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Floatability of Suspended Particles from Wastewater of Natural Stone Processing by Floc-Flotation in Mechanical Cell

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Abstract: Flocculation and flotation methods have been applied individually to achieve the removal of suspended particles from wastewater. However, in this study, the floc-flotation method where these two methods are applied together in a mechanically agitated cell and the use of combinations of different type surfactant and polymer to achieve floatability of fine particles are investigated. The conventional and floc-flotation experiments were carried out to determine the effect of different parameters such as collector type and dosage, pH, flotation time, condition time and polymer/surfactant interactions on floatability of very fine particles. When the results of floc-flotation and conventional flotation were compared, it has been found that higher floatability values were achieved with floc flotation. It can be obtained 96.8% floatability of particles in presence of anionic surfactant (Aero 845, 750 g/t) and nonionic polymer (SPP N 134, 30 g/t) at pH 8 and 5 min. conditioning time for 5 min. flotation time by floc flotation in the mechanical cell.

Keywords: Floc-flotation, Mechanical Flotation cell, Fine particle, Wastewater

Mekanik Flotasyon Hücresinde Flok-flotasyonu ile Doğaltaş işleme atıksuyundan süspanse tanelerin yüzdürülmesi

Öz: Flokülasyon ve flotasyon yöntemleri, atık sulardan süspanse tanelerin uzaklaştırılmasında ayrı ayrı uygulanan yöntemlerdir. Bununla birlikte bu çalışmada mekanik flotasyon hücresinde bu iki yöntemin birlikte uygulandığı flok flotasyonu yöntemi ve ince tanelerin yüzebilirliğini sağlamak için farkı tip toplayıcı ve polimer kombinasyonlarının kullanılması araştırılmıştır. Klasik flotasyon ve flok-flotasyonu deneyleri, toplayıcı tipi ve dozajı, pH, flotasyon süresi, koşullandırma süresi ve polimer/ toplayıcı etkileşimleri gibi parametrelerin ince tanelerin yüzebilirliği üzerine etkilerini belirlemek için yürütülmüştür. Klasik ve flok-flotasyonu deney sonuçları karşılaştırıldığında, flok-flotasyonu ile çok daha yüksek yüzdürme değerlerine ulaşıldığı bulunmuştur. Mekanik hücrede flok-flotasyonu ile pH 8'de 5 dakika koşullandırma ve 5 dakika flotasyon süreleri için anyonik toplayıcı (Aero 845, 750 g/t) ve iyonlaşmayan polimer (SPP N 134, 30 g/t) kombinasyonunda tanelerin yüzdürme verimi %96.8 olarak elde edilmiştir.

Anahtar kelimeler: Flok-flotasyonu, Mekanik Flotasyon hücresi, İnce tane, Atıksu

1. Introduction

The wastewaters exiting mining and many other industries contain harmful pollutants such as chemical substances, suspended solids, fats, colloids [1]. These waters are discharged to the nearest receiving environment and cause pollution. Therefore, the reuse or recycle of process waters is an important issue and water recycling technologies are required. Removal of very fine particles from

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mining wastewaters is one of the major challenges for mine operators. Not only very fine particles in wastewater cause turbidity of the water but also it causes problems in the recycle/reuse of the contaminated wastewater at plants. As a result, a larger volume of fresh water is used in plants increasing the costs associated with raw materials and wastewater treatment [2].

Turkey has 40% of world natural stone reserves and is a major marble exporter. There are also numerous natural stone processing plants in Turkey [3]. However, natural stone (marble, travertine, granite, basalt, etc.) processing plants are one of the places where water is used too much. In a medium-sized plant (The daily average production, 30-100 tons), 50-150 m3 /day water is used for cutting, washing and polishing processes and wastewater containing high suspended solids is obtained [4]. Therefore, reuse of process water is of great importance. Recently, an important issue for the natural stone industry is the formation of fine particles (<0.2 mm) during cutting and polishing in slab or tile production. The fine particles in the wastewater cause scratches on the surface of the natural stone during the polishing and cleaning processes. In addition, the suspended particles may result in pipe clogging [3-5]. Therefore, water to be used again should be treated through a good treatment method.

Natural stone processing wastewater is characterized by high suspended solids and the wastewater is generally treated by sedimentation with flocculation method. The purpose of the flocculation process is to form flocs from finely dispersed particles with the help of natural or synthetic polymeric substances which are referred to as flocculant (Figure 1-a). There are many types of polymeric flocculant used in various solid-liquid separation processes. These flocculants are characterized by their ionic nature: cationic, anionic and non-ionic. Ionic polymers usually can be called polyelectrolytes. The most important characteristics of synthetic flocculants are molecular weight and charge density. At present, synthetic polymeric flocculants are commonly used in the flocculation process [2]. They are highly efficient but are mostly non-biodegradable, expensive and may be toxic. On the other hand, bio-polymer flocculants extracted from plant materials are natural organic flocculants that consist of natural polymers or polysaccharide complexes formed from the sugars of different monosaccharides such as starch, cellulose, chitosan, natural gums. The advantages of natural polymers are non-toxic, readily available from renewable natural resources and biodegradable. Their efficiency is low, thus they are not widely used [1-2]. In the literature, a number of researches have been carried out to remove suspended particles from wastewater by using synthetic flocculants and determine the flocculation behavior of the natural stone suspensions [5-12]. However, at most of them, only marble effluents were used. The researches have reported that high molecular weight anionic flocculants are commonly used in the dewatering of natural stone processing wastewater and gave the best result on the settling rate and water clarity.

In recent years, as an alternative to sedimentation and filtration, the flotation method is also used for solid-liquid separation in wastewater treatment [13-14]. Flotation is a method based on the adherence of particles with bubbles and their collection in a foamed layer (Figure 1-b). In mineral flotation, the particles are quite large, typically having diameters of 50 μm or more. Thus, while the bubbles should generally be about 1 mm large, the solids content in the pulp is normally high, 25% by weight, and a high selectivity between particles is desirable [15-16]. Conversely, in wastewater treatment, the particles are typically small (diameter less than 20 μm), close to natural buoyancy, and are present in highly diluted concentrations. The method is used to selectively separate particles from other particles in mineral flotation, while it used to float all particles present in water treatment [17-18]. The flotation process for wastewater treatment is rapidly broadening their applications in different areas and is applied to removing very fine (-20 μm) and ultra-fine particles (-5 μm), colloidal (-1 μm) dispersions, organisms and oily emulsions in environmental applications [19]. Many flotation technologies were improved in wastewater treatment and developments have been still continuing. The dissolved air flotation (DAF) one of these methods is commonly used in the treatment of wastewater [20]. Currently, other flotation techniques such as electro flotation, carrier flotation, induced air flotation (IAF), shear flocculation, floc-flotation have been developed for wastewater treatment [21].

A research in which the flotation method was applied to treat natural stone processing wastewater not been found in the literature. However, in this study, it was investigated floatability of very fine natural stone particles from wastewater with conventional flotation and floc-flotation. Mechanically agitated flotation cell has been applied first time to treatment of natural stone wastewaters. The effect of different parameters such as collector type and dosage, pH, flotation time and condition time on floatability of very fine particles was investigated in the conventional flotation. Then, floc-flotation experiments performed to increase the floatability of fine particles in mechanic cells. Floc flotation involves the use of surfactants and polymers simultaneously. The presence of surfactant and polymer together in the suspension can lead to unavoidable interactions between the two species. Depending on the charge of the surfactant in relation to the charges of the polymer and particle surface, flotation can be enhanced, activate or depress. Therefore, it was also investigated the effect of surfactant and polymer interactions on particle floatability in the floc-flotation.

2. Floc-Flotation

In very fine mineral processing and wastewater treatment, flotation technologies based on particle aggregation are realized through floc flotation including shear flocculation, selective polymer flocculation, carrier flotation, etc. The rate of removal of very fine particles (<20 μm) by flotation is rather slow; however, aggregation of particles can lead to more rapid removal. Flotation of fine particles after they have been aggregated by flocculation or coagulation is called floc flotation [22-26]. Flotation of fine particles (Shear flocculation) after they have been aggregated by coagulation or flocculation has a vast potential for treating the slime problems experienced in the processing of some mineral ores Aggregation of fines particles (Floc flotation) by coagulation or flocculation followed by flotation is a process that is already applied to wastewater treatment. In floc flotation, fine particles are flocculated with a polymer and then added a collector that is able to interact with the flocs, rendering the flocs sufficiently hydrophobic for their attachment to air bubbles (Figure 1-c). The flocs adhering to air bubbles are floated readily, avoiding the problem of low probability of collision and attachment of fine particles to air bubbles. Rapid floc flotation is of great importance in mineral flotation and effluent treatment with or without water reuse [27].

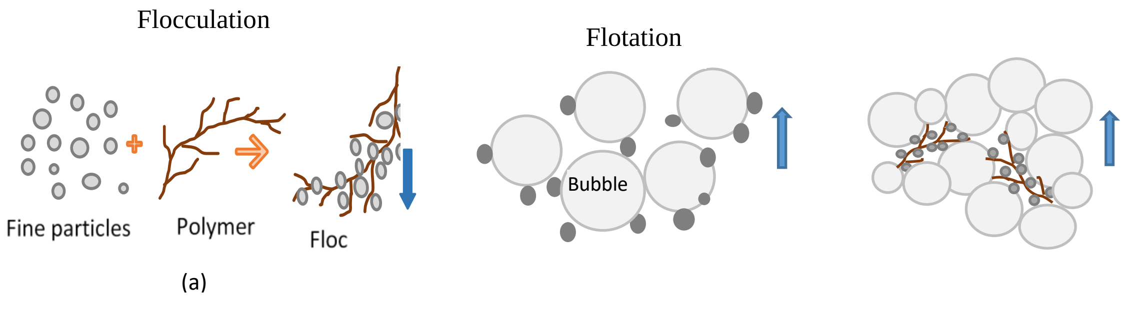

Figure 1. Schematic diagrams of flocculation, flotation, and floc-flotation processes

3. Experimental studies
3.1. Materials

In the experiments, model suspension of fine particles was prepared by using the waste powders taken from a natural stone processing plant (Turkey). Natural stone powders were sieved to -20 micron and dried. The particle size distribution measures of the sample were determined using a Malvern Mastersizer 2000 and d90, d50 and d10 diameters were measured as 27.71 µm, 8.84 µm, and 1.29 µm, respectively. A mineralogical (XRD) and chemical analysis (XRF) of the sample are given in Table 1 and Figure 2. According to the XRD analysis, the majority of the natural stone powders are consisting of calcite and quartz; the sample also contains albite, muscovite and microline minerals. Chemical analysis results showed that the CaO and SiO_2 content of the sample was 42.32% and 16.81%, respectively.

Table 1. Quantitative analysis of the sample

CaO%	**SiO_2%**	**Al_2O_3%**	**K_2O%**	**Na_2O%**	**MgO%**	**Fe_2O_3%**	**TiO_2%**	**LOI%**
42.32	16.81	3.5	1.14	1.06	0.87	0.73	0.09	33.48

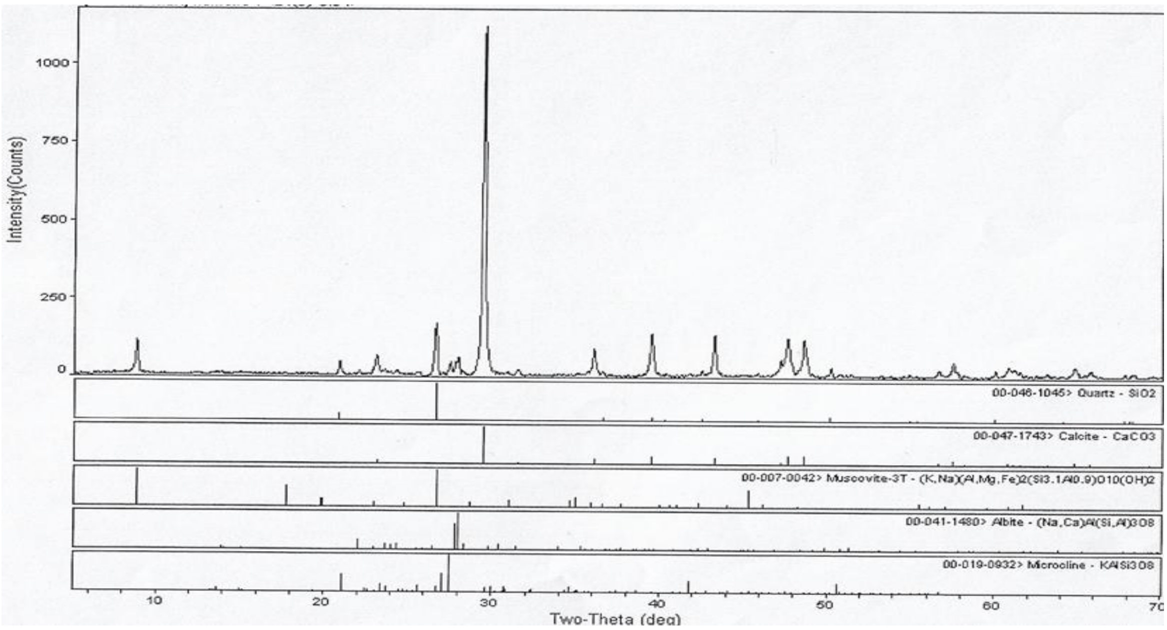

Figure 2. X-ray diffraction pattern of sample

Zeta-potential of fine particles (natural stone powders) was measured as a function of pH. The data is given in Figure 3. The zeta potential measurements of particles were accomplished by a Malvern Nano-Z model device using the laser Doppler velocimeter principle. The highest zeta potential (–36.6 mV) was obtained at pH 11, which shows that the suspension was stable. The zeta potential of suspension was about –13.3 mV at natural pH 8 that the particles tend to form aggregates easily in suspension. The experiments were performed with prepared suspensions in a 1% solid ratio. The properties of the suspensions are given in Table 2. The initial turbidity of suspension (12000 as NTU unit) was measured using the HF Scientific turbidimeter. PH, conductivity, and suspension temperature were measured with Orion 5 star multimeter. The natural pH and conductivity of the suspension were 8 and 580 µS / cm, respectively. The hardness of the water was determined by a volumetric method.

Table 3 presents the chemical reagents used in experiments. Aeromine 3030 C is an amine type, cationic collector. Aero 704 and Aero 727 are fatty acids with anionic properties. Aero 845 is a sulfosuccinamate type collector in the oxyhydryl group and has anionic properties. Aerofroth 65 (AF 65), a mixture of polyglycols, was used as a frother reagent.

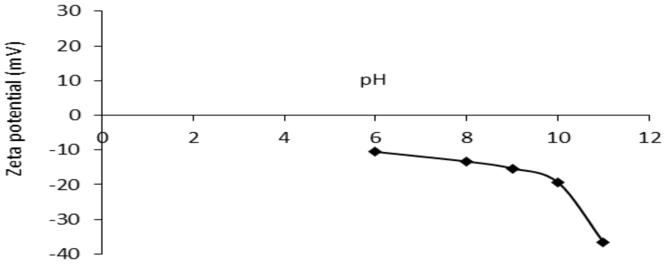

Figure 3. Zeta-potential measurement of fine particles

Table 2. Properties of wastewater

pH	8
Solid ratio	1%
Turbidity	12000 NTU
Conductivity	580 µS/cm
Temperature	17.9 ^{o}C
Water hardness	356 mg/L $CaCO_3$
Particle size	-20 µm

According to the ionization, three types of polymers were used. SPP 508 is an anionic polymer with high molecular weight and 28% of charge density. Enfloc 440C (cationic polymer) has high molecular weight and 55% of charge density. SPPN 134 is a non-ionizing polymer with medium molecular weight. SPP polymers were supplied by the Superkim and Enfloc polymer provided by ECS Chemistry.

Table 3. Chemical reagents used in experiments

	Commercial name	*Type*	***Supplier***
Surfactant	Aeromine 3030C	Cationic collector	Cytec
	Aero 704	Anionic collector	Cytec
	Aero 727	Anionic collector	Cytec
	Aero 845	Anionic collector	Cytec
Frother	Aerofroth 65	Frother	Cytec
Flocculant	SPP 508	Anionic polymer	Superkim
	Enfloc 440C	Cationic polymer	ECS Chemical
	SPP N 134	Nonionic polymer	Superkim

3.2. Method

Figure 4 shows the laboratory scale mechanical flotation cell (Denver) used in the study. It consists of a rectangular 1-liter cell and vertical impeller. Wastewater suspension and reagents were mixed in the cell for certain conditioning times before conventional and floc-flotation experiments. Then, the experiments have been carried out at conditions given in Table 4. In the experimental studies, the solid ratio, the type, and dosage of frother and polymer dosage were kept constant. The effects of pH, flotation time, conditioning time, polymer type, collector type and dosage on fine particle floatability were investigated. Since the amounts of collector reagents used in the experiments were determined based on the recommended dose ranges in the literature [28] the value ranges for each reagent dosage were different. The dosage of polymers was determined previously [29]. In flotation studies, firstly suspension was mixed with the 30 g/t polymer for 2 min. The polymer dosage and agitation time used in the preparation stage of the flocculated particles were the same for all experiments. Then the surfactant was added and was conditioned. Finally, 30 mg/L frother was added and after 2-minute air was delivered to the cell. The experiments aimed to float all the particles through air bubbles from the wastewater suspension containing 1% solids. Thus, to determine the effect of parameters on the floatability of fine particles, the floating and sinking particles were collected separately, dried and then weighed. The floatability (%) of the fine particles was calculated by using the amounts of these products obtained at the end of the flotation time.

Figure 4. Laboratory type Denver flotation cell

Table 4 Experimental conditions

Solid ratio	1%
pH	6, 8 and 10
Flotation time	2 and 5 minute
Condition time	2, 5 and 10 minute
Frother type and dosage	30 mg/L (Aerofroth 65)
Polymer type and dosage	30 g/t (Anionic, SPP 508) 30 g/t (Cationic, Enfloc 440 C) 30 g/t (Nonionic, SPP N 134)
Collector type and dosage	100, 250 and 500 g/t (Aeromine 3030 C) 500, 1000 and1500 g/t (Aero 704) 500, 1000 and 1500 g/t (Aero 727) 100, 500 and 750 g/t (Aero 845)

4. Results

4.1 Conventional flotation experiments

Since the wastewater suspension used in the experiments consists of mainly marble and granite bearing mineral particles such as calcite, quartz, albite, muscovite, microline (Figure 2), experiments have been carried out to determine optimum collector type and dosage for their floating. In conventional flotation experiments, the effects of a cationic collector concentration and concentration of different type anionic collectors have been investigated. Flotation tests were performed for 2 and 5 min. flotation times according to the different dosages of each collector at pH (8). The results are given in Figure 5. It is seen in all graphs (Figure 5a-d) that the flotation time affects the floatability and increasing flotation time from 2 min. to 5 min. increases flotation recovery. Figure 5 (a) shows the results of experiments with different amounts of the cationic collector (Aeroamine 3030 C). The highest removal efficiency was obtained as 70.7% by using 500 g/t collector concentration at 5 min. flotation time. Similarly, according to the results obtained with the anionic collectors, it was obtained 87.4% floatability with 1500 g/t Aero 727 (Fig. 5b); 69% floatability when used 1500 g/t Aero 704 (Figure 5c) for 5 min. flotation time. The highest floatability (91.8%) is possible with 750 g/t dosage of Aero 845 with 5 min. flotation time (Figure 5d).

The low flotation recovery of fine particles with very small mass and high surface area is due to the low collision and adhesion possibilities of the air bubbles. Therefore, the residence time (flotation time) in the flotation cell is another important parameter affecting the fine particle floatability. Figure 5 (a-d) also shows the effects of flotation time on flotation efficiency for each type of collector. As shown in Figure 5, higher floatability values were reached in the 5 min. flotation period than those obtained by the 2 min. of flotation time. Because increased residence time increases the probability of collision and adhesion of the particles to bubbles and even re-adhesion chance of detached particles from bubbles is provided with increased flotation time.

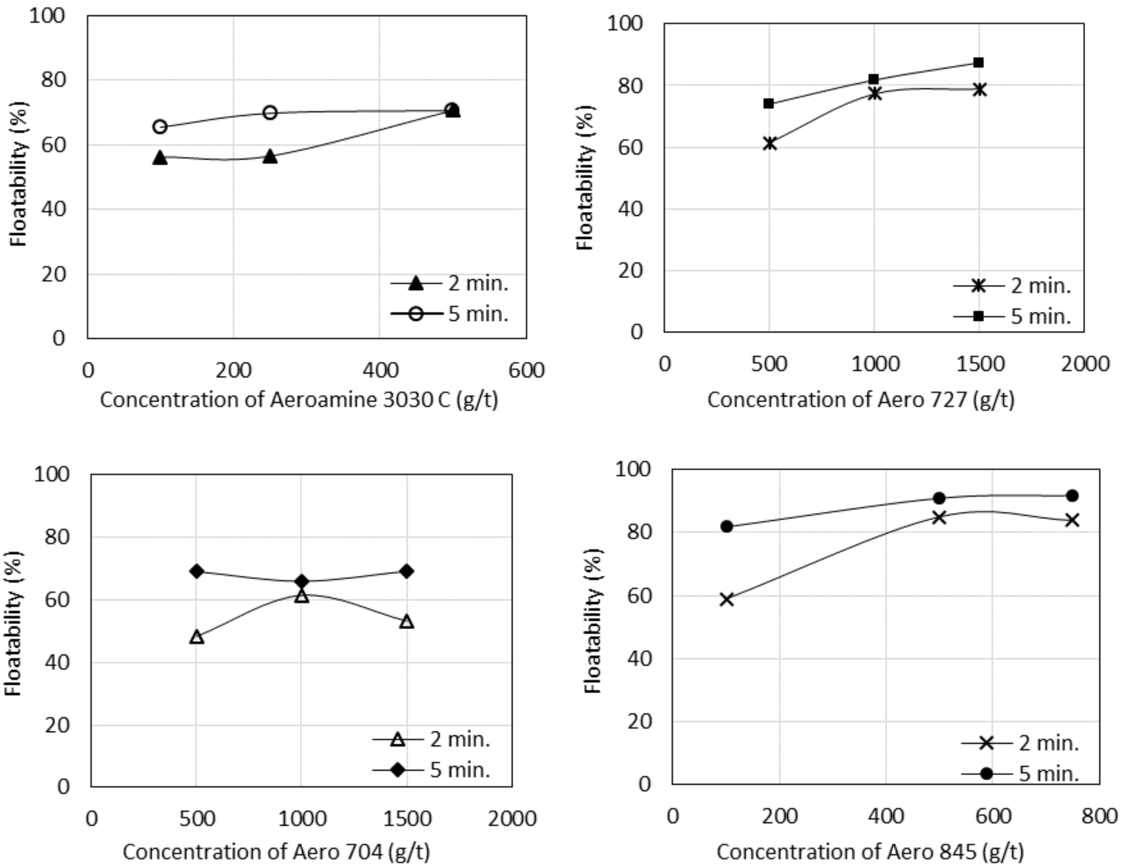

Figure 5. Effect on floatability of fine particles of surfactants concentrations and flotation times

Particle removal efficiencies of the different type surfactants for 2 and 5 min. flotation times were compared and the results are illustrated in Figure 6. As seen, the highest floatability was reached with the Aero 845 (anionic surfactant) for both 2 and 5 min. flotation period. As seen in Figure 6, it is possible to reach almost the same flotation recovery obtained with Aero 845. However, almost two times collector dosage of Aero 725 (1500 g/t) is needed for the same recovery obtained by Aero 845 (750 g/t). According to these results, Aero 845 is more adsorbed on the surface of particles than the other collectors and thus hydrophobic particles float easily with bubbles.

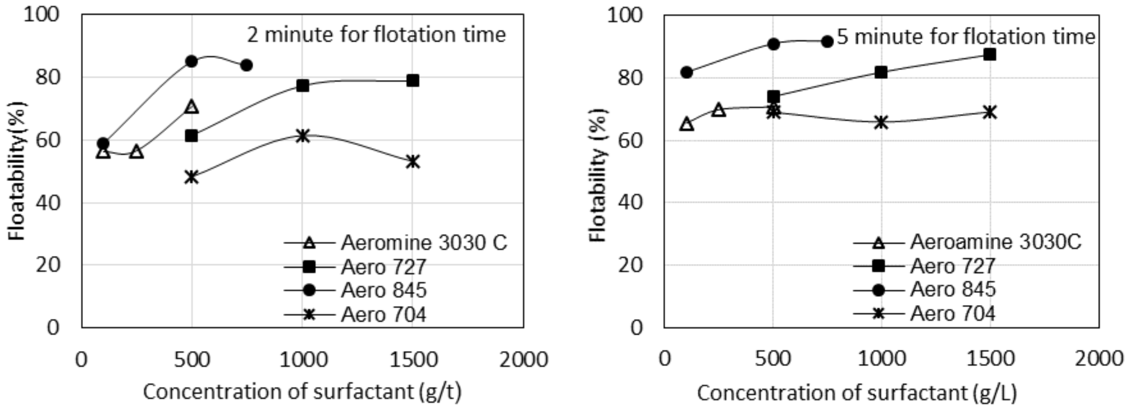

Figure 6. Effect on floatability of fine particles of surfactant type for 2 and 5 minute flotation times

The pH of the suspension is one of the important variables affecting the surface charge and stabilization of particles and also adsorption of surfactant onto particles in the suspension. Therefore, it is primarily necessary to determine the optimum pH value in flotation. The results of the conventional flotation test which examines the effect of pH on floatability of wastewater particles are given in Figure 7. The experiments were carried out at 2 min. conditioning time with the fixed dosages of Aero 845 (750 g/t) and Aero 727 (1500 g/t), in which the highest flotation recoveries were obtained. When the results of experiments performed at pH 6, 8 and 10 were examined, it was determined that higher floatation recoveries were obtained for Aero 727 at pH 8 compared to pH values at both flotation times. The same results were obtained with Aero 845 for 2 min. of flotation time but better recovery value was obtained at pH 6 for 5 min. time.

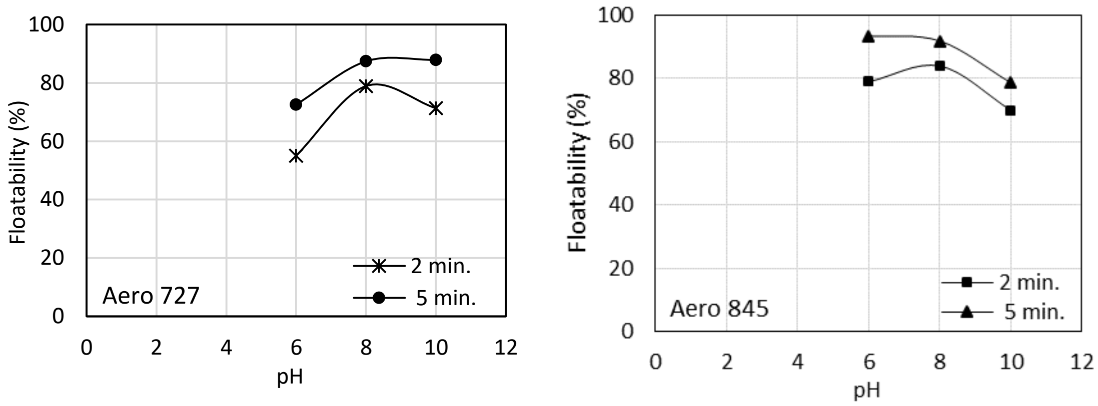

Figure 7. Effect on floatability of fine particles of pH

Since the surface area of the fine particles is very high, the reagent consumption will be higher. Therefore, it is important to determine the optimum conditioning time required for the adsorbing of the surfactants on the surface of particles in the suspension. The effect of conditioning time on the floatability is presented in Figure 8. In the experiments, the dosage of the collectors (Aero 845, 750 g/t and Aero 727, 1500 g/t) and the pH (8) were kept constant. When the results were examined (Figure 8), it was observed that for Aero 727 the conditioning time has little effect on the flotation efficiency for both 2 and 5 min. of flotation time and the 2 min. conditioning time was the most appropriate time. For the Aero 845 reagent, it was determined that the conditioning time influences the floatability and the better recovery was obtained with 5 min. of conditioning time and it is chosen as optimum.

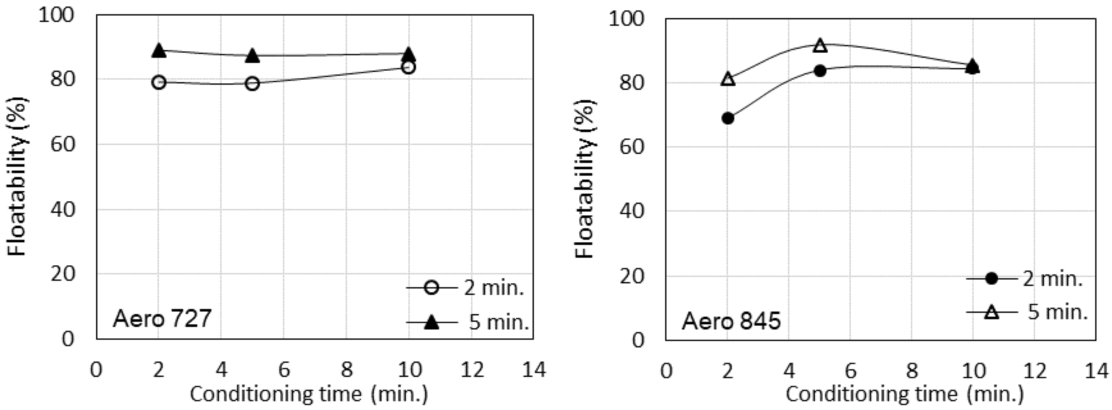

Figure 8. Effect on floatability of fine particles of condition time

4.2 Floc-Flotation Experiments

Floc-flotation experiments performed to increase the floatability of fine particles in mechanically agitated flotation cells. The results were compared with conventional flotation test results. The effect of combinations of different surfactant and polymer type on particle floatability was investigated. In the experiments, aeromine 3030C (500 g/t) was used as a cationic collector and aero 845 (750 g/t) was chosen as anionic collector and they are tested with different type polymers (anionic, cationic and nonionic) in floc-flotation experiments. The dosage of 30 g/t was taken as the same for all polymers. The experiments were carried out at the natural pH (8) and 5 min. conditioning time.

In Table 5, the results of the floc-flotation experiments which were used different polymers with the cationic collector (Aeroamine 3030 C) and conventional flotation experiment for 5 min. flotation time is given. The floatability values compared in the form of column graphs are also presented in Figure 9. In the conventional flotation, the floatability of 70.7% with the cationic collector was obtained for 5 min. flotation time. As seen, higher floatability values were obtained with floc flotation compared to conventional flotation. Floatability of fine particles flocculated with the other polymers is enhanced markedly because of the high probability of collision and attachment of large particles to bubbles. In the case of using cationic surfactant and nonionic polymer, flotation recovery increased to 84.5%. Floc flotation in the presence of cationic surfactant and nonionic polymer increases the floatability of the fine particles by about 20% for 5 min. flotation time, in comparison with the corresponding conventional flotation.

In floc flotation, the presence of surfactant and polymer together in the suspension can lead to unavoidable interactions between two chemical reagents. The effects of such interactions on floatability of particles are important. Depending on the charge of the polymer in relation to the charge of the surfactant and particles, the polymers used in floc-flotation can enhance, activate or depress floatability. The polymer and surfactant interactions are highly dependent on the chemical nature of the polymer and the surfactant [30]. Electrostatic and hydrophobic forces are primarily responsible for the different interaction behavior of surfactants with the charged and uncharged polymers. It has been shown that in the floc flotation, the lowest floatability (80%) was obtained in the presence of cationic surfactant and cationic polymer. The main reason for this finding is due to the adsorption of surfactant on the surface of the particles decreased because of the electrostatic repulsion between the same charged surfactant and the polymer. Therefore, the flotation of particles was slightly depressed.

The size of flocs can be dependent on the amount of the polymer used [31-32]. Floc flotation requires low-density flocs with a narrow size distribution. Polymer bridging and charge neutralization are common mechanisms in flocculation. The most important feature of anionic polymer and polymer bridging mechanism is the production of large size flocs. The cationic flocculants adsorb on the surfaces of negatively charged particles by means of a charge neutralization mechanism due to the electrostatic attraction between the counter-charged ionic groups. In the case of a non-ionic polymer, the most likely mechanism of adsorption is hydrogen bonds. In previous studies, it has been reported that the flocs obtained with cationic and nonionic polymers are relatively small in size [33-35]. Since the amount of polymers used in the experiments was very low, it was observed that small size flocs were obtained. Therefore, it was observed that the size of flocs did not play an important role in the floc-flotation.

Table 5. The results of floc-flotation experiments (Cationic collector (Aeroamine 3030 C)/different type polymers) and conventional flotation experiment in mechanically agitated flotation cell

		Floatability for 5 min. time (%)
Conventional flotation test	Aeroamine 3030 C (Without polymer)	70,7
Floc-flotation test	Aeroamine 3030 C / Anionic polymer (SPP 508)	83,1
	Aeroamine 3030 C / Cationic polymer (Enfloc 440 C)	80
	Aeroamine 3030 C / Nonionic polymer (SPP N 134)	84,5

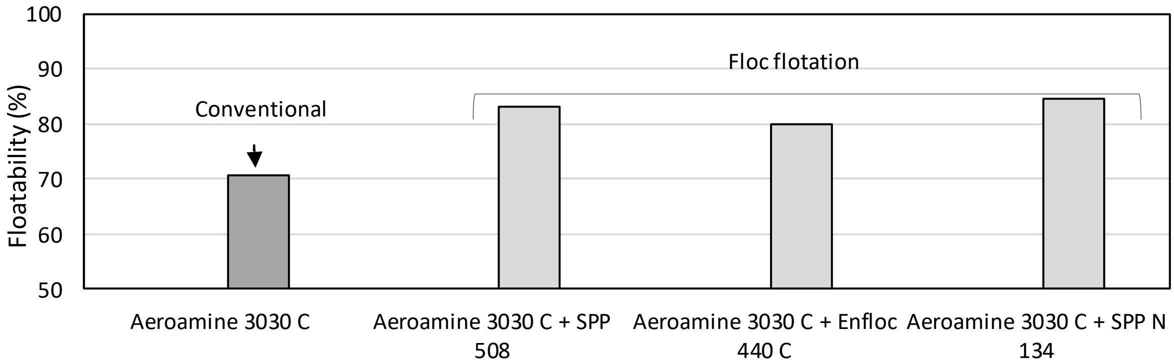

Figure 9. Floatability efficiency of particles from suspension as a function of different reagent conditions (cationic collector+different type polymers).

As shown in Table 6, floatability of 91.8% with the anionic collector (Aero 845) was obtained for 5 min. flotation time by conventional flotation. Floc flotation slightly increased the floatability of the fine particles about 5% in the presence of anionic surfactant and nonionic polymer, the highest floatability was achieved to 96.8% (Figure 10). As mentioned earlier, the electrostatic forces between the anionic surfactant and the anionic polymer having the same charge had a negative effect on the flotation efficiency. When compared with the results obtained with other polymers, low floatability value (92.4%) was achieved in the presence of anionic surfactant and an anionic polymer.

All compared results of conventional and floc flotation experiments that used anionic and cationic surfactants with different polymers were given in Fig. 11. It was seen that increasing flotation time from 2 min. to 5 min. increases the flotation recovery for both conventional and floc flotation.

Table 6. The results of floc-flotation experiments (anionic collector (Aero 845)/different type polymers) and conventional flotation experiment in mechanically agitated flotation cell.

		Floatability for 5 min. time (%)
Conventional flotation test	Aero 845 (Without polymer)	91,8
Floc-flotation test	Aero 845 / Anionic polymer (SPP 508)	92,4
	Aero 845/ Cationic polymer (Enfloc 440 C)	95
	Aero 845/ Nonionic polymer (SPP N 134)	96,8

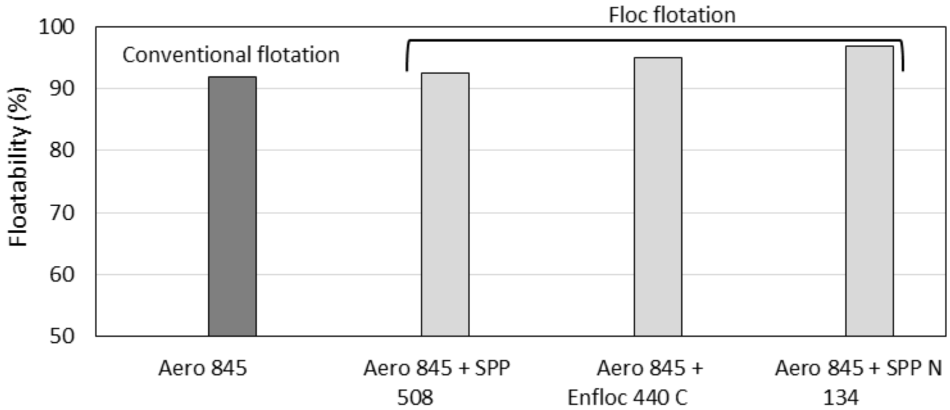

Figure 10. Floatability efficiency of particles from suspension as a function of different reagent conditions (anionic collector+different type polymers)

The higher floatability values were obtained with the anionic surfactant compared to the cationic surfactant. It was found that the floatability efficiency was increased to approximately 97% when a combination of the anionic surfactant and nonionic polymer was applied.

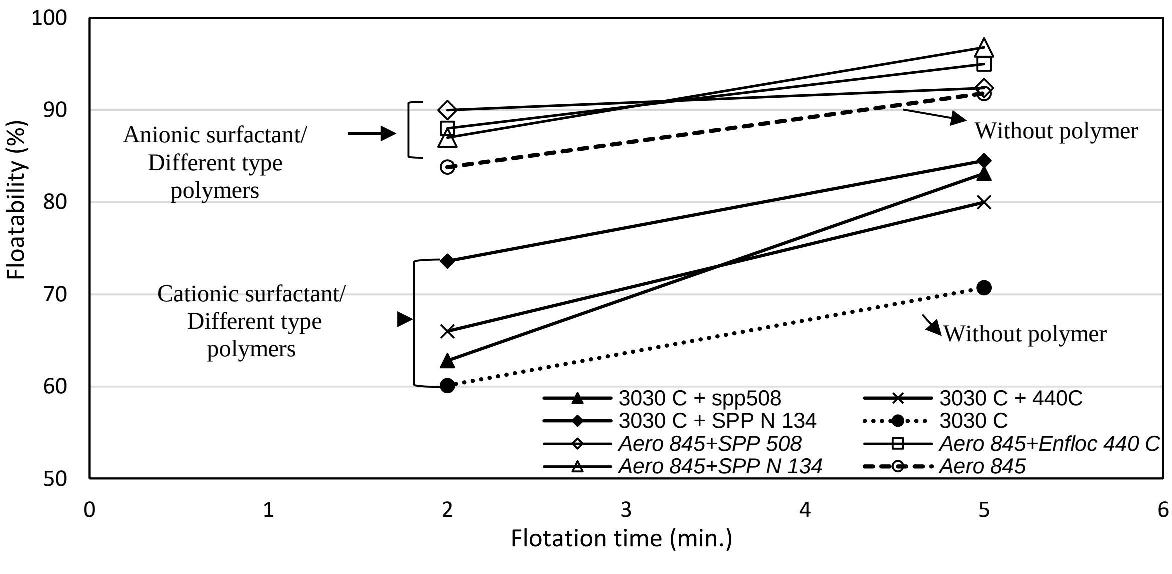

Figure 11. Comparative results of conventional and floc flotation experiments using anionic and cationic surfactants with different polymers

5. Conclusions

Floatability of very fine natural stone particles from wastewater in a mechanical flotation cell was investigated. Conventional flotation and floc-flotation were applied for the removal of fine particles from suspension. The effect of different parameters such as collector type and dosage, pH, flotation time and condition time on floatability of very fine particles has been determined by the conventional flotation. Floc-flotation experiments performed to increase the floatability of fine particles. The use of anionic and cationic surfactant to float particles which had been flocculated with different type polymers was investigated.

The floatability of particles was improved by aggregating the particles with a polymer prior to flotation. When the results of floc flotation and conventional flotation were compared, it has been found that higher floatability values were achieved with floc flotation. It was obtained 96.8% floatability of particles in presence of anionic surfactant (Aero 845, 750 g/t) and nonionic polymer (SPP N 134, 30 g/t) at natural pH (8) and 5 min. conditioning time for 5 min. flotation time by floc flotation in the mechanical cell.

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