been shown by Chenu *et al* (1987). They used a clay suspension concentration of 0.5%. Based on the adsorption maximum (25 mg g^{-1}) and the equilibrium concentration of dextran (approximately 1.25 mg ml^{-1}; final volume 20 ml), the polymer/clay ratio at the adsorption maximum in Chenu's study is estimated to be 0.3. This corresponds to the polymer/clay ratio where sedimentation was not enhanced for either the T500 or T2000 dextran. Therefore, the absence of particle rearrangement in the presence of dextran in the study of Chenu *et al* (1987) may be attributed to an excessive polymer concentration. Flocculation of the clays did not occur and the dextran polymers would have stabilized the clay suspension.

SUMMARY

The addition of dextran of differing molecular weight to Ca- and Mg- saturated montmorillonite suspensions resulted in enhanced sedimentation of the Ca-montmorillonite but not the Mg-montmorillonite. The absence of marked flocculation of Mg-saturated montmorillonite may be caused by a greater collapse upon adsorption of the polymer on the clay surface com-

pared to that of the Ca system. The result would be an adsorbed polymer with a greater proportion of segments existing as trains and fewer as loops and tails.

Addition of salt to clay suspensions after the introduction of the T500 dextran resulted in the removal of the polymer effect which was evident when no salt had been added. The control, where no dextran had been added, sedimented at a similar rate as the suspensions with dextran plus 0.01 M CaCl$_2$. The salt either disrupted polymer-clay interactions or prevented the polymer effect. The coagulation of the dextran-clay complexes had a greater effect on sedimentation than the addition of polymer with no additional salt.

Coagulation of the Ca-montmorillonite clay suspension by addition of salt prior to addition of the polymer resulted in enhanced sedimentation at all polymer concentrations. The approach of clay particles through the action of the coagulant facilitated particle bridging by the T500 polymer and therefore flocculation was possible. However, in the case of the Mg-montmorillonite coagulation of the suspension did not result in enhanced flocculation relative to the control. This suggests that the polymer was adsorbed with a greater proportion of collapsed segments in the Mg-montmorillonite suspension. The adsorbed polymer was not able to flocculate the coagulated suspension, probably due to fewer and shorter loops and tails extending away from the clay surface.

ACKNOWLEDGMENTS

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