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Research Paper / Makale

Removal of Suspended Particles from Wastewater by Conventional Flotation and Floc-flotation

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Abstract: In this study, the application of conventional flotation and floc-flotation methods to the wastewater containing suspended particles have been investigated. The experiments were carried out in two stages in the Jameson flotation Cell. The results obtained from the two methods were compared. The effect of flotation time and polymer type on the fine particle removal efficiency and residual turbidity was also determined. As a result of experimental studies, it was found that higher particle removal recovery and lower residual turbidity were obtained by floc-flotation compared to conventional flotation. Increasing flotation time increased fine particle floatability and decreased residual turbidity. In 1st stage of the experiments, 96.6% of the floatability recoveries and 304 NTU residual turbidity value was obtained with the combination of anionic polymer (SPP 508) and anionic surfactant (Aero 845) at the end of the 10 minutes flotation time. After 10 minutes of 2nd stage experiments, over 98% of floatability recoveries were obtained by floc flotation. The residual turbidity value of wastewater was decreased from the initial turbidity value of 12000 NTU to 78 NTU by anionic polymer, to 39 NTU by cationic polymer, and 27 NTU by a nonionic polymer.

Keywords: Wastewater treatment; Flocculation; Flotation; Floc-flotation

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Klasik Flotasyon ve Flok-Flotasyonu ile Atıksudan Süspanse Tanelerin Uzaklaştırılması

Öz: Bu çalışmada, askıda tane içeren atıksuya klasik flotasyon ve flok-flotasyonu yöntemlerinin uygulanması araştırılmıştır. Deneyler, Jameson flotasyon hücresinde iki aşamada yürütülmüştür. İki yöntemden elde edilen sonuçlar karşılaştırılmıştır. Aynı zamanda, ince tane uzaklaştırma etkinliği ve kalan bulanıklık üzerine flotasyon süresi ve polimer tipinin etkisi belirlenmiştir. Deneysel çalışmalar sonucunda, klasik flotasyona kıyasla flok-flotasyonu ile daha yüksek tane uzaklaştırma verimi ve daha düşük kalan bulanıklık değerlerinin elde edildiği bulunmuştur. Flotasyon süresini arttırmak, ince tanelerin yüzebilirliğini arttırmış ve bulanıklığı azaltmıştır. 1. aşama deneylerinde, 10 dak.flotasyon süresi sonunda anyonik polimer (SPP 508) ve anyonik toplayıcının (Aero 845) birlikte kullanılması ile %96.6 flotasyon verimi ve 304 NTU bulanıklık değeri elde edilmiştir. 2. Aşama deneylerinde flok-flotasyonu ile 10 dak. sonunda %98'in üzerinde yüzdürme verimleri elde edilmiştir. Atıksuyun kalan bulanıklık değeri, başlangıç bulanıklık değeri olan 12000 NTU'dan 78 NTU'ya anyonik polimer ile, 39 NTU'ya katyonik polimer ile ve 27 NTU'ya iyonlaşmayan polimer ile düşürülmüştür.

Anahtar Kelimeler: Atıksu arıtımı; Flokülasyon; Flotasyon; Flok-flotasonu

1. Introduction

Mining, metallurgical, petroleum and chemical industries generally produce wastewaters containing suspended particles, emulsified oils, process chemicals, organic and other impurities to be removed. Very fine particles are stable and remain suspended in suspensions, thus cause pollution in water into which they are discharged or reduced re-circulation water in processing plants. Wastewater

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<u>Bu makaleye atıf yapmak için</u> Taşdemir, T., Başaran, H. K., "Klasik Flotasyon Ve Flok-Flotasyonu İle Atıksudan Süspanse Tanelerin Uzaklaştırılması", El-Cezerî Fen ve Mühendislik Dergisi 2021, 8(1); 421-431. ORCID ID: ^a0000-0001-7817-1182; ^b0000-0002-3850-1332 treatment is required and must result in improved solid-liquid rapid separation, improved water quality. Many separation methods such as sedimentation filtration and flotation are widely used. All of these methods are largely dependent on the size of the particles to be removed. It is necessary to increase the particle size by coagulation /flocculation processes to achieve effective solid-liquid separation [1-2].

Flocculation and flotation processes have been used for the removal of fine suspended particles from wastewater and mineral slurries for a long time [3]. Flocculation is usually a necessary pretreatment step. The flocculation aim is to form aggregates or flocs from finely dispersed particles with natural or synthetic flocculants which are referred to as polymer. There are many types of polymer used in dewatering. Polymers are defined by their ionic nature (Anionic, cationic, and nonionic) and the important properties of polymers are molecular weight and charge density [4-5].

Flotation is a process in which mineral particles are removed selectively from suspension in water through attachment to air bubbles that have a common industrial practice in mineral processing. Recently, it is also being extended to wastewater treatment [6-8]. In wastewater treatment, this process is not required to be selective, as it aims to remove all particles from the liquid phase [9].

Particles in wastewater mostly small (from $-20 \ \mu m$ to colloidal sizes, $-1 \ \mu m$) and close to neutral buoyancy. The rate of removal of fine particles by flotation is rather slow, for particles smaller than 20 μm , low flotation recoveries typically obtained [10]. The main reason for the low floatation ratio of the fine particles is primarily due to the low mass and high surface area, which leads to a low collision and the possibility of particles adhering to air bubbles. The fine particle recovery in flotation can be improved either by decreasing the size of the air bubbles or by increasing the particle size [11-14]. The small bubbles can be produced via several methods such as dissolved air flotation (DAF, bubbles formed by the release of pressure in water saturated with air at higher pressure), induced air flotation (IAF, bubbles formed by injection along with high-speed agitation) electro flotation (microbubbles produced by electrolysis of a conducting solution) [15].

Increasing the particle size and thus increasing the rate of collection by the air bubbles can be achieved through several aggregation methods. 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. Flotation of fine particles after they have been aggregated by flocculation or coagulation is called floc flotation [16-21]. In floc flotation, fine particles are flocculated with a polymer and then added a collector that can interact with the flocs, rendering the flocs sufficiently hydrophobic for their attachment to air bubbles. The flocs adhering to air bubbles are floated readily, avoiding the problem of the 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.

In this study, Jameson cell has been applied first time in the literature to the treatment of natural stone wastewaters. Experiments were carried out as conventional flotation (without polymer) and floc flotation method in a laboratory-type Jameson cell by using wastewaters containing -20 μ m natural stone powders. In the experiments, the effects of flotation time and polymer type on removal efficiency and residual turbidity were investigated.

2. Materials and Method

2.1. Material

The sample used in the experiments was obtained from a natural stone factory located in Eskişehir, Turkey. In the factory, the wastewater collected after cutting is subjected to solid-liquid separation in the sedimentation tank by natural sedimentation (without the addition of flocculant) and the sludge is discharged into the waste site of the factory. A sufficient amount of natural stone sludge was taken from this area and then the sample was sieved by 20-micron sieve. In the conventional and floc-flotation experiments, wastewater samples prepared with -20 microns natural stone powder were used. The properties of wastewater are given in Table 1.

рН	Solid ratio	Initial Turbidity	Conductivity	Water hardness	Particle size
8	1%	12 000 NTU	580 µS/cm	356 mg/L CaCO ₃	-20 micron

Table 1.	Properties	of wastewater
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Natural sedimentation analyzes of the wastewater sample were made by measuring the turbidity values and taking samples at certain times in 500 mL measure. Figure 1 shows the time versus residual turbidity values of the sedimentation test. As can be seen, the residual turbidity of the sample decreased to 40 NTU with natural sedimentation after more than 5 hours.



Figure 1. Sedimentation analyses of wastewater sample

The chemical composition of the natural stone powder was analyzed by a X-ray fluorescence (XRF) using a Rigaku ZSX Primus device. A mineralogical analysis of particles was characterized by X-ray diffraction (XRD) using a Rigaku Rint-2000 model diffractometer.



Figure 2. X-ray diffraction pattern

XRD and XRF results are given in Figure 2 and Table 2, respectively. Most of the powders are composed of calcite mineral and the sample also contains quartz, albite, muscovite, microline minerals (Figure 2). According to the results of the chemical analysis given in Table 2, approximately 42% of the sample is composed of calcium oxide and about 17% is SiO₂.

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CaO%	SiO ₂ %	$Al_2O_3\%$	K ₂ O%	Na ₂ O%	MgO%	Fe ₂ O ₃ %	TiO ₂ %	LOI%
42.32	16.81	3.5	1.14	1.06	0.87	0.73	0.09	33.48

 Table 2. Chemical analysis

Electrokinetic measurements of suspended particles in wastewater were performed by using the Malvern Nano-Z model zetameters. Figure 3 shows the zeta potential of the sample as a function of pH. Suspended particles in wastewater are negatively charged in the pH range 6-11.



Figure 3. Zeta-potential measurement of natural stone powders

The commercial names and properties of reagents used in the experiments are given in Table 3. 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. These reagents were provided by Cytec. Three types of polymers were used. SPP 508 is an anionic polymer with high molecular weight and 28% of charge density. Enfloc 440C, a cationic polymer, has high molecular weight and 55% of charge density. SPPN 134 is a non-ionizing polymer. SPP polymers were supplied by the Superkim and Enfloc polymer provided by ECS Chemistry.

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Commercial name	Туре	Supplier
SPP 508	Anionic polymer	Superkim
Enfloc 440C	Cationic polymer	ECS Chem.
SPP N 134	Nonionic polymer	Superkim
Aero 845	Anionic surfactant	Cytec
Aerorfoth 65	Frother	Cytec

 Table 3. Used Reagents

2.2. Method

Conventional and floc-flotation experiments were carried out by the Jameson flotation cell produced at Multiphase Processes Centre, Newcastle University, and established at Mineral Processing Laboratory of Eskişehir Osmangazi University (Figure 4). The Jameson Cell is a device for bubble and particle contact where a plunging jet naturally entrains air, achieving a high void [22]. It consists of a downcomer (21 mm), separation cell (145 mm), and a conical type nozzle (3 mm). There are flowmeter and pressure gauge in the feed line and airflow rotometer in the airline. Conditioning tank with a stirrer and peristaltic pumps mounted in the cell. Pumps can adjust the flow rate for feeding and recirculating of wastewater. Wastewater was conditioned with chemical

reagents in the tank before conventional and floc-flotation. After conditioning, the suspension is delivered to the nozzle under pressure by a peristaltic pump.



Figure 4. Jameson flotation cell

Conventional and floc flotation experiments in the Jameson Cell were carried out in two stages. At the end of the 1st stage of the flotation test, the wastewater from here was remixed with the reagents in the conditioning tank and then the 2nd stage flotation experiments were carried out. The test conditions are given in Table 4. In the experiments, the solid ratio, pH, surfactant, and frother dosage, condition time were kept constant.

Solid ratio	1%
Feed flow rate	6.5 L/min
рН	8 (natural)
Condition time	2 minute
Frother type and dosage	Aerofroth 65 (30 ppm)
Surfactant type and dosage	Anionic, Aero 845 (750 gr/t)
Flotation time	2, 5 and 10 minute
	Anionic, SPP 508 (0.3 mg/L)
Polymer type and dosage	Cationic, Enfloc 440 C (0.3 mg/L)
	Nonionic, SPP N 134 (0.3 mg/L)

Table 4.	Experimental	conditions
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The effects of polymer type and flotation time on flotation efficiency were investigated by using combinations of different types of polymer and anionic collector. Experiments aim to remove all the particles floating through air bubbles from the wastewater sample containing 1% solids. In each experiment, 20 liters of wastewater were filled into the tank and conditioned by adding the required

amount of reagents (polymer, surfactant, and frother) and then fed to the cell by a pump. At the end of the 2, 5, and 10 minute flotation times, flocs/fine particles removed from the upper part of the cell were dried and weighed. The flotation recovery of the fine particles was calculated by using the amounts of solid obtained.

Continuous gravimetric analysis is difficult in the wastewater treatment process so that indirect methods like turbidity measurement are used. Turbidity can be determined with simple optical methods, which is a quantitative measure of remaining suspended particles, organic and inorganic matter, soluble colored organic compounds, and other microscopic organisms. Therefore, in the tests, residual turbidity measurements were performed to determine the cleaning efficiency of wastewater. The residual turbidity values of the cleaned wastewater (as NTU unit) were measured using the HF Scientific turbidimeter (Figure 5a). The pH, conductivity, temperature measurements of natural stone powder suspension were measured with Orion 5 star multimeter device (Figure 5b).



Figure 5. The HF Turbidimeter (a) and Orion 5 star multimeter (b)

3. Experimental Results

3.1. First Stage Experiments

The dosage of the surfactant (750 g/t) and polymers (0.3 mg/L) used in the experiments were determined in a preliminary study and was taken as a constant [23]. In the experiments, it is aimed to reach the highest flotation recovery and to reduce the wastewater turbidity. Figure 6 shows the effect of different reagent conditions and flotation time on flotation efficiency in 1st stage experiments of conventional and floc-flotation. As seen in figure 6(a), the increase in the flotation time increased the flotation recovery. Floatability values obtained from 1st stage tests of conventional and floc flotation were similar. Compared to the test results using cationic and nonionic polymers or without polymer, high floatability was obtained with anionic polymer for all flotation times.

In wastewater treatment, it is important to determine the optimum flotation time to achieve high floatability efficiency in the floatation of very fine particles. Figure 6 (b) shows the flotation recovery values obtained for 2, 5, and 10 minute flotation times and column charts of comparative results. Time on floatability efficiency has a significant effect. For a flotation time of 2 minutes, while 56.1% floatability was obtained with conventional flotation, the highest flotation recovery obtained by floc flotation was 59.8% in the experiment using anionic polymer. At the end of the flotation time of 10 minutes, 91.8% flotation recovery was obtained with conventional flotation. It was also achieved more than 95% recoveries in all tests of floc flotation. The highest floatability efficiency of 1st stage experiments was 96.6%, which was obtained by anionic polymer and the optimum time was determined to be 10 minutes.



Figure 6. Effect of different reagent conditions and flotation time on flotation removal efficiency in 1st stage experiments of conventional and floc-flotation

The results of the residual turbidity of wastewater according to the flotation time and reagent conditions (no polymer and different type polymers) are given in Figure 7. As seen, much lower turbidity values were obtained by floc flotation compared to conventional flotation. The flocculation of the particles before flotation and the higher probability of adhesion to air bubbles of flocs during flotation contributed to the reduction of wastewater turbidity. Similarly, wastewater residual turbidity decreased with increasing flotation time (Figure 7 a). In conventional flotation, for the flotation times of 2, 5, and 10 minutes, 1612 NTU, 1200 NTU, and 780 NTU turbidity values were obtained respectively (Figure 7b). In floc flotation, the initial turbidity value (12000 NTU) was reduced to 304 NTU with an anionic polymer, to 375 NTU with a cationic polymer, and 400 NTU with a nonionic polymer. It is also evident in the column graph of Figure 7 (b) that in the 1st stage experiments the flotation time significantly affects the residual turbidity in the presence or absence of the polymer.



Figure 7. Effect of different reagent conditions and flotation time on residual turbidity of wastewater in 1st stage experiments of conventional and floc-flotation

In the 1st stage experiments performed in the combination of anionic flocculant (SPP 508) and anionic collectors (Aero 845), the best result obtained is that, for flotation time of 10 minutes, the initial turbidity value decreased from 12 000 NTU to 304 NTU with 96.6% floatability efficiency. Başaran and Taşdemir (2014) previously determined the flocculation characteristics of the sample

used in the experiments and the lowest turbidity values were obtained with the anionic polymer (SPP 508) [24]. At the same time, they observed that the flocs formed with the anionic polymer have a large compact structure. Similar results have been reported in other studies [25-27]. Ensuring good flocculation and robust floc with anionic polymer has contributed to the increase of particle removal efficiency in flotation.

3.2. Second Stage Experiments

Second stage experiments were performed to re-float the fine particles that could not be removed from the wastewater in the first stage experiments. The same experimental conditions in the first stage were also applied in the second stage. Figure 8(a-b) shows the effect of different reagent conditions and flotation times on flotation recovery in 2nd stage experiments of conventional and floc-flotation. When the results are compared, it is possible to say that higher floatability efficiency is obtained by floc flotation than conventional flotation. In the second stage experiments, there are obvious differences between conventional and flock flotation test results for the removal of fine particles from wastewater. The increase in the flotation time increased the flotation recovery in conventional flotation, however, flotation time had little effect on recovery in floc flotation (Figure 8 a).

Figure 8 (b) shows data and column graphs obtained at the end of 2, 5, and 10 minute flotation times. As can be seen, while 96.4% flotation recovery was achieved in conventional flotation, recoveries were higher than 98% for all conditions in floc flotation. The recycling of wastewater in the cell (operation of the cell in two stages) increased the removal efficiency of particles. Also, it can be said that high recovery is due to the production of small size bubbles in the Jameson cell and the formation of the intensity bubble and particle/floc contact. Previous studies on this subject have revealed that 95% of the suspended solids, oil, and grease, algae, precipitated phosphorus can be removed with the Jameson flotation cell [28-30].



Figure 8. Effect of different reagent conditions and flotation time on flotation recovery in 2nd stage experiments of conventional and floc-flotation

The measured residual turbidity values of the wastewater according to the flotation times are given in Figure 9. it has been shown that increasing the flotation time reduces residual turbidity of wastewater. The lowest turbidity value obtained at the end of the flotation time of 10 minutes in conventional flotation was 293 NTU. Applying the same flotation time, the residual turbidity value of wastewater was decreased to 78 NTU by anionic flocculant, to 39 NTU by cationic flocculant, and 27 NTU by nonionic flocculant. In Figure 10, general comparative results of the first and second stage experiments are presented together and better results were obtained in the second stage experiments. It was determined that flotation time has a significant effect on the residual turbidity and flotation efficiency in the 1st stage experiments.



Figure 9. Effect of different reagent conditions and flotation time on residual turbidity of wastewater in 2nd stage experiments of conventional and floc-flotation

However, there was very little effect of the time in the 2nd stage experiments. If very low turbidity values and flotation efficiency above 98% are desired, wastewater treatment should be applied in two stages in the Jameson Cell.



Figure 10. Comparative results of 1st Stage and 2nd Stage experiments in Jameson cell

3. Conclusions

Flocculation and flotation methods are effective methods that are widely used in wastewater treatment. In this study, the first time in literature, conventional flotation and floc flotation methods were applied to remove fine particles in the wastewater of natural stone by Jameson flotation cell and the results were evaluated comparatively. The effects of flotation time and the use of different

reagents on particle floatability and residual turbidity were also investigated. The experiments were carried out in two stages and the following results were obtained:

- Higher flotation recoveries and lower wastewater turbidity were obtained by floc flotation compared to conventional flotation.
- It was determined that flotation time significantly affected the floatability efficiency in conventional and floc flotation in the first stage experiments and increasing the flotation time increased the floatability efficiency.
- It was found that polymer type had an important effect on fine particles removal from wastewater in floc flotation.
- In the 1st stage experiments performed in the combination of anionic flocculant (SPP 508) and anionic collectors (Aero 845), the best result obtained is that, for flotation time of 10 minutes, the initial turbidity value decreased from 12 000 NTU to 304 NTU with 96.6% floatability efficiency.
- In the 2nd stage experiments, the lowest turbidity value obtained at the end of the flotation time of 10 minutes in conventional flotation was 293 NTU. The residual turbidity value of wastewater was decreased to 78 NTU by anionic flocculant, to 39 NTU by cationic flocculant, and 27 NTU by nonionic flocculant.

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