

## Determination of Various Flocculants' Performance in Flocculation of Lignite Waste Pulps

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In this study, the performance of the different flocculants, in the flocculation of the pulps prepared using lignite samples, has been investigated taking into consideration floc settling rate, supernatant turbidity and filtration. In the experiments, their anionic, cationic, non-ionic flocculants and mixtures have been used. For the high settling rate and low turbidity the best results have been obtained with non-ionic flocculants. Regarding the filtration anionic flocculant has given the best result.

**Key words:** Flocculation, filtration, settling rate, environmental pollution.

### INTRODUCTION

Fine and ultrafine particles are always present in mineral processing as a result of either production or size reduction processes. Especially, the amount of the fine particles is continuously increased due to both natural and structural faults in coal seams; mechanized production methods and fine sized coal are required in some usage industries (areas) such as liquefaction, gasification. Fine particles cause several problems in some processes such as transportation, enrichment, dewatering and storage. It has been reported that the effluent of coal preparation plants consisted of 20 to 80% coal by weight<sup>1</sup>. In the coal preparation plants, fine particles which are not recovered lead to both energy losses and several environmental problems. Effluent of plants must be treated to remove solid particles in order to refuse the problems seen in both effluent area and reuse in the plants.

Solid-liquid separation processes play a major role in most resource and many of the manufacturing industries and the coal industry is no exception. Since almost all existing coal preparation processes involving water as the carrying medium, solid particles/water suspensions present one of the main problems for the solid/liquid separation engineer. Separation of solids from suspending liquids generally involves the application of an external driving force to the system.

The particle size plays a controlling role in separation processes. Most of the

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solid/liquid separation aids are water soluble chemicals. They include simple salts producing ions such as  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Al}^{3+}$  and polymers. The former are usually referred to as coagulants and the latter as flocculants<sup>2</sup>. Coagulation is affected by positive (or negative) ions neutralizing opposite charges on the solid particles thus overcoming their resistance to approach each other and form larger aggregates. On the other hand, flocculants produce aggregation by physically "tying" particle together. The water soluble, "long-molecule" polymers in water absorb simultaneously on different particles by what is postulated by many authors as a "bridging" mechanism. In the flocculation process, high molecular weight flocculants are generally used. Flocculants are generally far more efficient in particle aggregation than coagulants. Flocculants lead to flocculation by increasing the settling rate of particles because of the bridging among the particles<sup>3-5</sup>. In the flocculation settling rates (sedimentation rates) are the simplest and most commonly used.

Three types of performance criteria are described as turbidity of supernatant, floc settling rate and compressibility of sediment in order to express a flocculant performance<sup>6</sup>. These type of criteria (one or some) are important in different application areas. For any kind of flocculation process, the choice of flocculant (polymer) is dependent on a lot of factors such as cost, supply, solubility and ease of use.

In this study, anionic, cationic and non-ionic flocculants individually and as a mixture have been evaluated in order to flocculate lignite samples prepared from Yozgat Ayridam lignite.

## EXPERIMENTAL

**Preparation of sample:** The coal tailing sample taken from Yozgat-Ayridam Coal Management was used in all experimental studies. The coal sample consisted of 55% ash. All of the samples were ground into  $-100\ \mu\text{m}$  by using ball mill. The sample was wet screened.

The size distribution of the sample is given in Table-1.

TABLE 1  
SIZE DISTRIBUTION OF THE SAMPLE

Particle size ( $\mu\text{m}$ )	Weight (%)	Cumulative under size passing (%)
-100 + 63	12.07	100.00
-63 + 45	16.04	87.93
45 + 38	17.85	71.89
38 + 20	28.93	54.04
20	25.61	25.61

**Mineralogical and chemical analysis of the sample:** According to XRD results of the sample, it was determined that main mineral matters are kaolinite,

pyrite, calcite and quartz. The sample consisted of 55% ash and 1.19% sulphur on dry basis.

**Flocculants and their properties:** In the experiments, superfloc type flocculants produced by Cyanamid Company were used. Flocculants were prepared as 0.01% solution by weight. Some properties<sup>7</sup> of flocculants were shown in Table-2.

TABLE 2  
SOME PROPERTIES OF FLOCCULANTS

Flocculant	Form	Type	Molecular weight ( $\times 10^6$ )
N-100	Powder	Non-ionic	5-15
A-150	Powder	Anionic	5-15
C-521	Emulsion	Cationic	2-6

**Flocculation experimental procedure:** Settling rate tests were carried out following a standardised procedure<sup>8</sup>. A number of 500 mL pyrex graduated cylinders having an inner diameter of 55 mm and a height of about 300 mm at the 500 mL mark (initial settling point) were used. In case of dual polymer addition, the order of addition of flocculants is given as shown in Figs. 2-4.

The desired concentrations of flocculant were added by means of a syringe to the cylinder containing the sample. After the mixing was completed, the time and the level of slurry-supernatant liquid interface (mud-line) were recorded and subsequently plotted. In the experiments, an adjustable speed magnetic mixer and distilled water were used.

In the earlier study, the effect of various parameters such as pH, mixing time, mixing rate, flocculation concentration was investigated. It was found that the optimum pH value of flocculants was to be 8 for all of the used flocculants<sup>9</sup>. Therefore, the experiment was performed at pH 8. NaOH was used as a regulation of pH. In addition, in the turbidity studies, Jenway 6035 type turbidimeter was used.

Experimental conditions are as follows:

pH	8
mixing speed	500 rpm
solid ratio	2.5% by weight
stirring time	1 min

**Filtration experimental procedure:** Filtration experiments were performed with pulps which were flocculated at optimum flocculant dosage. The experiments were performed by using vacuum filter and filtration paper (Toyo No. 5B) under the constant pressure (0.80 atmosphere). The diameter of pore size for the filtration is 0.45  $\mu\text{m}$ . In the experiments the filtrate volume vs. time was determined.

The form of presentation of the results is explained schematically in Fig. 1<sup>2, 10</sup>. In this plot, the abscissa represents the filtration volume "V" collected in time

"t" while the ordinate represents the ratio "t/V". The resulting plot is ideally a straight line of the form  $dt/dV = KP + B^{11-13}$ .

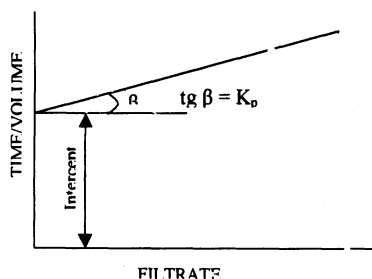


Fig. 1. Schematic representation of Filtration data

## RESULTS AND DISCUSSION

**Evaluation of flocculation experimental results:** In order to evaluate the experimental results, the settling rate and turbidity values have been considered. The settling rate was determined from the slope of straight line which indicates the change of interface height depending on time, as mentioned in the literature<sup>14, 15</sup>. The turbidity values were determined in supernatant part taken from the surface of the settling unit, after settling was completed. The main factor of success and economy of the flocculation process is flocculant concentration. Therefore, the effect of flocculant concentration on the settling rate and turbidity was investigated.

The settling rate and turbidity variations vs. flocculant concentration were shown in Figs. 2 and 3 respectively.

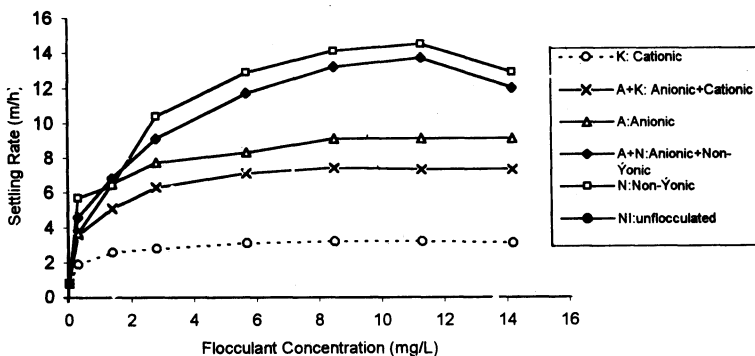


Fig. 2. Change of settling rate at different flocculant concentrations

As shown in Fig. 2; generally, as the concentration increases, the settling rate increases. Even at low concentrations, the settling rate is rather high than natural settling rate (0.852 m/h). At the same concentrations, the high settling rate was obtained by using non-ionic flocculants. At low concentrations of flocculants decreasing of the settling rate was based on adequate flocculant bridging among the particles. As the concentration of flocculants increases, increase in the settling rate is due to the formation of large sized flocs. The decrease in the settling rate

at high concentrations (12 mg/L) (particularly nonionic and nonionic + anionic flocculants) was based on inadequate flocculant distribution and change in the order of flocculant in the pulp. At 2 mg/L and much higher concentrations, the settling rate is invariable by using anionic, cationic and anionic + cationic flocculants. This case was based on adsorption of the added flocculants on the surface of flocs. The findings mentioned above comply with literature<sup>16, 17</sup>.

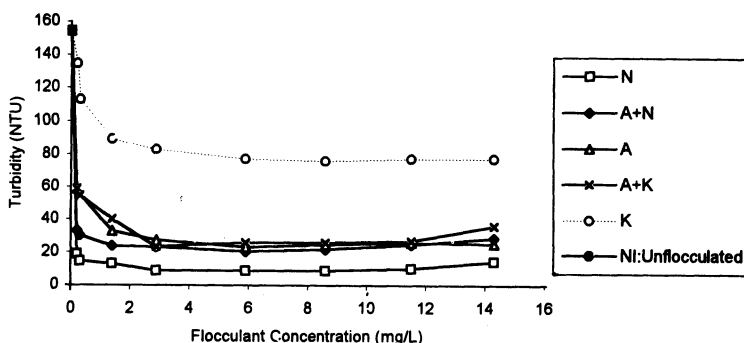


Fig. 3. Change of flocculant concentration vs. turbidity

As can be seen in Fig. 3, the lowest turbidity of supernatant was obtained with non-ionic flocculant. The turbidity of supernatant was the highest even at high concentration of cationic flocculants. The turbidity of supernatant was the lowest values at about 3 mg/L concentration all of the flocculants. However, the partly increase of turbidity values of the supernatant were observed at very high concentration of flocculants. This can be due to both the bonding of each other of flocculants molecule with hydrophobic interaction and the adsorption of adding flocculant over the flocs occurred before. Optimum flocculant concentrations were determined according to both settling rate and turbidity values. The optimum concentrations of flocculants are as follows:

Flocculant	Concentration (mg/L)
Cationic	2.85
Anionic + Cationic	5.70
Anionic	5.70
Non-ionic	8.55
Anionic + Non-ionic	8.55

As mentioned above results show that optimum flocculant concentration is given different values for the varying flocculants. As shown in Figs. 2 and 3, the lowest turbidity and the highest settling rate values were obtained by using non-ionic flocculant.

**Evaluation of filtration results:** The results of filtration experimental study were evaluated as mentioned in literature<sup>2, 12, 13</sup>.

Main filtration equation is described as mentioned below:

$$\frac{dt}{dv} = KpV + B$$

In this equation;  $K_p$  and  $B$  are constants where

$$K_p = r\mu W/2A^2P, \quad B = \mu R_m/AP$$

In this equations:

- $r$  = Specific cake resistance ( $m \text{ kg}^{-1}$ )
- $\mu$  = Filtered viscosity ( $N \text{ s m}^{-2}$ )
- $W$  = Solid concentration ( $kg \text{ m}^{-3}$ )
- $A$  = Filtrate area ( $m^2$ )
- $P$  = Total pressure drop ( $N \text{ m}^{-2}$ )
- $R_m$  = Filter medium resistance ( $m^{-1}$ )
- $V$  = Filtrate volume ( $m^3$ )
- $t$  = Time (s)

As shown in basic filtration equations, the intercept,  $B$ , of the straight line is directly proportional to  $R_m$  (filter medium resistance) and the slope,  $K_p$ , is directly proportional to  $r$  (specific cake resistance). For the filtration process at the constant pressure;  $\mu$ ,  $W$ ,  $A$  and  $P$  are kept constant. So, the value of intercept ( $B$ ) of  $dt/dV$  axis and straight line is directly proportional to filter medium resistance and the slope of the line is directly proportional to cake specific resistance.

Therefore, it can simply be stated that the high intercept and the large slope imply the poor filtration characteristics.

As shown in Fig. 4, as compared to unflocculated pulp, all flocculants gave the more efficient filtration characteristic pulps.

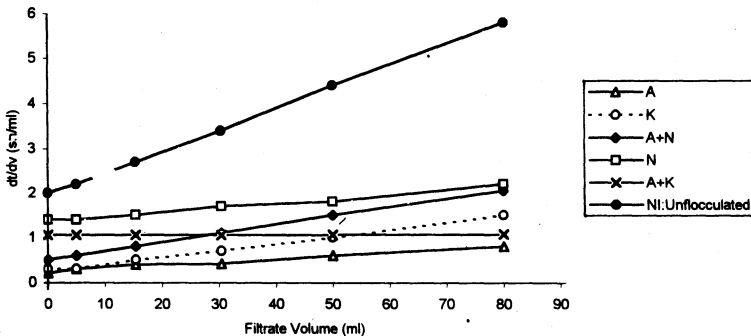


Fig. 4. Filtration curves of sample pulp using various flocculants

With unflocculated pulps, the intercept value  $dt/dV$  axis and the straight line is higher and the slope of the straight line is larger. Besides, the anionic flocculant has the lowest intercept and the smallest slope.

Anionic flocculants gave the best results in the filtration. It was attributed to both the less deformation of the flocs under the pressure and the limited compression of them. No doubt, the different filtration behaviours of flocs change depending on both the order of flocs and the structure of flocculants and flocs.

## Conclusions

- All of the flocculants increase the settling rate. While the higher increase of settling rate was obtained by using non-ionic flocculants, the lower one

was obtained by using cationic flocculants. The lower settling rate with cationic flocculant was attributed to the low molecular weight.

- The increased order of the turbidity values were found to be non-ionic < mixture of flocculants < anionic < cationic flocculant.
- The best filtration properties were obtained with the anionic flocculant. It was found that the resistance of cake and filter medium were considerably reduced by using anionic flocculant.
- No doubt, because of the high cost of flocculant, the economical criteria are also considered for choosing of flocculants.

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