FLOCCULATION STUDY OF NATURAL QUARTZ SAMPLE USING ANIONIC POLYACRYLAMIDE

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Abstract: The waste sludge generated during processing of iron ore in the Omarska mine (The Republic of Srpska, Bosnia and Herzegovina) is fine-grained (<15μm), containing relatively high concentrations of iron, and quartz as its major impurity. The flocculation behaviour of the primary natural raw "quartz" sample from Omarska mine was studied in the present paper. This sample is composed of major quartz which dominates over minor contents of clay minerals and feldspars, and contain 92.9% of SiO_2. Particle size distribution analysis confirm that it is present as fine and ultra-fine particles. The zeta potential of quartz depends on pH value. Settling experiments were performed by using three different dispersants (Na-hexamethaphosphate, Na-pyrophosphate and Na-silicate), and anionic polyacrylamide as flocculants. The best results were achieved with Na-hexametaphosphate (1000 g/t) and anionic polyacrylamide A 100. The effect of a flocculant on the settling rate depends on solid concentration. Settling rates increase significantly with the increase of the liquid component in both cases (natural settling and hindered settling by addition of a flocculant).

Keywords: Quartz, iron ore, sludge, flocculation, settling rates.

1. INTRODUCTION

Utilization of low-grade ores or waste materials from mineral processing has become important today, because high-grade ores have already been largely exploited in the past. The problem is that they are very complex systems, composed of several intimately mixed minerals, and usually with very fine particles. Fine and ultrafine particles cannot be separated by using conventional methods, such as gravity concentration and magnetic separation. The waste sludge generated during processing of iron ore in the Omarska mine (The Republic of Srpska, Bosnia & Herzegovina) is fine-grained (<15μm), containing relatively high concentrations of iron, and with quartz as its major impurity. Namely, our previous results showed that sludge samples are composed of major goethite-FeO(OH) and quartz, less clay minerals, and minor magnetite and todorokite [1].

Due to the very slow settling of fine and ultrafine particles, the particles need to be coarsened to produce more rapid settling rate. Selective flocculation is one of the promising techniques applicable to the separation of sludge fraction [2-15]. This technique is based on the preferential adsorption of an appropriate reagent on the mineral particles to be flocculated. The disadvantage of this technique is the difficulty of assuming a selective action on the desired minerals in complex systems such as mixtures of natural minerals, because hetero-coagulation often occurs in such systems [16–17]. The selective separation is difficult and depends on the individual components of the sludge and their behaviour [18–21].

Our earlier results of sludge testing with anionic polyacrylamide as the flocculant have shown that, despite to the good results regarding the stability of dispersion and flocculation, selectivity has not been achieved [22]. Therefore, our present research has been focused at the physic-chemical properties of individual components of sludge. At such way, we recently also studied natural raw „clay" sample [23].

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The aim of this study was to examine the chosen natural minerals that have been previously identified from the primary natural raw "quartz" sample I from Omarska mine, i.e. major quartz which dominantes over minor contents of clay minerals (of illite-sericite – kaolinite composition) and feldspars [24], and their behaviour in the presence of different dispersants and anionic polyacrylamides as flocculants. The dispersion-flocculation properties, such as: natural settling; settling with the addition of a flocculant; effect of solid concentration; effect of the addition of different dispersants concentration and zeta potential variations were studied. The study of the mineral composition and its connection with some properties important for the quartz flocculation process, was processed.

2. MATERIALS AND METHODS

2.1. Materials

In this paper a primary natural raw material sample from Omarska mine, labeled as „quartz I" [24] was studied. It has the following mineral composition: major quartz (~91%) which dominates over clay minerals of illite-sericite – kaolinite composition (~6%) and feldspars (~3%); with chemical composition (in mass %): SiO_2 92.90, Al_2O_3 3.28, Fe 0.74, LOI 1.62; and density of 2.663 (g/cm^3).

All reagents used were of analytical grade, and they were prepared as solutions in distilled water. The sodium hexametaphosphate (SHMP), sodium pyrophosphate (SPP) and sodium silicate (SS) manufactured by Lach-Ner, s.r.o. (Czech Republic), were used as dispersants. Anionic polyacrylamide (PAM) type SUPERFLOC A100, manufactured by Kemira, was used as the flocculant.

Preparation of the flocculant for all experiments was carried out in the same way according to the instructions provided by the manufacturer of reagents [25]. 0.1M NaOH was used as pH modifier.

2.2. Methods

2.2.1. Particle size distribution analysis

Particle size distribution of the sample was performed using Malvern analyser in the laboratory Global Research and Development, Mining and Mineral Processing, Maizières-lès-Metz, France. Data were obtained on Mastersizer 2000, at dry and wet samples.

2.2.2. Settling dispersion and flocculation experiments

The natural settling, and settling with the addition of dispersant and the flocculant, were determined. The natural settling was examined at 3 different pH values (4, 7 and 10.5). Also, different solid/liquid relationships (marked as S:L) of: 1:7; 1:9.7; and 1:19.7 were used in the same conditions. For settling, graduated glass cylinder with volume of 100 ml was used. The flocculation experiments were performed at a pulp density with 3 different solid/liquid ratios (i.e.: 1:7; 1:9.7; and 1:19.7), at the pH=7, which is average pH value in real conditions.

For the flocculation tests it was used: (i) Beaker of 1000 ml volume for better visualization of the dispersion; (ii) 69 g of dry sample in 500 ml of distilled water for the experiment (which is adequate to the simulation of natural industrial conditions); (iii) 30 and 50 g/t of the flocculant A100; and (iv) 50, 100, 200 and 1000 g/t of dispersants SHMP and SPP; and 300, 500 and 1000 g/t of SS. The effect of different dispersants on the stabilization of system was studied by conducting the dispersion tests in 1000 ml graduated glass cylinder. Suspension of 12.5 % solids by wt. was mixed for 2 minutes, while maintaining the pH at 10.5 value. Desired dispersant dose was added to the suspension, mixed five minutes, and left to settle. It was separated float from sink, after ten minutes, and dried both of them at 105° C in oven. For flocculation tests, the sample was mixed for two minutes. After adjusting pH value at 10.5, the suspension continued to mix for two minutes, and after that, a dispersant was added. The suspension was mixed up for another five minutes. After addition of the flocculant, sample was mixed for five minutes, and left to settle for one minute, separated floating from sinking part, and dried both of them at 105° C in an oven.

2.2.3. Zeta potential measurements

Zeta potential measurements were performed using a ZM3-D-G meter, Zeta Meter system 3.0+, with direct video imaging from Zeta Meter Inc., USA; at Universidad Federal de Minas Gerais, Belo Horizonte, Brazil. The measurements were carried out according to the following procedure: samples were classified through the sedimentation in test tubes of 250 ml, with a mineral concentration of 80 mg/l to reach a particle size below 10 μm. Distilled water was used during the sedimentation procedure

with the natural minerals, and the dispersant reagents solutions were used for the remaining sedimentation tests. The pH of the mineral suspensions, with or without dispersant reagents, was adjusted at the beginning of the sedimentation procedure. Before each test, the completely opened Zeta Meter cell was first washed intensively with tap water, and after that with distilled water. Before each measurement, the platinum and the molybdenum electrodes were washed with distilled water. The voltage used in the test was always the highest possible voltage that did not generate vortex due to the heating of the suspension during the measurements.

3. RESULTS AND DISCUSSION
3.1. Particle size distribution analyzis

In the flocculation process of the fine particles, the size diameter range has a significant role. So called "fine sludges" have particles smaller than 10 μm. The result of particle size distribution analyses of the quartz sample is given in Figure 1. It is clearly seen that high mass percentage (about 50%) belongs to the finest classes, i.e. to those bellow 9 μm. Furthermore, almost 80% of the sample belongs to the classes bellow 25 μm. This data indicates that the quartz is in a stable suspension, and the solid phase is in a dispersed state under the influence of molecular forces. Because of very slow settling of such fine particles, the particles need to be coarsened to produce more rapid settling rate.
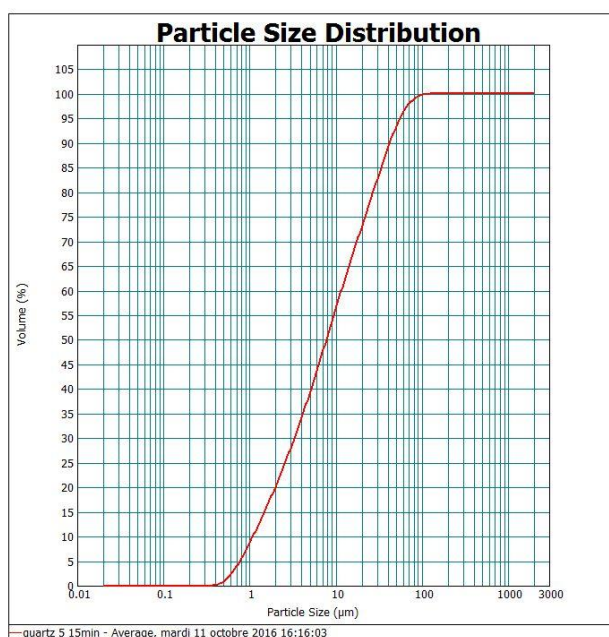

Figure 1. Particle size distribution of quartz

3.2. Settling, disspersion and flocculation studies

One of the basic parameters for the selective flocculation process is dispersive and flocculating behaviour of the individual components of the system. A prerequisite for successful flocculation is good particle dispersion. The main goal of initial settling, dispersion and flocculation studies was to obtain the behaviour of natural quartz sample, defined as the major impurity in sludge from Omarska mine. The experiments were conducted on the system with different S/L ratios, with different dispersants, and with or without the flocculant, as well.

These experiments aim to demonstrate the possible impact of the different input parameters on the behaviour of settling rate: naturally and also in the presence of different dispersants and flocculants. Furthermore, effect of solid concentration was examined by using different solid/liquid ratios.

3.2.1. Natural settling at different pH and with different S/L ratios

The settling rate depends on size and shape of the grain, the fluid and grain density, and in particular, the questions related to the flocculation or dispersion of the smallest classes resulting from the charge of the grain or Braun's motion. At the beginning of precipitation, in diluted pulp and under free fall conditions, deposition rate is relatively large and constant without interference from nearby particles (unhindered settling). As the concentration of particles in a suspension is increased, particles are closer together and they no longer settle independently of one to another. There is also an influence of settling particles on flow of liquid. This results in a reduced particle-settling velocity and the effect is known as hindered settling.

In Figure 2, natural settling curves of quartz at different pH values are shown. The effect of pH value is clearly seen on the settling behaviour of quartz. Quartz particles do not precipitate at pH=10, because of increasing of surface charge due to electronegative SiO⁻ groups at quartz surfaces. This phenomenon causes an increasing of the repulsive forces.

Natural settling of quartz with different ratios of S:L were examined at the pH=7, which is average pH value in real conditions. The settling curves are shown in Figure 3. Generally, there is no big difference in settling behaviour between these three different solid/liquid ratios. At the beginning, settling rate is rapid in all three cases. Also, samples

with the smallest solid/liquid ratios have the highest settling rates, which is in accordance with some other studies [20,22,23,26].
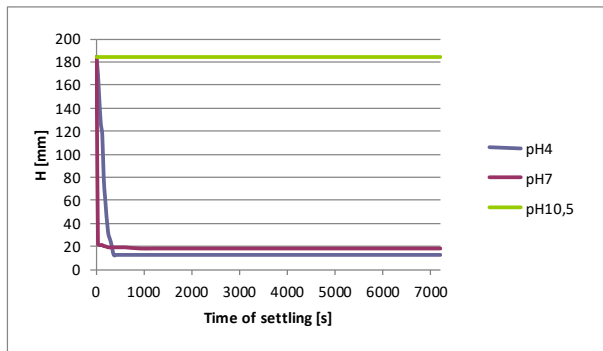

Figure 2. Natural settling of quartz at different pH values
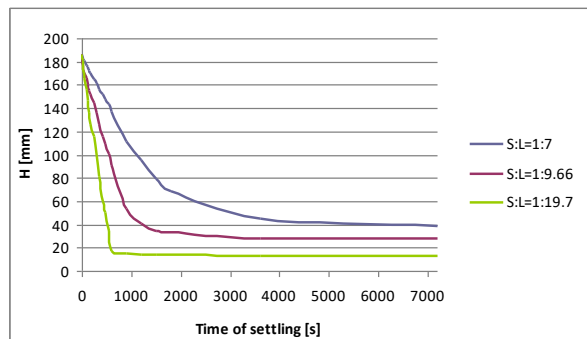

Figure 3. Natural settling of quartz with different solid/liquid ratios

3.2.2. Settling with different S/L ratios with an additional flocculant A100

In Figure 4, settling curves with different S/L ratios, and with an additional anionic flocculant A100, are shown. For all three different ratios, the effect of the flocculant addition can be clearly seen,

because the quartz particles are settling much faster than the natural settling (Figure 3), and regardless to the S/L ratio. The settling starts only after 30 seconds from the beginning.
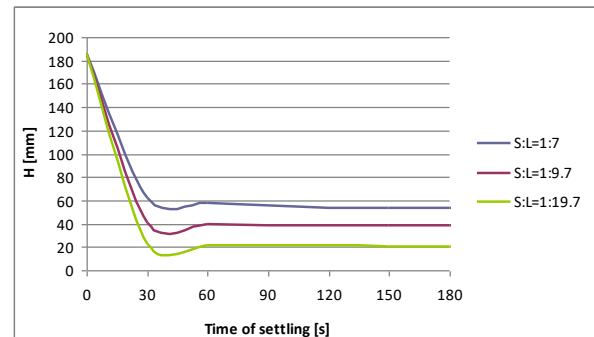

Figure 4. Flocculation settling of quartz with an additional flocculant A100 and with different solid/liquid ratios

Critical and hindered settling rates of quartz sample are shown in Table 1. The critical rate (V_{av}) in this paper is calculated as: $V_{av} = H/t_{cr}$ (mm/s), where H is height of the clear zone during critical time, and t_{cr} is deposition time required to achieve a critical deposition point. The hindered settling rate was calculated according to Richardson-Zaki equation [26]. This equation describes a method of calculating the sedimentation rate in a liquid-solid system as a function of the free falling rate of a single particle (terminal settling) and the concentration of particles. From these results, it can be seen that samples with the smallest solid/liquid ratios have the highest settling rates in both cases. With the addition of the flocculant, these rates increase significantly with the increase of the liquid component in both of the samples.

Table 1. Critical and hindered settling rate (mm/s) of quartz

S:L (% of Solid)	Quartz			
	natural settling mm/s	settling with flocculant mm/s	terminal settling mm/s	hindered settling mm/s
1:7 (12.5)	0.071	4.130	0.570	0.446
1:9.7 (9.4)	0.120	4.800	0.570	0.477
1:19.7 (4.8)	0.280	5.400	0.570	0.522

3.2.3. Dispersion studies

A prerequisite for successful flocculation is good particle dispersion. The dispersion studies were carried out with different inorganic reagents: sodium hexametaphosphate (SHMP), sodium-pyrophosphate (SPP) and sodium-silicate (SS) with dosage of

SHMP and SPP of 50, 100, 200 and 1000 g/t; and with 300, 500 and 1000 g/t of SS. Due to the close similarity of the preliminary results between dispersants SHMP and SPP, we decided to exclude the results with dispersant SPP from this study. Dispersion behaviour of natural quartz sample was tested by monitoring the weight distribution and

elemental composition determination in floating and sinking parts. The results of the distribution of masses after one or two grinding and elemental composition between sinking and floating parts are presented in Figure 5 and Table 2. Several conclusions can be drawn from the obtained results. First, both dispersants significantly increase the mass fraction of the floating part (better dispersion).

This is especially pronounced after the second milling. Out of the two other reagents studied, it is seen that there are no big differences between SHMP and SS in the amount of sinking and floating parts. According to the results obtained, 1000 g/t sodium hexametaphosphate consumption was used in further studies.
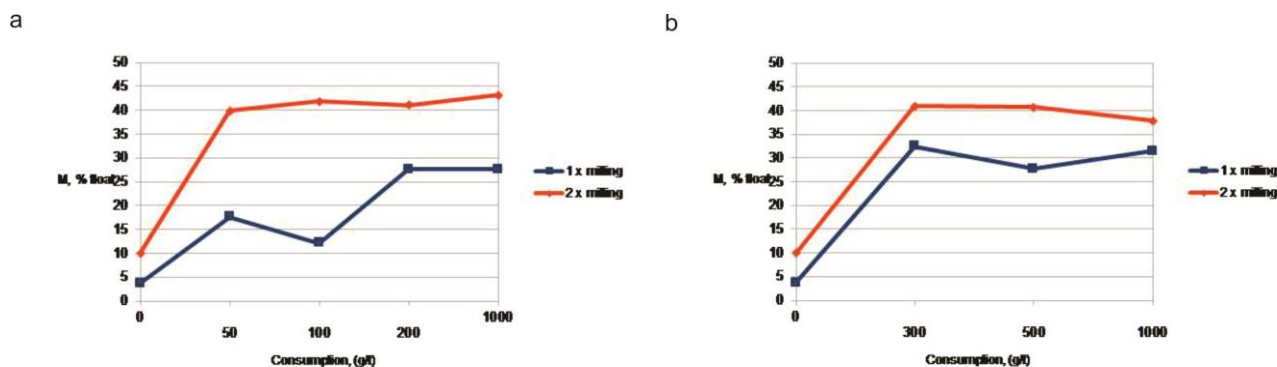

Figure 5. Quartz with dispersants: a) SHMP; and b) SS

Table 2. Elemental composition of natural quartz (in %) in presence of SHMP and SS dispersants (in g/t)

Element		SHMP 2xmill				SS 2xmill		
		50	100	200	1000	300	500	1000
SiO_2	Float	87.6	87	86.9	88.1	88.4	88.1	87.2
	Sink	94.7	95.2	94.9	95.4	95.5	95.2	95
Al_2O_3	Float	5.56	5.64	5.98	5.26	5.3	5.34	5.64
	Sink	1.80	1.62	1.74	1.76	1.84	1.8	1.82
Fe	Float	0.93	0.95	1.06	0.9	1.03	0.95	0.98
	Sink	0.87	0.83	0.83	0.84	0.89	0.81	0.75
Na_2O	Float	0.964	0.988	0.77	0.889	0.759	0.772	0.847
	Sink	0.127	0.102	0.118	0.103	0.11	0.099	0.11
K_2O	Float	0.852	0.865	0.894	0.806	0.813	0.815	0.857
	Sink	0.28	0.255	0.271	0.276	0.282	0.279	0.284
MgO	Float	0.23	0.24	0.27	0.23	0.23	0.24	0.24
	Sink	0.08	0.07	0.08	0.08	0.08	0.08	0.08
LOI	Float	2.87	3.27	2.83	2.71	2.7	2.63	2.7
	Sink	0.89	0.82	0.79	0.79	0.88	0.77	0.87

The elemental composition from Table 2 shows that the distribution of SiO_2 is similar in floating and sinking parts, while the largest difference is in the content of other elements, which probably originate from clay minerals (i.e. Al_2O_3, Na_2O, K_2O and MgO). This indicates that the clay's particles, as impurities of natural quartz sample, almost completely flow into the floating part.

3.2.4. Flocculation studies

The flocculant was selected at the basis of the earlier initial flocculation study of the sludge from

Omarska mine [22]. In this Chapter the influence of different consumption of the flocculant on flocculation behaviour of quartz was tested, as one of the individual components of the sludge. Initially, flocculation behaviour was tested by monitoring the weight distribution in floating and sinking parts. Interestingly, after the addition of the flocculant all the mass was in the sinking part. For those reasons, there is no elemental analysis of floating and sinking parts after the addition of the flocculant.

According to the earlier clay behaviour study [23], an increase of the flocculant consumption causes an increase in the sinking part, but elemental

analysis showed that clay-derived elements are more prevalent in the floating part, whereas quartz prevails in the sinking part. Given these observations, we can assume that, under alkaline conditions, A100 is a more efficient flocculant for quartz than for clay. This may be important information regarding the behaviour of the two basic impurities in the Omarska sludge for the selective flocculation process.
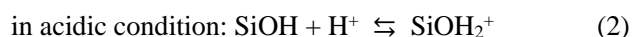
3.3. Zeta potential study

In this Chapter we discuss the charging behaviour of natural quartz mineral, as for the quartz itself, as well as for the quartz in the presence of the different surfactants, which can be used to provide useful information about the quartz surface properties. This study was done by measuring of zeta potential in the various combinations of absence/presence of the three dispersants: sodium-hexametaphosphate (SHMP), sodium-pyrophosphate (SPP), sodium-silicate (SS); and also in the absence/presence of the anionic polyacrylamide A100 (PAM) flocculant, as well.

A good particle dispersion is a prerequisite for the mineral separation process. The stability of dispersion of fine grained minerals is directly dependent on zeta potential (ζ-potential) of particles. Zeta (ζ) potential is a measure of the magnitude of the electrostatic or charge repulsion/attraction between particles. The higher ζ-potential's absolute value produces a well-dispersed suspension. Isoelectric point (IEP) represents condition when the value of zeta potential is zero. The pH value corresponds to the presence of equal numbers of oppositely charged groups on the mineral surface. Particles with an IEP < pH=7 have acidic character, whereas those with an IEP > pH=7 are alkaline. When a metal oxide is placed in water, the surface of oxide will hydrate, resulting in the formation of metal hydroxide. Surface charges of metal oxides in water depends moderately on pH value. The concentration of OH⁻ and H⁺ ions in the solution effects the degree of the (-MOH) group dissociation, and thus determines the potential of the surface. According to well-known DLVO theory (Derjaguin, Landau, Verwey, Overbeek), the stability of a suspension of colloidal particles is determined by the balance between the electrostatic interaction and the van der Waals interaction between particles. Since the van der Waals interaction remains relatively constant in aqueous media, the stability of the system depends on the magnitude of the electrical repulsion [27-29].

It is well known that SiO_2 is an acidic oxide and the pH of an aqueous suspension of silica is slightly acidic. It depends on the crystal structure, atomic density of particles and presence of contaminants of the SiO_2 surface. When a quartz interacts with water, (-SiO) groups are hydrated and form (-SiOH) groups on the crystal edge surface:

$$2(\text{-SiO}) + H_2O \leftrightharpoons 2(\text{-SiOH}) \qquad (1)$$

The concentration of OH⁻ and H⁺ ions in the solution effects to a degree the (-SiOH) group dissociation, and thus determines the potential of the silicon surface. According to the zeta potential measurements observed, there is a development of a net particle negative surface charge as a function of increasing pH, because the charge density of the surface of the quartz in aqueous solutions is due to proton adsorption and desorption reactions of the surface hydroxyl groups, i.e. the silanol group, SiOH:

in acidic condition: $SiOH + H^+ \leftrightharpoons SiOH_2^+$ (2)

in alkaline condition: $SiOH + OH^- \leftrightharpoons SiO^- + H_2O$ (3)

An increase of pH (i.e. concentration of the OH⁻ ions), increases the silicon surface negative potential and thus stabilizes quartz in aqueous suspensions, which confirm the results obtained in Chapter 3.2.1., Figure 2.

Zeta-potential curves for natural mineral raw sample of quartz in absence/presence of three dispersants (SHMP, SPP and SS), are shown at Figure 6. The zeta potential curve for natural quartz is similar to various curves available in the literature [30]. The zeta potential is about 0 mV (IEP) for quartz at around pH=2.6, in the studied pH range in this paper of 2.1–11.8.
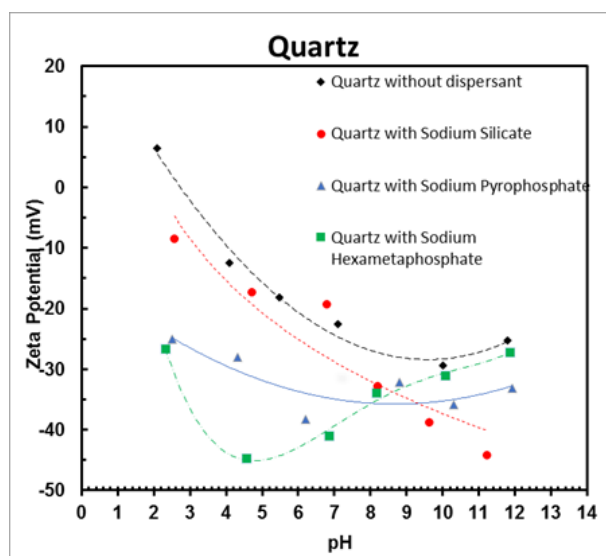

Figure 6. Zeta potential of the quartz samples in absence/presence of different dispersants

From Figure 6 it can be clearly seen that, despite the fact that the IEP of quartz with sodium silicate could be estimated close to pH=2, there is not an IEP for quartz when the poly-phosphates were used and the surface charge remains negative throughout the studied pH range. There exists a tendency for all of the curves to move to a lower negative charge region when dispersants are added. It can be noticed that quartz sample in presence of SHMP, shows a variation of zeta potential as a function of pH in the range from about -26.7 to -44.7 mV, in presence of SPP from about -25.0 to -38.2 mV, and in presence of SS from about -8.6 to -44.3 mV. The presence of SHMP causes low increase of negative zeta potential magnitudes, only from -29 mV (without SHMP) to -31mV (with SHMP), at pH=10. This pH value was chosen on basis of our previous research, because at pH=10 the biggest difference is between the zeta potential of quartz and other minerals present in the sludge from Omarska mine [22,25].

Specific adsorption of surfactant ions leads to surface charging and modification of surface properties. The energy barrier can be eliminated by neutralization of surface charge due to the adsorption of a suitable flocculant onto the particle surface and bridging between the particles. Flocculant adsorption can be measured by changing of the zeta potential after adsorption. If the polymer is adsorbed, there is a change in the surface of the particles, and thus the change in the zeta potential.
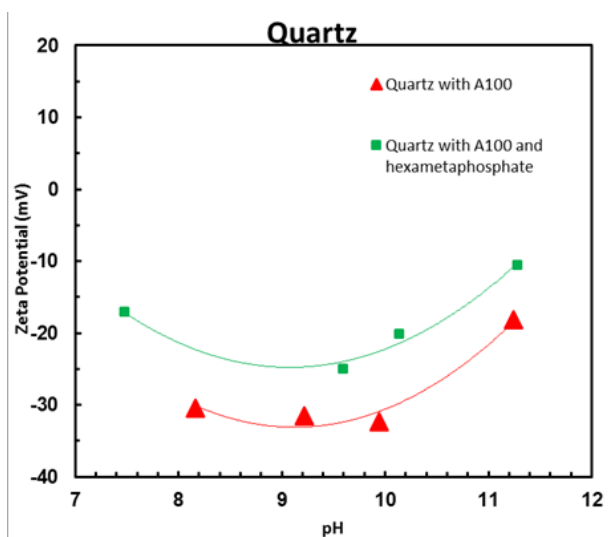

Figure 7. Zeta potentials of quartz: with PAM (A100); and with PAM (A100) and SHMP.

Figure 7 shows zeta potential curves as a function of pH for the same studied samples in presence of anionic polyacrylamide (PAM, A100), and with/without SHMP dispersant. It is obvious that the used anionic polyacrylamide (PAM) causes changes on the surface of the tested quartz sample. It is clearly seen that presence of the anionic PAM and SHMP significantly reduces the zeta potential in the highly alkaline region (about pH value of 10-11), indicating favourable conditions for flocculation. This means that the negative surfaces of quartz are covered with polymer molecules and that they have less negative surface charge than their initial conditions. Adsorption of polymer molecules on mineral particles is governed mainly by three types of bonding: electrostatic, hydrogen and covalent bonding, as well as their combinations. There is no evidence that the electrostatic character is dominant. It can be concluded from Figs. 6 and 7 that all quartz samples without/with reagents are negatively charged within the whole studied pH range, but the zeta potential value is relatively low, indicating favourable flocculation conditions. This agrees with the results of the flocculation tests (Chapter 3.2.4.).

We can also conclude that used anionic polyacrylamide is an applicable quartz flocculant, especially in highly alkaline medium. Also, adsorption of PAM on mineral particles is of combined character, less electrostatic and rather another type of bonding-probably hydrogen bonding between the polymer molecules and the hydroxylic groups on the surface of quartz minerals.

4. CONCLUSION

The settling behaviour of the natural quartz mineral from Omarska mine, which is important in dispersion and flocculation processes, was studied. Particle size distribution analyses have shown that quartz samples are present as fine and ultra-fine particles with sizes less than about 10 μm, which makes this system very complex for separation processes.

The rates of natural settling of the quartz depends on pH value and S/L ratios. An increase of pH (i.e. concentration of the OH$^-$ ions), increases the surface negative potential and thus stabilizes quartz in aqueous suspensions. So, at pH=10, there is no natural settling of quartz. Samples with smallest solid concentration of the quartz have a highest settling rates. The settling rate is obviously improved by using the anionic polyacrylamide flocculant. The effect of the flocculant depends both on the mineral composition and on the S/L ratios.

The dispersion tests show that all of the used dispersants cause the stable dispersion of the quartz, that they also depend on pH value, and the best

results were achieved with 1000 g/t consumption of dispersants.

According to the results of the flocculation tests, anionic polyacrylamide is an applicable flocculant of quartz. After the addition of the flocculant, all of the mass goes to the sinking part.

The influence of the mineral composition of the quartz on the surface charge can be seen from the zeta potential values. IEP of natural quartz was obtained at the pH values of 2.6. After the addition of dispersants, the surface charge remained negative throughout all of the studied pH values, and for all of the samples. The zeta potential values depend on the pH value. An increase of pH value (i.e. concentration of the OH^- ions), increases the silicon surface negative potential, and thus stabilizes quartz in aqueous suspensions, which confirms the results obtained in settling tests.

The presence of the anionic PAM A100 and SHMP as dispersants, significantly reduces the zeta potential and indicate favourable conditions for flocculation, particularly at pH range above 10-11. Adsorption of PAM on mineral particles is of combined character, less electrostatic and rather another type of bonding, probably hydrogen bonding between the polymer molecules and the hydroxylic groups on the surface of quartz minerals.

Finally, anionic PAM A100 is more efficient flocculant for quartz than for clay under high alkaline conditions. This may be important information about the behaviour of the two basic impurities in the Omarska sludge for the selective flocculation process.

5. ACKNOWLEDGMENTS

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ПРОУЧАВАЊА ФЛОКУЛАЦИЈЕ УЗОРКА ПРИРОДНОГ КВАРЦА У ПРИСУСТВУ АНЈОНСКОГ ПОЛИАКРИЛАМИДА

Сажетак: Отпадни муљ који настаје у процесу припреме жељезне руде у Руднику Омарска (Република Српска, БиХ) је фине гранулације (< 15 μm), садржи релативно висок проценат жељеза и кварц као главну нечистоћу. У овом раду је представљено истраживање понашања у процесу флокулације ровног природног узорка кварца из Рудника Омарска. Узорак се састоји од кварца који доминира, уз присутност минерала глине и фелдспата, са садржајем SiO₂ од 92,9%. Гранулометријска анализа је показала да се ради о финим и ултрафиним честицама. Зета-потенцијал зависи од pH вриједности. Експерименти таложења су вршени уз присуство три дисперзанта (Na-хексаметафосфат, Na-пирофосфат и Na-силикат и анјонског полиакриламида као флокуланта. Најбољи резултати су постигнути са Na-хексаметафосфатом (1000 g/t) и анјонским полиакриламидом A 100. Утицај флокуланта на брзину таложења зависи од концентрације чврсте супстанце. Брзина

таложења значајно расте са разрјеђењем система у оба случаја (природно таложење и таложење у присуству флокуланта).

Кључне ријечи: Кварц, жељезна руда, муљ, флокулација, брзина таложења.

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