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DETERMINATION OF FLOCCULATION AND ADSORPTION- DESORPTION CHARACTERISTICS OF Na-FELDSPAR CONCENTRATE IN THE PRESENCE OF DIFFERENT POLYMERS

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Feldspars are among the most important mineral groups on the earth. Although the group of feldspar has 20 minerals, only nine of them are well known. These minerals make up the largest percentage of minerals in nature. Esan albite beneficiation plant faces serious disposal problems in dewatering the minus 20 μm albite concentrate. Flocculation studies conducted with albite in the presence of various charged and nonionic polymers indicate that nonionic polymers exhibit the best performance. Electrokinetic studies on pure albite shows that it is negatively charged throughout the entire pH and the isoelectric point of the mineral is below pH 2. Adsorption of the nonionic polymer (N-300) is found to increase with increasing pH and concentration of the polymer. A hydrogen bonding mechanism between the polymer carbonyl (-C=O) or the amide (-NH₂) groups and the surface oxygen species is proposed to be responsible for the adsorption of nonionic polymer onto albite.

key words: albite, flocculation, zeta potential, adsorption, nonionic polymer

INTRODUCTION

For several decades, inorganic electrolytes, natural polymers and synthetic, high molecular weight polymers have been used as coagulants or flocculants for clarification of effluents, acid mine drainage and treatment of paper, textile, sugar and

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other industrial waste water processing applications. Research has shown that interactions between the functional groups on particulate surfaces and those on polymers are responsible for adsorption and subsequent flocculation, and can be appropriately controlled by tailoring polymers with specific functional groups suitable for particular applications. The effectiveness of a polymer for a given flocculation system depends on several parameters, e.g. the polymer dosage, amount adsorbed on the solid surfaces, molecular weight of the polymer, particle size, shape, porosity, ionic strength of the system, electrokinetic properties and more importantly, conformation of the polymer at the solid-liquid interface. According to desorption tests, no significant change was observed for the albite-non-ionic polymer system.

Polymer molecules can be made to selectively adsorb onto particles by a) adjusting the chemical composition of the suspending media, b) introducing into polymer, active functional groups that will form complexes or salts with the metal atoms on the surfaces of the desired minerals, and c) using depressants, such as sodium silicate, that would adsorb on the undesired mineral surface, thereby preventing adsorption of the polymers by using activators that induce adsorption of polymers on desired minerals (Akers, 1975). Adsorption of polymers on solid surfaces is dependent on polymer properties, such as molecular weight and configuration, distribution of functional groups, solid properties, such as surface charge and oxidation states, and solution properties such as ionic strength, temperature and solvent power for the polymer. The various forces responsible for the adsorption of the polymers on the mineral surfaces result primarily from three types of bonding, namely electrostatic, hydrogen and covalent bonding. The solution-chemistry conditions, the rheology of the solid suspension and the polymer properties dictate the nature of interactions (Chou and Wollast. 1985).

Often several mechanisms may be operating at the same time controlling the adsorption/flocculation behaviour system. Long chained polymers can be adsorbed onto the grains from several locations along the chain in a single polymer molecule and can also be adsorbed onto more than one grain. In such cases, the grains are bridged by the adsorbed polymers. This mechanism plays an important role in a number of applications. In order for the bridging flocculation to form, the polymer should be of large molecular weight, and a large part of the polymer chain should be adsorbed without interaction with the grain surface. Moreover, the adsorbed amount should not be high, so that a larger part of the grain surface remains free (Çelik et al., 1991). Only in such a case, the free part of the grain surface and extended part of the polymer that is adsorbed onto another grain come into mutual contact. When over-adsorption of a polymer occurs, there remains no free surface for bridging to take place, and bridging is further prevented because of steric impulsion. Therefore, in order for bridging flocculation to occur, there is an optimum polymer dosage, the value of which depends generally on grain concentration.

Albite ($\text{NaAlSi}_3\text{O}_8$), one of the minerals in the plagioclase feldspars series, is characterized by a lustre appearance with white and grey colours, less frequently greenish and yellowish (Potter, 1985). Albite is usually found together with quartz, mica, and sometimes iron oxides, rutile, and tourmaline. Feldspars is principally used in the manufacture of glass and ceramics as a flux or source of alumina. Beneficiation of albite is primarily accomplished by a number of separation methods including magnetic, electrostatic and flotation. Although the concentration scheme is usually dependent on the quality of the end product, flotation is almost invariably the most popular beneficiation method. While anionic collectors such as oleic acid is used to collect colored impurities, e.g., iron oxides and rutile, cationic collectors are used to float feldspar at pH 2 while depressing quartz with HF (Hill et al., 1969; Warren, Kitchener, 1972). Important reserves of feldspar ores are available in western Turkey. Turkish feldspars, especially albite, contain relatively high levels of TiO_2 and Fe_2O_3 as their main impurities. Mineralogical investigations on the main ores show mainly albite and quartz, mica and microcline with secondary minerals such as anatase, rutile, ilmenite, sphene, magnetite, hematite, limonite, epidote, grona, biotite, phlogopite, pyrite and zirconium minerals (Kangal et.al.1998, 2001)The largest feldspar reserves of Turkey are located in the Çine-Milas district. Çine-Milas has significant quantities of albite with much as 400 million tons of reserves represented with average qualities (Kangal et al. 2001) Esan-Eczacıbaşı Milas Albite concentrator produces glass and ceramic quality albite with a production capacity of 150,000 Mg/g. The ore is reduced to -0.5 mm in size and a fatty acid type collector is used to float the color impurities at pH 8-10. The minus 0.5 mm flotation concentrate is then passed through a combination of hydrocyclone/spiral classifier to obtain three products, i.e. 0.5x0.1 mm,0.1x0.02 mm and -0.02 mm. The latter two products are fed into the settling cones where the solids is densified with the aid of flocculants followed by dewatering by di sc filters. The overtlow is recirculated back to the plant after it is clarified.

Investigations in the plant have revealed that the flocculation regime used in the dewatering circuit is not appropriate and thus leads to slow settling rates and in turn to inefficiencies in the plant operation. No literature of relevance was found on the subject of albite flocculation. It is therefore the objective of this study to test the performance of various acrylamide based polymers in settling the minus 20 μm albite concentrate and find out the optimum operating conditions. Towards this aim, a systematic study has been also initiated to understand the way albite interacts with nonionic polymer (Kursun, 2000).

EXPERIMENTAL

MATERIALS

The sample used in the flocculation tests was collected from the fine (-20 μm) settling cone product in the Esan-Eczacıbaşı Albite concentrator. The chemical composition of the sample is presented in Table 1. Ultrapure albite crystals received from the same deposit was ground in an agate mortar to obtain a sample of -74 μm in size. This sample was used for both adsorption and zeta potential measurements. While experiments associated with pure albite were conducted in distilled water, the actual plant water was used in the flocculation tests.

Table 1. Analysis of -20 μm albite concentrate

Item	Weight %
SiO ₂	71.9
Al ₂ O ₃	17.6
Na ₂ O	9.8
K ₂ O	0.3
TiO ₂	0.07
Fe ₂ O ₃	0.14
CaO	0.10
MgO	0.20

The anionic (A-120, A-95, and A-130), cationic (C521), and nonionic (N-I00, N-200 and N-300) flocculants were all received from Cytec Industries Inc. and specified to have molecular weights of $3\text{-}15 \times 10^6$, $3\text{-}4 \times 10^5$, and $3\text{-}4 \times 10^6$, respectively. Tannic acid, NaCl, NaOH and HCl were all Fluka made certified chemicals.

METHODS

The flocculation tests were performed in a one dm^3 graduated cylinder. The settling tests, apart from studies on solids concentration, were done by adding 200 g of -20 μm material into the cylinder, mixed thoroughly and falling of the interface height was recorded as a function of time. Zeta potential measurements were conducted by means of Zeta Meter 3.0 which is equipped with a microprocessor unit capable of directly measuring the average zeta potential and its standard deviation. 100 mg - 20 μm pure albite was added into 100 ml of distilled water and conditioned for 10 min. The suspension was kept stationary for 3 min and the average of ten particles was taken as the zeta potential. Details of the measurement procedure is given elsewhere (Celik and Yasar, 1995; Kursun et al., 1996). Adsorption tests were carried out in 20 ml-glass scintillation vials. A sample of 1 g pure albite was added to 15 ml of polymer solution

of desired concentration. The supernatant was subjected to polymer analysis described below. Polymer analysis was made by a nephelometric method developed by Attia and Rubio (1975) for low concentrations of polyethylene oxide and polyacrylamide flocculants. A series of nonionic polymer solutions of known concentrations (5 ml) in the range of 0-3 parts/ 10^6 (ppm) were treated with 40 ml of 0.1 M NaCl solution and 5 ml of 0.1% tannic acid solution in 50 ml volumetric flask. The mixture was shaken for 1 h and the turbidity of the resultant solution was measured by a Shimadzu UV/Vis spectrophotometer at 625 nm wavelength. The linear portion of the calibration curve in the range of 0.5-2.5 mg/kg was used for calculating the residual polymer concentrations.

RESULTS AND DISCUSSION

FLOCCULATION STUDIES

Figure 1 presents the height of the interface in the absence of polymer as a function of time at different pulp densities as defined by percent solids by weight. The plant water was used as the medium at its natural pH. It is apparent that as the pulp density increases the settling rate, as defined by the initial slope in the sedimentation curve, and also the interface height decrease. Despite higher sedimentation rates at low pulp densities, considering both conditions at the plant and economic capacity of the operation, 20 % pulp density was considered suitable for performing the subsequent flocculation tests. It is also evident in Figure 1 that most of the sedimentation occurs during the first 15 min of the settling for all pulp densities. However, it is interesting to note that, in the absence of any polymer addition, the upper portion of the suspension appeared to be rather hazy indicating that under natural conditions a long retention time is required to achieve a clear supernatant.

Figures 2-5 illustrate the effect of various type of flocculants on the elapsed settling time as determined by the interface height. The amount of polymer is 5 g/Mg for all polymers used. Compared to the anionic and cationic polymers, the nonionic polymers yields the best settling condition. The settling rate further increases with increasing the degree of nonionicity, Figures 2-5 also further show that the settling time is drastically reduced in the presence of polymer. Settling is almost over in less than five min. The same improvement was observed in the supernatant as the cloudiness was substantially reduced in the order of effectiveness shown in Figures 2. A similar enhancement was also noted upon increasing the amount of polymer up to a certain concentration above which destabilisation of the suspension was observed as reported in most flocculation studies.

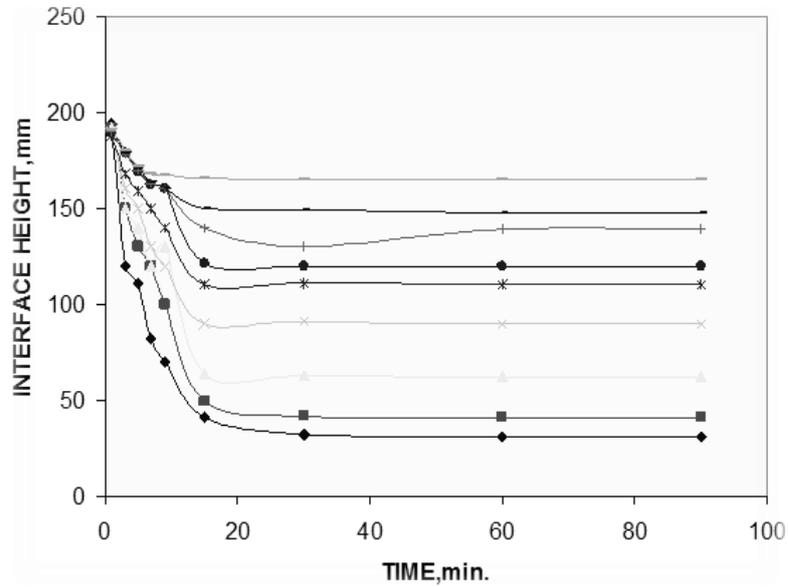


Fig. 1. Settling behaviour of $-20\mu\text{m}$ albite concentrate against time in the absence of flocculant at different pulp densities

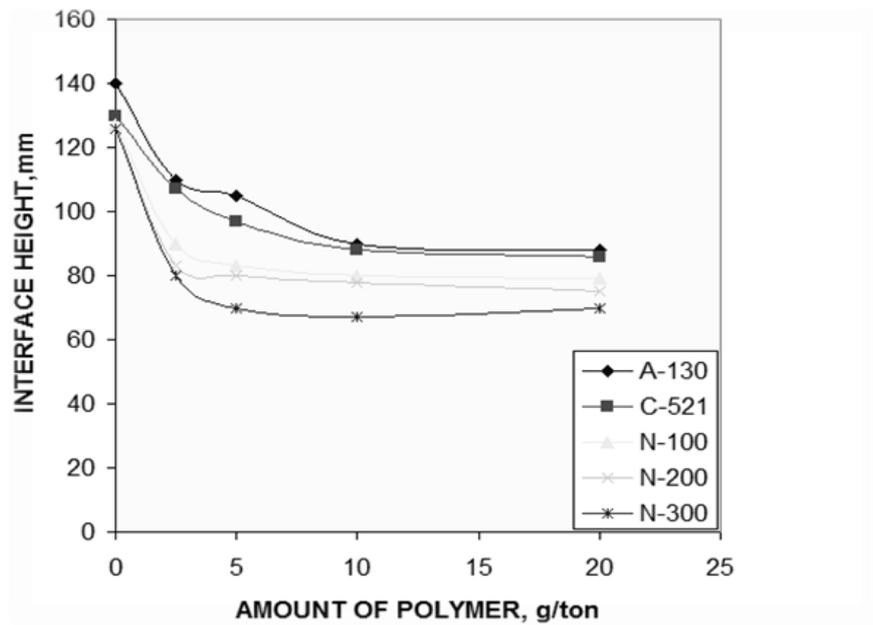


Fig. 2. Variation of interface height with the amount of polymer for $20\mu\text{m}$ albite concentrate up to 5 min of settling time

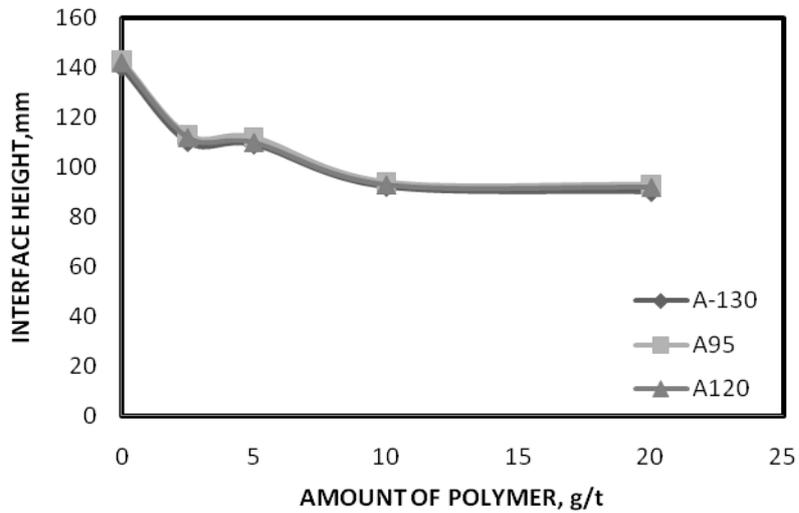


Fig. 3. Variation of interface height with the amount of anionic polymer for 20 µm albite concentrate up on 5 min of settling time

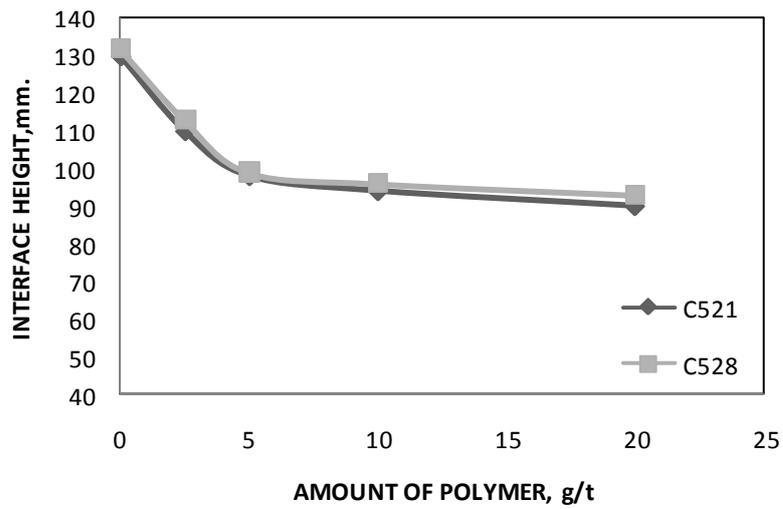


Fig. 4. Variation of interface height with the amount of cationic polymer for 20 µm albite concentrate up on 5 min of settling time

Figure 5 exhibits the settling behaviour of albite in the absence and presence of polymer as a function of pH. It is clear that, due to scattering, no significant effect of pH is apparent both in the presence and absence of polymer.

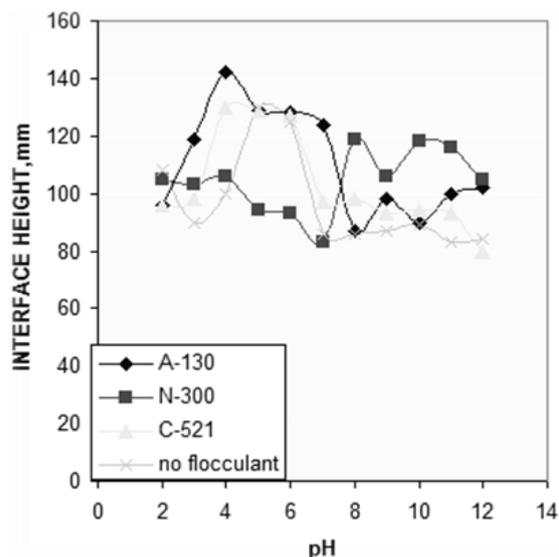


Fig. 5. Settling behaviour of -20 μm albite concentrate versus pH with different flocculant (settling time 5 min, polymer concentration 5 g/Mg)

EQUILIBRATION OF ALBITE IN WATER

The plagioclase feldspars form a complete solid solution series from pure albite ($\text{NaAlSi}_3\text{O}_8$) to pure anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$). Considerable potassium may be present toward the albite end of the series (Hurlbut, 1971). Therefore, it is more likely that albite should be written as $(\text{Na},\text{K})\text{AlSi}_3\text{O}_8$. The dissolution behaviour of albite has been systematically studied by Chou and Wollast (1985). Helgeson et al. (1984) suggest that the rate of hydrolysis of feldspar is highly pH dependent and results from the following reactions:

at low pH < 2.9



at intermediate pH (2.9-8.0)



at high pH > 8.0



Stumm and Morgan (1981), based on coordination chemistry, suggest that the factorial orders on H^+ and ligands given in Eq. 3 are compatible with a direct dependence on the degree of surface protonation or on the concentration of ligand surface complexes. Stumm et al. (1983) also propose the existence of charged

complexes at mineral surfaces. In both Eqs. 1 and 2 hydronium (H_3O^+) ions can replace Na^+ or K^+ Wikby (1972) has demonstrated that the alkali ions are replaced by H^+ rather than H_3O^+ . This hypothesis is further supported by the fact that the volume occupied by alkali ions in albite is rather small compared to the large H_3O^+ (Chou, Wollast, 1985). The most important factors affecting the rate of dissolution of albite are pH and the concentration of dissolved Al. The rate dependence on pH exhibited remarkable similarities to the solubility curves of Al compounds and also Al showed the largest inhibiting effect on the dissolution rate. Chou and Wollast (1985) thus suggest the formation of surface complexes involving both H^+ and Al.

DISSOLUTION BEHAVIOR OF ALBITE

Figure 6 presents the dissolution kinetics of albite in water as a function time at initial pH values of 3, 11, and natural. The natural pH of albite was found to be 8.1. When the suspension is mixed with distilled water of about pH 6, it takes approximately 15 min for albite mineral to reach its equilibrium pH of 8.1. When the initial pH is adjusted to 3 or 11 it takes about 15 min to approach one pH unit to the natural pH but several additional hours to reach its equilibrium pH. In other words, albite suspensions are buffered relatively strongly in both acidic and alkali media. This feature becomes important in the interpretation of adsorption results.

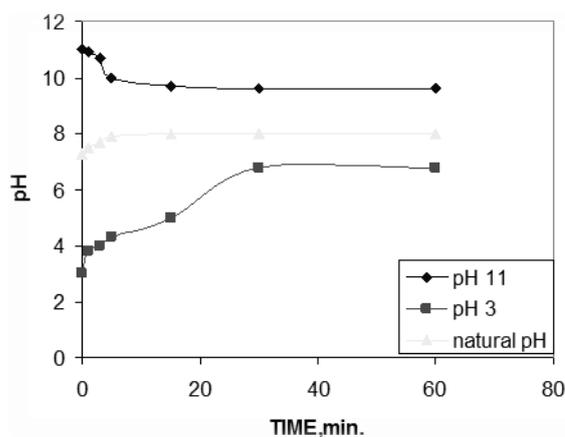


Fig. 6. Dissolution kinetics of pure albite in water at different initial pH values

ELECTROKINETIC BEHAVIOR OF ALBITE

Albite, as most other silicate minerals, exhibit negative zeta potentials throughout the practical pH range of 2 to 12. The zero point charge (zpc) of albite is below pH 2,

as shown in Figure 7. This is in line with the zpc of many silicate minerals including quartz (Fuerstenau and Palmer, 1976). However, no electrokinetic data on albite was found in the literature.

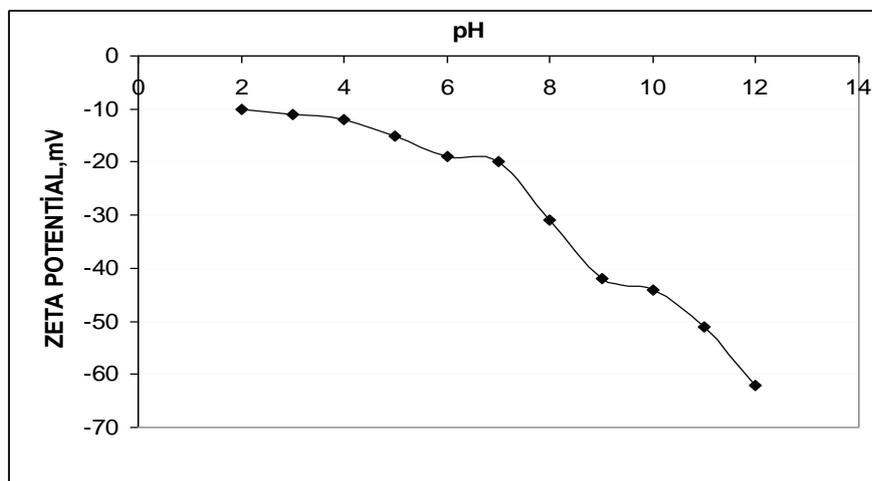


Fig. 7. Zeta potential of albite as a function of pH in water

The zeta potential of albite as a function of N-300 nonionic polymer concentration is given in Figure 8. The zeta potential gradually decreases with increasing N-300 concentration, i.e. it becomes less negative. The albite suspension was found to flocculate at 1 mg/kg N-300 concentration and measurements became increasingly difficult above this value.

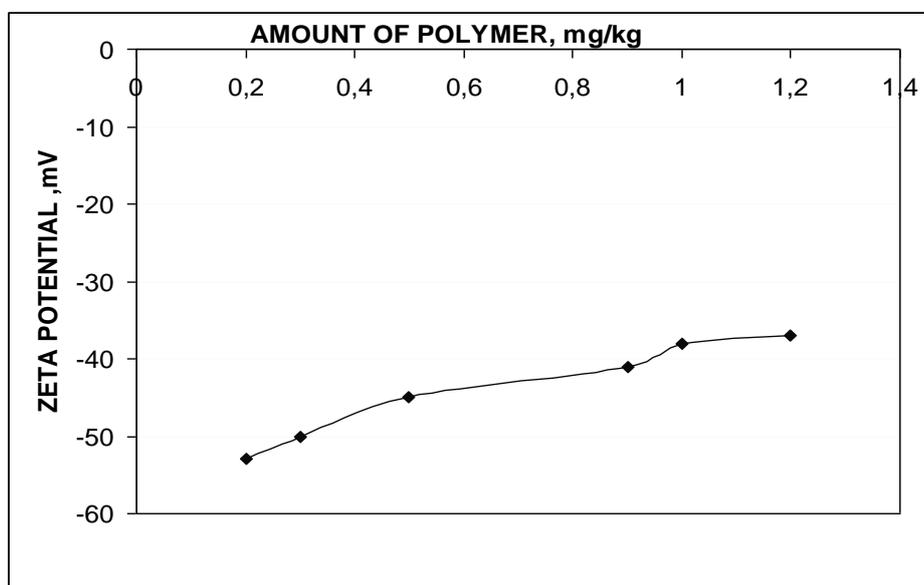


Fig. 8. Variation of zeta potential with N-300 concentration

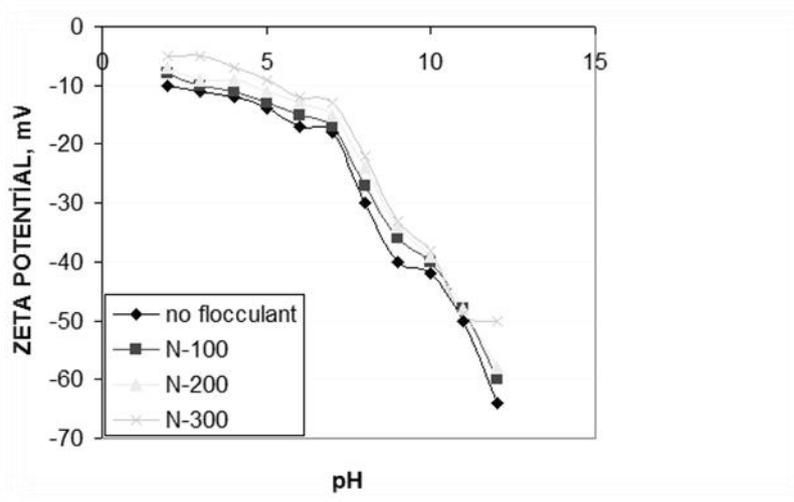


Fig. 9. Zeta potential versus pH profile for albite with 1 mg/kg and without polymer addition

The dependence of pH on zeta potential of albite in the presence and absence of different nonionic polymers is presented in Figure 9. It is clear that the addition of polymer makes the surface of albite less negative at all pH values with N-300 being the most effective. This indicates that as the nonionicity increases more of negative charges on the albite surface are tied up with the polymer.

ADSORPTION OF POLYMER ON ALBITE

Adsorption is a rate dependent phenomenon. Polymers, unlike low molecular weight compounds, are made up of thousands of units. Adsorption kinetics is therefore not only dependent on the conducted at surface properties of minerals but also on those of chemicals as well. For a polymer to adsorb it is sufficient to attach its one or more of segments to the solid. The dependence of adsorption on conditioning time is given in Figure 10 at natural pH of albite. The equilibrium is practically attained after about one hour and this is maintained with only a slight drift. One hour of equilibrium time was used in the subsequent adsorption experiments.

Flocculation and adsorption are both dependent on the solids concentration. In Figure 1, flocculation was shown to drastically change with the amounts of solid in solution. Similarly, adsorption also changes with solids concentration, as shown in Figure 11. It is seen that adsorption decreases with increasing the solids concentration and the curve flattens out at above about $5\text{g}/100\text{ dm}^3$. For the sake of convenience all adsorption tests were thus conducted at $1\text{g}/15\text{ ml}$ solids concentration. It should be noted that flocculation is generally preceded by polymer uptake but not all polymer

adsorption leads to flocculation (Yarar, 1982). Polymer conformation whether straight or coiled plays a significant role in the magnitude of final flocculation. High molecular weight polymers are more akin to coiling particularly under high salinity and temperature conditions (Celik et al., 1991).

Since pH was pointed out as an important parameter in the dissolution of albite, the dependence of adsorption of N-300 was studied as a function of pH. This is shown in Figure 12. Evidently, the adsorption density increases with increasing pH and it reaches a constant value above pH 9 where it becomes difficult to adjust pH. Adjusting the initial pH of the solution to pH 12 resulted in a pH value of 9.3. High adsorption density at neutral and high pH correlates well with the flocculation and settling tests discussed previously.

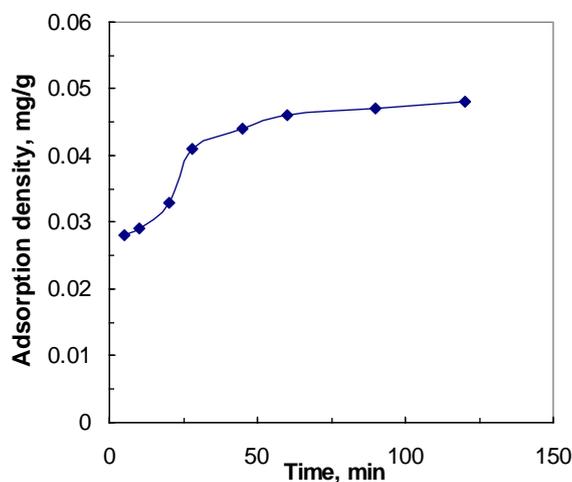


Fig. 10. Dependence of adsorption of 10 mg/kg N-300 on albite against conditioning time at natural pH

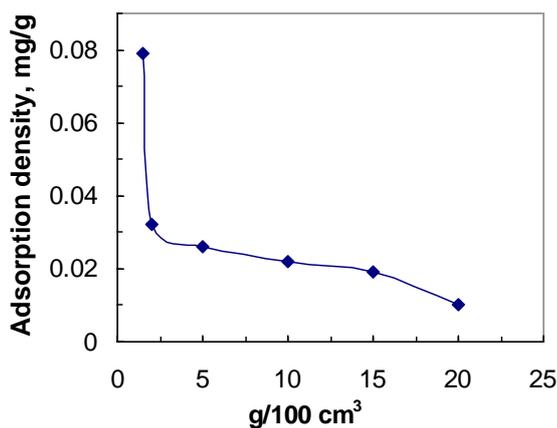


Fig. 11. Adsorption density as a function of solid concentration at 10 mg/kg N-300 concentration

Adsorption isotherm of N-300 onto albite is presented in Figure 13 where the adsorption density is plotted against the residual polymer concentration. Adsorption is found to increase with an increase in polymer concentration and reaches a plateau value at about 10 mg/kg N-300 concentration. The adsorption isotherms appears to be of a typical Langmuir type.

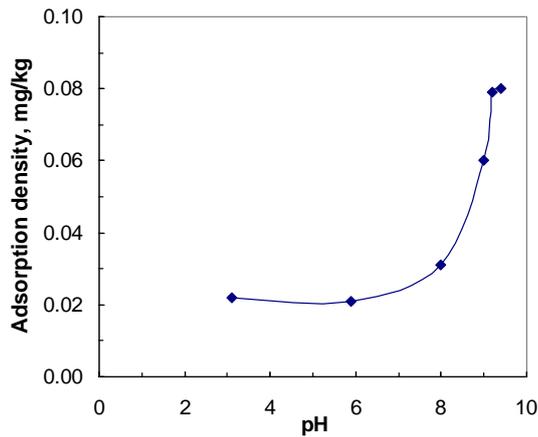


Fig. 12. Effect of pH on the adsorption of 10 mg/kg N-300 onto albite

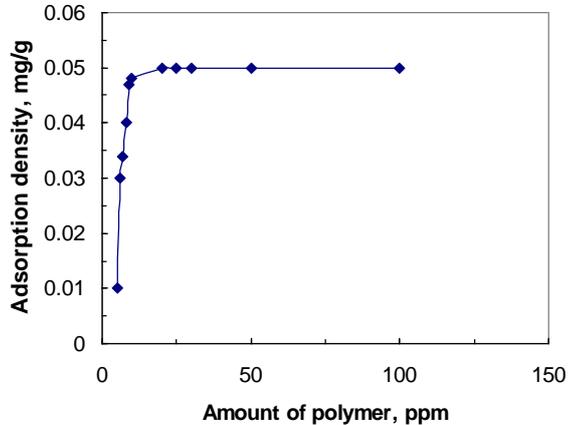


Fig. 13. Adsorption of N-300 onto albite at natural pH

DESORPTION EXPERIMENTS

Desorption tests were carried out by replacing the removed portion of the supernatant (2/4 ml) with a supernatant solution of the same salinity and adjusted pH to match the original supernatant. The desorption cycle was continued until the

polymer concentration was below detection limit of the polymer analyses technique. As a result of the desorption tests, no significant change was observed for the albite-nonionic polymer system.

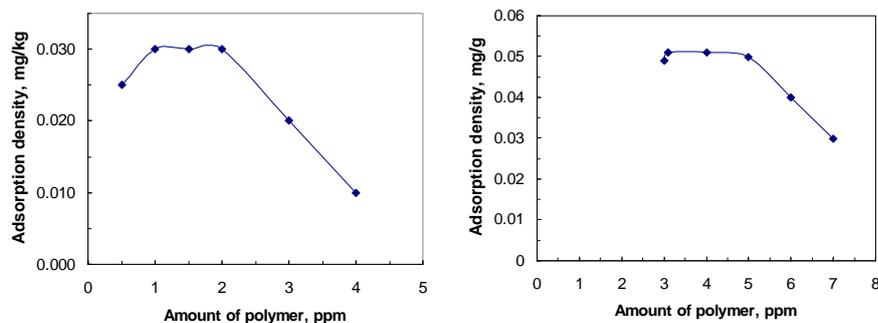


Fig. 14. Desorption of 5 ppm polymer concentration Fig. 15. Desorption of 10 ppm polymer concentration

ADSORPTION MECHANISM OF NONIONIC POLYMER ON ALBITE

Several mechanisms of polymer adsorption have been suggested including electrostatic interaction, hydrogen bonding, chemical interactions and hydrophobic bonding (Slater and Kitchener, 1966). Since the polymer under discussion is nonionic in nature, the electrostatic mechanism can be ruled out. Hydrophobic bonding requires that the surface of mineral be modified by a non-polymeric surfactant and thus can be excluded as well. The latter two mechanisms are therefore possible candidates. Hydrogen bonding occurs through binding of hydrogen to an electronegative element such as F, N, S and O. This is a widely accepted mechanism for freshly ground natural silica (Griot and Kitchener, 1965) and clays (Hollander et al., 1981) with nonionic polymers. Hydrogen bonding can occur between the polymer carbonyl ($-C=O$) or the amide ($-NH_2$) groups and the hydrogen bonding with surface oxygen species. The sharing of the hydrogen bonding between the two electronegative oxygen species induces the adsorption. Flocculation then follows by interparticle bridging if the electrostatic repulsion between the particles is not strong. Since albite is a sodium form of silicate mineral and the polymer is nonionic, it appears that hydrogen bonding is the only plausible mechanism in the system. As shown in Equations 1 through 3, albite acquires a positive charge below pH 2.9 and thus is not as much amenable to adsorption of N-300. This is clearly seen in Figure 12 where adsorption remains at its lowest level above pH 3, adsorption progressively increases due to formation of silanol groups and reaches its highest level above pH 8 where additional negatively charged surface complexes form. The decrease in the zeta potential of surface with the addition of polymer shown in Figures 8 and 9 is in

agreement with the above explanation. It appears that positive sites are depleted upon addition of the polymer.

CONCLUSIONS

Experimental studies are apparent that as the pulp density increases the settling rate, as defined by the initial slope in the sedimentation curve, and also the interface height decrease. Despite higher sedimentation rates at low pulp densities, considering both conditions at the plant and economic capacity of the operation, 20 % pulp density was considered suitable for performing the subsequent flocculation tests. Flocculation studies conducted with albite in the presence of various charged and nonionic polymers indicate that nonionic polymers exhibit the best performance. Increasing the degree of nonionicity improves the degree of settling. Electrokinetic studies conducted with pure albite shows that albite is negatively charged throughout the entire pH and the isoelectric point of the mineral is below pH. Adsorption of the nonionic polymer is found to increase with increasing the pH and concentration of the polymer. A hydrogen bonding mechanism between the polymer carbonyl (-C=O) or the amide (-NH₂) groups and the surface oxygen species is proposed to be responsible for the adsorption nonionic polymer onto albite. According to desorption tests, no significant change was observed in the system.

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Zakład Wzbogacania Albitu Esan boryka się z poważnym problemem, jakim jest odwodnienie koncentratu zawierającego ziarna albitu poniżej 20 µm. Badania nad flokulacją zawiesiny albitu z udziałem różnych polielektrolitów i niejonowych polimerów wykazały, że niejonowe polimery wykazują najlepsze działanie flokulujące. Badania elektroforetyczne przeprowadzono na czystym albitcie wykazały, że cząstki tego minerału są ujemnie naładowane w całym badanym zakresie pH, a punkt izoelektryczny jest położony poniżej pH =2. Adsorpcja niejonowego polimeru N-300 wzrastała wraz ze wzrostem pH oraz wraz ze wzrostem stężenia polimeru. Zaproponowano mechanizm wiązania oparty na wiązaniu

wodorowym między grupami -C=O lub -NH_4 polimeru i tlenowymi grupami na powierzchni ziaren albitu. Mechanizm ten jest odpowiedzialny za adsorpcję niejonowego polimeru na albitcie.

słowa kluczowe: albit, flokulacja, potencjał dzeta, adsorpcja, polimery niejonowe