

## COMPARISON OF THE INFLUENCE OF POLYACRYLAMIDE AND SODIUM OLEATE ON SELECTIVE FLOCCULATION OF GOETHITE FROM SLUDGE

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### Abstract

*The paper represents a part of research conducted with the aim of examining the possibility of applying selective flocculation of goethite from sludge that occurs in the process of iron ore production. The aim of this study was to compare the influence of polyacrylamide and sodium oleate as a flocculant on the settling behavior of goethite under different conditions. The paper presents the results of the research on the influence of the flocculants based on polyacrylamide and sodium oleate, as well as pH values on the settling rate of a natural goethite sample. The results showed that the settling rate of goethite was influenced by pH value and the type of flocculant, while the concentration of flocculant did not significantly affect the settling rate. Measurements of zeta potential showed the effect of flocculants on the surface of the goethite.*

**Key words:** Goethite; Flocculation; Settling rate; Zeta potential.

### 1. Introduction

In recent years, generation of fines and slimes during mining and mineral processing has become an important problem, both economically and ecologically. The large quantity of these fines/slimes are often discarded as waste into tailing ponds. In the mineral processing, many of wastes contain colloidal stable dispersions. These are, as a rule, complex systems, which contain several mineral species. Fine iron ores most often contain clay, silica, and other minerals as gangue material along with iron minerals. For these reasons, a number of new approaches to recover the fine and ultrafine particles have been investigated, including selective flocculation.

Processing of iron ore in Omarska mine is carried out by following methods: washing, sieving, grading, and magnetic concentration. The large quantity of fine sized (<15 µm) sludge disposed as final waste in the beneficiation plants of the Omarska mine (Republic of Srpska, Bosnia and Herzegovina) is an important economically and ecologically problem. The sludge samples are mostly composed of goethite-FeO(OH) and quartz, less clay minerals (mostly illite-sericite), and the least part is formed of magnetite and todorokite [1, 2].

One of the most investigated techniques for removing of gangue from fine iron ores is selective

flocculation [2-11]. Generally, flocculation is the process in which fine particles, dispersed in water or other liquid, aggregate under the influence of binding compounds called flocculants [12]. A flocculant molecule bind particles into aggregates, after adsorption of different parts of a flocculant chain simultaneously on several particles. Newly formed aggregates of fine particles, called flocs, have considerably larger size than initial particles, so due to gravity forces, they settle much faster than single particles [2]. Selective separation of mineral components is achieved in flocculation process by addition of specific flocculants with/without dispersants and manipulation of solution conditions (pH, solid /liquid ratio, a flocculant dose) to produce different surface properties for mineral components. The flocculation behaviour of mineral components depends on the surface properties. Selective flocculation studies of iron ore were carried out using different flocculants, such as starch [13-17], polyacrylamide [18-22], oleate [23-27], etc., but mostly on hematite. The characteristics of ores/slimes from different localities differ substantially and the knowledge about fine goethite ore processing is still relatively poor [28-30].

The reason for this research is that the selectivity of anionic polyacrylamide has not been achieved in the flocculation of waste sludge from the Omarska mine.

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These results suggest that it is necessary to further examine the individual components and their behavior in a complex system such as sludge, in order to determine the best parameters (conditions, reagents, etc.) for selective flocculation [2, 32, 33].

This paper presents a part of research of individual components of waste sludge from the Omarska mine. The polyacrylamide and sodium oleate efficiency in goethite removal from aqueous solutions was investigated on a sample of natural goethite from Omarska mine. In order to compare of the two flocculants, a study of the flocculation goethite suspensions using anionic polyacrylamide and sodium oleate as flocculants was made by determining the settling rates of the suspensions and the amount of suspended solids. All experiments were performed as a function of the pH value and flocculant dosage.

## 2. Materials and Methods

### 2.1. Materials

#### 2.1.1. Goethite

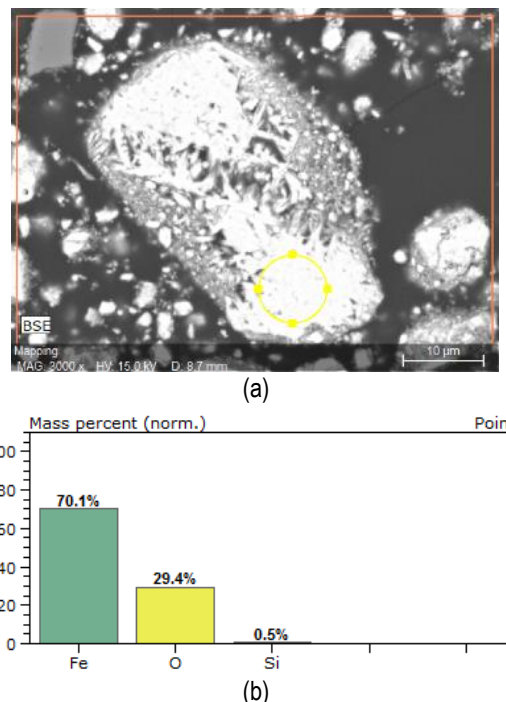
Representative iron ore from Omarska mine, labeled as "limonite ore" was handpicked. The "limonite ore" samples are mostly composed of goethite -  $\text{FeO}(\text{OH})$  which dominate over hematite ( $\text{Fe}_2\text{O}_3$ ), and with minor contents of magnetite ( $\text{Fe}_3\text{O}_4$ ), quartz ( $\text{SiO}_2$ ) and clay minerals. The XRPD, FTIR, and SEM-EDS analyses were recently performed for obtaining more precise mineral determination [31-33]. The density of the sample was also determined:  $3.940 \text{ g/cm}^3$ . Chemical composition obtained by wet analysis of natural goethite from Omarska mine is given in Table 1 and typical SEM image and chemical composition obtained by X-ray microanalysis (EDS) from selected point is given in Figure 1.

**Table 1** Chemical composition of the goethite from Omarska mine

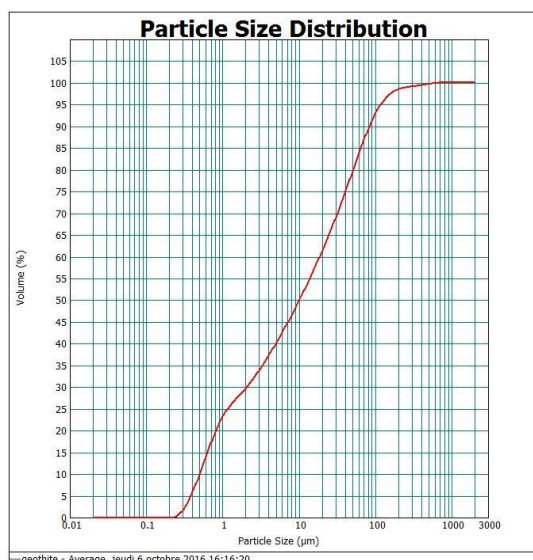
Sample	Chemical compositions (in mass %)					
	Fe	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	CaO	MgO	$\text{TiO}_2$
Goethite	57.16	4.44	0.59	0.03	0.22	<0.01

Sample	Chemical compositions (in mass %)					
	P	Mn	S	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$	LOI
Goethite	0.078	1.18	0.007	0.009	0.033	10.86



**Figure 1** SEM image of goethite ore from Omarska mine (a) with EDS data of selected point (b)



**Figure 2** Particle size distribution of goethite

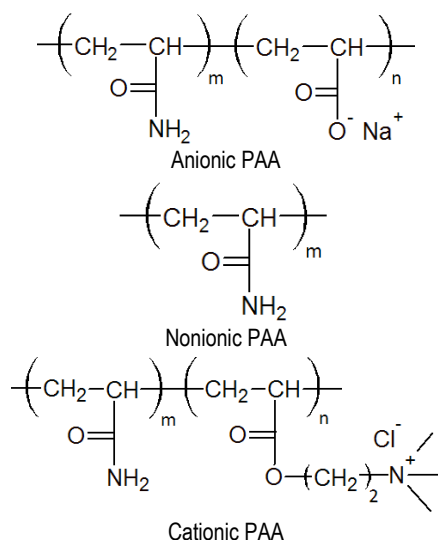
The result of particle size distribution analyzes of the goethite sample is given in Figure 2. It is clearly seen that: 80% was below  $50 \mu\text{m}$ , 65% was below  $25 \mu\text{m}$  and the highest mass percentage (about 50%) belonged to

the finest classes, i.e. to those below 10  $\mu\text{m}$ . This confirmed that the limonite ore from Omarska was fine-grained, which was one of the reasons for the large amount of waste sludge.

### 2.1.2. Flocculants

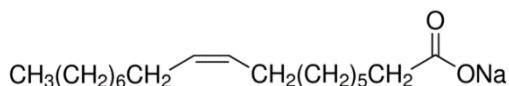
This work is a continuation of previous research, so the same reagents were used and the experimental procedure was the same, as previously described [2].

Polyacrylamide and its derivative are widely used as thickening agent, super absorbent, soil conditioner, flocculating agent, suspending agent, etc. There are three types of PAM, cationic, nonionic, and anionic (Figure 3). Flocculation with high molecular weight polymers was formed by the association of particles due to the adsorption of molecules or micelles of polymers (flocculants) which with their hydrocarbon radicals form, as already mentioned, "bridged" between particles [12].



**Figure 3** Polyacrylamide structure

Sodium oleate (cis-9-octadecenoic acid salt) is already a well-known collector in flotation of iron ore, especially hematite. Also, the use of sodium oleate as a flocculant is known in the literature. Oleate can act as a flocculant over a wide pH range depending on its concentration [25-26].



**Figure 4** Structure of sodium oleate

## 2.2. Methods

### 2.2.1. Settling and flocculation studies

The natural settling, and settling with the addition of sodium oleate (SO) and polyacrylamide (PAM) as flocculant, were determined. The flocculation experiments were performed at a pulp density with 13.8% of solid, which was adequate to the simulation of natural industrial conditions. To examine the effect of flocculant dosage the following were used: (i) Beaker of 100 ml volume, (ii) 13.8 g of dry sample in 100 ml of distilled water for the experiment; (iii) 250 g/t, 500 g/t, 1000 g/t of sodium oleate (SO), and (iv) 50 g/t, 100 g/t, 250 g/t of PAM. To compare the efficacy of two flocculants the following were used: (i) Beaker of 100 ml volume, (ii) 13.8 g of dry sample in 100 ml of distilled water for the experiment; and (iii) 250 g/t of SO and 250 g/t of PAM. All of the tests were performed at the pH=4, pH=7, and pH=10.5. For flocculation tests, the sample was mixed for two minutes. After adjusting pH value, the suspension continued to mix for two minutes. After addition of flocculant, sample was mixed for five minutes, and left to settle for one minute, separated float from sink, dried both of them at 105° C in oven and weighed the sediment material [2, 32, 33].

### 2.2.2. Zeta Potential Measurements

The electrokinetic (zeta) potential of minerals was determined by electrophoresis, by measuring the electrophoretic mobility of mineral particles with a Malvern Zetasizer Nano Z device. The Zetasizer Nano Z system is intended for measuring zeta potential and electrophoretic mobility in aqueous and organic dispersions using the laser effect of Doppler microelectrophoresis. Laser wavelength was 633 nm. Preparation of minerals for testing was performed under the following constant conditions:

- Mass of the mineral sample in each individual experiment 0.010 g, and
- Volume of solution in which conditioning was performed 100 ml.

## 3. Results and discussion

### 3.1. Settling and flocculation studies

In solid-liquid systems, the settling velocity of the solids depend on size and shape of the grain, the fluid

and grain density, charge density, etc. The highest mass percentage (about 50%) of investigated sample belonged to the finest classes, i.e. to those below 10  $\mu\text{m}$ . As expected, decreasing a particle's size reduced its settling rate. For that reason, it is necessary to produce more rapid settling of fine particles by using flocculation technique.

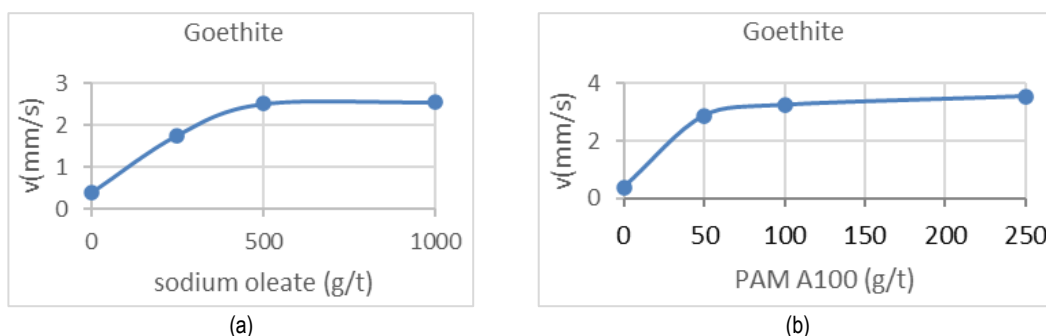
The main goal of settling and flocculation studies was to compare the behavior of natural goethite samples with or without two different flocculants (anionic polyacrylamide A100 and sodium oleate, SO). These experiments aimed to demonstrate the possible impact of different input flocculants on the behavior of settling rate on different pH value.

### 3.1.1. Influence of the type and dosage of flocculants on settling rates

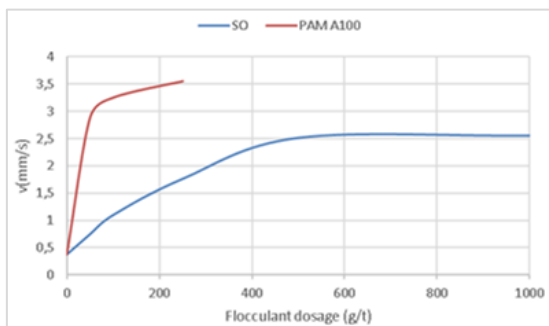
The effect of two flocculants was studied by comparing critical rates. The critical rate ( $V_{av}$ ) in this paper was calculated in the same way as we described in a previous paper [2].

The usual doses for each flocculant were used, taking into account the maximum doses recommended for each of them. The effect of different flocculants was studied by conducting settling tests at pH 7, which was an average pH value in real conditions.

Settling rates of the goethite suspensions depending on the dosage of two different flocculants are shown in Figure 5 (a) and (b) and Figure 6.



**Figure 5** Settling rates of the goethite suspensions depending on the amount added flocculant: sodium-oleate (a) and PAM A100 (b)

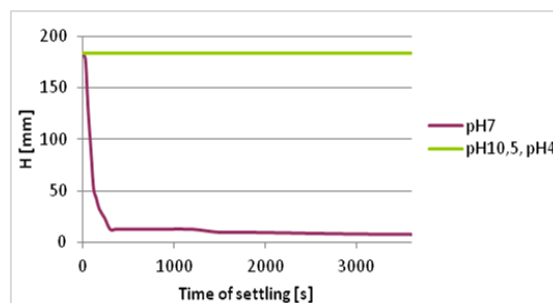


**Figure 6** Settling rates of the goethite suspensions for two flocculants comparatively

### 3.1.2. Influence of pH

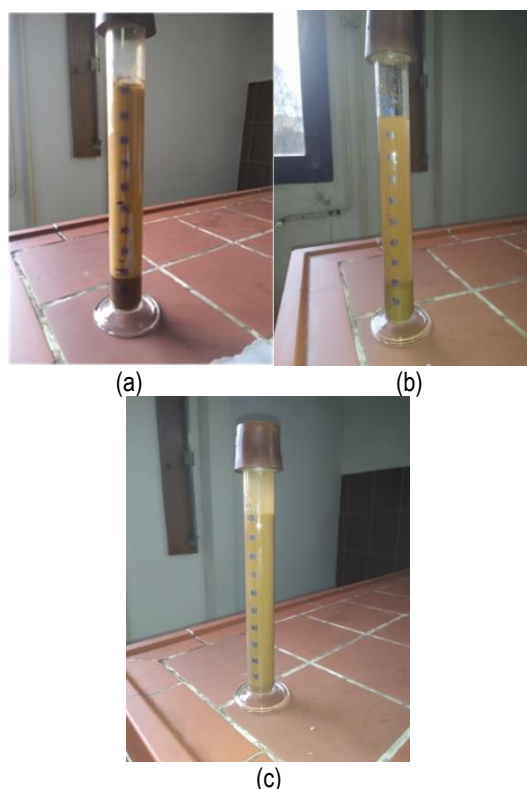
Generally, the stability of the metal oxide suspension strongly depends on the pH value, because their surface charge becomes increasingly positive as the pH decreases and becomes increasingly negative as the pH increases. The charge on most oxides in

aqueous systems is controlled by the acid-base chemistry of surface oxide and hydroxide species. The stability of the goethite suspension without flocculants strongly depends on the pH value. This is clearly seen in Figure 7 in which shows natural settling curves of goethite at different pH values. There was no settling at either pH 4 or pH 10, because the increase of surface charge caused the repulsive forces.



**Figure 7** Natural settling of goethite at different pH values [32]

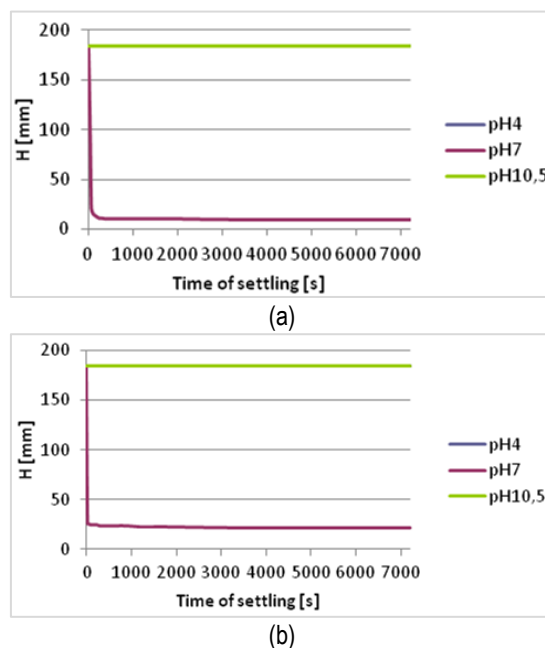
Influence of the type of flocculants on settling behavior of goethite depending on pH was also investigated. As an illustration, Figure 8 shows settling experiments with goethite in the presence of anionic polyacrylamide A100 at different pH values.



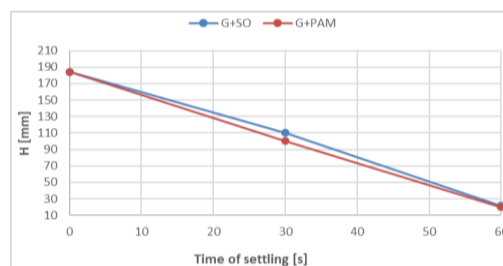
**Figure 8** Illustration of Goethite + PAM A100 settling at: pH=4 (a); pH=7 (b); (c) pH=10.5

Settling tests of goethite with different flocculants and different pH showed that it was not possible to monitor the deposition because the column of sediment was turbid, i.e. no clear deposition could be seen. At pH value of 7, it was possible to determine the settling time because the water column was clearer and the settling was visible.

The effect of flocculants was also highly dependent on pH (Figure 8 - 9), but there was no difference in the behavior of the suspension, regardless of used flocculant. Since in both cases the settling occurred very quickly (within 60 seconds) at pH=7, a slight difference between the action of the two flocculants was seen in this interval (Figure 10). Settling tests with additional different flocculants at different pH showed that none of the sample was precipitated at pH=4 and pH=10.5.



**Figure 9** Flocculation settling of goethite at different pH values: (a) with sodium-oleate; (b) with PAM A100

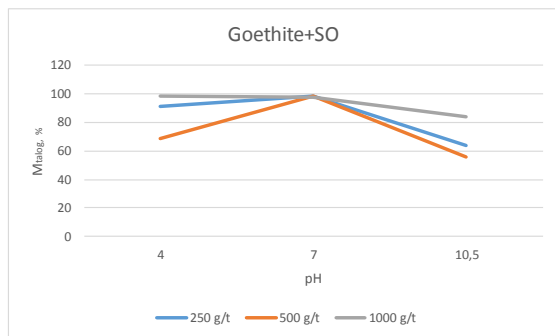


**Figure 10** Flocculation settling of goethite at pH 7 for two flocculants comparatively

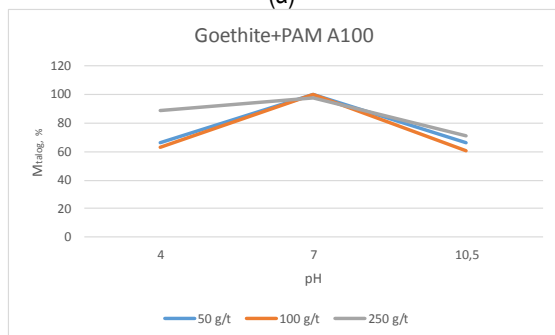
Since the positive charge of the goethite decreased with increasing the pH value, the occurrence of inefficient action of both flocculants in an alkaline medium was understandable. From the other side dependence of flocculation on pH could be due to the effect of pH on the surface characteristics of goethite in water as well as that on the anionic polyacrylamide and sodium oleate solution chemistry. Namely, for anionic polyacrylamide and sodium oleate, in the base medium, anionic species were predominate as well as for goethite. The settling in a neutral medium occurred because in the neutral pH (range of about 5-8) neutral surfaces predominated, both in goethite and in both flocculants (the un-dissociated oleic acid and weakly dissociated polyacrylamide).



A comparison of the amounts of suspended solids as a function of pH showed that the highest amounts were expected at pH=7 in both cases regardless of the dosage of flocculants (Figure 11-12).

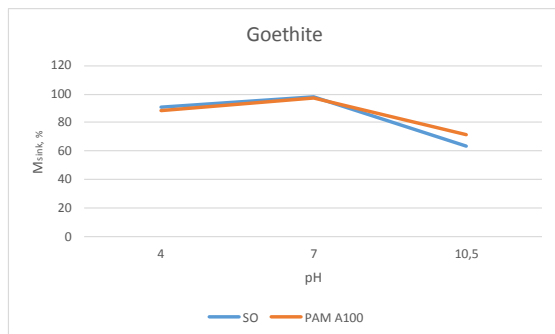


(a)



(b)

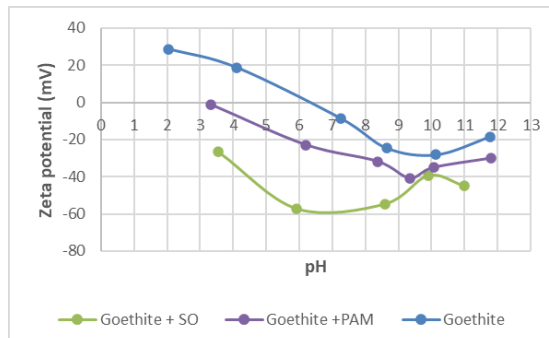
**Figure 11** Amount of suspended solids with different dosage of: sodium oleate (a); PAM (b)



**Figure 12** Amount of suspended solids with dosage of 250 g/t with sodium oleate and PAM A100 comparatively

### 3.2. Zeta potential study

The zeta potential of the goethite samples in presence/absence of two different flocculants is shown in Figure 13.



**Figure 13** Zeta potential of the goethite samples in presence/absence of different flocculants

The electro kinetic (zeta,  $\zeta$ ) potential of minerals is a potential at the hydrodynamic shear plane of the double electric layer mineral/solution. As a measure of the magnitude of the charge repulsion/attraction between particles, it is one of the most important parameters known to affect flocculation behavior. Isoelectric point (IEP) represents condition when value of zeta potential is zero. As a rule,  $\pm 30$  mV is taken as the limit value of the zeta potential required for dispersion stability. Namely, at that condition, the electrostatic repulsion between particles is typically strong enough to prevent particles attraction.

Zeta-potential curves for natural mineral raw sample of goethite was previously determined [2,32] and the IEP for natural goethite was observed at pH=6.6. The magnitude of the positive zeta potential goethite was maximized at pH=2 value, and the magnitude of the negative zeta potential was maximized at pH=10 value. That corresponded with settling behavior of goethite (Figure 7), because a higher zeta potential produced a stable dispersion. At pH 7 fast settling of goethite particles occurred. After interacting with flocculants, the changes in zeta potential of goethite surface were obvious, indicating that the both flocculants did interact with the goethite surface causing an increase of negative zeta potential magnitudes in both of the investigated cases, as it can be seen from Figure 13. Also, it can be seen that the surface charge remained negative throughout all of the investigated pH values, after addition flocculants. The difference of the zeta potentials of goethite after interaction with used flocculants indicated a more pronounced flocculation for goethite with PAM than for goethite with sodium oleate. That corresponded with settling rate comparison shown in Figure 6, where a significantly greater effect of PAM on the settling rate of goethite could be seen.

#### 4. Conclusion

The anionic polyacrylamide and sodium oleate efficiency in goethite removal from aqueous solutions was investigated on a sample of natural goethite from Omarska mine.

Comparison of the critical settling rates showed that the addition of the flocculant increased critical rates in both cases, but more significant in the case of using PAM A100.

The natural settling behavior of the goethite without flocculant strongly depended on the pH value, because their surface charge in aqueous systems was controlled by the acid-base chemistry of surface oxide and hydroxide species. There was not settling at either pH=4 or pH=10, because the increase of surface charge caused the repulsive forces.

The settling behavior of the goethite with additional flocculant was influenced more by the type of flocculants and pH value, than by the dosage of the flocculant.

The effect of anionic polyacrylamide and oleate as a flocculants was highly dependent on pH. This was expected because the species distribution of sodium oleate and the charge density of ionic polymers also depended on the pH conditions. Effect of anionic polyacrylamide and sodium oleate on the flocculation of goethite was noticeable at pH=7 only. A comparison of the amounts of suspended solids as a function of pH showed that the highest amounts were expected at pH=7 in both cases regardless of the dosage of flocculants.

After interacting with flocculants, the changes in zeta potential of goethite surface were obvious, indicating that the both flocculants did interact with the goethite surface causing an increase of negative zeta potential magnitudes in both of the investigated cases. The difference of the zeta potentials of goethite after interaction with used flocculants indicated a more pronounced flocculation for goethite with PAM than for goethite with sodium oleate.

The highest settling rate of goethite and the highest amount of suspended solids were achieved under the following conditions: at pH=7 and the addition of anionic polyacrylamide with dosage of 250 g/t.

In relation to selectivity, our unpublished results indicate greater selectivity of oleates, but the conclusion can be made only when all research with anionic polyacrylamide on other components of sludge (quartz and clay sample from Omarska mine) is completed.

#### Note

A part of this study was presented at the XIV International Mineral Processing and Recycling Conference, organized by the University of Belgrade, Technical Faculty in Bor, from 12<sup>th</sup> to 14<sup>th</sup> May 2021, Belgrade, Serbia.

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## POREĐENJE UTICAJA POLIAKRILAMIDA I NATRIJUM-OLEATA NA SELEKTIVNU FLOKULACIJU GETITA IZ TALOGA

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### Izvod

Ovaj rad predstavlja deo istraživanja sprovedenog sa ciljem da se ispita mogućnost primene selektivne flokulacije getita iz taloga koji nastaje tokom procesa proizvodnje rude gvožđa. Cilj istraživanja je bio da se uporedi uticaj poliakrilamida i natrijum-oleata, koji su korišćeni kao flokulanti, na taloženje getita pod različitim uslovima. U radu su prikazani rezultati istraživanja uticaja flokulanata na bazi poliakrilamida i natrijum-oleata, kao i pH vrednosti za brzinu taloženja prirodnog uzorka getita. Rezultati su pokazali da na brzinu taloženja getita utiču pH vrednost i vrsta flokulanta, dok koncentracija flokulanta nije imala značajan uticaj na brzinu taloženja. Merenje zeta potencijala je pokazalo da postoji dejstvo flokulanta na površini getita.

**Ključne reči:** Getit; Flokulant; Brzina taloženja; Zeta potencijal.