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DEWATERING OF BARITE CLAY WASTEWATER BY INORGANIC COAGULANTS AND CO-POLYMER FLOCCULANTS

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Abstract: This work is focused on dewatering of barite-containing clay waste from a barite beneficiation plant by coagulation and flocculation to enhance the dewatering rate and recycling of process water. A flocculation performance of co-polymers (Superfloc A-130 and A-100) was compared with coagulants ($\text{Al}_2\text{SO}_4 \cdot 18\text{H}_2\text{O}$, CaCl_2 and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$). The best results were obtained with an anionic co-polymer, i.e. Superfloc A-130. The settling rate increased 635-fold in the case of using anionic co-polymer Superfloc A-130 in comparison with the natural settling rate of the same wastewater. In laboratory tests, an optimum thickener design was developed for the barite clay wastewater. In this paper, sedimentation studies on the barite clay wastes are presented for the first time, both as the laboratory studies and industrial applications.

Keywords: *dewatering, sedimentation, inorganic coagulant, co-polymer flocculants, wastewater, barite*

Introduction

Clarifications of municipal and industrial wastewaters by solid-liquid separation techniques as well as removal of suspended particles are challenging problems for plants and people because of growing environmental consciousness. On the other hand, there are very limited number of studies reporting in the literature on flocculation and settling properties of mineral industry wastewaters containing high specific gravity mineral such as manganese and chromite. Solid-liquid separation or dewatering processes generally involve unique set of challenges because of the presence of colloidal particles with different sizes, shapes, and specific gravities as well as solution chemistry of their environments. Currently, the most common method for solid-liquid separation is the use of thickeners, in which the particulates settle out to form a bed, leaving a clear supernatant, which can be recycled back to the plant. The settling rate during solid-liquid separation within the thickener is important, so

that this influences the size of the required thickener and the rate at which water can be recycled back to the plant (Kominek and Lash, 1979; Nasser and James, 2006; Cengiz, et al., 2009; Deniz, 2013).

In the mineral industry, many of these wastes contain minerals such as clay, which due to the small particle sizes and charge properties are difficult in order to settling and consolidation. The conventional method used to colloidal particles (e.g. kaolinite) involves inorganic salts-aided coagulation or high molecular weight polymer-aided flocculation and subsequent sedimentation in thickeners (Naser and James, 2006).

The addition of coagulants is used to reduce the overall negative surface charge that surrounds each particle. The coagulants enable the weak van der Waals forces to dominate, which results in attraction between particles causing the colloidal clay-rich suspension to settle out. This is a result of the coagulants neutralizing the surface charge of clay. Two different coagulation mechanisms are used in order to efficiently settle out slimes: inorganic salts and poly-electrolyte coagulation. The most common electrolyte coagulants are aluminum, ferric and calcium salts (Fowler and Morkel, 2010).

Whereas inorganic salts promote coagulation by charge neutralization and double-layer compression, the organic polymers promote solid-liquid separation by the bridging mechanism, sometimes in combination with charge patch effects. Flocculation of fine particles may occur by polymer bridging, charge neutralization, polymer-particle surface complex formation and depletion flocculation, or by a combination of these mechanisms (Gregory, 1985; Ersoy, 2011).

Nowadays, the flocculants used for the flocculation process are non-ionic, anionic and cationic polymers. The non-ionic and anionic polymers cause to form big, fast sedimentation and fairly compact flocs. However, it is preferable to use higher molecular weight anionic or non-ionic polymers over cationic flocculants for the sedimentation process. It is known that the non-ionic polymers are usually used in acidic slurries and the high molecular weight anionic polymers are used in alkali slurries (Sabah and Yeşilkaya, 2000).

The several studies have shown that high molecular weight anionic polymers (e.g. polyacrylamide) are commonly used in settling of negatively charged particulate clay (Scheiner and Wilmen, 1987; Richardson and Connely, 1988; Singh et al., 1999; Hogg, 2000; Abd-WI Rahman, 2000; Patience et al., 2003; Ozkan, 2003; Tao et al., 2010; Kursun, 2010; Sabah and Aciksoz, 2012; Kurama and Karaguzel, 2012). In that case, the polymer bridging mechanism is of primary importance, whereas charge neutralization will be of secondary or little importance (Patience et al., 2003). The use of high molecular weight anionic polyacrylamide in flocculating negatively charged particles have the advantage of being more effective than cationic polymers by increasing the settling rate and producing a distinct sediment structure, and in addition, re-stabilization of clay particles by excessive polymer adsorption driven by strong electrostatic attraction may be avoided. The electrostatic repulsion between the clay particles and the anionic polyacrylamide allows only limited polymer adsorption. On

the other hand, the polymer molecule expansion arising from the charge repulsion produces loops and tails, which lead to formation of large open structure flocs. This effect has been seen to be effective in the flocculating negatively charged clay dispersions (Somasundaran and Moudgil, 1988).

A barite beneficiation plant belonging to Baser Mining Co. (Sarkikaragac/Isparta) in Turkey has a capacity of 40 Mg/h. The barite slime is concentrated with multi gravity separator (MGS) and shaking tables to increase its quality. Six settling ponds (Fig. 1) are used in the beneficiation plant to separate the slime from water by natural sedimentation (Deniz, 2000). The barite-containing clay wastewater in the ponds does not contain appreciable amount of chemical pollutant and the amount of clean water, which is needed by the plant. However, it cannot be cleaned due to very slow settling rate of solid ingredients in the settling ponds.

In this study, dewatering of barite clay wastewaters belonging to Baser Mining Co. from Isparta (Turkey) was investigated using the sedimentation technique. This technique was found to be a suitable solution for fresh water for barite beneficiation plant.



Fig. 1. View of settling ponds sand tailing area in the Başer barite beneficiation plant

Material and methods

Material

During beneficiation of Isparta (Turkey) barite ores, a fine particle fraction ($-106\ \mu\text{m}$) is separated by cyclones as a dilute (7% solids by weight) aqueous slurry. The solids are a mixture of clay minerals, barite and other fine divided minerals, such as: orthoclase, calcite and quartz. This slurry is pumped into large impoundment areas for the natural settling. Due to the colloidal nature and slow consolidation characteristics of such clays, a large storage area is required. In order to place these clays, together

with the sand tailings (from a jigging process), back into the original mine cut without building aboveground dams, the clays would have to be dewatered to over 50% solids by weight. Unfortunately, decades are needed for clays to dewater to such high solid level in the conventional settling areas. Thus, a huge volume of water and large areas of land are tied-up by impounding these clays for a long period.

Methods

The chemical composition of particulate tailings was analyzed by an X-ray fluorescence, and a particle size distribution was determined using Malvern Mastersizer Particle Size Analyzer (Mastersizer 2000). The mineral composition was determined by an X-ray diffraction (XRD). Electrokinetic (zeta potential) measurements were obtained using a Zeta-Meter 3.0 device. The pH was measured with a lab pH-meter.

The flocculation and coagulation experiments were carried out using a mechanical mixer with a speed control. The tests were conducted on original barite clay wastewater samples. For each test, a 500 cm³ of original slime wastewater containing 7% wt./wt. solids was placed in a 600 cm³ glass measuring cylinder and stirred for 2 minutes at a rotational speed of 500 rpm to ensure homogeneous dispersion. The required amount of coagulant and polymer solution was added continuously into the suspension during the stirring, which was stopped after the optimum mixing time.

A base case natural settling test on the samples was performed in order to highlight the problem areas of material type and depths. The coagulants were Fe³⁺, Ca²⁺ and Al³⁺ salts. The inorganic coagulants solution, distilled water and 10 g of dry sample were mixed together and then stirred at 500 rpm for 30 min. The anionic polyacrylamide flocculants, Superfloc A-100 and A-130, an acrylamide–acrylic acid random copolymer were obtained courtesy of Cytec Industries, USA. Superfloc A-100 and A-130 have a molecular weight of 12·10⁶ g/mol and the content of acrylic acid monomer of approximately 30% and 35%, respectively. The addition of 0.5% by weight polymer solution was made up by adding a dry polymer powder to the vortex of a stirred solution followed by high speed mixing for 1 h. The solution was mixed for 12 h. The solution was then rested for a further 12 h prior to use. The suspensions were diluted in tap water.

After addition of the coagulant and flocculant, the suspension was mixed for 20 sec, and then the prepared clay suspensions were transferred to the 600 cm³ measuring cylinders (60 mm in diameter). The height of the slurry and water interface was recorded as a function of time and used to calculate the settling rates of coagulated and flocculated suspension.

Results and Discussion

Chemical and mineralogical analysis

The chemical compositions of the associated minerals in the barite tailing were determined by an XRF method as shown in Table 1. As expected, the tailing is mainly composed of SiO_2 , Al_2O_3 and CaO .

Table 1. Chemical compositions of barite tailing

Components	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	BaSO_4	LOI*
Amount, %	60.17	12.84	6.66	3.85	1.06	9.79	1.44	3.41	5.05	0.78

*LOI: Loss-on-ignition

The XRD analysis of the barite tailings indicated that the main minerals are chlorite, serisite, barite, orthoclase, calcite and quartz (Fig. 2). According to the chemical analysis data and the XRD results, the original tailings, theoretically, contain approximately 45% clay minerals (serisite, chlorite), 14% calcite, 5% orthoclase and 6% quartz, with the percentage of barite being about 5%.

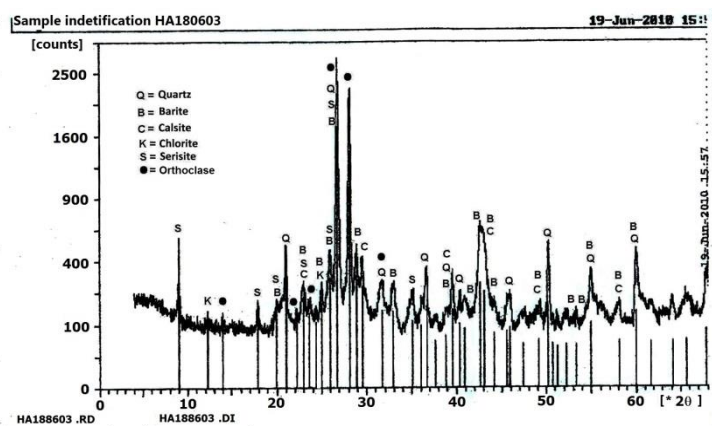


Fig. 2. XRD patterns of barite tailing elements

Particle size distribution

The average particle size determined from the Gaudin- Schuhman type of plot was $9.47 \mu\text{m}$, and the percentage of slime size ($<20 \mu\text{m}$) constituted 74% of the overall material, which indicates the presence of very high specific surfaces area. Based on classification of Wentworth (1922), although the percent of particles in clay size accounts for 25% ($<4 \mu\text{m}$), the percentage of particles in the silt size is 72% ($4\text{--}95 \mu\text{m}$). Those particles in the sand size were 3% ($>95 \mu\text{m}$).

Zeta potential tests

Because of the high ionic strength (or high conductivity) of barite tailing, the zeta potential of particles from the wastewater was not measured directly. Instead of this, the original tailing was dried at first, and then the test solution was prepared with distilled water and the solid particles of 106 μm in size. The zeta potential of this solution was measured at different pH values. Figure 3 presents the effect of pH on the zeta potential of barite tailings. As apparent, the tailings exhibit negative charge at all practical pH values with no apparent zero point of charge. The highest zeta potential (28.4 mV) measured in the neutral pH (8) indicates that the suspension is quite stable. The pH of solution was adjusted by adding HCl and NaOH. The relatively moderate zeta potential values obtained particularly at high pH are in a good agreement with the presented data by Tunçan (1995) for different types of clay minerals. It indicates that there is a higher percentage of chlorite and sericite than kaolinite and illite in the barite plant tailings.

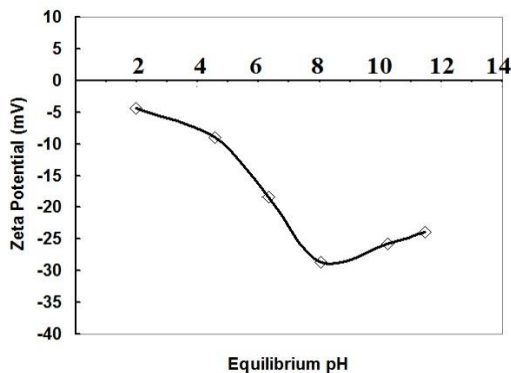


Fig. 3. Zeta potential-pH profile for the particles from barite tailing

Sedimentation tests

The coagulant tests were performed in the presence of three types of inorganic coagulants at different dosages (Figs. 4-6). It is found that aluminium sulphate ($\text{Al}_2\text{SO}_4 \cdot 18\text{H}_2\text{O}$), calcium chloride (CaCl_2) and iron(III) chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), alone, are not effective for the sedimentation processes. As it can be seen from Fig. 7, the settling rate increased with increasing coagulant dosage for three coagulant types. For coagulants, settling rate reached a maximum at around 2400 ($\text{Al}_2\text{SO}_4 \cdot 18\text{H}_2\text{O}$), 4800 (CaCl_2) and 1800 ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) g/Mg dosage, and then began to decrease with increasing coagulant dosage. Unfortunately, as it was expected, sedimentation depending on electrostatic and van der Waals attraction forces did not occur by expectation that adding cations such as Al^{3+} , Ca^{2+} and Fe^{3+} to negatively charged solution would decrease the zeta potential of particles. The reason for this is that the Al^{3+} , Ca^{2+} and Fe^{3+} cations alone are not effective coagulants and they form a series of

hydrolysis reactions in the colloidal systems, where the solids concentration and alkalinity are low. Since these cations are amphoteric, they behave as acid in alkali medium and vice versa. The reactions dependent upon this property show repetitive hydrolysis of cations.

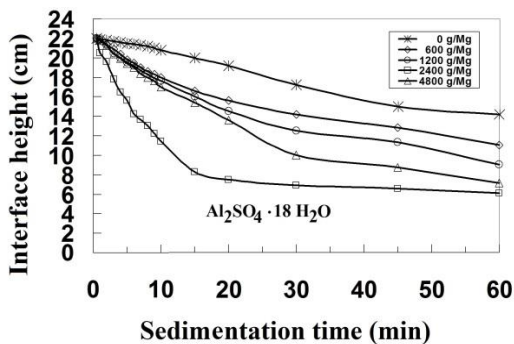


Fig. 4. Sedimentation curves of barite tailing elements for different doses of aluminum sulphate

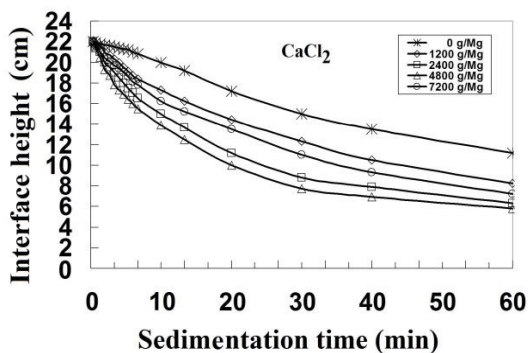


Fig. 5. Sedimentation curves of barite tailing elements for different doses of calcium chloride

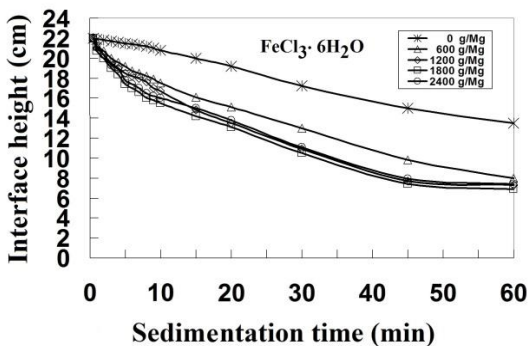


Fig. 6. Sedimentation curves of barite tailing elements for different doses of ferric chloride

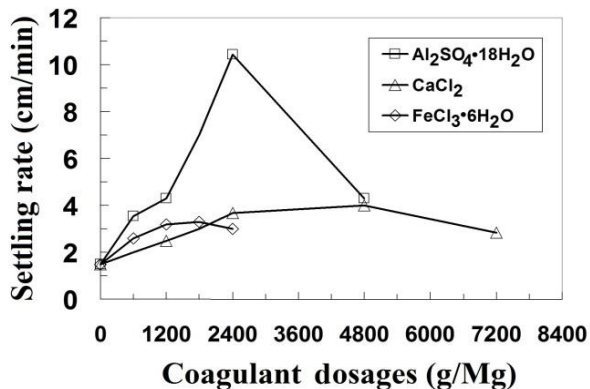


Fig. 7. Effect of flocculant dosages on settling rate for different coagulants

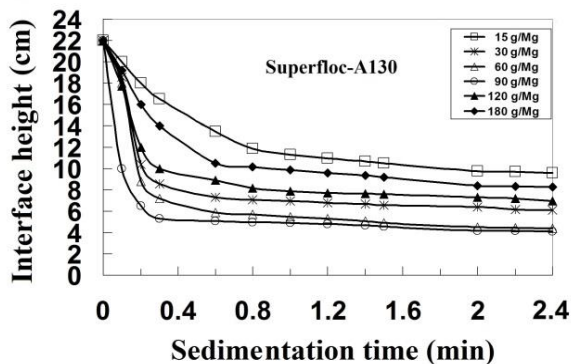


Fig. 8. Sedimentation curves of barite tailing elements for different doses of Superfloc A-130

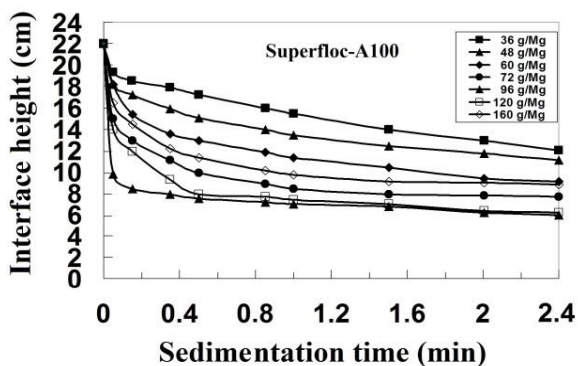


Fig. 9. Sedimentation curves of barite tailing elements for different doses of Superfloc A-100

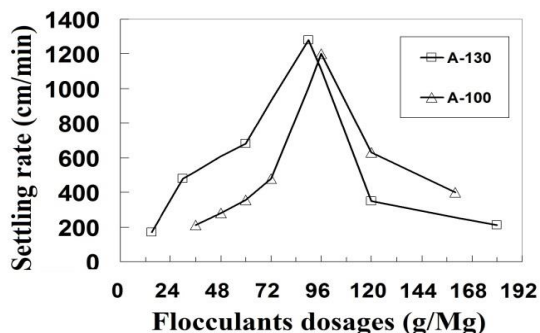


Fig. 10. Effect of flocculant dosages on settling rate for different flocculant types

The flocculation tests were performed in the presence of two types of polymer flocculants (Figs. 8 and 9) at different dosages. Figures 8 and 9 show the effect of polymer type and its dosage on the settling rate of particulate clays, while Figure 10 the relationship between settling rate and flocculant dosage. For Superfloc A-130 and Superfloc A-100, the settling rate reached the maximum for 90 and 96 g/Mg polymer dosage, respectively, and then began to decrease with increasing in the polymer dosage. At low dosages, the floc size is expected to be very small because of insufficient amount of polymer adsorption on the particles. The increase in the amount of adsorbed polymer results in incorporation of more suspended particles in the floc and in turn enlargement of the floc size, leading to the enhanced settling rate. Figure 10 clearly shows that the Superfloc A-130 settling rates are higher than for Superfloc A-100 flocculant in the most polymer dosages. In other words, for the same polymer dosages (90-96 g/Mg) Superfloc A-130 produces larger flocs. In addition, Superfloc A-130 shows much better performance than inorganic coagulants. For instance, whereas the highest settling rate of Superfloc A-130 has 1275 cm/min and A-100 has 1200 cm/min, on the other hand, $\text{Al}_2\text{SO}_4 \cdot 18\text{H}_2\text{O}$ has 10.5 cm/min, CaCl_2 4 cm/min and $\text{Fe}_2\text{Cl}_3 \cdot 6\text{H}_2\text{O}$ 3.2 cm/min settling rates.

Figure 11 provides the photographic description of settling behaviour of coagulant and flocculant dosed in the 600 cm³ measuring cylinder sets. The natural settling behaviour of barite clay wastewater is shown in Fig. 11(a). Under this condition, there is no clear distinct interface between the accumulated sediment bed and overlying water, thereby the volume fraction at the top of the accumulated sediment cannot be considered as the gel point (see Fig. 11(a)). When the particles are coagulated, the van der Waals and other colloidal forces are dominant and the coagulated particles settle together forming the interface between the coagulated sediment network and supernatant (see Figs. 11(b-d)). It can be seen from Figs. 11(e) and 11(f), that there is a very rapid settlement, leading to the formation of a coarsely structured matrix of deposited solids, with using flocculant. The settlement properties of this solids column appear to be largely insensitive to dose. Through time this layer slowly reduces in height as water is gradually released due to compressive settlement of the solids

matrix. Because of low particle charges, an effective flocculation of tailings, which contain high amounts of serisite and chlorite with anionic Superfloc A-100 and A-130 can be explained by the presence of electrostatic attraction forces and /or bonds of particles with the polymer bridges and van der Waals forces. Although polymers and particles have the same charges, better flocs obtained with Superfloc A-130 rather than Superfloc A-100 indicate that the particles are coated with the polymer bridges. Again, with the use of anionic polymer, a very high amount of anions adsorbed onto cations sites such as calcium, magnesium with the resultant electrostatic attraction forces and thus strong flocs are obtained.

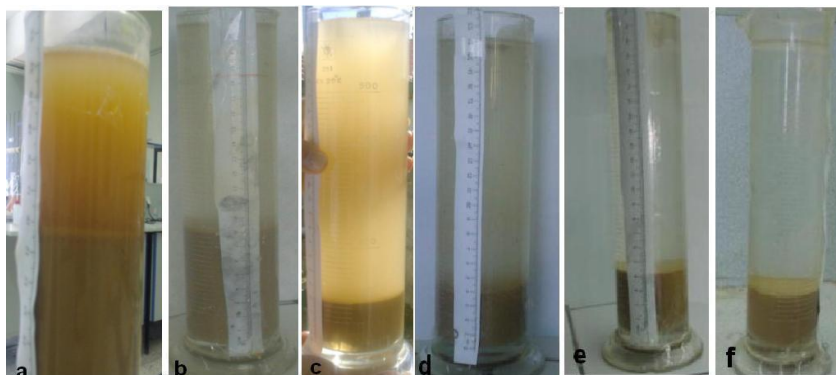


Fig. 11. Settling test after 30 min for various samples studied: (a) natural settling, (b) iron chloride, (c) calcium chloride, (d) aluminum sulphate, (e) Superfloc A-100 and (f) Superfloc A-130

Calculations of thickener area

The thickener design was used by Talmage and Fitch (1958) to determine the limiting flux, and thus to predict the unit area for specific thickening applications. The value of the area (A) was developed by the following equation (Talmage and Fitch, 1958):

$$A = \frac{Q \cdot t_u}{C_0 \cdot H_0}, \quad (1)$$

where A is the thickener area, m^2 , Q feed solid rate (9.49 Mg/h), t_u maximum time for underflow concentration (h), H_0 initial height (0.22 m), C_0 initial concentration (73 kg/m^3).

From the definition point of view, the thickener capacity was defined as the amount of the clear solution overflowing of slime slurry. In this work, the thickener area was calculated for each coagulant and flocculant type (Table 2). Table 2 summarized the data of the above factors, affecting flocculation behaviour of slime.

Table 2. Effect of coagulant and flocculant dose on the thickener area

Name	Dose g/Mg	Sediment rate mm/min	Thickener Area (m ²)
None	0.0	20.4	963
Al ₂ SO ₄ ·6H ₂ O	600	36	723
	1200	41	410
	2400	105	141
	4800	44	336
	1200	25	591
CaCl ₂	2400	36	410
	4800	40	369
	7200	28	528
	600	26	568
FeCl ₃ ·6H ₂ O	1200	30	492
	1800	32	462
	2400	30	492
Superfloc A-130	15	1700	8.69
	30	4800	3.08
	60	6850	2.16
	90	12750	1.15
	120	3500	4.22
	180	2200	6.71
Superfloc A-100	36	2150	6.87
	48	2650	5.57
	60	3500	4.22
	72	5000	2.95
	96	12000	1.23
	120	6250	2.36
	156	4100	3.60

According to the above calculations, a thickener was designed (Fig. 12). The thickener was realized for 130 m³/h wastewater treatment with the installed motor power of 40 kW. The flocculant consumption (Superfloc A-130) and transmittance of the reclaimed water were in a good agreement with the laboratory studies. The operating cost of the facility excluding labor is less than two cents (penny) per m³ of reclaimed water. For a comparison, one can take the cost of fresh municipal water from a municipality which is around ten cents per m³. The facility is able to reclaim approximately 86% of the wastewater and it has been run without any problems.



Fig. 12. Wastewater treatment plant belonging to the Baser barite beneficiation plant

Conclusions

Based on this study, the following conclusions can be made:

- X-ray and XRD analysis showed that the material used in this work is composed of serisite and chlorite,
- the measured electrical surface charges on the barite tailing are evaluated. It is observed that natural sedimentation of mineral particles in pulps is difficult,
- the electrical surface charges of clay minerals are negative (-), so, the change of OH^- and H^+ ion concentration of the pulp does not affect the sedimentation process,
- natural settling of suspended solids was found to be very slow and it took approximately 6 h of settling. The present study showed that the supernatant after 6 h of settling was not free from suspended particulate clays. The average settling rate was estimated to be 20.4 mm/min,
- the cations (Al^{3+} , Ca^{2+} and Fe^{3+}) can be used to improve the settling rate of barite tailings. However, the cations had a poor effect on sedimentation. The cation Al^{3+} proved to be the most effective coagulant in this test work,
- the flocculant settling tests work produced the highest settling rate. Also polyacrylamides are bound to the surface of particles with electrostatic attraction forces along with polymer bridges,
- the flocculation of clays (the net negative charge) by anionic polyacrylamides was explained in the light of interaction of the carboxylate group ($-\text{COOH}$) with positive site edges of clay minerals,
- the Superfloc A-130 flocculant had the best results in settling the slimes. The dosage of 90 g/Mg of polyacrylamide flocculant showed the best settling rate.
- clarification of barite tailing by polyacrylamide flocculants was achieved within 1-2 min against the natural settling time of 6-8 h.

The results showed that a simple solid-liquid separation system may reclaim the water input very efficiently. The simultaneous recovery of clay particulates from the

barite beneficiation plant effluent is essential from the economic point of view, as well as for the water pollution control.

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