

MODELLING OF MAGNESITE FLOTATIONS WITH TWO DIFFERENT COLLECTORS: BIOCOLLECTOR AND OLEATE

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Keywords

Bacillus subtilis
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Abstract

As environmental concerns grow, flotation researches, particularly for the recovery of fine-grained ores, encourage "bioflotation" studies, in which biological origin alternatives are used instead of traditional flotation reactivities. While bioflotation applications starting from the pyrite mineral have spread to many carbonate and oxide minerals over time, and biomaterials used as a bioreagent have diversified from the microorganism itself to its metabolites and even cell components.

In this article, the use of "surfactin" derived from Bacillus subtilis as a bio-collector in the flotation of magnesite was investigated. The results of bioflotation studies were compared to those of oleate, traditional magnesite collector. Moreover, process models were created with statistical design methods, and the verification results of the optimization studies using model data showed that these models were statistically strong.

İKİ FARKLI TOPLAYICI İLE YAPILAN MANYEZİT FLOTASYON SÜREÇLERİNİN MODELLENMESİ: BİYOTOPLAYICI VE OLEAT

Anahtar Kelimeler

Bacillus subtilis
Biyoflotasyon
Manyezit flotasyonu

Öz

Çevresel kaygıların artması, özellikle ince taneli cevherlerin geri kazanılması için yapılan flotasyon araştırmaları, geleneksel flotasyon reaktifleri yerine biyolojik kökenli alternatiflerin kullanıldığı "biyoflotasyon" çalışmalarını teşvik etmektedir. Pirit

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Sümfaktin İstatistiksel deney tasarımı mineralinden başlayan biyoflotasyon uygulamaları zamanla birçok karbonatlı ve oksitli minerallere yayılmış, reaktif olarak kullanılan biyolojik maddeler ise mikroorganizmanın kendisinden metabolitlerine ve hatta hücre bileşenlerine kadar çeşitlenmiştir. Bu makalede, Bacillus subtilis'ten elde edilen "sümfaktin" in manyezit flotasyonunda biyotoplayıcı olarak kullanımı araştırılmıştır. Biyoflotasyon çalışmalarının sonuçları, geleneksel manyezit toplayıcısı olan oleatin sonuçlarıyla karşılaştırılmıştır. Ayrıca istatistiksel tasarım yöntemleri ile süreç modelleri oluşturulmuş ve model verileri kullanılarak yapılan optimizasyon çalışmalarının doğrulama sonuçları bu modellerin istatistiksel olarak güçlü modeller olduğunu göstermiştir.

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1. Introduction

The gradual depletion of the world's mineral resources has long driven researchers to develop technologies to prolong the life of these resources and at the same time make them economically viable. Today, the flotation method is widely used in fine sized minerals, particularly metal sulfides. However, rapid industrialization has threatened life on Earth by polluting the environment, and as a result, mining, ore preparation, and particularly flotation areas have begun to seek more eco-friendly processes, as in all industrial applications. Thanks to the advancements in the field of biotechnology, it has been discovered that several microbial surface-active metabolites reduce the surface tension (Gautam and Tyagi, 2006). Thus, their use in flotation and flocculation methods provided numerous benefits in terms of environmental aspects and even process costs (Behera and Mulaba-Bafubiandi, 2017; Smith and Miettinen, 2006; Otsuki, 2016). Flotation processes that use microorganisms, metabolites, or microbial components instead of traditional reagents are referred to as "bioflotation" applications (Dwyer, Bruckard, Rea and Holmes, 2012).

In fact, studies using *At. ferrooxidans* as a pyrite depressant are the first bioflotation experiments encountered in the literature (Atkins, Bridgwood, Davis, and Pooley, 1987; Davis and Atkins, 1988; El Zeky and Attia, 1987; Townsley, Atkins, and Davis, 1987). These studies have expanded from the pyrite mineral (Amini, Hosseini, Oliazadeh and Kolahdoozan, 2009; Attia, Elzaky and Ismail, 1993; Mehrabani, Mousavi and Noaparast, 2011; Nagaoka, Ohmura and Saiki, 1999) to variety of carbonate (Botero, Torem and de Mesquita, 2008; Kim, Park, Choi, Gomez-Flores, Han, Choi and Kim, 2015; Zheng, Arps and Smith, 2001), silicate (Farahat, Hirajima, Sasaki, Aiba and Doi, 2008; Natarajan and Padukone, 2012; Yang, Feng, Li, Wang and Teng, 2014; Zhao, Wu, Zhang, Zhu and Tan, 2017), and oxide (Farghaly, Abdel-Khalek, Abdel-Khalek, Selim and Abdullah, 2020; Sarvamangala and Natarajan, 2011; Yang, Li, Tang, Wang and Ma, 2013) minerals. The

bioreagents used in this process have evolved from microorganisms (Consuegra, Kutschke, Rudolph and Pollmann, 2020; Lopez, Merma, Torem and Pino, 2015; Merma, Torem, Moran and Monte, 2013) to its metabolites (Gholami and Khoshdast, 2020; Sanwani, Chaerun, Mirahati and Wahyuningsih, 2016; Yehia, Khalek and Ammar, 2017) and in more recently, to their cellular components (Bleeze, Zhao and Harmer, 2018; La Vars, Quinton and Harmer, 2021; Santhiya, Subramanian and Natarajan, 2002; Vasanthakumar, Ravishankar and Subramanian, 2014).

Among these metabolites "surfactin" is one of the most powerful biosurfactant molecule produced by the culture broth of *Bacillus* spp (Arima, Kakinuma and Tamura, 1968). At low concentrations, it has been proved to reduce the surface tension of water from 71.6 to 28 mNm⁻¹. (Ohno, Ano and Shoda, 1995). In a recent study, the frothing properties of a biosurfactant obtained from *Lactobacillus pentosus*, including surfactin, were evaluated (Vecino, Devesa-Rey, Cruz and Moldes, 2013). The results of this research indicated that, the adsorption properties of the biosurfactant make it a potential frothing agent in flotation. According to similar research, surfactin's exceptional frothing properties were demonstrated by its greater ability to form and stabilize foam at concentrations as low as 0.05 mg/mL (Razafindralambo, Paquot, Baniel, Popineau, Hbid, Jacques and Thonart, 1996). Furthermore, a study demonstrates bioflotation as a promising alternative cleaning technology for the recovery of hematite from iron ore tailings by using a biosurfactant extracted from *Rhodococcus opacus* as a "green" collector (Pereira, Hacha, Torem, Merma, Silvas and Abhilash, 2021). Additional flotation studies also revealed that *R. opacus* has a significant potential as a biocollector of calcite and magnesite (Botero, Torem and de Mesquita, 2007).

Although culturing microorganisms is a costly and time-dependent process, bioflotation applications still have the potential to make this process more profitable on the industrial scale (Banat, 1995; Kinnunen, Miettinen and Bomberg, 2020; Otsuki, 2016; Rahman and Gakpe, 2008; Souza, Vessoni-Penna and de Souza Oliveira,

2014; Varjani and Upasani, 2016; Whang, Liu, Ma and Cheng, 2008). There are still some obstacles to overcome, such as making bioreagent production more economical and easier in large amounts.

In this study, the usability of "surfactin", which has been reported to be used only as a biofrother in the literature, as a magnesite biocollector was investigated. Also, the results of bioflotation were compared with the classical magnesite flotation data obtained with oleate. In all flotation experiments, mathematical models of the process were created by using one of the experimental design methods, Central Composite Design (CCD), the estimation of the optimum conditions and the results were obtained, and the verification experiments were also carried out.

2. Materials and Methods

All analyses and experiments for magnesite flotation were performed comparatively in two parallel sets for surfactin and oleate.

The authors declared that research and publication ethics were followed in this study.

2.1. Materials

Magnesite ore with a purity of 96.4% was obtained from the Eskişehir-Çukurhisar region for this study. X-ray fluorescence spectrometry (XRF Panalytical Zetium, Malvern Panalytical Inc., UK) and X-ray diffraction (XRD Panalytical Empyrean, Malvern Panalytical Inc., UK) analyses were carried out in ESOGU ARUM laboratories to determine the content of magnesite samples. Chemical composition of magnesite studied indicates that 46.108% MgO, 0.171% SiO₂, 0.220% Al₂O₃, 0.475% CaO, 0.070% CuO, 0.034% NiO, 0.045% Fe₂O₃, 52.887% loss of ignition. The results of XRD analysis of magnesite sample are represented in Figure 1.

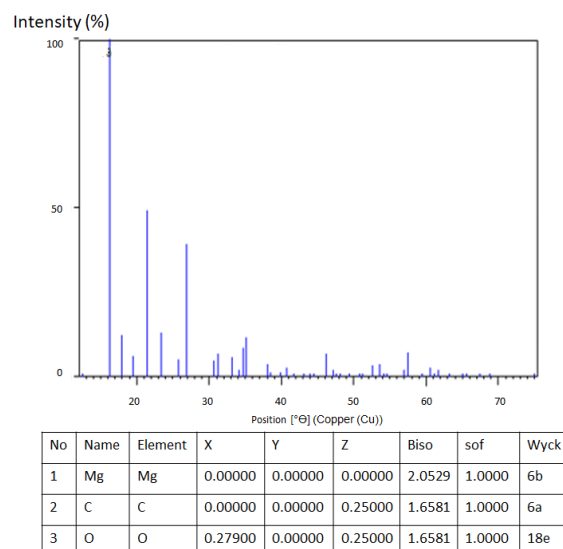


Figure 1. XRD Pattern of Magnesite

Magnesite sample was ground to -0.150 mm by closed-circuit dry grinding-sieving in a laboratory-scale jar type ceramic ball mill to avoid contamination.

Surfactin, a bioreagent obtained from *Bacillus subtilis*, and sodium oleate (SigmaAldrich, Germany) were used as collectors in magnesite flotation. The pH of the experiments was adjusted using analytical grade NaOH and H₂SO₄. Because of surfactin's low water solubility and high molecular weight, organic solvents are usually required to increase its solubility. The solvent used for surfactin purification was ethyl acetate, and the surfactin/ethyl acetate ratio was calculated to be 1/25. Frothing reagent was not used in flotation experiments since surfactin (Shaligram and Singhal, 2010) and oleate had frothing properties.

2.2. Methods

2.2.1. Biosurfactant Extraction

Surfactin is a bacterial cyclic lipopeptide which include both hydrophilic and hydrophobic parts. General chemical formula of surfactin is C₅₃H₉₃N₇O₁₃.

Following the instructions of the study by (Cakmak, Gungormedi, Dikmen, Celik and Cabuk, 2017), *Bacillus subtilis* was used for the production of the biosurfactant. The cell free supernatant containing the biosurfactant was adjusted to pH 2.0 with the addition of HCl (2 M) and stored at 4 °C overnight for the extraction. The solution was centrifuged (10.000 rpm at 4°C for 10 min) and the precipitate was dissolved in etyl acetate. A vacuum concentrator (Labconco Centrivap Concentrator, Labconco Inc., USA) was used to concentrate the final solution.

2.2.2. Flotation Experiments

Flotation experiments were divided into two groups: bio flotation with surfactin and classical flotation studies with oleate. Flotation experiments were carried out in a Denver laboratory scale mechanically stirred flotation machine. Since a high purity magnesite sample was used in the experiments, the results were based solely on magnesite weight yield (WY). Equation 1 was used to calculate magnesite WY.

$$WY = 100 \frac{C}{F} \tag{1}$$

where, WY is the magnesite weight yield (%); C is amount of the floating material (g); F is amount of the feed (g).

In both sets of experiments, conditioning time was kept constant at 8 min and air velocity 5 l/min. Froth was collected through 150 sec. In experiments using oleate, the temperature was kept constant at 26-28 °C while ambient temperature was applied at surfactin flotation.

Central Composite Design (CCD) method, one of the statistical experimental design methods, was applied in all systematic experiments. The α coefficient used in the calculation of the axial points was chosen as 1.6, which is one of the values suggested by the software used. Three parameters were chosen to examine the effect on the amount of weight yield in systematic experiments and to create a mathematical model of the process based on these parameters. Parameters whose effects were examined along with their levels are given in Table 1. According to the chosen method, a total of 20 experiments were performed for each reagent ($2^3+2 \times 3+2 \times 3$). The results of the experimental studies were subjected to analysis of variance (ANOVA) and the statistical significance of the effects of the parameters was evaluated within the 95% confidence interval. The terms with no statistically significant effect at the 95% confidence interval were removed from the model and a "reduced ANOVA Table" was obtained. While creating the reduced ANOVA Table, the main effect terms were

kept in the model even if the effect was insignificant. Thus, the model equations depending on the parameters examined for the effect of the flotation process were obtained.

Table 1
The Examined Effects of the Parameters and Their Levels

Factors		Levels				
Parameters		$-\alpha$	-1	0	+1	$+\alpha$
A	Solid ratio (%)	21.6	25	30	35	38.41
B	pH	6.5	7.5	9	10.5	11.5
C	Collector amount (g/t)					
	Surfactin	65	200	400	600	735
	Oleate	140	1500	3500	5500	6900

After the mathematical models were created, the optimum conditions at the studied levels of the parameters and the magnesite weight yield to be obtained under these conditions were estimated for each experimental set by using convenient module of the software, and the verification experiments were also performed.

3. Results and Discussion

3.1. Flotation Experiments

Table 2 shows the magnesite weight yield results from two sets of magnesite flotation experiments, in which surfactin and oleate were used as collectors, as well as the experiment parameters and levels.

Table 2

Flotation Experiments Results

Exp. No	Parameters and Levels				Weight Yield (%)	
	Solid Ratio (%)	pH	Collector Amount (g/t)		Surfactin	Oleate
1	25	7.5	Surfactin 200	Oleate 1500	42.03	41.7
2	35	7.5	200	1500	49.61	51.1
3	25	10.5	200	1500	39.10	43.8
4	35	10.5	200	1500	51.41	51.3
5	25	7.5	600	5500	55.75	69.2
6	35	7.5	600	5500	60.43	71.9
7	25	10.5	600	5500	53.21	64.7
8	35	10.5	600	5500	60.05	66.1
9	21.60	9.0	400	3500	41.81	45.6
10	38.40	9.0	400	3500	56.57	61.2
11	30	6.5	400	3500	58.39	67.1
12	30	11.5	400	3500	57.18	70.0
13	30	9.0	65	140	29.83	18.9
14	30	9.0	735	6900	63.23	68.9
15	30	9.0	400	3500	54.85	49.3
16	30	9.0	400	3500	50.47	53.4
17	30	9.0	400	3500	52.18	55.7
18	30	9.0	400	3500	48.76	52.1
19	30	9.0	400	3500	50.01	51.9
20	30	9.0	400	3500	48.53	53.0

The reduced ANOVA table obtained from the statistical analyses is also shown in Table 3 for both sets. This Table also includes the coefficient of determination (R^2), and predicted R^2 ($P-R^2$) values of the mathematical models obtained. The consistency of a model with experimental data is represented by R^2 value which is the proportion of the variance in the dependent variable. The compatibility of the model predictions with the experimental data for both surfactin and oleate flotation experimental sets is also shown graphically in Figures 2a and b, respectively. The power of a model is determined by $P-R^2$ which is a measure of how well the model predicts a response value (Khuri and Mukhopadhyay, 2010).

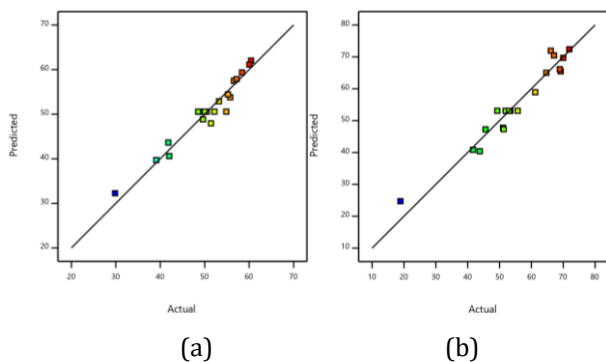


Figure 2. Consistency of a model with experimental data (a) for surfactin (b) for oleate

Table 3

Analysis of Variance Summary (ANOVA) Results.

Source	Surfactin	Oleate	
	p-value	p-value	
Model	<0.0001	<0.0001	significant
A-Solid Ratio	<0.0001	0.0027	significant
B-pH	0.4525	0.8132	
C-Collector Amount	<0.0001	<0.0001	significant
B ²	0.0002	<0.0001	significant
C ²	0.0004	0.0107	significant
Lack of fit	0.7042	0.0771	insignificant
R ²	0.9435	0.9441	
P.R ²	0.8776	0.8278	

Mathematical models of two magnesite flotation sets were also developed. Equation 2 and 3 show the models for magnesite flotation with surfactin (for coded and actual values, respectively), and Equation 4 and 5 show the models for magnesite flotation with oleate (for coded and actual values, respectively). Proposed models by software for surfactin and oleate are significant as p values of models are less than 0.05. p values of Lack of

fit for all models are greater than 0.05 which indicates experimental error is statistically insignificant.

$$WY (\%) = 50.57 + 4.12 A - 0.4456 B + 6.59 C + 2.84 B^2 - 2.55 C^2 \quad (2)$$

$$WY (\%) = 107.53942 + 0.823517 \text{ Solid Ratio} - 23.04859 \text{ pH} + 0.083944 (\text{Collector Amount}) + 1.26397 \text{ pH}^2 - 0.000064 (\text{Collector Amount})^2 \quad (3)$$

$$WY (\%) = 53.09 + 3.46 A - 0.2287 B + 12.31 C + 6.01 B^2 - 2.71 C^2 \quad (4)$$

$$WY (\%) = 220.12936 + 0.691755 \text{ Solid Ratio} - 48.20809 \text{ pH} + 0.010893 (\text{Collector Amount}) + 2.66976 \text{ pH}^2 - 6.7703 \times 10^{-7} (\text{Collector Amount})^2 \quad (5)$$

When the parameter effects and model terms are examined individually, the absence of the interaction term indicates that there is no significant interaction

between the parameters whose effects are investigated in the studied range. It is also noteworthy that the effect of pH is similar in both reagents.

3.2. Verification Results

The possible optimum conditions and estimated weight yield values for magnesite flotation obtained from the model equations created with the help of the relevant module of the software are given in the Table 4. Afterwards, a verification test was performed by using estimated optimum conditions. The same Table also includes the weight yield estimations and the verification results obtained under these conditions. As can be seen from Table 4 verification test results fell in estimated values that indicates the validity and adequacy of the predicted models.

Table 4

Optimum Conditions, Estimated and Experimental Results.

	Parameter		Weight Yield (%)			Exp. Result
			Estimated Values			
	Parameter	Value	Minimum	Average	Maximum	
1 (Surfactin)	A (Solid Ratio; %)	35	59.58	62.01	64.46	63.15
	B (pH)	7,5				
	C (Coll. Amount; g/t)	600				
2 (Oleate)	A (Solid Ratio; %)	35	67.91	71.93	75.95	68.52
	B (pH)	10,5				
	C (Coll. Amount; g/t)	5500				

4. Conclusions

In this study, the usability of surfactin, which is used as a biocollector, in magnesite flotation was investigated. In addition, the obtained results were compared with the results of a set of classical magnesite flotation using oleate to demonstrate the efficacy of surfactin.

In the design method used, the parameters were studied for 3 levels (-1; 0; +1 as coded levels). When the results for the studied levels of the parameters are examined, it can be seen that increasing the amount of reagent in both flotations increases the weight yield and the maximum yields were obtained in the amount of collector coded as +1. Maximum yields were approximately 60.4% at 600 g/t collector amount for surfactin, while approximately 72% at 5500 g/t collector amount for oleate. Considering the possible effect of using more amount of collectors (+alpha collector levels; 735 g/t for surfactin; 6900 g/t for oleate), it can be seen that the increase in the amount of surfactin increases the weight yield (to 63.23%), while the increase in the amount of oleate affects the weight

yield negatively (approximately 69%). It was thought that the reason for this effect in oleate was that the use of excessive amounts of reagents caused micelle formation and adversely affected the process. The result obtained in surfactin showed that higher weight yield could be obtained with the increasing reagent amounts. When comparing the two flotation reagents in terms of collector amount: oleate consumes 9 times more than surfactin that was quite remarkable. Regarding the flotation temperature, surfactin works effectively at ambient temperatures, whereas oleate requires a higher temperature, such as 27°C. Although oleate has a significant cost advantage in terms of reagents, the fact that surfactin was used in much lower amounts per unit ore, that it works independently of temperature, and, most importantly, that it is eco-friendly, makes it more prominent. These data, however, must be clarified through detailed feasibility analyses. Nonetheless, novel strategies for increasing surfactin yields are required to reduce production costs.

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Contribution of Researchers

Author1, Author2, and Author3 contributed to literature research and experimental studies. Author4 and Author5 contributed to biosurfactant production and funding acquisition. Author6 and Author7 contributed to project consultancy and evaluation of the results.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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