NÖHÜ Müh. Bilim. Derg. / NOHU J. Eng. Sci., 2024; 13(1), 149-155

Niğde Ömer Halisdemir Üniversitesi Mühendislik Bilimleri Dergisi
Niğde Ömer Halisdemir University Journal of Engineering Sciences

Araştırma makalesi / Research article

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Flotation of low-rank coal slimes

Düşük dereceli kömür şlamlarının flotasyonu

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Abstract

During coal production, a significant amount of fine coal dust is generated. Utilizing these high-ash coals is of great importance for the national economy. This study aims to recover the slime of the coal washing plant located in the Kütahya-Tuncbilek region by flotation. The experimental parameters studied include the collector dosage, dispersant dosage, frother dosage, flotation time, air flow rate, and solid ratio. Flotation performance was evaluated by determining the ash, calorific value, sulfur, volatile matter, fixed carbon, and combustible recovery values of the products obtained at the end of the experiment. As a result of the experimental studies, the highest combustible matter recovery value was obtained using 2000 g/t sodium silicate, 2000 g/t diesel oil, and 100 g/t MIBC with a solid ratio of 10%, a flotation time of 4 minutes, and an air flow rate of 4 L/min. Thus, marketable coal was produced with 6312 kcal/kg calorific value, 0.95% sulfur, and 22.4% ash content.

Keywords: Enrichment, Flotation, Lignite, Low-rank coal, Slime,

Öz

Kömür üretimi sırasında yüksek miktarlarda ince boyutlu toz kömür ortaya çıkmaktadır. Yüksek kül içerikli bu kömürlerin değerlendirilmesi ülke ekonomisi açısından büyük önem taşımaktadır. Bu çalışmada Kütahya-Tunçbilek bölgesinde bulunan lavvar tesisindeki tikiner altı şlamlı malzemenin flotasyonla zenginleştirilerek ekonomiye kazandırılması amaçlanmıştır. Toplayıcı miktarı, köpürtücü miktarı, flotasyon süresi, hava akış hızı ve katı oranı çalışılan deneysel parametrelerdir. Flotasyon performansı, deney sonunda elde edilen ürünler üzerinde kül, kalori, kükürt, uçucu madde, sabit karbon ve verim değerleri belirlenerek değerlendirilmiştir. Gerçekleştirilen deneysel çalışmalar sonucunda, 4 L/d hava akış hızında, 4 dakika flotasyon süresinde, %10 katı oranı, 2000 g/t Sodyum silikat, 2000 g/t mazot ve 100 g/t MIBC kullanılarak gerçekleştirilen en yüksek verim değerine sahip deney sonucu ile 6312 kcal/Kg ısıl değere sahip %0.95 kükürt ve %22.4 kül içerikli satılabilir kömür elde edilmiştir.

Anahtar kelimeler: Zenginleştirme, Flotasyon, Linyit, Düşük dereceli kömür, Şlam,

1 Introduction

In recent years, with the continuous development of coal mining and the transition to mechanized methods, the amount of coal slime produced in coal preparation plants has increased significantly. Coal slime is often used as a fuel for power generation, resulting in significant waste of resources and environmental pollution [1]. The industrial use of coal slime has been significantly limited due to its high moisture content, high viscosity, high ash content and low calorific value [2,3]. In order to use this part of the coal efficiently and to reduce pollution, the slime is usually dried to increase the calorific value or enriched by flotation to reduce the gangue content [4,5]. Flotation has been widely used in the enrichment of fine-sized coals for decades [1]. However, it is difficult to separate coal gangue, which consists mainly of quartz, kaolin and montmorillonite, by flotation due to the abundance of oxygen-containing functional groups on the low-grade coal surface [3,6]. Fine slime containing high amounts of clay tends to coat the surface of coal particles by electrostatic attraction and reduce surface hydrophobicity, resulting in a low recovery rate of combustible materials. In addition, due to their lightness and small particle size, clay minerals are dragged into the concentrate by the mechanical transport mechanism, further reducing the flotation efficiency. Sodium silicate is a widely used dispersant in coal flotation to suppress quartz and silicate minerals [7].

About half of all global coal deposits are bituminous coal and lignite, which are very abundant in China, Australia, Turkey, India and the northern United States. Lignite mining is of great importance today [6]. Lignite, one of the main riches of our country, is evaluated by both the state and the private sector, as it is both an energy raw material and an important source of industry. Turkish lignites are low rank coals of which calorific values varies between 1000 and 4200 Kcal/kg. The upper calorific value of lignite coal in the Afşin-Elbistan basin, where the largest reserve is located, is 1000-1400 kcal/kg, while the lower calorific value is in the range of 900-1250 kcal/kg [8].

In the coal washing plant located in the Tuncbilek district of Kütahya province, which is the subject of this study, coal is produced by the open pit method from the inefficient veins of a mine whose production was terminated because it was not economical at the time (Figure 1). The seam structure contains an average of 3 meters of coal within an 8 meter thickness. This structure shows a distribution of 30 cm clayey formation and 25 cm coal and has a very intermediate

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Geliş / Recieved: 08.07.2023 Kabul / Accepted: 09.11.2023 Yayımlanma / Published: 15.01.2024
doi: 10.28948/ngumuh.1324175

slice structure. Since the excavator mouths of the machines are larger than the seam, the intermediate cutting is taken without being completely separated. Coal enrichment is carried out at the coal washing plant. Additionally, there is a decrease in coal washing recovery due to low open pit efficiency. Approximately 100000 tons of saleable coal is obtained for an average of 500000 tons of raw coal production. There is a slime loss of around 21% in the plant [9]. Considering that the slime loss is equivalent to coal washing recovery, the main objective of this study is to investigate the recovery of fine-sized Tunçbilek lignite coal through flotation and increase the plant's efficiency to prevent cost losses arising from these losses.

Figure 1. Coal seams of the plant

2 Material and method

2.1 Material

The coal, whose characteristics are given in Table 1, is being fed to the washing plant for enrichment after being extracted from the coal mine. The washing plant is operating at efficiency levels of around 20% in parallel with the mine's efficiency, which is 25%. Considering the market share of the powdered coal in the plant, which is currently sized between 10 and 0.2 mm, this coal is mixed with the slime to evaluate the slime. In plant, powder coal, which has an average combustible matter recovery of 8.5% and a calorific value of 3575 kcal/kg, accounts for 41.5% of the total sales. Depending on the formation of seams, the recovery of powdered coal taken from the plant may decrease. The tailing in the plant ranges from 50% to 59%. It is thought that the efficiency of clean coal can be increased by flotation of the slime in the plant and thus the losses can be compensated.

Table 1. Properties of raw coal in plant

	Original Coal	Dry coal (in air)	Dry Coal
Ash (%)	42	57	60
Volatile matter (%)	19.24	25.95	27.35
Total sulfur (%)	0.99	1.33	1.40
Upper cal. value (kcal/kg)	1750	-	3307
Lower Cal. Value kcal/kg)	1501	-	2166

In the experimental studies, the slime of 75 kg taken from downstream of the thickener in the washing plant was used (Table 2). Due to the open pit and washing plant conditions, the samples were taken at different times, homogenized, and used in the studies. Then, the sample was divided by the rectangular method: one part was used in the experiments, and the other part was stored. The particle size of approximately 80% of the samples was below 0.2 mm. In the experiments, diesel oil, methyl Isobutyl Carbinol (MIBC), and sodium silicate were used as a collector, frother, and dispersant, respectively. Tap water was used in the study.

Table 2. Properties of the slime used in experimental studies

	Original Coal	Dry Coal
Lower calorific value (KCal/Kg)	504	1040
Upper calorific value (KCal/Kg)	757	1130
Volatile matter (%)	13.57	19.45
Total sulfur (%)	0.60	1.71
Ash (%)	34.83	72.84

2.2 Method

The experiments were done in a 1L cell using a Denver-type flotation machine having an air compressor providing variable air flow rates. The experiments were carried out at the natural pH (7.8) of the coal suspension and at a stirring speed of 1450 rpm. The water and coal mixture prepared according to the desired solid ratios were stirred for 3 minutes for a homogeneous pulp mixture. Then, dispersant, collector and frother were added respectively, air was supplied to the system and froth was removed at certain flotation times. Conditioning times for the collector and frother were determined as 3 minutes. At the end of the experiment, the obtained products were filtered and dried in an oven (at 105 °C), and the ash contents of the concentrate were determined to calculate the combustible recovery values (Equation 1).

$$\text{Combustible recovery (\%)} = \frac{M_c \times (100-A_c)}{M_f \times (100-A_f)} \times 100 \qquad (1)$$

Where, Ac and Af are the ash content of clean coal and feed; Mc and Mf are the mass of clean coal and feed, respectively.

The flotation performance was evaluated by determining the ash, calorific value, sulfur, volatile matter, fixed carbon, and combustible recovery values on the obtained products at the end of the experiment. Leco AC-350 model calorimeter device was used for calorific value analysis, Leco TGA-701 for volatile matter determination, and Leco SC-144 DR carbon-sulfur analysis device for sulfur content and fixed carbon analysis. The experimental studies were carried out depending on variables including the concentration of collector, dispersant and frother, airflow rate, and solid %. (Table 3).

Table 3. Experimental parameters

Parameters	
MIBC (g/t)	50, 75, 100, 150, 200
Sodium Silicate (g/t)	750, 1000, 2000, 4000, 6000, 7000
Diesel oil (g/t)	800, 1000, 2000, 4000, 6000, 10000
Solid ratio (%)	10, 15, 20, 25
Air flow rate (L/min^{-1})	4, 6, 7, 8, 9, 10

3 Results and discussion

3.1 Optimization experiments

Firstly, preliminary experiments were conducted to test the floatability of the slime and to determine the average working range of the studied flotation chemicals (collector, dispersant, frother) and experimental conditions (air flow rate). In the experimental studies carried out at this stage, the flotation performance was evaluated by the percentage of float product (Figure 2). In the preliminary experiments, the working ranges of the collector and dispersant concentrations were tested in the range of 800-10000 g/t for the collector and 750-7000 g/t for the dispersant. As a result, floated matter recoveries in the range of 23% to 41% and 35% to 40% were obtained, respectively (Figure 2a-b). For low-grade coal, the strong surface hydrophilicity of oxygen functional groups makes the collector to adsorb on coal surface difficult, so high float product recovery cannot be achieved. The maximum floated product recoveries were obtained at collector and dispersant concentrations of 2000 g/ton. Further addition of the collector decreased the amount of floated matter (Figure 2a). It is known that the addition of the collector above the optimum concentration leads to a negative effect on flotation efficiency due to layered adsorption on the mineral surface. Furthermore, the addition of more than the optimum concentration of collector also causes the clays and associated particles to float, reducing the product quality [10]. Sodium silicate is one of the most important modifying chemicals in flotation. It is commonly used as a depressant but can also function as a dispersant. Soluble silicates are intensely hydrolyzed and interact with the mineral surface [11]. However, no effective increase was observed in the float product recovery with sodium silicate, and the addition of sodium silicate above the optimum concentration did not result in a significant change in float product recovery (Figure 2b). Similarly, Oats et al. [12] also found that dispersants did not significantly improve flotation efficiency in coal flotation.

In the preliminary experiments, frother concentration was tested in the range of 50-200 g/t, and recoveries in the range of 32-40% were achieved. The maximum recovery was obtained with a frother concentration of 100 g/ton (Figure 2c). Bubble size significantly influences the flotation efficiency, especially for very fine-sized coal. The flotation of fine particles requires large surface areas, so fine bubbles must be produced. On the other hand, the main purpose of using frother is not only to create small-sized bubbles but also to generate small and stable bubbles that do not rapidly coalesce with other bubbles. This situation varies depending on the type/concentration of frother used and the presence of fine-sized particles in the feed [13]. Apart from this, the airflow rate is another factor that affects the bubble sizes. The most important variable that increases the flow rate of froth is the air flow rate fed into the cell. In this case, it can be expected that the float product recovery will increase with the increasing air flow rate, but it is observed that there is a decrease in the float product recovery with the increase in the airflow rate. The highest float product recovery (41%) was obtained with the lowest air flow rate (4 L/d) (Figure 2d). It is thought that this situation may be caused by the increase in the airflow rate, increasing the bubble size, and, consequently, the decrease in the bubble surface area. The number of bubbles in the pulp increases with increasing airflow rate. This will increase the probability of collisions between bubbles and coal. In this case, the flotation efficiency can be expected to increase. However, a very high airflow rate will cause a decrease in the recovery rate of combustible material in flotation due to the increased bubble size. When the air flow rate exceeds a certain value, a decrease in efficiency is observed due to bubble coalescence. Additionally, fast airflow can cause instability in the flow level. Furthermore, fine slime particles can adhere to air bubbles, resulting in more ash and less clean coal [14]. It is also supported by the studies in the literature that increasing air flow rate leads to bubble coalescence beyond a certain threshold, disrupting the flow conditions and reducing flotation efficiency by decreasing the bubble surface area [10, 14]. In the second stage of the experimental studies, the effect of these variables on the product quality (ash-sulfur-volatile matter- calorie values-combustible matter recovery) in different experimental variable combinations was also evaluated.

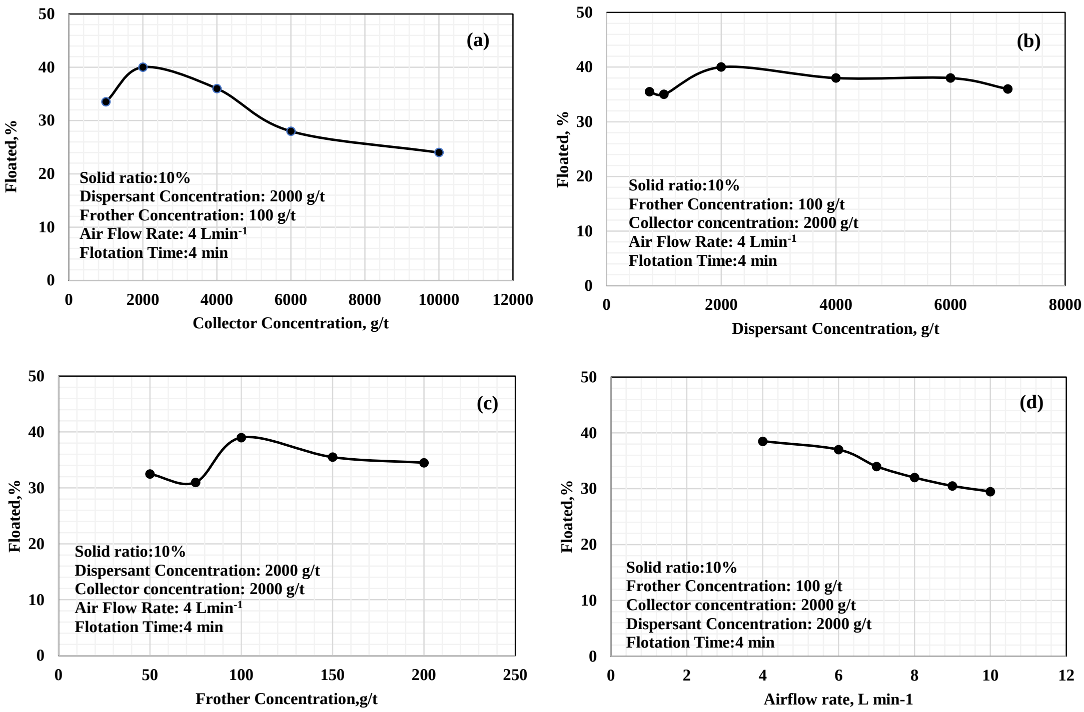

Figure 2. Effect of flotation variables on floatability of lignite slime

3.2 *Flotation experiment results*

The preliminary experiments conducted with the slime taken from the thickener in the coal preparation plant showed that the material could be floated to some extent, even in small amounts. However, no evaluation was made in terms of product quality. In the second stage of the study, experiments were conducted with different combinations of experimental variables to investigate the potential of incorporating of the slime to the plant. At this stage, flotation performance is discussed in terms of combustible matter recovery and product quality (ash- sulfur- volatile matter-calorific values). Table 4 shows the flotation test results for different combinations of experimental variables.

According to the experimental studies, the optimum conditions were determined as follows: 10% solids ratio, 100 g/t MIBC, 2000 g/t sodium silicate, 2000 g/t diesel oil, 4 L/min airflow rate, and 4 min flotation time. As the amount of reagents studied and other variables increase under experimental conditions the samples obtained in the experiments tend to deviate from the optimum result and the product quality decreases (Table 4). Unlike the optimization studies that focus on determining the float product amount, this section also includes evaluating flotation time and solid ratio as additional experimental variables. The quality of the product obtained is expected to decrease with the increasing flotation time. However, long flotation times have also been tried since the aim is to obtain a product with a minimum of 2500 kcal/kg and above. Optimum results were obtained in the studies performed with 10% solids ratio, and while the ash content increased with increasing solids ratio, the calorific value and combustible matter recovery decreased (Figure 3). A significant decrease in efficiency was observed at high solid concentrations due to the coal slime used in the study having very fine particle size and containing a significant amount of clay as the gangue. It was also determined in the previous flotation studies that a lower pulp density should be used for fine coal particles [15,17]. In addition, an increased solid ratio may lead to a decrease in efficiency due to the effect of intra-cellular airflow.

Consequently, under optimum conditions, clean coal with a calorific value of 6312 kcal/kg, sulfur content of 0.95%, ash content of 22.44 % on an dry basis was obtained from slimy material with a calorific value of 1130 kcal/kg, sulfur content of 1.71% and ash content of 72.8% on the dry basis. However, in these conditions, obtaining a larger amount of product at an acceptable calorific value seems possible by extending the flotation time. Lignite surface is rich in polar functional groups such as C-O, O-H, and CO= groups. Lignite is much more hydrophilic than other coals due to the abundance of oxygenated functional groups, cracks and micropores on its surface. Therefore, achieving satisfactory flotation efficiency for lignite is challenging. These groups can combine with water molecules via hydrogen bonds and cause the formation of a thicker hydration film on the surface. The formed hydration layer limits the adsorption of collectors on the surface [18]. In addition, exposure to oxidation of coal slimes, which are exposed to outdoor effects such as sun, rain, and wind for a long time, also leads to low flotation efficiency. Furthermore, the degree of slimming also significantly affects the flotation properties. Slimes cause low flotation efficiencies by covering the surface of minerals, preventing direct contact between collectors and air bubbles [14].

Table 4. Flotation test results for different experimental variable combinations

Solid Ratio %	Amount of MIBC g/t	Amount of Sodium Silicate, g/t	Amount of Diesel oil, g/t	Airflow Rate $Lmin^{-1}$	Flotation time, min	Sulfur, %	Amount of Volatile matter, %	Ash, %	Fixed Carbon, %	Calorific Value kcal/kg	Combustible matter Recovery, %
10%	100	2000	2000	4	4	0.95	34.02	22.44	41.46	6312	41
	100	4000	2000	4	4	1.27	21.24	27.45	43.91	5188	38
	150	4000	4000	6	6	1.32	17.99	28.96	30.20	4140	39
	150	6000	4000	6	6	1.61	18.88	32.53	28.88	4042	40
	200	6000	6000	7	7	0.94	15.24	34.35	41.12	3901	40
	200	7000	6000	7	7	1.68	25.37	35.24	27.24	3817	36
	75	7000	800	8	8	1.55	22.65	35.90	34.56	3761	37
	75	750	800	8	8	1.14	26.79	33.25	29.22	3617	34
	50	750	10000	9	9	1.21	28.70	38.41	26.68	3601	38
	50	1000	1000	10	10	1.21	28.26	35.00	28.98	3532	32
15%	100	2000	2000	4	4	1.26	27.30	36.08	28.96	3757	33
	100	4000	2000	4	4	1.68	27.82	37.15	31.77	3633	33
	150	4000	4000	6	6	1.11	28.04	37.99	30.86	3615	30
	150	6000	4000	6	6	0.98	29.87	38.06	29.86	3211	38
	200	6000	6000	7	7	0.96	26.71	38.37	31.66	3121	36
	200	7000	6000	7	7	0.80	24.90	38.71	17.81	3057	35
	75	7000	800	8	8	1.10	20.72	38.36	32.23	2986	32
	75	750	800	8	8	0.99	20.45	38.28	25.44	2887	26
	50	750	10000	9	9	1.11	25.36	36.46	27.19	2711	21
	50	1000	1000	10	10	1.02	19.99	39.01	28.85	2651	24
20%	100	2000	2000	4	4	1.44	22.08	39.98	23.79	2922	24
	100	4000	2000	4	4	1.05	22.49	40.71	28.97	2894	23
	150	4000	4000	6	6	0.98	23.58	41.92	30.16	2885	23
	150	6000	4000	6	6	1.26	26.51	42.15	30.50	2817	34
	200	6000	6000	7	7	1.13	24.08	42.39	27.86	2803	30
	200	7000	6000	7	7	0.86	24.10	42.50	29.30	2788	36
	75	7000	800	8	8	0.86	17.74	43.30	32.42	2774	17
	75	750	800	8	8	0.85	18.21	43.46	31.66	2690	18
	50	750	10000	9	9	0.91	19.90	44.58	30.14	2470	16
	50	1000	1000	10	10	1.15	21.14	45.28	27.15	2385	20
25%	100	2000	2000	4	4	1.01	25.65	45.39	24.56	2752	26
	100	4000	2000	4	4	1.29	17.29	45.45	29.35	2706	22
	150	4000	4000	6	6	1.15	25.99	45.46	27.91	2680	23
	150	6.000	4000	6	6	1.00	20.64	46.47	28.20	2590	23
	200	6000	6000	7	7	1.01	22.38	46.85	24.92	2533	26
	200	7000	6000	7	7	0.98	25.95	47.32	23.05	2495	28
	75	7000	800	8	8	0.99	27.30	48.52	31.05	2461	14
	200	750	10000	9	9	0.92	28.40	48.84	17.45	2310	19
	75	1000	4000	6	10	0.98	23.92	41.60	31.82	2619	19

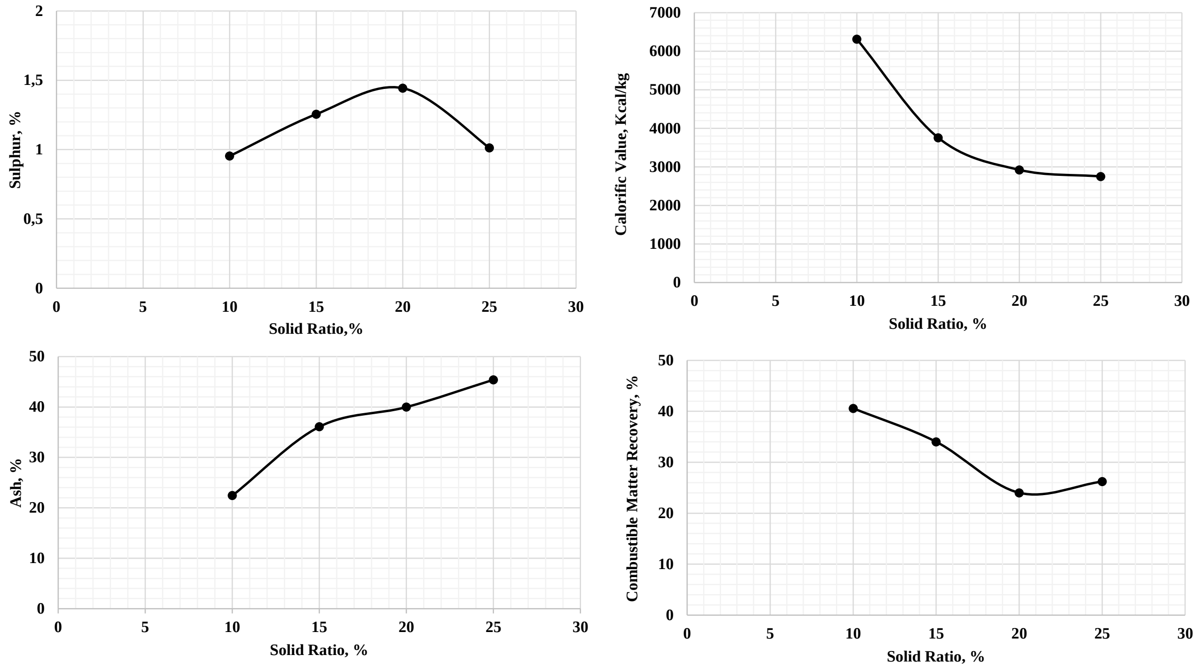

Figure 3. Variation of sulfur, calorie, ash values and combustible matter recovery of concentrate depending on solid ratio (Collector concentration: 2000 g/t; Dispersant concentration: 2000 g/t; frother concentration: 100 g/t; Air flow rate: 4 $Lmin^{-1}$; flotation time 4 min)

4 Conclusion

This study aimed to enrich the slime below the thickener by flotation method in a coal washing plant located in the Tunçbilek district of Kütahya province and contribute it to the economy. The loss of coal at the same level of efficiency as the plant and its effect on operational efficiency causes a decrease in profitability. Pre-flotation tests, where the flotation performance was evaluated based solely on the float product recovery were carried out at varying concentrations of frother, collector, and dispersant, and varying air flow rates in order to test the floatability of the slime. As a result, coal was floated at approximately 40% recovery.

Following the preliminary optimization studies, different combinations of flotation experiments examining the interaction of all studied experimental variables (collector concentration, dispersant concentration, frother concentration, solid ratio, air flow rate, and flotation time) were carried out. At this stage, the flotation performance was evaluated by determining the ash, calories, sulfur, volatile matter, moisture, fixed carbon and recovery values on the products obtained at the end of the experiment. As a result of the experimental studies, the highest efficiency value was obtained under the following conditions: an airflow rate of 4 L/min, a flotation duration of 4 minutes, a solid ratio of 10%, 2000 g/t sodium silicate, 2000 g/t diesel oil, and 100 g/t MIBC. The resulting marketable coal had a sulfur content of 0.95%, an ash content of 22.4%, a calorific value of 6312 kcal/Kg, and a combustible matter recovery of 41.22%. For different experimental conditions, it was determined that coal can be obtained in the range of 2300-6300 kcal/kg average calorific value. As a result, it was concluded that approximately 90000 tons of sludge loss in this plant, which washes approximately 500000 tons of coal per year, may have the potential to be brought into the economy.

Acknowledgement

This study was supported by the Coordinatorship of Selcuk University Scientific Research Projects. (Grant No: 17201106).

Conflict of interest

The authors declare that there is no conflict of interest.

Similarity rate (iThendicate): 15%

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