MODELLING OF SOME EFFECTIVE PARAMETERS IN FINE COAL FLOTATION

Derya Öz AKSOY[1*], Sabiha KOCA[2], Hüseyin KOCA[3]

[1,2]Mining Engineering Department, Eskisehir Osmangazi University, Turkey
[3]Porsuk Technical College, Eskisehir Technical University, Turkey

Anahtar Kelimeler

Flotasyon, Merkezi Kompozit Tasarım, Flotasyon Parametrelerinin Modellenmesi, Düşük Ranklı Linyit

Öz

Türkiye'nin ham petrol ithalatına %90, doğal gaz ithalatına %99 ve kömür ithalatına %95 oranında bağımlı olduğu bilinmektedir. Türkiye'nin en önemli enerji kaynağı yaklaşık 20 milyar ton olan linyitlerdir. Enerjide dışa bağımlılığın yüksek olmasına rağmen, Türkiye'nin elektrik üretiminin yalnızca %14,21'i yerli linyit kaynaklarından elde edilmektedir. Yerli linyitlerden çok düşük miktarda elektrik elde edilmesinin başlıca nedenlerinden biri, bu rezervlerin önemli bir kısmının düşük ranklı olması, yüksek kül ve yüksek kükürt içeriğine sahip olması ve arıtılmadan kullanıldığında ciddi çevre sorunlarına yol açmasıdır.

Bu çalışmada, düşük ranklı linyitlerin kül içeriğini azaltarak santrallerde kullanımını artırmak için bir dizi flotasyon deneyi gerçekleştirilmiştir. Deneylerin tasarımı, deneysel sonuçların analizi ve matematiksel modellerin oluşturulması Merkezi Kompozit Tasarım Yöntemi kullanılarak yapılmıştır. Deneysel sonuçlar, numunelerin kül içeriğinin %52,47'den %29,37'ye düştüğünü ve yanıcı geri kazanımının %32,50 olduğunu ortaya koymuştur.

Keywords

Froth Flotation, Central Composite Design, Modelling of Flotation Variables, Low-Rank Lignite.

Abstract

The most important energy resource of Turkey is lignites which counts for about 20 billion tones. Despite the high external dependency on energy, only 14.21% of Turkey's electricity production is obtained from domestic lignite resources. One of the main reasons why very low amounts of electricity are received from domestic lignites is that a significant portion of these reserves are low-rank, have high ash and sulfur content, and cause severe environmental problems if utilized without purification.

In this work, to reduce the ash content of these lignite's thus increasing their utilization in power plants, a series of flotation experiments were carried out. Designing experiments, analysing experimental results, and creating the mathematical models were performed using the Central Composite Design Method. The experimental results revealed that the ash content of the samples was reduced from 52.47% to 29.37% with a combustible recovery of 32.50%.

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* Sorumlu yazar: deryaoz@gmail.com
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1. INTRODUCTION

Turkey has insufficient reserves of crude oil, hard coal and natural gas and is dependent on foreign countries by 90%, 95% and 99%, respectively (https://enerji.gov.tr/info-bank-energy).

Lignite reserves, the most important fossil fuel source in Turkey, are approximately 20 billion tons. 79.1 million tons of lignite were produced in Turkey in 2022. All of these were excavated from domestic sources, and 83.5% of this amount was utilized in electricity generation in thermal power plants (https://enerji.gov.tr/info-bank-energy).

Only 14.21% of Turkey's electricity production is obtained from domestically produced coal (TEİAŞ, 2023). The main reason why domestic energy resources are used at such a low rate in Turkey is that a significant portion of these resources have low-rank, high ash and sulfur content and cause serious environmental problems if used without enrichment (TMMOB, 2024). Approximately 80% of Turkey's lignite reserves have a calorific value below 2500 Kcal/kg, with minimum and maximum values ranging from 1000 to 4200 Kcal/kg. The average sulfur content is above 2%, and the average calorific value is around 1250 Kcal/kg. Therefore, the use of these lignites in energy production without cleaning causes serious negative effects on the environment. This leads to restrictions on their utilization.

In countries such as China, Australia and the USA, froth flotation is widely used on an industrial scale for fine lignite (<0.5 mm) cleaning (Lynch, Watt, Finch and Harbort, 2007; Dube, 2012). There are many studies in the literature on reducing the ash content of lignite (Jia, Harris and Fuerstenau, 2000; Xia, Yang and Zhu, 2012; Oh-Hyung, Min-Kyu, Byoung-Gon, Nimal and Chul-Hyun., 2014) and removing sulfur (Yoon, Lagno, Lutrell and Mielczarski, 1991; Aksoy, Aytar, Toptas, Çabuk, Koca and Koca, 2014; Aksoy, Ozdemir, Aytar, Koca, Çabuk, Koca and Brito-Prada, 2022) by froth flotation.

In this work, to reduce the ash content of these lignites, the enrichment studies are carried out by the flotation method. The effects of process variables such as collector type and amount, solid ratio, pH and flotation time on weight yield, ash content and combustible recovery were optimized. This study was planned using the Central Composite Design (CCD) method which is one of the principal response surface methodologies. CCD is a widely used experimental design method, because of it can give more data with lower number of experiments (Öz Aksoy and Sağol, 2016). One of the most important advantages of the statistical method chosen in this study is the generation of model equations for the selected response variables. Furthermore, the interactions between the investigated parameters and the effects of these interactions on the response variable during the flotation process are interpreted.

2. MATERIALS AND METHOD

2.1. Sample

Samples taken from the preparation plant of Eskisehir region lignites were dried in the open air and separated into size fractions. The fractions larger than +0.053 mm were cleaned by physical methods in another project and the results were published elsewhere (Aksoy, Koca and Koca, 2012). In this project, it was determined that the fraction finer than -0.053 mm could not be enriched by physical methods. In the preliminary experiments, it was shown that the fine fraction could be cleaned by physicochemical methods. Therefore, this project was initiated to clean the fine fraction by froth flotation. The ash content of the studied sample was analyzed as 52.47%, the lower calorific value (LCV) as 2678 Kcal/kg and the sulfur content as 4.76%.

Analytical grade reagents were used in the flotation studies. Fuel oil (FO), Philflo (PF) and a mixture of Philflo+kerosene (PF+K) were used as collectors. Sodium silicate was utilized as a dispersant to prevent agglomeration of fines. Pine oil was used as a frother. A tailor-made reagent, Philflo, was manufactured and supplied by Chevron Philips Chemical Company, LP.

2.2. Method

Flotation experiments were carried out by using laboratory type Denver flotation machine with a volume of 1.5 liters cell at the Mineral Processing Laboratory of Eskisehir Osmangazi University. Before systematic experiments, a series of preliminary experiments were carried out to select the collector and to determine the constant and varied parameters and levels of varied parameters. Some parameters were kept constant. These are: Particle size, depressant type, frother type, agitation speed and flotation time.

Particle size: -0.053 mm,
Depressant: Na_2SiO_3,
Frother: Pine oil,
Agitation speed: 1200 rpm
Flotation time: 90 s.

In the preliminary experiments, it was determined that the sulfur content of fine size lignite's could not be reduced sufficiently. It was stated in the literature that the sulfur reduction in fine size lignite's by froth flotation was not achieved sufficiently due to slime coating (Andrews and Maczuga 1982; Lizama and Suziki 1987). For this reason, sulfur removal was not studied in this project and it was decided to use biological methods in another study.

In the systematic experiments, CCD, one of the statistical experimental design methods, was used to set up the experimental design and the analysis of the obtained results. The α factor was selected as 1 in CCD. Design Expert 10.0.6 Software was used for achieving the design matrix of the response variables. The levels of the varied parameters are given in Table 1.

Table 1. Parameters and Levels.

Parametre	Level		
	-1	0	1
A-Collector Amount (g/t)	1200	1700	2200
B-Solid Ratio (%)	8	13	18
C-pH	6	7,5	9
D-Depressant Amount (g/t)	500	1000	1500

The products obtained after the experiments, concentrate and waste, were dried and weighed. The ash and sulfur contents of the products and their combustible recovery (CR) were determined. The combustible recovery was calculated using Equation 1.

$$CR\ \% = \frac{\text{wt\% concentrate x (100−Ash content of concentrate)}}{\text{(100−Ash content of feed)}} \quad (1)$$

where wt% is the amount of concentrate (%). In this study, ash content and combustible recovery in the concentrate fraction are presented.
Varied parameters were evaluated according to 3 response variables:

1. Weight yield (amount of concentrate) (WY %),
2. Ash content of concentrate (Ash %)
3. Combustible recovery (CR %)

After the variance analysis of the results (according to a 95% confidence interval), statistically insignificant terms for each response variable were eliminated, and reduced variance analysis tables were prepared, and mathematical models were created.

3. RESULTS AND DISCUSSION

3.1. Determination of Collector Type

Fuel oil and kerosene are generally used in coal and lignite flotation. (Xia, Xie and Peng, 2015; Jia et al., 2000) In recent years, as suggested by some publications, a tailor-made synthetic collector, PhilFlo, was also used (Aksoy et al., 2014).

Before systematic experiments, a series of experiments were conducted to determine the type of collector. In these experiments, FO, PF and PF+K (2:1 w/w) were tested and the most effective collector was determined. The effect of the collector type on the ash content and combustible recovery of the concentrate is shown in Figure 1.

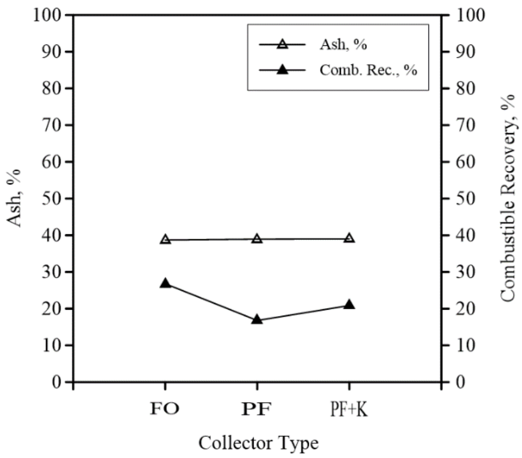


Figure 1. The Effects of Collector Type on Flotation Results

All three collectors used had the same effect on the ash content of the concentrate, reducing the ash content to less than 40%. The best result was obtained with fuel oil with an ash content of 38.69%. This corresponds to a reduction of more than 26%. When the results were examined in terms of combustible recovery, it was found that fuel oil gave the best result with 27.79%. Therefore, fuel oil was used as the collector in the following studies.

After the determination of the collector type, systematic experiments were planned according to Design Expert Software. The design matrix and the results of experimental studies are given in Table 2.

In the variance analysis of the results for the parameters whose effects were examined, the software suggested a quadratic model for all three models.

Reduced variance analysis, p values indicating the significance levels of the terms are given in Table 3, along with R^2, AR^2 and PR^2 values showing the model strength for the three response variables.

Mathematical models of response variables are given in Eqs. 2-4 according to the actual values of the factors.

$$WY\ (\%) = -26.10588 - 0.005882xA - 0.679808xB + 9.55950xC + 0.022818xD + 0.000807xAB + 0.001059xAC - 0.722796xC^2 - 0.000011xD^2 \quad (2)$$

$$Ash\ (\%) = 128.19264 + 0.064383xA - 5.59407xB - 28.58840xC + 0.000895xD + 0.000310xAB + 2.42x10^{-6}xAD + 0.222833xBC - 0.000782xCD - 0.00019xA^2 + 0.115173xB^2 + 1.68859xC^2 \quad (3)$$

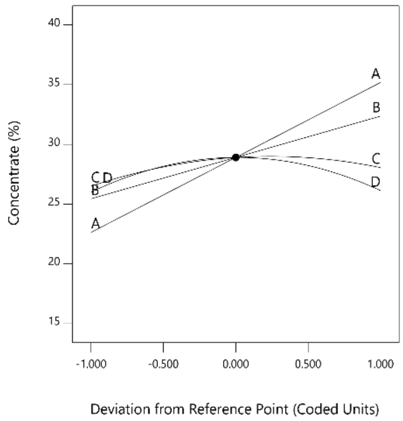
$$CR\ (\%) = -93.75336 + 0.001945xA - 0.483092xB + 25.46876xC + 0.030533xD + 0.000900xAB - 1.62100xC^2 - 0.000015D^2 \quad (4)$$

When the summary table of reduced variance analysis is examined, the models obtained for all three response variables are found to be statistically significant since their p-values are <0.05. The models are also statistically quite strong with R^2 values well above 90% and PR^2 values above 85%.

Interpretation of the results and graphical representations is given below one by one for each response variable.

3.2. The Response of Weight Yield

As can be seen from the reduced ANOVA Table (Table 3) and Figure 2a, the most effective parameters for weight yield are collector amount and solid ratio, and they are in interactions with each other. pH also significantly affects the weight yield and interacts with the collector amount. But no interaction was observed between pH and solid ratio. Figure 2a graphically illustrates the main effects of parameters for weight yield. The compatibility between the experimental data and the values estimated from the model is also given in Figure 2-b. As seen from this figure, predicted weight yield values represent over 97% of actual weight yield values (R^2=0.9701), indicating the consistency of the model. Apart from this, high values of P-R^2 (0.9370) and A-R^2 (0.9587) show the high power of prediction of this model.



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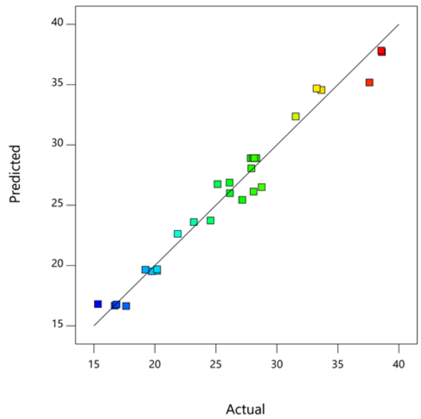

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Figure 2. a) Parameter Main Effect Graphs, B) Experimental Data-Model Prediction Fit Graph for the WY (R^2=0,9701).

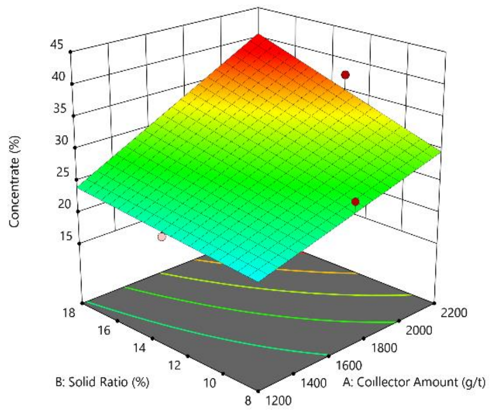
As seen in Figure 2 a, the collector amount and the solid ratio increased the weight yield and this increase continues linearly in the studied range. The increase in pH increased the floating product rate up to a certain level, but it caused a decrease above this level. The depressant amount showed a parabolic effect in the studied range, but this parameter was not significant in the variance analysis.

Table 2. Design matrix and results of the experiments for the three response variables.

No	A	B	C	D	WY (%)	Ash (%)	CR (%)
1	2200	18	6	500	33.65	39.60	42.76
2	1200	18	9	500	19.75	34.55	27.20
3	1200	18	6	500	20.18	34.76	27.70
4	1700	13	7.5	1000	28.24	37.81	36.95
5	1200	8	6	500	16.71	42.57	20.19
6	2200	18	9	500	38.61	40.93	47.98
7	1700	13	7.5	1000	28.16	36.92	37.37
8	2200	18	9	1500	38.56	41.08	47.80
9	2200	8	6	500	23.19	46.00	26.35
10	2200	8	9	1500	26.12	39.47	33.26
11	1200	18	6	1500	20.18	33.02	27.80
12	1200	8	6	1500	15.33	42.98	18.39
13	1700	13	7.5	1500	28.09	37.95	36.67
14	1700	13	7.5	1000	28.32	38.03	36.92
15	1200	18	9	1500	19.22	32.14	27.44
16	1700	13	7.5	1000	28.04	37.90	36.64
17	2200	13	7.5	1000	37.59	33.29	52.76
18	1700	13	6	1000	28.75	41.77	35.22
19	1700	8	7.5	1000	27.16	41.86	33.22
20	1700	18	7.5	1000	31.54	36.18	42.35
21	1700	13	7.5	500	26.15	37.05	34.63
22	1700	13	9	1000	27.91	38.11	36.34
23	1200	13	7.5	1000	21.87	29.37	32.50
24	1200	8	9	1500	16.85	32.56	23.91
25	2200	18	6	1500	33.26	41.57	40.89
26	1200	8	9	500	17.64	37.78	23.09
27	1700	13	7.5	1000	27.86	37.70	36.52
28	2200	8	9	500	25.14	40.74	31.34
29	2200	8	6	1500	24.56	45.99	27.91
30	1700	13	7.5	1000	28.15	37.50	37.02

Table 3. Reduced variance analysis summary (ANOVA)

	WY %	Ash %	CR %
	P Value	P Value	P-Value
Model	<0.0001	<0.0001	<0.0001
A	<0.0001	<0.0001	<0.0001
B	<0.0001	<0.0001	<0.0001
C	0.0184	<0.0001	0.0043
D	0.8356	0.1275	0.7751
AB	<0.0001	0.0093	0.0008
AC	0.0225	-	-
AD	-	0.0336	-
BC	-	<0.0001	-
BD	-	-	-
CD	-	0.0410	-
A2	-	<0.0001	-
B2	-	0.0002	-
C2	0.0293	<0.0001	0.0077
D2	0.0005	-	0.0060
R^2	0.9701	0.9554	0.9393
AR^2	0.9587	0.9281	0.9199
PR^2	0.9370	0.8520	0.8963



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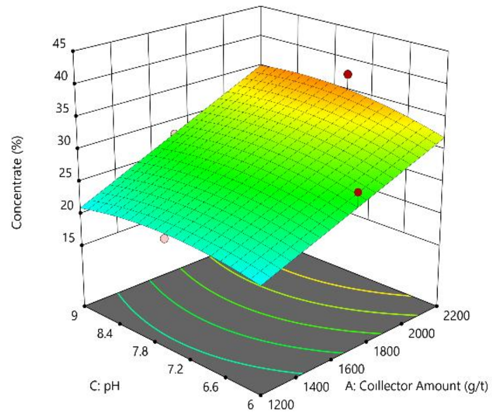


(b)

Figure 3. Interaction Graphs For The Amount Of Concentrate. a) Collector Amount/Solid Ratio (A/B), b) Collector Amount/pH (A/C).

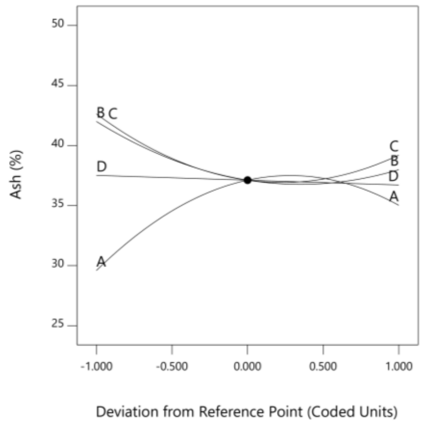
Interaction graphs for collector amount/solid ratio and collector amount/pH in three dimensions are given in Figure 3a and 3b, respectively. When the interaction graphs are examined, it is seen that the increase in the collector amount increases the weight yield, but this increase is much faster at high solid ratios. In parallel with this, the collector amount/pH interaction, the increase in the collector amount increases the weight yield at all pH values, but this increase is faster at high pH values.

3.3. The Response of Ash Content

Model equality and variance analysis were given in Table 3. The main effect graphs of the studied parameters are given in Figure 4-a. The graph showing the compatibility between the experimental data and the values estimated from the model is given in Figure 4-b. This figure shows that the predicted ash content of concentrate values represents over 95% of actual ash content values (R^2=0.9554), indicating high consistency of the model. The values of P-R^2 (0.8520) and A-R^2 (0.9281) are also quite high and show the high power of prediction of this model.

As can be seen from the reduced variance analysis, all the studied parameters except the depressant amount and the interactions between collector amount/solid ratio, collector amount/depressant amount, solid ratio/pH and pH/depressant amount are statistically significant. Interaction graphs for all 4 interactions in three dimensions are given in Figure 5 a, b, c and d, respectively.

As seen in Figure 4a, the three parameters (A, B and C) showed a parabolic effect. Among these, the solid ratio and pH first decreased and then increased the ash; the other parameter, the collector amount first increased and then decreased the ash. When the interaction graphs were examined (Figure 5), similar to the first response variable, all the parameters affect the ash content significantly.



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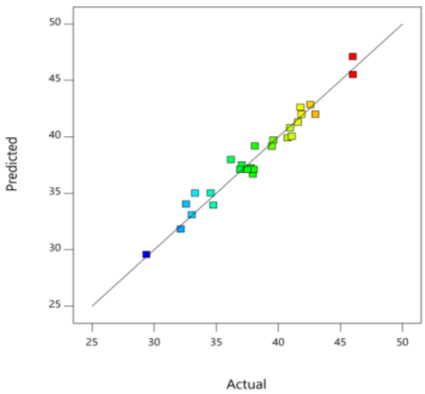

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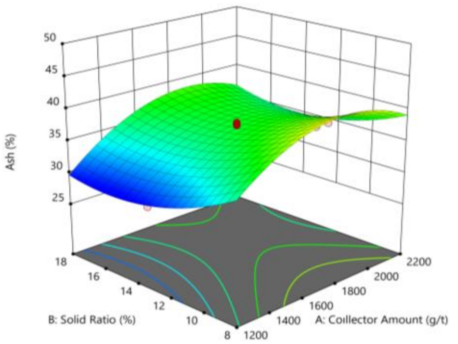
Figure 4. a) Parameter main effect graphs, b) Experimental data-model prediction fit graph for the ash (R^2=0,9554)



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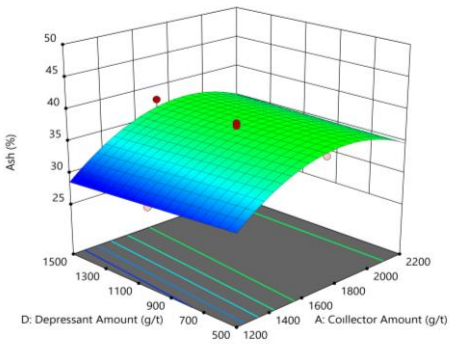

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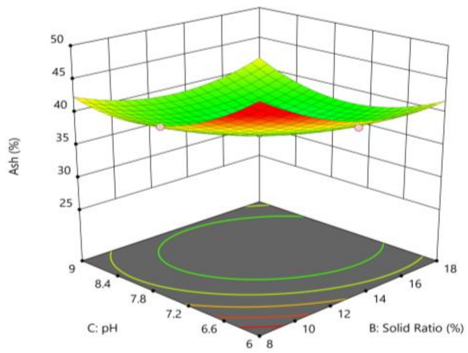

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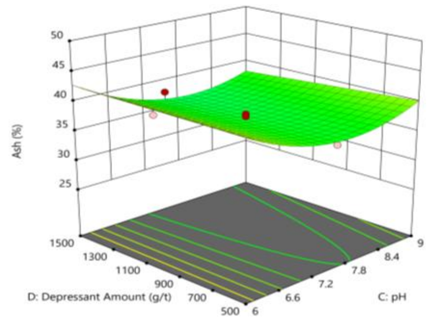


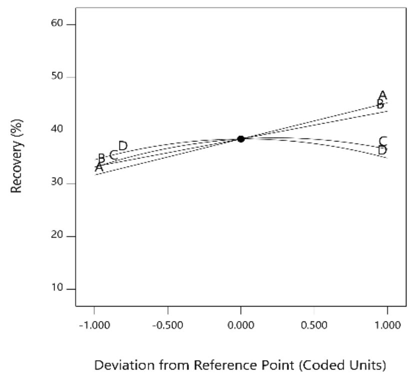
(d)

Figure 5. Interaction graphs for the ash. a) collector amount/solid ratio (A/B), b) collector amount/depressant amount (A/D), c) solid ratio/pH(B/C), d) pH/depressant amount (C/D)

3.4. The Response of Combustible Recovery

As can be seen from the reduced ANOVA Table (Table 3) and Figure 6a, the most effective parameters for combustible recovery are collector amount and solid ratio, and they are in interactions with each other. pH also affects the combustible recovery, significantly. But no interaction was observed between pH and other parameters. Depressant amount and its interactions with other parameters are found to be insignificant. Figure 6a graphically illustrates the main effects of parameters for combustible recovery. The compatibility between the experimental data and the values estimated from the model is also given in Figure 6b. As can be seen from this figure, predicted combustible recovery values represent nearly 94% of actual combustible recovery values (R^2=0.9393), indicating the consistency of the model. Apart from this, high values of P-R^2 (0.8963) and A-R^2 (0.9199) show the high power of prediction of this model.

As can be understood from Figure 6a, while the collector amount and solid ratio showed a linear effect on the combustible recovery in the studied range, pH and depressant amount caused a parabolic effect.



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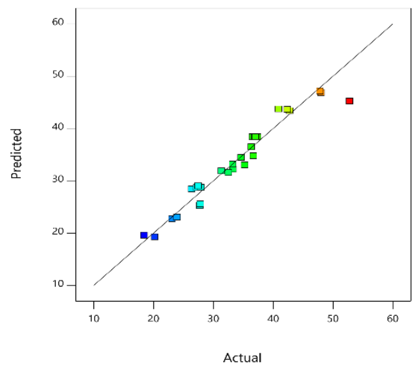

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Figure 6. a) Parameter Main Effect Graphs, B) Experimental Data-Model Prediction Fit Graph for Combustible Recovery. (R^2=0,9393).

Interaction graphs for collector amount/solid ratio interaction in three dimensions are given in Figure 7. When the interaction graph was examined, an increase in collector amount at high solid ratio increased the combustible recovery rapidly; however, the increase remained at a very low level at low solid ratio.

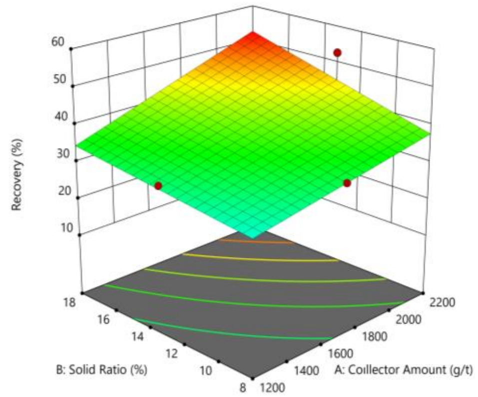

Figure 7. A-B Interaction Graphs (A-B) for the Combustible Recovery.

4. CONCLUSIONS

Firstly, fuel oil, PhilFlo and the mixture of Philflo+kerosene were tested as collectors, and fuel oil produced the best results among them. Afterwards, modelling studies were carried out by using fuel oil as a collector.

According to the results of experimental design and their variance analysis presented in Tables 2 and 3, regression models for weight yield, ash content of concentrate and combustible recovery were created. The created models, given in Eqs 1-3, are found to be statistically significant as their p values are <0.05. The models are also statistically quite strong since R^2 values are well above 90% and PR^2 values are above 85%.

The R^2 value of the model for weight yield is 0.9701, indicating nearly 97% of the data deviation can be explained by the model, created. As can be seen from variance analysis, collector amount and solid ratio were found to be the most significant variables for weight yield, followed by pH. Linear and quadratic effects of pH were found to be significant on weight yield, while only the linear effect of collector amount and solid ratio were found to be significant. On the other hand, linear effects of depressant amount were found to be insignificant while quadratic effects of depressant amount were significant. The dual interaction effects between collector amount and solid ratio and collector amount and pH were significant.

The R^2 value of the model for ash content of concentrate is 0.9554 that indicates over 95% of the data deviation can be explained by the model. Variance analysis showed that collector amount, solid ratio and pH were found to be the most significant variables for ash content while depressant amount was insignificant. Linear and quadratic effects of collector amount, solid ratio and pH were found to be significant on ash content, while the linear or quadratic effects of depressant amount were found to be insignificant. The dual interaction effects between collector amount and solid ratio, collector amount and depressant amount, solid ratio and pH and pH and depressant amount were significant.

The R^2 value of the model for combustible recovery is 0.9393, indicating nearly 94% of the data deviation can be explained by the model, created. As can be seen from variance analysis, collector amount, solid ratio and pH were found to be the most significant variables for combustible recovery. Linear and quadratic effects of pH were found to be significant on combustible recovery, while only the linear effect of collector amount and solid ratio were found to be significant. Besides, linear effects of depressant amount were found to be insignificant while quadratic effects of depressant amount were significant. The dual interaction effects between collector amount and solid ratio were significant.

After modelling studies, the ash content of the samples was reduced from 52.47% to 29.37% with a 32.50% combustible recovery. Calorific value of the studied sample is increased from 1250 Kcal/kg to 2645 Kcal/kg.

Author Contributions

Derya Öz Aksoy and Sabiha Koca are responsible for the implementation of the planned experimental study. All three authors contributed equally to the planning of the experiments, interpretation of the results, and writing of the article.

Conflict of Interest

No conflict of interest was declared by the authors.

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