

Settling characteristics of coal preparation plant fine tailings using anionic polymers

Hasan Ciftci^{*,†} and Serhat Isik^{**}

^{*}Mineral Processing Division, Department of Mining Engineering, Suleyman Demirel University,
West Campus, Isparta TR 32260, Turkey

^{**}Imbat Coal Mining Co., Inc., Soma, Manisa, Turkey

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Abstract—The objective of solid-liquid separation in thickeners in coal preparation plants is to obtain both a clear supernatant liquid with a low turbidity for reuse and a dense slurry. It is important for the smooth operation of the plant to produce good-quality recirculation water. In this study, settling characteristics of coal tailings ($d_{80}=70\ \mu\text{m}$) taken from the thickener feed of Derekoy Coal Preparation Plant (Manisa, Turkey) were investigated with the use of anionic polymers. In the tests we determined the effects of the process parameters including polymer type, polymer dosage, temperature, suspension pH, and pulp density on the flocculation of the fine tailings of the coal preparation operation. Minefloc anionic polymer showed a better flocculation performance in comparison with other polymers. An optimum settling rate of 300 mm/min was reached at a dosage of 30 g/t-solids, a pulp density of 5%, pH 7.9, and temperature 25 °C using Minefloc polymer.

Keywords: Dewatering, Coal Tailing, Flocculation, Thickener, Settling

INTRODUCTION

Solid-liquid separation is a very important process and a significant time constraint in mineral and coal preparation plants. Many mineral and coal preparation procedures take place in wet environments, and the partial or complete removal of water from the concentrated materials is carried out by means of solid-liquid separation methods.

In coal preparation plants, solid-liquid separation takes place in settling tanks (thickeners). The aim is to obtain a clear liquid for reuse in processing and a thick mud; however, the procedure is complicated and problematic. The presence of suspended solids in the supernatant liquid can lead to a reduction in the efficiency of the coal processing. Amongst these problems are differences in particle size, shape and weight of the particles in suspension, different colloidal behaviors, environmental variables, ions passing into the solution, and the interaction of all these variables [1]. Wastewater produced from coal preparation plants contains high percentages of ultrafine particles and inorganic impurities, which are composed of coal particles and minerals such as kaolinite, illite, muscovite, and quartz. Fine solid particles, especially those smaller than 50 μm , settle quickly in the solid-liquid separation stage, and therefore obtaining a high solids ratio in the slurry is not possible with normal sedimentation methods. This is due to the ratio of solids, particle size, surface properties and density of the solid, and viscosity and consistency of the liquid [2,3]. Coagulation and flocculation can be used successfully to overcome these issues [4].

Suspensions containing ultrafine solid particles settle very slowly

in a thickener for solid-liquid separation. This means that larger thickeners are needed, or else the available capacity is reduced. To assist the settling process, polymeric flocculants are used to increase the settling rate and to achieve more efficient solid-liquid separation for smaller-sized particles.

Efficient and economical completion of solid-liquid separation is dependent upon the flocculant type used, flocculant quantity, suspension pH, and other parameters chosen to suit the flocculation method and the mineral characterization [5-9]. The flocculant quantity required is specified to be enough to cover half the surface area of the solid. If excess flocculant is added, large quantities of flocculant will be adsorbed onto the surface of the particles and will prevent the formation of bridges between the particles [10]. This situation negatively affects the success of flocculation.

There are numerous studies investigating the flocculation and filtration behaviour of coal tailings. Foshee et al. [11] used organic polyelectrolytes to achieve improved wastewater clarification in a coal preparation plant. The flocculation of coal with xanthate or polyethylene was investigated [12]. Menon et al. [13] investigated the effect of coal and pyrites on the settling behaviour of clays in wastewater. Attia and Yu [14] investigated the flocculation and filtration of coal slurries with the use of hydrophobic flocculants. Tadros et al. [15] investigated the influence of a polyelectrolyte, a non-ionic polymer and their mixtures on the rheology of coal/water suspensions. Akdemir et al. [16] studied the effects of polymeric flocculant on limestone and gas oil in the dewatering of fine-grained coal. Tao et al. [17] made a comparative flocculation-filtration investigation using vacuum, hyperbaric and centrifugal filters on an ultrafine clean coal slurry. A lignitic coal (from Yozgat, Turkey) was studied to identify the conditions for optimum flocculant type and dosage [7]. In the study by Sabah and Cengiz [18], the effects of ionic groups on the settling behavior of polyacrylamides in the coal preparation

[†]To whom correspondence should be addressed.

E-mail: hasanciftci@sdu.edu.tr, ciftci_h@yahoo.com

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plant tailings were studied. Sabah and Erkan [19] investigated the interaction mechanism between various types of flocculants and multi-component fine coal tailings suspensions. Alam et al. [20] studied the effects of preconditioning tailings with varying flocculants and dosages on filtration kinetics. Ofori et al. [21] examined the effect of floc structure on coal tailings dewatering and the impact of shear-induced modification of floc network structure on additional sediment consolidation and densification. The colloidal and dewatering behaviors of four coal processing tailings were investigated in the presence of high-molecular-weight anionic poly-electrolytes [22].

Understanding of the interaction mechanism between polymeric flocculants and solid particles in suspensions and the associated flocculation mechanism is of great practical and fundamental importance in mineral processing and interface science [23]. The optimization of process parameters such as suspension pH, polymer type, and dosage is important in achieving the desired settling rate and water clarity values [24,25]. In this study we examined dewatering of the coal preparation operation plant tailings containing fine solid particles by the use of anionic flocculants, and the effect of process parameters including flocculant type, flocculant dosage, pH, temperature, and pulp density on the settling of solid particles for plant wastewater feeding into the thickener at the coal preparation plant.

MATERIALS AND METHODS

1. Materials

The samples used in the experimental studies were taken from the feed into the thickener at the Ciftay-Ozdoganlar A.S. Derekoy Coal Preparation Plant (Manisa, Turkey) for four days at representative set intervals. Physical, chemical, and mineralogical tests were carried out to determine the properties of the fine coal tailings samples. Chemical and mineralogical tests of the samples were at the accredited and certified General Headquarters Laboratory of the General Directorate of the Mineral Research & Exploration (MTA, Turkey). The physical and chemical analysis values of coal tailings samples, based on dry base, are given in Table 1.

Wet sieve analysis results of coal tailings samples from the thickener feed are given in Table 2. D_{80} and D_{50} of coal tailings samples

Table 1. Characterizations of the coal preparation tailings

Pulp density (original suspension) (%)	7.32
Natural pH of pulp	7.9
Ash content (%)	59.77
Total sulfur content (%)	0.68
Carbon content (%)	10.51
Volatile matter (%)	29.72
Lower calorific value (kcal/kg)	1772
Higher calorific value (kcal/kg)	1870
d_{80} (μm)	70
SiO_2 (%)	51.4
Al_2O_3 (%)	23.6
CaO (%)	14.3
Fe_2O_3 (%)	3.2
K_2O (%)	1.7
MgO (%)	1.6
TiO_2 (%)	0.6
Na_2O (%)	0.3
Others	3.3

from the thickener feed were below 70 μm and 20 μm , respectively (Table 2). X-Ray diffractometer (XRD) analysis was carried out on representative samples to determine the mineralogical composition of the tailings samples. The XRD analysis indicated that the samples contained coal, along with quartz (SiO_2), calcite (CaCO_3), chlorite ($(\text{Mg,Fe,Al})_6(\text{Si,Al})_4\text{O}_{10}(\text{OH})_8$), microcline (KAlSi_3O_8), and beidellite ($\text{NaO.5Al}_2(\text{Si}_3.5\text{Al}_{0.5})\text{O}_{10}(\text{OH})_2 \cdot n(\text{H}_2\text{O})$) (Fig. 1).

2. Method

In the study, we used six anionic flocculants commonly used in industry (Minefloc Lot X 4119, Brentamer A 2030, Brentamer A 2530, BIFLOC AN 254, Akkim Akua END 5220, and Sedifloc 750 AHM) to carry out the tests.

Flocculation experiments were carried out in a Velp model JLT6 jar test device with six mixers. In the experiments, 600 ml beakers were used with millimeter scales. Prior to flocculation experiments, a stock solution (0.1% w/v) of each flocculant was prepared with distilled water.

In experiments to research the effect of pH, sodium hydroxide

Table 2. Particle size and ash distributions of the coal preparation plant tailings on air dried basis

Particle size (μm)	Amount (wt%)	Ash (%)	Cumulative undersize (wt%)	Cumulative oversize (wt%)	Ash distribution (%)
+300	3.87	8.83	100.00	3.87	0.61
−300+212	2.58	15.82	96.13	6.45	0.73
−212+150	3.33	24.80	93.55	9.78	1.48
−150+106	5.82	29.88	90.22	15.6	3.10
−106+75	3.36	36.54	84.4	18.96	2.19
−75+53	6.1	39.63	81.04	25.06	4.31
−53+45	1.01	42.53	74.94	26.07	0.77
−45+38	4.26	45.97	73.93	30.33	3.49
−38+25	3.31	48.34	69.67	33.64	2.86
−25	66.36	67.95	66.36	100.00	80.46
Total	100.00	56.04			

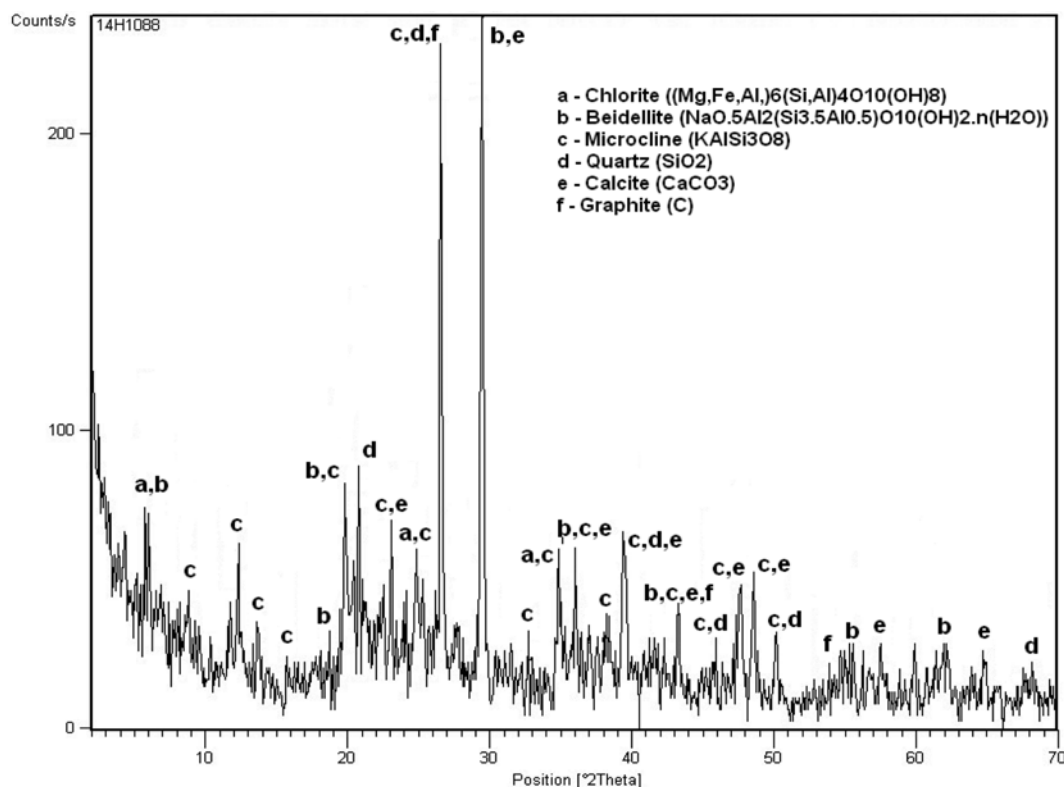


Fig. 1. XRD pattern of the coal preparation plant tailings.

(NaOH) and sulfuric acid (H_2SO_4) were used to obtain the preferred pH. A Hanna model HI 9321 pH meter was used to determine the pH of the solution. Following the flocculation experiments, turbidity measurements were carried out with a WTW Turb model 550 turbidity meter.

Approximately 100 liters of samples collected from the preparation plant thickener feed contained 7.32% (w/w) solids. Flocculation tests were carried out at 5% (w/w) solids concentration. For every experiment, a 600-ml beaker was filled with a sample of 500 ml of a homogeneous mixture (5% solids). The beakers containing the 500-ml samples were placed in the jar test device and mixed continuously for 2 minutes at 300 rpm. Following this, the desired amount from flocculant solution was added to the suspension and mixed for 2 minutes at 100 rpm. After mixing, the beakers were allowed to stand undisturbed and the heights of the slurry/water interfaces were recorded over time. Following the flocculation process and after waiting 15 minutes, 20-ml samples of the supernatant liquid were taken for turbidity measurement, and the turbidity was recorded with the turbidity meter.

RESULTS AND DISCUSSION

1. Effect of Polymer Dosage

To study the effect of flocculant dosage on the coal tailings slime samples following flocculation, the anionic flocculants Akkim Akua 5220 (F1), Brentag Brentamer A 2030 (F2), Brentag Brentamer A 2530 (F3), Bifloc AN 254 (F4), Minefloc Lot X 4119 (F5), 3F Sedi-floc 750 AHM (F6) were used to complete the flocculation experi-

ments and record the settling rates in the experiments, and changes to turbidity values were observed.

In the experiments, the settling rate increased up to a flocculant dosage of 30 g/t-solids, but remained stable above this dosage (Fig. 2(a)). The floc size was very small at low flocculant dosages due to insufficient polymer adsorption onto the particles, resulting in a poor settling rate. With increasing flocculant dosage, the amount of adsorbed polymer increased, resulting in the incorporation of more suspended particles into the flocs and enlargement of the floc size, leading to an enhanced settling rate [25]. The optimum dosage of the flocculants studied was therefore 30 g/t-solids, as the settling rate remained stable and the turbidity values showed only a small increase beyond this flocculant dosage.

Anionic flocculants bond to solid particles with their long hydrocarbon chains and form bridges between the grains to form a floc. In systems with kaolinite-type clay minerals, hydrogen bonds are formed with the surfaces to make the stable colloidal grains unstable [26].

In proportions above the optimum dosage, there is no space left on the particle surfaces for the flocculant bonding to occur, and therefore fewer bonds are formed, affecting the flocculation in a negative manner [9]. This situation is also known as a steric disability at high dosages [10,27,28].

An increase in flocculant amount can result in a decrease in turbidity; however, at dosages above the optimum proportion of 30 g/t-solids determined for settling rate, turbidity decreased. Generally, the lowest turbidity values in all the flocculants were observed at a flocculant dosage of 50 g/t-solids at the 68.0-97.7 NTU

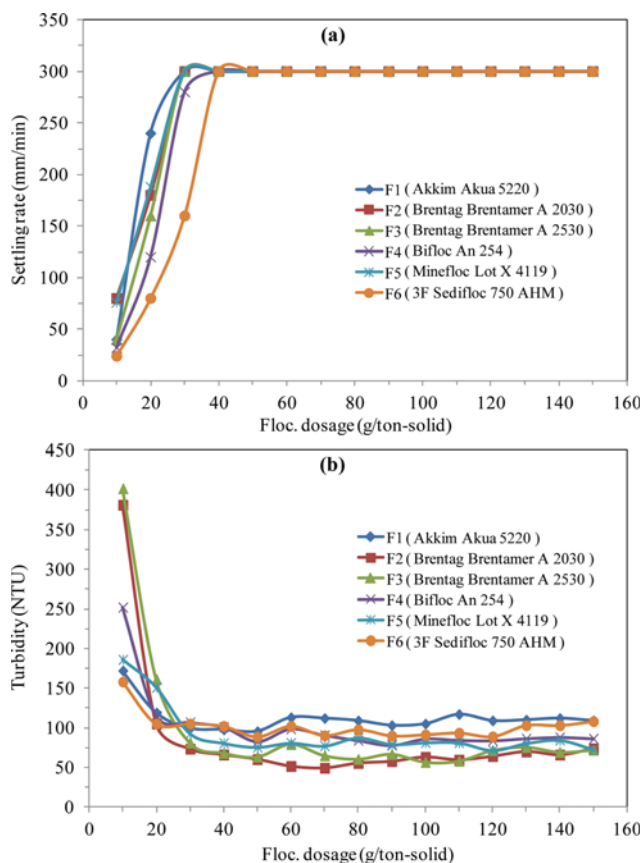


Fig. 2. Effects of polymer type and dosage on settling rate (a) and turbidity (b) [pulp density: 5%, stirring rate: 100 rpm, pH: 7.9, temperature: 25 °C].

interval (Fig. 2(b)). In the presence of Brentag Brentamer A 2030 (F2) and Brentag Brentamer A 2530 (F3) flocculants at a dosage of 10 g/t-solids, although the settling rate was higher than for other samples, turbidity values at this dosage were considerably higher than for the other flocculants (Figs. 2(a) and 2(b)).

2. Effect of pH

Suspension pH affects the stability of particles and also the ionization of flocculants, and it is closely linked to hydrolysis and suspension arrangement. Therefore suspension pH in the flocculation process is very important [26].

Akkim Akua 5220 (F1), Brentag Brentamer A 2030 (F2), and Minefloc Lot X 4119 (F5) which performed better in settling rate and turbidity in comparison to the others, were used, and experiments were carried out at pH 2-12 to study the effect of pH on the flocculation process in experiments where the effect of flocculant dosage was studied. The changes in settling rates and turbidity values obtained from the experiments are shown in Figs. 3-5.

The rate of settling was considerably reduced when the medium had acidic pH in the flocculation tests, but the turbidity was also significantly lower. The samples taken from the thickener feed with pH values higher than the original pH (7.9) had high rates of settling, but the turbidity was also significantly high. In the experiment at pH 6 with the Brentag Brentamer A 2030 (F2) polymer, settling rates of over 120 mm/min were observed with a flocculant

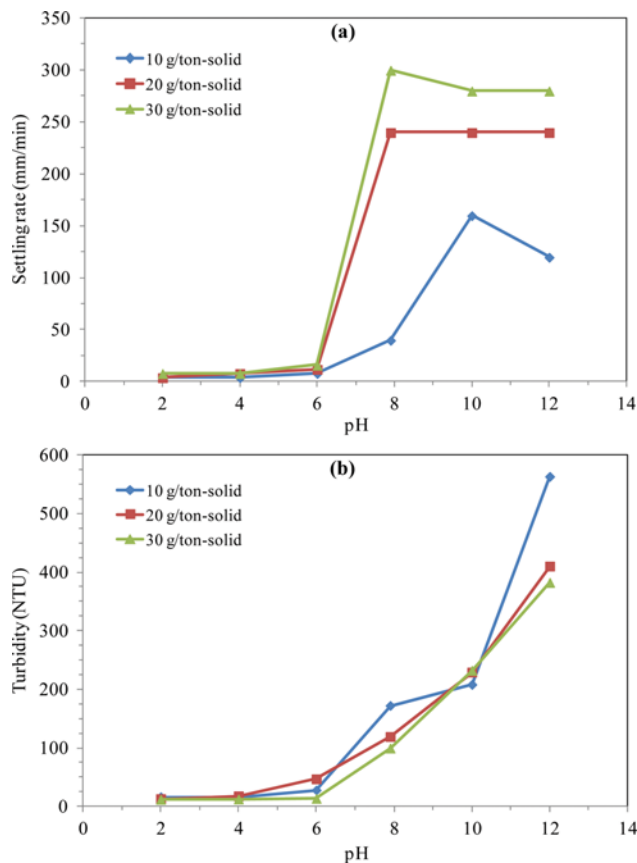


Fig. 3. Effects of suspension pH on settling rate (a) and turbidity (b) in the flocculation tests using Akkim Akua 5220 (F1) polymer [pulp density: 5%, stirring rate: 100 rpm, temperature: 25 °C].

dosage of 30 g/t-solids (Fig. 4(a)), whilst other flocculants showed lower settling rates at the same pH (Figs. 3(a) and 5(a)).

Suspension pH plays a key role in the activation of flocculation in the medium. A non-ionic form of a polymer can obtain positive (+) or negative (-) charges in acidic and basic pHs and change to ionic (charged) form [29,30]. This situation increases the electrostatic interaction between the polymer and the particle. Also, pH directly affects whether or not the solid particles in the suspension will become charged [10].

Although settling rates are low in acidic mediums, turbidity values are also low, and therefore flocculation is better, while in basic mediums, although the settling rate increases, the turbidity values are high and therefore flocculation efficiency falls considerably. In an acidic medium, the number of carbonyl groups ($-C=O^-$) in the molecules decreases. Therefore, the ability of the anionic flocculants to form flocs decreases, and bridge formation becomes weaker due to coagulation in the groups [18].

The decrease in turbidity in an acidic medium results in large aggregates from side and surface interactions of colloidal-sized clay minerals. Also, because coal and quartz particles have lower zeta potentials in an acidic medium, with the effect of van der Waals forces, the interaction between the particles increases. Settling rate increases in neutral or weakly alkaline pH as the bridges formed

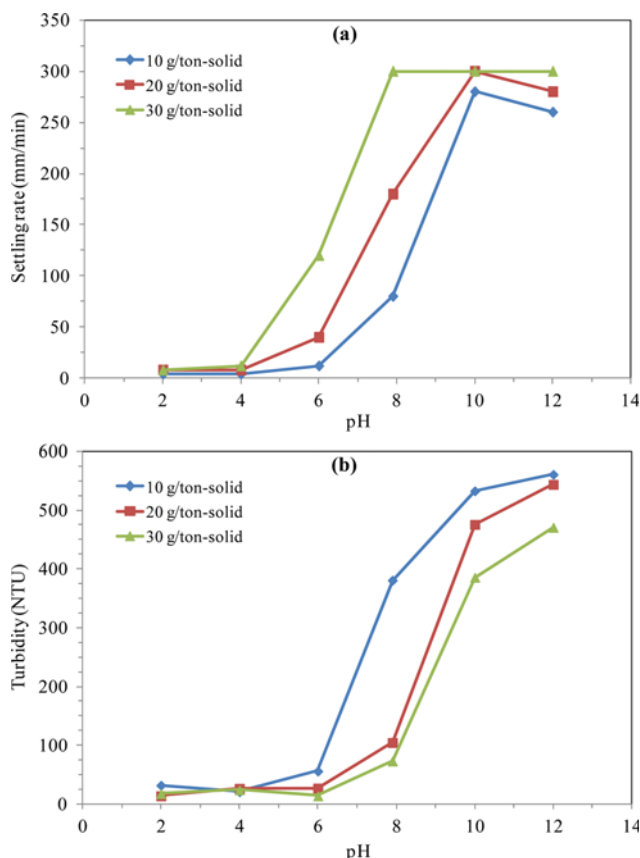


Fig. 4. Effects of suspension pH on settling rate (a) and turbidity (b) in the flocculation tests using Brentag Brentamer A 2030 (F2) polymer [pulp density: 5%, stirring rate: 100 rpm, temperature: 25 °C].

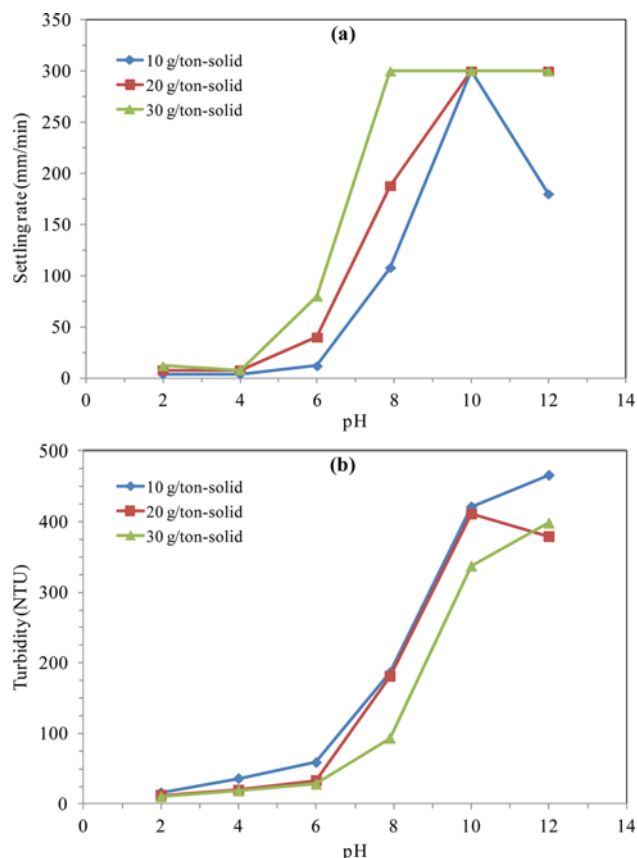


Fig. 5. Effects of suspension pH on settling rate (a) and turbidity (b) in the flocculation tests using Minefloc Lot X 4119 (F5) polymer [pulp density: 5%, stirring rate: 100 rpm, temperature: 25 °C].

between the anionic polymers are started with particles of high molecular mass, and therefore large flocs are formed. Generally, the pH at which functional groups of anionic flocculants become active varies from 6 to 13, with anionic groups becoming ionized below pH 6 and above pH 13, and thus reducing flocculation efficiency [26].

As the pH value increases in the medium, the increasing zeta potential and the ability of the particles in suspension to repel one another increases, and coal suspension becomes more stable. Also, when the coal surfaces have a high negative potential, flocculants carrying a negative charge have more difficulty in adsorbing to these surfaces. Therefore, these effects result in reduction in flocculation efficiency [31].

3. Effect of Temperature

To study the effect of temperature upon flocculation of the coal suspension, experiments were carried out at 10–40 °C with Minefloc Lot X 4119 (F5). The settling rates obtained and changes in turbidity are given in Fig. 6. The preferred suspension temperature was achieved with a water bath.

The suspension temperature affects the activation of the flocculant molecule. Also, the viscosities of the suspensions achieved with the flocculants vary with temperature [10]. The highest settlement rate was observed at a room temperature of 25 °C (Fig. 6(a)). Also,

in experiments with different flocculant dosages (10–30 g/t-solids) at 25 °C, the suspension turbidity was generally lower than at other temperatures (Fig. 6(b)).

4. Effect of Pulp Density

Other parameters investigated to study the effect of solids ratio on flocculation of the coal suspension showed that the best flocculant for the application was the Minefloc Lot X 4119 (F5) with anionic character. It was used at 2.5–15% solids in experiments, and the settling rates and changes to turbidity values are shown in Fig. 7. In experiments to study the effect of solids ratio on flocculation, the original pH and room temperature were maintained, and flocculant dosage was increased until high rates of settling were obtained. It is apparent from Fig. 7(a) that the initial settling rates decrease with increasing pulp density. As a result of these experiments, when solids ratios were 2.5%, 5%, and 7.5%, even small flocculant dosage (50 g/t-solids) had high settling rates, and as the solids ratio increased, so did the turbidity values (Figs. 7(a) and 7(b)). Higher solids ratios, such as 10% solids, did not achieve flocculation when the flocculant dosage was 100 g/t-solids or below. At 12.5% and 15% solids (w/w), the settling rates increased remarkably after the dosage of 175 g/t-solids and 325 g/t-solids, respectively. Also, for flocculation to be achieved at 12.5% and 15% solids, the flocculant dosage was increased to 200 g/t-solids and 400 g/t-

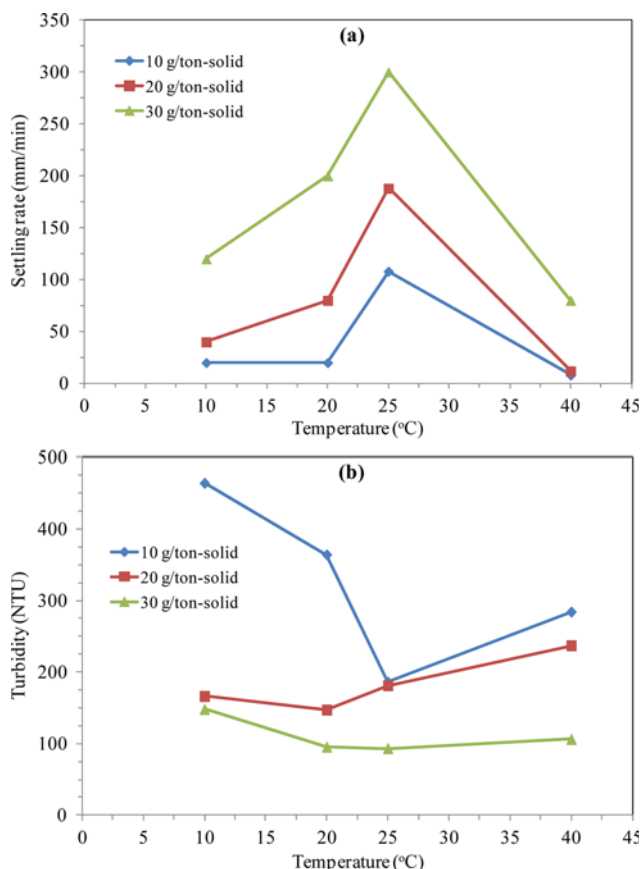


Fig. 6. Effects of temperature on settling rate (a) and turbidity (b) in the flocculation tests using Minefloc Lot X 4119 (F5) polymer [pulp density: 5%, stirring rate: 100 rpm, pH: 7.9].

solids, respectively, with high rates of settling and low turbidity values. As solids ratio increased, so did the amount of flocs formed during flocculation.

It can be seen from the study that solids ratio is an important parameter in the flocculation of the coal tailings slime, and that as the solids ratio increases, the required flocculant dosage increases in a logarithmic manner. When the solids ratio increases, the number of particles in the medium increases and as the particles become closer together, the electrostatic repulsion force of same-charged particles increases. Therefore, the number of polymer chains becomes insufficient for flocculation, and, as a result, the flocculant dosage needs to be increased. From this perspective, for a more efficient flocculation, when the solids ratio is increased, the flocculant amount needs to be increased considerably more. Moreover, a decreasing rate in settling is generally observed at higher pulp densities due to greater buoyancy forces as well as lesser ease of liquid trickling through the particles [32].

CONCLUSIONS

Flocculation experiments carried out on tailings samples collected from the Derekoy Processing plant thickener feed demonstrated that the settling rates of solid particles were high when anionic flocculants were used with the suspension at its original pH and

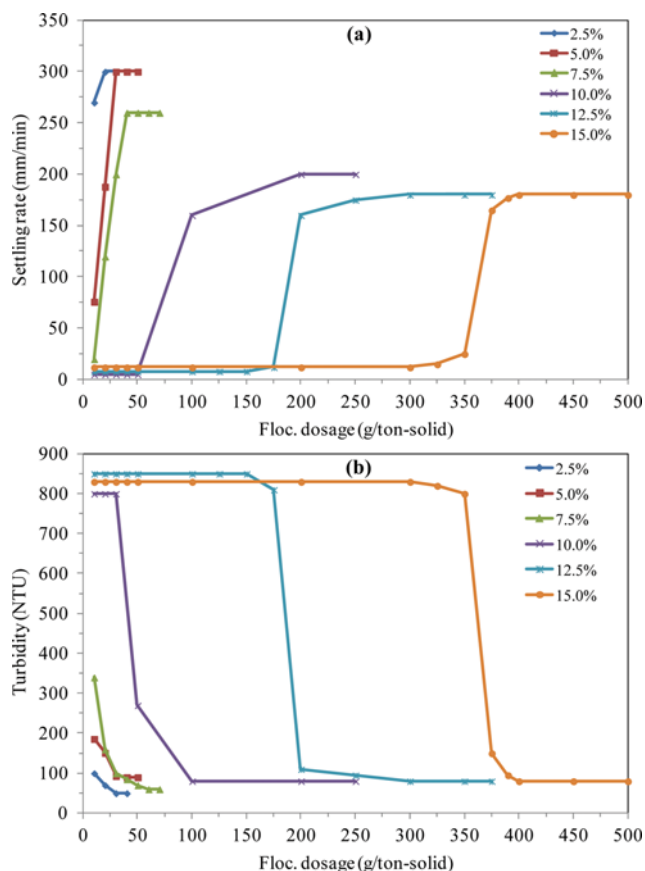


Fig. 7. Effects of pulp density on settling rate (a) and turbidity (b) in the flocculation tests using Minefloc Lot X 4119 (F5) polymer [stirring rate: 100 rpm, pH: 7.9, temperature: 25 °C].

that even low dosages (30 g/t-solids) increased the efficiency of flocculation.

In basic mediums, although settling rates were high because the negative zeta potential of the particles increased the interparticle repulsion force increased, and thus clay particles were more dispersed and high turbidity values were obtained. In a weakly acidic medium, the best flocculation performance was seen from Brentag Brentamer A 2030 (F2). In the flocculation experiments at different temperatures (10–40 °C), the highest settling rates were at 25 °C.

The flocculation experiments on tailings samples collected from the coal processing plant thickener feed also demonstrated that the anionic flocculants displayed the highest efficiency in a neutral or weakly basic (pH 7–8) medium at 25 °C. As the solids ratio increased, the number of particles in the medium increased, and as the particles became closer together, the electrostatic repulsion force of same-charged particles increased. Therefore the number of polymer chains became insufficient for flocculation, and, as a result, the flocculant amount needed to be increased. Therefore, for efficient flocculation when the solids ratio is increased, the flocculant dosage needs to be increased considerably more.

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