

Iranian Journal of Chemical Engineering

Journal Homepage: www.ijche.com

pISSN: 1735-5397 eISSN: 2008-2355

**Full Length Article** 

# Flocculation Behavior of Ultrafine Quartz and Magnetite as the Main Mineral Components of Iron Ore Tailings

S. Ghasemi<sup>1</sup>, A. Behnamfard<sup>1\*</sup>, R. Arjmand<sup>2</sup>

<sup>1</sup> Faculty of Engineering, University of Birjand, Birjand, Iran <sup>2</sup> Opal Parsian Sangan Industrial and Mineral Co. (OPSIM), Khaf, Iran

#### ARTICLE INFO

Article history: Received: 2020-12-02 Accepted: 2021-05-19 Available online: 2022-02-06

#### Keywords:

Iron Ore Tailing, Mineral Components, Quartz and Magnetite, Flocculation Behavior, Settling Rate

#### ABSTRACT

The thickening of the iron ore tailings allows process water to be partially recovered and recirculated, also it reduces the fresh water consumption, which results in lower operating costs and less environmental impacts. The settling characteristics of the mineral components of an iron ore tailing in the thickening process may be different under various pulp conditions. Hence, the study of the characteristics of the mineral components of the iron ore tailings separately can provide very useful information about the thickening of an iron ore tailing. In this research, the settling behavior of the main mineral components of iron ore tailings including quartz and magnetite have been investigated under various operational conditions. The quartz and magnetite showed different settling behaviors, so as the maximum settling rate of quartz was achieved under different pulp conditions than that of magnetite was. There was a big difference between the maximum settling rates of quartz and magnetite, as the maximum settling rates of quartz and magnetite were 197 and 873 m/h respectively. In the thickening of an iron ore tailing, the pulp conditions must be set based on the settling behavior of the mineral component with the lowest settling rate.

DOI: 10.22034/ijche.2021.260225.1362 URL: http://www.ijche.com/article\_144318.html

#### 1. Introduction

The water consumption in mineral processing plants is more than twice of the feed ore tonnage [1]. Hence, assessing the availability of the adequate and reliable water supply for a mineral processing plant to work effectively is essential, especially in areas with water shortage due to the unfavorable climate [2]. In the mineral processing plants, the valuable minerals are separated from the non-valuable minerals (i.e., gangue minerals) by physical separation methods such as the gravity separation, magnetic separation, etc. [3]. The tailings of a mineral processing plant are

\*Corresponding author: behnamfard@birjnad.ac.ir (A. Behnamfard)

mostly discharged as concentrated pulps or slurries, so dewatering plays an important role in reducing the fresh water consumption [4]. The first and main step of dewatering tailings in the mineral processing plants is thickening by using a thickener. The use of thickeners near the mineral processing plant allows to partially recover and recirculate process water at relatively low costs, also helps reduce the fresh water consumption, which lowers operating costs and reduces the ground water pollution [5]. In the thickening process, the settling rate of the flocculated particles is the most significant parameter to evaluate the settling characteristics since it directly affects the capacity of the thickeners [6].

Eswaraiah et al. showed that the settling rate of ultrafine iron slimes increases several times by using flocculants [7]. Shi et al. also observed that the settling velocity of iron tailings is lower during the natural sedimentation and the settling velocity is obviously improved by adding polyacrylamide flocculant in the pulp material pretreatment [8].

Various parameters affect the flocculation process. The dosage of the flocculant has a great influence on the flocculation process. The settling rate usually increases by increasing the dosage of the flocculant up to a certain value which is the optimum dosage [9, 10]. At this dosage of the flocculant which is the maximum amount of the flocculant, half the area of the mineral is covered with the flocculant and the mineral can be utilized for flocculation [9, 10]. At the dosages of the flocculant more than the optimum one, some layers of polymer will cover the surface of the particle. It makes the suspension very stable which makes the separation difficult [9, 10]. The agitation of the flocculant suspension also affects the flocculation process in a way that

some flocs may break due to the excessive agitation. It is clear that the settling rate reduces as the smaller flocs form [11, 12]. The floc breakage produces new particle surfaces in the solution which increase the capacity of flocculation by the adsorption of the flocculant onto the mineral surface, but the efficiency of flocculation decreases due to repulsion as a result of the excess adsorption [11, 12]. The flocculant molecular weight also affects the flocculation process. The flocculants with higher molecular weights usually show better performance in the flocculation process since after the adsorption of the flocculants with high molecular weights, they can be far from the mineral surface and it takes them more time to reach equilibrium [13-15]. Flocculants with similar molecular weights may show different flocculation performances depending on the properties of the flocculant and pulp. Hence, the molecular weight is not the sole requirement for a good flocculation [13-15]. The mineral particle size has also a great influence on the flocculation process that the degree of flocculation increases by decreasing the mineral particle size [16]. Minerals with particle sizes of more than 100 µm usually do not agglomerate well unless high molecular weight flocculants being applied. Minerals with particle sizes of less than 50 µm normally show a good flocculation behavior [16]. The pH of the solution can also affect the flocculation process. The type and amount of the mineral surface charge are dependent on the pH of the solution [17]. Furthermore, the degree of the ionization of the flocculants and the polymer chain charge are also dependent on the pH of the solution [17].

Sadangi et al. studied the effect of the concentration of the solid, dosage of the flocculant, and pH of the solution on the flocculation and settling rate of a low grade goethetic-hematite iron ore with the particle size of less than 45  $\mu$ m [18]. They observed that at the 10 % concentration of the solid, the minimum required dosage of the flocculant to achieve a 2 m/hr settling rate was 10 g/t. They also observed that with the increase in the percentage of the solid, the settling rate of particles decreased. At the 30 % concentration of the solid, the required dose of the flocculant was around 60 g/t. Once, the concentration of solid particles increases more than 30 %, the settling rate becomes very low even after adding the flocculant [18].

Arjmand et al. studied the effect of various parameters of the process including the dosage of the anionic flocculant (5-20 g/t solid), concentration of the slurry solid (3-12 %) and pH (4-12) on the settling rate of the tailing of an iron ore processing plant [19]. The results revealed that the settling rate increased by increasing the pH value of the pulp. Furthermore, the settling rate increased by decreasing the concentration of the slurry solid. The dosage of the flocculant had a considerable effect on the flocculation performance and a significant improvement in the settling rate was observed by increasing the dosage of the polymer [19].

Yang et al studied the process of the flocculation of the iron ore fine tailings in an alkaline environment. They reported that in the alkaline environment, the surface of tailings particles was negatively charged, which led to the anionic flocculant having been combined with tailings particles only by bridging. The adsorption of  $Ca^{2+}$  ions on tailings particles caused a strong electric double layer compression and promoted the aggregation of tailings particles [20].

Findings about the flocculation behavior of the iron ore tailings have been the subject of several researches [18-20], but the study of the flocculation behavior of the mineral components of the iron ore tailings separately has not attracted any attention up to now. The iron ore tailings are usually composed of various minerals such as iron ore minerals (magnetite and hematite), quartz, calcite, etc. [18, 19]. These minerals have different settling rates since they have different specific gravities, surface chemistries, chemical compositions, and mineral lattices. The focus of this research is on studying the flocculation behavior of quartz and magnetite as the main mineral components of the iron ore tailings. By finding the optimum conditions for the settling of the mineral components of the iron ore tailings, it will be possible to improve the dewatering process of the iron ore tailings.

# 2. Materials and methods

# 2.1. Materials

Analytical grade reagents of calcium nitrate tetrahydrate, sodium hydroxide and hydrochloric acid were obtained from Merck anionic Company. The Polyacrylamide flocculant with the trade name of A26 was obtained from Akhtar Shimi Co., Yazd, Iran. It has the molecular weight of  $18 \times 10^6$  g/mol. Another anionic flocculant with the molecular weight of  $800 \times 10^6$  was obtained from Anhui Tianrun Chemicals Co., Ltd, China. The Sodium Silicate solution with the Na<sub>2</sub>O/SiO<sub>2</sub> ratio of 2.5 and the concentration of 45 % was obtained from Silicate Gostar Co., Esfahan, Iran.

The high purity quartz was obtained from a quartz mine in Nehbandan, east of Iran. The sample was crushed, by a laboratory jaw crusher, to particles sized less than 2 mm and then the milling of the sample was performed by a ceramic ball mill. Afterwards, the sample was washed with distilled water several times, dried and used in the experiments. The high purity magnetite sample was obtained from Sangan Iron Ore Complex (SIOC), Iran.

# 2.2. Experimental methodology

The mineralogical composition of the quartz sample was determined through X-Ray Diffraction (XRD) tests by the Philips-Xpert Pro. X-ray Diffractometer. In order to determine the total content of iron in the magnetite sample, an exact amount of the dissolved in concentrated sample was hydrochloric acid at elevated temperatures, and then Fe(III) was reduced to Fe(II) by tin chloride [21, 22]. The solution volume reached the mark by adding distilled water and sulfuric acid. The extra amount of tin chloride was neutralized by mercury chloride. After adding phosphoric acid, the entire iron of the solution was measured by titration using potassium dichromate as the titrant and sodium diphenyl sulfonate as the indicator [21, 22]. For analysing FeO, a similar method of the total iron analysis was applied, with the difference that tin chloride was not added to the leach solution [23, 24]. The grade of sulfur in the magnetite sample was determined by the LECO CS-400 Carbon Sulfur Analyzer, ROMQUEST TECHNOLOGIES CORP., Canada. The particle size distribution of the quartz and magnetite samples was determined by the Malvern Instruments Mastersizer 3000 Particle Size Analyzer.

The settling experiments were performed in the 50 mL graduated cylinder with the height of 17.5 cm and diameter of 2.2 cm. An accurate amount of the magnetite or quartz sample was added to the graduated cylinder and a pulp was prepared by the addition of distilled water until reaching near the mark. The pH of the pulp was adjusted at a predetermined value by the addition of a 1 M sodium hydroxide or hydrochloric acid solution. The concentration of Ca<sup>2+</sup> ion was adjusted at a predetermined amount by adding a specific volume of a calcium nitrate solution. The calcium nitrate solution was previously prepared by adding an exact amount of calcium nitrate tetrahydrate into the 100 ml volumetric flask and raising its volume to the mark with distilled water. An exact volume of sodium silicate was added to the graduated cylinder and mixed thoroughly by inverting the cylinder four times. A flocculant solution with the concentration of 52 mg/L was prepared by adding an accurate amount of the flocculant into the beaker containing 200 mL of distilled water and agitating the solution for one hour by a mechanical stirrer at the constant rotation speed of 200 rpm. An accurate volume of the flocculant solution was added to the cylinder and mixing was performed by gently inverting the cylinder ten times and the cylinder immediately was placed on the table and the settling time was recorded. The settling time is the time between the beginning and when the mud line is fixed for the first time. The settling rate can be calculated with the determination of the settling time and sedimentation height.

The effects of the molecular weight and concentration of the flocculant, pH, the percentage of the solid, the concentrations of the  $Ca^{2+}$  ion and sodium silicate on the settling rate of quartz and magnetite were investigated. Table 1 shows the factors and their levels for the study of the settling rates of quartz and magnetite.

# 2.3. Statistical analysis

In statistics, fractional factorial designs are experimental designs consisting of a carefully chosen fraction (subset) of the experimental runs of a full factorial design [11]. Fractional designs are expressed using the notation L<sup>k-p</sup>, where L is the number of levels of each factor

investigated, k is the number of factors investigated, and p describes the size of the fraction of the full factorial used [11]. In this research, the design of experiments was performed by a  $2^{6-1}$  fractional factorial design. A  $2^{6-1}$  design is 1/2 of a two level, six factor

factorial design. The design of experiments was carried out by the Minitab 17 software package. Table 2 shows the test conditions for the study of the settling rates of quartz and magnetite.

#### Table 1

Factors and	their	levels	for the	e studv	of the	settling	rates of	duartz and	l magnetite.
								1	

levels	Low (-)	High (+)	
Factor			
Flocculant conc. (mg/L)	0.26	3.12	
pH	1 (for quartz); 3 (for magnetite)	11	
Solid weight percent (%)	3	10	
$Ca^{2+}$ ion conc. (mg/L)	0	80	
Sodium silicate conc. (mL/L)	0	40	
Flocculant molecular weight	$800 \times 10^6$ with trade name of	$18 \times 10^6$ with trade name of	
(g/mol)	Tianrun	A26	

#### Table 2

Consequence and conditions of experiments for the study of the settling rates of quartz and magnetite.

		Flocculant	Flocculant				Sodium
Std.	Run	molecular	conc.	pН	Weight	Ca <sup>2+</sup> conc.	silicate conc.
order	order	weight	(mg/L)		(%)	(mg/L)	(mL/L)
2	1	+	-	-	-	-	+
9	2	-	-	-	+	-	+
14	3	+	-	+	+	-	+
3	4	-	+	-	-	-	+
6	5	+	-	+	-	-	-
10	6	+	-	-	+	-	-
16	7	+	+	+	+	-	-
15	8	-	+	+	+	-	+
7	9	-	+	+	-	-	-
26	10	+	-	-	+	+	+
8	11	+	+	+	-	-	+
25	12	-	-	-	+	+	-
30	13	+	-	+	+	+	-
28	14	+	+	-	+	+	-
31	15	-	+	+	+	+	-
17	16	-	-	-	-	+	+
32	17	+	+	+	+	+	+
20	18	+	+	-	-	+	+
24	19	+	+	+	-	+	-
5	20	-	-	+	-	-	+
21	21	-	-	+	-	+	-

27	22	-	+	-	+	+	+
23	23	-	+	+	-	+	+
19	24	-	+	-	-	+	-
12	25	+	+	-	+	-	+
22	26	+	-	+	-	+	+
29	27	-	-	+	+	+	+
18	28	+	-	-	-	+	-
13	29	-	-	+	+	-	-
11	30	-	+	-	+	-	-
4	31	+	+	-	-	-	-
1	32	-	-	-	-	-	-

#### 3. Results and discussion

# 3.1. Flocculation studies of quartz

#### 3.1.1. Characterization of quartz

Table 3 shows the chemical analysis of quartz.

Table 3								
Chemical analysis of the quartz sample determined by XRF.								
Oxide type	SiO <sub>2</sub>	$Al_2O_3$	CaO	MgO	TiO <sub>2</sub>			
Percent	96.68	2.32	0.38	< 0.1	< 0.1			
Oxide type	Fe <sub>2</sub> O <sub>3</sub>	$P_2O_5$	$SO_3$	Na <sub>2</sub> O	K <sub>2</sub> O			
Percent	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1			

The particle size distribution curve and the cumulative particle size distribution curve of the quar tz sample have been shown in Figures 1(a) and (b) respectively. The values of d10, d50, d80 and d90 of the quartz sample are 2.0, 11.6, 26.5 and 36.0  $\mu$ m respectively.

As it can be seen, nearly 97 % of the sample is

composed of SiO<sub>2</sub> which indicates the high

purity of the quartz sample.



**Figure 1.** a) The particle size distribution and b) The cumulative particle size distribution curves of the quartz and magnetite samples.

#### 3.1.2. Settling studies

Figure 2 shows the effect of the process parameters on the settling rate of quartz. As it

can be seen, the settling rate of quartz is higher by using Tianrun instead of A26 as the flocculant. Tianraun had a greater molecular weight than A26. By using higher molecular weight flocculants, more mineral particles have this chance to adsorb on the flocculant. Hence, it takes more time to reach the equilibrium which results in increasing the efficiency of flocculation due to increasing the floc size and number of collisions [13-15]. It can be seen that by increasing the concentration of the flocculant from 0.26 mg/L to 3.12 mg/L, the settling rate of quartz increases from 51 to 76 m/h. Increasing the dosage of the flocculant to its optimum increases the efficiency of flocculation [9, 10]. The settling rate of the quartz sample increases by increasing the pH value of the solution from 1 to 11. This indicates that the performance of the anionic flocculant is better at basic pH values than acidic ones. It can be due to this fact that the opening of the polymeric chains of the anionic flocculant increases at the basic pH values since the ionization of the carboxylate groups (-COOH) of the anionic flocculant is performed at the basic pH values [25, 26]. The better performance of the anionic flocculant at the basic pH values also indicates that the electrostatic interaction between the anionic flocculant and the negative surface of quartz is not the case since the PZC (point of zero charge) for quartz is less than 3 [27]. The adsorption of the anionic flocculant on the surface of quartz at basic pH values can be due to the hydrogen bonding between the silanol groups on the surface of quartz and the amide (-NH<sub>2</sub>) groups of the flocculant [26, 29]. A nearly 30 % decrease in the settling rate of quartz was observed by having increased the percentage of the solid from 3 to 11. It may be due to the change in the pulp viscosity since the pulp viscosity increases by increasing the pulp density [30]. Also, the mixing of the flocculant and quartz cannot be performed properly in the pulps with high percentages of the solid [30]. The settling rate of quartz increases by increasing the concentration of  $Ca^{2+}$  ions through the addition of the calcium nitrate salt. It is due to this fact that the  $Ca^{2+}$ ions in the pulp are adsorbed electrostatically by the negatively charged surfaces which results in increasing the density of the positive charge of the quartz surface [31]. The adsorption of the anionic flocculant on the surfaces with positive charges increases which results in the increasing of the settling rate of quartz. The settling rate of quartz decreases in the presence of sodium silicate. Sodium silicate is usually applied as the depressant and depresses the silicate minerals in the flotation flocculation operations and [32]. The adsorption of sodium silicate on the quartz surface builds a barrier on the quartz surface which results in the decreasing of the flocculant adsorption and disturbance to the flocculation process.

Figure 3 shows the two-way interaction plots. As it can be seen, the interaction between the concentration and pH of  $Ca^{2+}$  is so considerable that the presence of the  $Ca^{2+}$  ion in the pulp at the pH value of 11 results in the considerably increasing of the settling rate, while it has a negligible effect at the pH value of 1. It is due to this fact that the ZPC for quartz is less than 3 and the adsorption of  $Ca^{2+}$  on the quartz surface is electrostatically favorable at basic pH values [33]. This increases the density of the negative charge of the quartz surface and results in the more adsorption of the anionic flocculant.

In the presence of sodium silicate,  $Ca^{2+}$  has no significant effect on the settling rate of quartz, whereas it has a significant effect in the absence of sodium silicate. This indicates that the activity of sodium silicate towards the quartz surface is higher than that of  $Ca^{2+}$ towards the quartz surface which restricts the adsorption of Ca<sup>2+</sup> ions.

The two-way interaction between the molecular weight and pH of the flocculant shows that the A26 flocculant (i.e., the flocculant with lower molecular weight) is not

sensitive to the pH of the pulp while the Tianrun flocculant (i.e., the flocculant with higher molecular weight) has a better performance at the pH value of 11.



Figure 2. The effect of main parameters on the settling rate of quartz.



Figure 3. The effects of the interaction between parameters for the study of the settling rate of quartz.

Figures 4(a) and (b) show the Pareto chart and normal plot of the standardized effects for the study of the settling rate of quartz. As it can be seen in Figure 4(a), the concentration of sodium silicate has the highest effect on the settling rate of quartz. The effects of the concentration of the flocculant, percentage of the solid, and concentration and pH of the Ca<sup>2+</sup> ion on the settling rate of quartz are significant although in a decreasing order in consequence. The molecular weight of the flocculant has no significant effect on the settling rate of quartz. Among the two-way interactions, the interaction between the pH and  $Ca^{2+}$  ion has the highest effect and after that the interaction between sodium silicate and  $Ca^{2+}$ . Among the three-way interactions, the interaction between the molecular weight of the flocculant, the concentration of the flocculant and the percentage of the solid has the highest effect on the settling rate of quartz.

Figure 4(b) shows the normal plot of

standardized effects for the study of the settling rate of quartz. In this chart significant and insignificant effects have been shown by red square and blue circle symbols respectively. In this plot, the symbols which are on the right side of the red line have positive effects and the symbols which are on the left side of the red line have negative effects on the settling rate of quartz. As it can be seen, the concentration of the flocculant has the most positive effect and the concentration of sodium silicate has the most negative effect on the settling rate of quartz.



Figure 4. The a) Pareto chart and b) normal plot of the standardized effects on the settling rate of quartz.

The maximum settling rate of quartz was determined using a Minitab Response Optimizer. It was determined to be 197 m/h. This settling rate was approved by the experimental tests. The maximum settling rate of quartz was achieved under the conditions of: the Tianrun flocculant with the concentration of 3.12 mg/L, the pH value of 11, 10 percent of the solid , the concentration of 80 mg/L of the Ca<sup>2+</sup> ion and without the addition of

sodium silicate.

## **3.2. Flocculation studies of magnetite 3.2.1. Characterization of the sample**

The total contents of Fe and FeO in the magnetite sample were determined by the titration method having used potassium dichromate as the titrant and diphenylamine as the indicator. The grades of the total Fe and FeO in the sample were determined to be 67.5 % and 28.32 % respectively. The grade of FeO of the sample indicates that the amount of the magnetite mineral is 91.25 %. The content of sulfur of the magnetite sample was determined to be 0.49 %.

The particle size distribution and cumulative particle size distribution curves for the magnetite sample have been shown in Figures 1(a) and (b) respectively. The d10, d50, d80 and d90 values of the sample were determined to be 5.6, 24.5, 47.1 and 61.9  $\mu$ m respectively.

# 3.2.2. Settling studies

Figure 5 shows the effect of different parameters on the settling rate of magnetite. As it can be seen a higher settling rate has been obtained by A26 flocculant in comparison with the Tianrun flocculant. This indicates that a flocculant with a lower molecular weight and more anionic sites has a better performance in the flocculation of magnetite. The settling rate of the magnetite sample increases from 369 to 505 m/h by increasing the concentration of the flocculant from 0.26 mg/L to 3.12 mg/L. Increasing the dosage of the flocculant to its optimum increases the degree of flocculation which increases the flocculation performance [9, 10]. The settling rate of magnetite decreases from 457 to 417 m/h by increasing the pH value of the pulp from 3 to 11. The

effect of the pH of the pulp on the settling rate of magnetite can be explained by the density of the positive charge of the magnetite surface. The PZC of magnetite is around 6.5 [34, 35]. Therefore, the surface charge of magnetite is positive at the pH value of 3 and it is negative at the pH value of 11. Hence, the adsorption of the anionic flocculant on the magnetite surface is favorable at the pH value of 3 due to the electrostatic interactions. The settling rate of magnetite decreases from 466 to 408 m/h by increasing the percentage of the solid from 3 to 10. It may be due to the viscosity of the pulp being increased by increasing the percentage of the solid. The settling rates of magnetite in the absence and presence of 80 mg/L of  $Ca^{2+}$ were 456 and 418 m/hrespectively. The surface chemistry of magnetite is changed due to the adsorption or precipitation of Ca<sup>2+</sup> ions which restrict the adsorption of the flocculant. The settling rate of the magnetite sample in the absence and presence of 40 mL/L of sodium silicate is 496 and 378 m/h respectively. Therefore, the settling rate of magnetite decreases by the addition of sodium silicate into the pulp. This may be due to the preadsorption of sodium silicate on the magnetite surface which results in decreasing the adsorption of the flocculant on the magnetite surface.



Figure 5. The effect of main parameters on the settling rate of magnetite.

Figure 6 shows the interaction plots for the study of the settling rate of magnetite. The

interaction plot between  $Ca^{2+}$  and pH shows that the presence of the  $Ca^{2+}$  ion in the pulp at

the pH value of 3 has no effect on the settling rate of magnetite while it retards the settling rate of magnetite at the pH value of 11. The surface charge of magnetite is positive at the pH value of 3 and it is negative at the pH value of 11 [34, 35]. Therefore, the adsorption of the  $Ca^{2+}$  ion on the magnetite surface is not performed at the pH value of 3 due to the electrostatic repulsion forces while it is favorable at the pH value of 11. The adsorption of Ca<sup>2+</sup> on the magnetite surface at the pH value of 11 occurs due to the electrostatic interactions which results in changing the chemistry magnetite. surface of The interaction plot between the molecular weight and concentration of the flocculant shows that the settling rate of magnetite increases by increasing the concentration of the flocculant, while by using the A26 flocculant this increase is more significant. The interaction plot between the concentration of the flocculant and the Ca<sup>2+</sup> ion and also sodium silicate shows that the settling rate of magnetite increases by increasing the concentration of the flocculant but the presence of the  $Ca^{2+}$  ion or sodium silicate has a negative effect.

Figure 7(a) shows the Pareto chart for the main effects and two and three-way interactions for the study of the settling rate of

magnetite. It can be seen that the concentration of the flocculant has the most significant effect on the settling rate of magnetite. The effect of main parameters reduces in the order of silicate sodium, the percentage of the solid, pH, the molecular weight of the flocculant and the presence of the  $Ca^{2+}$  ion. Among the two-way interactions, the interaction between the molecular weight of the flocculant and sodium silicate has the most significant effect on the settling rate of magnetite. Figure 7(b) shows the normal plot of the standardized effects for the study of the settling rate of magnetite. As it can be seen, the concentration of the flocculant the most positive effect and the has concentration of sodium silicate has the most negative effect on the settling rate of magnetite.

The maximum settling rate of magnetite was determined by a Minitab response surface optimizer. It was determined to be 873 m/h under the following conditions: the concentration of 3.12 mg/L of the A26 flocculant, the pH value of 11, 3 percent of the solid, the concentration of 40 mL/L of sodium silicate and without the addition of Ca<sup>2+</sup> ions. A sedimentation test was arranged under thoes conditions and nearly the same settling rate of magnetite was observed.



Figure 6. The interaction effects of parameters for the study of the settling rate of magnetite.



Figure 7. The a) Pareto chart and b) normal plot of the standardized effects on the settling rate of magnetite.

# **3.3.** Comparison of the settling characteristics of quartz and magnetite

In the previous sections the effects of process parameters including the molecular weight and concentration of the flocculant, pH, the percentage of the solid, and the concentrations of  $Ca^{2+}$  and sodium silicate on the settling rate of quartz and magnetite were studied. By comparing the effects of process parameters on the settling rates of quartz and magnetite, it is clear that increasing the concentration of the flocculant from 0.26 to 3.12 mg/l increases the settling rate of both quartz and magnetite minerals. The effects of the percentage of the solid and the addition of sodium silicate on the flocculation of magnetite are also similar to thoes on quartz, so both settling rates of quartz and magnetite decrease by increasing the percentage of the solid from 3 to 10 and by adding 40 mL/L of the sodium silicate solution into the pulp.

Among the studied process parameters, three parameters including the molecular weight of the flocculant, pH of the solution and addition of  $Ca^{2+}$  had adverse effects on the settling rates of quartz and magnetite. The settling rate of quartz increases by increasing the pH value of the solution (from the acidic to basic pH values) and the addition of  $Ca^{2+}$ , while the settling rate of magnetite decreases by

increasing the pH of the solution and the addition of  $Ca^{2+}$ . A flocculant with a lower molecular weight and more anionic sites has a better performance in the flocculation of magnetite, but the molecular weight of the flocculant has no significant effect on the flocculation of quartz. It is clear that the mineral components of iron ore tailings have different flocculation behaviors.

The maximum settling rates of quartz and magnetite were showed to be 197 and 873 m/h respectively. It is clear that there is a big difference between the maximum settling rate of quartz and that of magnetite, so as the maximum settling rate of quartz is quite lower than that of magnetite. The settling rates of quartz and magnetite are dependent on their specific gravities and flocculation behaviors. The specific gravities of magnetite and quartz are 5.2 and 2.65 respectively. In order to observe quartz and magnetite flocs which formed during the flocculation process under the test conditions of run 18, some of the quartz and magnetite pulps after flocculation were poured on a microscope slide and they were studied under the refractive light by a microscope polarizing with the total magnification of 100X ( $10X \times 10X$ ). Figures 8(a) and (b) show the images of magnetite and quartz flocs respectively. It can be seen that a proper flocculation of magnetite particles has been performed but the flocculation of quartz particles has been done poorly. Hence, in the thickening of iron ore tailings, the flocculation of quartz has more importance than that of magnetite and the condition of pulps must be set to achieve the maximum settling rate of quartz.



(a)

(b)

Figure 8. The microscope image of a) magnetite and b) quartz flocs with the total magnification of 100X  $(10X \times 10X)$ 

# 4. Conclusions

In this research, the effects of various process parameters on the main mineral components of the iron ore tailings including quartz and magnetite have been investigated separately. The molecular weight of the flocculant had a negligible effect on the settling rate of quartz. The settling rate of quartz increased from 51 to 76 m/h by increasing the concentration of the flocculant from 0.26 mg/L to 3.12mg/L. It also increased from 57.66 m/h to 69.96 m/h by increasing the pH value of the solution from 1 to 11. A nearly 30 % decrease in the settling rate of quartz was observed by increasing the percentage of the solid from 3 to 10 %. A 33 % increase in the settling rate of quartz was observed by the addition of 80 mg/L of Ca<sup>2+</sup> into the pulp. The settling rate of quartz decreased from 79.83 to 47.79 by the addition of 40 mL/L of sodium silicate into the pulp.

A higher settling rate of magnetite has been obtained for the A26 flocculant in comparison with the Tianrun flocculant. The settling rate of magnetite increased from 369 to 505 m/h by increasing the concentration of the flocculant from 0.26 mg/L to 3.12 mg/L. It decreased from 457 to 417 m/h by increasing the pH value of the pulp from 3 to 11. It also decreased from 456 to 418 m/h by the addition of 80 mg/L of  $Ca^{2+}$  into the pulp. The effect of the percentage of the solid on the flocculation of magnetite was similar to that of quartz and it decreased from 466 to 408 m/h by having increased the percentage of the solid from 3 to 10. The settling rates of magnetite in the absence and presence of 40 mL/L of sodium silicate were 496 and 378 m/h respectively. The concentration of the flocculant had the most positive effect on the settling rates of quartz and magnetite and the concentration of sodium silicate had the most negative effect on both of them.

The maximum settling rate of quartz was determined to be 197 m/h. It was achieved under the conditions of: the Tianrun flocculant with the concentration of 3.12 mg/L, the pH value of 11, 10 percent of the solid, the concentration of 80 mg/L of the Ca<sup>2+</sup> ion and without the addition of sodium silicate. The maximum settling rate of magnetite was determined to be 873 m/h under the following conditions: the concentration of 3.12 mg/L of the A26 flocculant, the pH value of 11, 3 percent of the solid, the concentration of 40 mL/L of sodium silicate and without the addition of Ca<sup>2+</sup> ions.

This research shows that there is a big difference between the maximum settling rates of quartz and magnetite. Hence, the mineralogical composition of the iron ore tailings has great influence on the thickening process. The maximum settling rate of an iron ore tailing can be determined based on the settling rate of the mineral component with the lowest settling rate. Hence, in order to achieve the maximum settling rate of an iron ore tailing, the pulp condition must be set based on the optimum settling condition of the mineral with the lowest settling rate, which is quartz in this regard.

# Acknowledgement

The authors express their deep gratitude to the Iranian Mines & Mining Industries Development & Renovation organization (IMIDRO) for bearing some research costs.

# References

- [1] Ihle, C. F. and Kracht, W., "The relevance of water recirculation in large scale mineral processing plants with a remote water supply", *J. Cleaner Prod.*, **177**, 34 (2018).
- [2] Northey, S. A., Mudd, G. M., Werner, T. T., Haque, N. and Yellishetty, M., "Sustainable water management and improved corporate reporting in mining", *Water Resour. Ind.*, 21, 100104 (2019).
- [3] Grammatikopoulos, T. and Downing, S., "The disruptive role of process mineralogy in geology and mineral processing industry", *Asp. Min. Miner. Sci.*, 5, 571 (2020).
- [4] Chaedir, B. A., Kurnia, J. C., Sasmito, A.
  P. and Mujumdar, A. S., "Advances in dewatering and drying in mineral processing", *Drying Technol.*, **39** (11), 1 (2021).
- [5] Jiao, H., Wu, Y., Wang, W., Chen, X., Wang, Y., Liu, J. and Feng, W., "The micro-scale mechanism of metal mine tailings thickening concentration improved by shearing in gravity thickener", J. Renewable Mater., 9 (4),

637 (2021).

- [6] Nguyen, C. V., Dinh, E., Doi, A., Nguyen, T. V. and Nguyen, A. V., "Accurate, fully automated determination of the initial settling rate of flocculated suspensions", *Miner. Eng.*, 164, 106823 (2021).
- [7] Eswaraiah, C., Biswal, S. K. and Mishra,
  B. K., "Settling characteristics of ultrafine iron ore slimes", *Int. J. Min. Met. Mater.*, 19 (2), 95 (2012).
- [8] Shi, Z. X., Zhou, X. L., Luo, W. B., Liu, Z. Q., Sun, D. Y. and Zhang, X. M., "Different slurry concentration on settling effect of the iron tailings," *Adv. Mater. Res.*, 634-638, 3325 (2013).
- [9] Jeldres, R. I., Jeldres, M., MacIver, M. R., Pawlik, M., Robles, P. and Toro, N., "Analysis of kaolin flocculation in seawater by optical backscattering measurements: Effect of flocculant management and liquor conditions", *Minerals*, **10** (4), 317 (2020).
- [10] Sun, Y., Zhou, S., Sun, W., Zhu, S. and Zheng, H., "Flocculation activity and evaluation of chitosan-based flocculant CMCTS-gP (AM-CA) for heavy metal removal", *Sep. Purif. Technol.*, 241, 116737 (2020).
- [11] BinAhmed, S., Ayoub, G., Al-Hindi, M. and Azizi, F., "The effect of fast mixing conditions on the coagulation– flocculation process of highly turbid suspensions using liquid bittern coagulant", *Desalin. Water Treat.*, **53** (12), 3388 (2015).
- [12] Quezada, G. R., Ayala, L., Leiva, W. H., Toro, N., Toledo, P. G., Robles, P. and Jeldres, R. I., "Describing mining tailing flocculation in seawater by population balance models: Effect of mixing intensity", *Metals*, **10** (2), 240 (2020).
- [13] Costine, A., Cox, J., Travaglini, S.,

Lubansky, A., Fawell, P. and Misslitz, H., "Variations in the molecular weight response of anionic polyacrylamides under different flocculation conditions", *Chem. Eng. Sci.*, **176**, 127 (2018).

- [14] Guo, K., Gao, B., Pan, J., Shen, X., Liu, C., Yue, Q. and Xu, X., "Effects of charge density and molecular weight of papermaking sludge-based flocculant on its decolorization efficiencies", *Sci. Total Environ.*, **723**, 138136 (2020).
- [15] Metaxas, A. E., Panwar, V., Olson, R. L. and Dutcher, C. S., "Ionic strength and polyelectrolyte molecular weight effects on floc formation and growth in Taylor– Couette flows", *Soft Matter.*, **17** (5), 1246 (2021).
- [16] Grabsch, A. F., Yahyaei, M. and Fawell, P. D., "Number-sensitive particle size measurements for monitoring flocculation responses to different grinding conditions", *Miner. Eng.*, **145**, 106088 (2020).
- [17] Chen, R., Fan, Y., Dong, X., Ma, X., Feng, Z., Chang, M. and Li, N., "Impact of pH on interaction between the polymeric flocculant and ultrafine coal with atomic force microscopy (AFM)", *Colloids Surf. A*, 622, 126698 (2021).
- [18] Sadangi, J. K., Sahoo, A. K., Sushobhan, B. R. and Choudhury, N., "Effect of anionic flocculant on settling rate of iron ore ultra-fines", *Mater. Today: Proc.*, 30, 316 (2020).
- [19] Arjmand, R., Massinaei, M. and Behnamfard, A., "Improving flocculation and dewatering performance of iron tailings thickeners," *J. Water Process Eng.*, **31**, 100873 (2019).
- [20] Yang, Y., Wu, A., Klein, B. and Wang, H., "Effect of primary flocculant type on a two-step flocculation process on iron ore

fine tailings under alkaline environment", *Miner. Eng.*, **132**, 14 (2019).

- [21] ISO 2597-1:2006, Iron ores -Determination of total iron content - Part 1: Titrimetric method after tin(II) chloride reduction, ISO Publications, Geneva, Switzerland, (2006).
- [22] Mohassab, Y., Elzohiery, M., Chen, F. and Sohn, H. Y., "Determination of total iron content in iron ore and DRI: Titrimetric method versus ICP-OES analysis", *Proceedings of EPD Congress* 2016, Springer, Cham, pp. 125-133, (2016).
- [23] Andrade, S., Hypolito, R., Ulbrich, H. H. and Silva, M. L., "Iron (II) oxide determination in rocks and minerals", *Chem. Geol.*, **182** (1), 85 (2002).
- [24] Maxwell, J. A., "Rock and mineral analysis", In: Elving, P. J., Kolthoff, I. M. (Eds.), Chemical Analysis, Wiley-Interscience, New York, pp. 419-421, (1968).
- [25] Pillai, J., "Flocculants and coagulants: The keys to water and waste management in aggregate production", Nalco Company, Illinois, USA, (1997).
- [26] Wiśniewska, M., Urban, T., Grządka, E., Zarko, V. I. and Gun'ko, V. M., "Comparison of adsorption affinity of polyacrylic acid for surfaces of mixed silica–alumina", *Colloid Polym. Sci.*, 292 (3), 699 (2014).
- [27] Yao, J., Yin, W. and Gong, E., "Depressing effect of fine hydrophilic particles on magnesite reverse flotation", *Int. J. Miner. Process.*, 149, 84 (2016).
- [28] Nasser, M. S. and James, A. E., "The effect of polyacrylamide charge density

and molecular weight on the flocculation and sedimentation behaviour of kaolinite suspension", *Sep. Purif. Technol.*, **52** (2), 241 (2006).

- [29] Graveling, G. J., Ragnarsdottir, K. V., Allen, G. C., Eastman, J., Brady, P. V., Balsley, S. D. and Skuse, D. R., "Controls on polyacrylamide adsorption to quartz, kaolinite, and feldspar", *Geochim. Cosm. Acta*, **61** (17), 3515 (1997).
- [30] Ibrahim, S. S. and Abdel-Khalek, N. A., "The action of different types of corn starch on the flocculation of phosphate slimes", *Miner. Eng.*, **5** (8), 907 (1992).
- [31] Lelis, D. F., Leão, V. A. and Lima, R. M. F., "Effect of EDTA on quartz and hematite flotation with starch/amine in an aqueous solution containing Mn<sup>2+</sup> ions", *REM: Int. Eng. J.*, 69 (4), 479 (2016).
- [32] Ma, M., "The significance of dosing sequence in the flocculation of hematite", *Chem. Eng. Sci.*, 73, 51 (2012).
- [33] Krouti, O., Chval, Z., Skelton, A. A. and Predota, M., "Computer simulations of quartz (101)–Water interface over a range of pH values", *J. Phys. Chem. C*, **119**, 9274 (2015).
- [34] Prakash, S., Das, B. and Venugopal, R., "Magnetic separation of calcite using selective magnetite coating", *Magnetic. Electr. Sep.*, 10, 1 (1990).
- [35] Lu, D., Hu, Y., Li, Y., Jiang, T., Sun, W. and Wang, Y., "Reverse flotation of ultrafine magnetic concentrate by using mixed anionic/cationic collectors", *Physicochem. Probl. Miner. Process.*, 53 (2), 724 (2017).