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DETERMINING OPTIMAL CONDITIONS FOR LIGNITE FLOTATION BY DESIGN OF EXPERIMENTS AND THE HALBICH UPGRADING CURVE

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Abstract: In this study conditions for flotation of low rank coal (lignite of Tuncbilek, Turkey) were investigated in detail. The experiments were performed using the 3-variable 2-level (2^3) full factorial experimental design with four base point replicates, and the results were analyzed by the regression model, Fischer test (F-test) and Halbich's upgrading curve for the responses of ash content (or combustible matter grade) and the combustible matter recovery. The results obtained from the analysis indicated that while every factor considerably affected the combustible matter recovery, both collector (kerosene) and frother (AF65) significantly influenced the ash contents of the carbonaceous matter products. The only effective mutual interaction influencing recovery was caused by the kerosene-aeration interaction, while the interaction of kerosene-aeration and kerosene-AF65 and interactions of all factors (kerosene-AF65-aeration) were significant for the ash content of the products. Basing on the grade–recovery Halbich upgrading curve, regression model and a criterion for optimum of flotation results, it was found that a coal product with combustible matter grade of 91.09% and 71% combustible matter recovery can be obtained provided that it is processed at the higher level of kerosene (3 kg/Mg), higher level of frother AF65 (40 ppm) and lower aeration rate level of (0.16 cm/s).

Key words: *low rank coal, flotation, design of experiment, upgrading curves*

Introduction

Every year in Turkey teragrams of lignite having particle size of finer than 0.5 mm are discharged to the slurry pond after coal processing which not only causes economic losses but also significant environmental problems. Continuous and mechanized mining operations are a source of these fine coal particles which are not easily separated from their gangue minerals by the use of physical separation methods such as heavy media separation, spirals, and screens etc. Meanwhile, flotation process has a great potential for efficient beneficiation of these coals. Additionally, lignite type coals

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especially have problematic flotation behavior, not as the high rank coals (Aplan, 1976; Polat et al., 2003). Many studies have been performed to investigate flotation of low rank coals to obtain the optimum working parameters (Naik et al., 2004; 2005; Jia et al., 2000; Mohanty and Honaker, 1999). Some of these studies have also been performed for the Turkish coals (Aktaş and Woodburn, 1995; Ateşok et al., 2001; Cebeci, 2002; Akdemir and Sönmez, 2003, Vapur et al., 2010). All these studies clearly show the importance of chemical factors that are type of collector, frother, and pH on flotation performance. Moreover, some parameters such as aeration, hold-up, and power input can also affect the flotation performance of coals (Kowalczuk et al., 2011; Mohanty and Honaker, 1999).

The parameters and their effects on the combustible matter recovery and other performance characteristics are innumerable. Lately, Kelebek et al. (2008) carried out a study to improve flotation of coal particles obtained from Tuncbilek lignite which is one of the biggest lignite reserves in Turkey. They determined the effect of collector type and pH and stated the necessity for determining the effect of frother (AF65) on Tuncbilek lignite flotation.

The procedure of factorial design of experiments is commonly used to examine the parameters controlling separation processes. The factorial design for performing experiments has many advantages over classical methods of treating one-variable-a-time (Box et al., 1978).The number of experiments and amount of material are reduced and time is saved by the use of factorial design. In addition, the analysis done on the results is easier while the experimental error is reduced. It can also helpful in determination of the optimal condition of the process.

Studies based on statistical approaches usually treat, incorrectly, grade and recovery independently. Therefore, it is necessary to use the so-called upgrading curves (Drzymala, 2006; 2007), that are plotted as quality vs. either quantity or quality of separation products (Drzymala et al., 2012). One of frequently used upgrading curves is the Halbich grade- recovery curve (Drzymala et al., 2012). The Halbich plot is practical and useful as well as has many advantages over other upgrading curves because of using recovery and grade, which are generally used in industrial, liberation, kinetic and theoretical studies (Drzymala et al., 2012).

The aim of this study is to report a procedure of determination of interactions between basic parameters of the process and optimal flotation conditions for the low rank coal of Tuncbilek region by means of factorial design and the Halbich plot.

Materials and methods

Materials

The lignite sample used in this study was obtained from the Tunçbilek underground mine, which is 50 km away from the city centre of Kutahya in Turkey. The sample, which is a sub-bituminous coal (Gülen et al., 2012), was first reduced to finer than 600 μm using a laboratory type jaw crusher and roll mill, respectively. The d_{80} of the samples was about 450 μm, and the ash content of the sample was found to be 12.13% (87.87% combustible matter). For the flotation tests kerosene, sodium silicate, and Aerofroth 65 (AF65), which is a polygylicol type frother obtained from Cytec and having chemical formula of $H(PO)_{6,5}OH$ (Melo, 2001 and Laskowski et al., 2003) were used as collector, depressant, and frother, respectively. The pH of the coal suspension was 7.5. The experiments were carried out at 23 ^{o}C. Local tap water was used in all flotation experiments.

Methods

Flotation tests

The flotation experiments were carried out in the Jameson flotation cell (JFC) which has a 25 mm downcomer equipped with a 6 mm nozzle.

For the flotation experiments, a 500 g sample of coal was added into a conditioning tank, and mixed with tap water at 1300 rpm for 5 min. Then, the desired amount of sodium silicate depressant (700 g/Mg) and kerosene as the collector were added to the conditioning tank and mixed for 10 min. Finally, a desired amount of frother (AF65) was added into the tank and mixed for 1 min. The feed solid concentration was 4% by weight.

After the conditioning process, the slurry was pumped to the nozzle on the top of the downcomer with a high pressure of 110 kPa. The pressure was tried to be fixed at the lowest degree to inhibit turbulence increase in the JFC. The working conditions for the JFC have been explained in detail in studies carried out by Evans et al. (1995) and lately by Şahbaz et al. (2013).

Experimental design

In this study, the flotation tests were performed by using the full factorial center point repeated experimental design. Three important parameters: collector, frother and aeration rate were chosen as independent variables (design factors), and two levels of these variables with their base points were used to generate data for 2^3 factorial design. The variables and their levels are given in Table 1. The 2^3 factorial design including the factors, levels and values are also given in Table 1.

Table 1. Variables and their levels

Flotation Factors	Low level (-1)	Mid Level (0)	High Level (+1)
A, Collector, g/Mg	500	1750	3000
B, Frother, ppm	10	25	40
C, Aeration rate (J_g), cm/s	0.60	0.95	1.30

In the design matrix, the higher level was designated as “+1” while the lower one and mid-point were designated as “–1” and “0”, respectively. The following equation (Eq. 1) was used to obtain coded units (X_{coded}) from the actual values (X_{actual}).

$$A_{\text{coded}} = \frac{X_{\text{actual}} - X_{\text{mean}}}{\frac{X_{\text{high}} - X_{\text{low}}}{2}} \tag{1}$$

where X_{high} is the highest value of any factor, X_{low} is the lowest value, and X_{mean} is the arithmetic value of X_{high} and X_{low}.

In the study, the combustible matter recovery and the ash content were chosen as responses. The main effect of any factor is the change in response produced by varying the level of the factors. It can be calculated by the below equation:

$$\text{Main effect of factor } X = (\text{Average response at } X_{high}) - (\text{Average response at } X_{low}) \tag{2}$$

Additionally, it is very important to find out the interaction effects which occur when the difference in response between levels of any factor is not the same at all levels of the other factor. This effect can only be found by the use of the statistical design given by Eq. 3

$$\begin{aligned}\text{Interaction effects of } X_1 \text{ and } X_2 = {} & (\text{Average response at } X_1X_{2\text{high}}) \\ & - (\text{Average response at } X_1X_{2\text{low}})\,.\end{aligned} \tag{3}$$

A full factorial 2^3 design with four base points can geometrically be shown by a cube. In this case, responses can be placed at the corners and X, Y, and Z axes representing factor *A*, *B* and *C*, respectively. The origin is the center of the cube, and every side of cube is equivalent to two units, +1 and −1. Design expert statistical software (www.statease.com) was used to determine the main and interaction effects with the confidence interval of 95%. The Fisher test and probability values were used to analyze the results statistically. In this analysis, the results were compared according to their *F* and *p*-values. The *F*-value indicates the model significance. It is used for determining the model navigation in the design space. The greater the F-value, the greater is the parameter effect on a response. In addition, the values of *p*-value is also used to determine the effectiveness of the parameters, and the *p*-value of less than 0.05 indicates that the model terms are significant at 95% confidence level.

Eight experiments were carried out for this determination using Eq. 4

$$NoE = 2^k, \tag{4}$$

where *NoE* is the number of the experiments, and *k* is the number of variables. To supply statistical significance and to estimate the variance (σ^2) and error, additional four experiments were performed at the base level. The variance of the main and interaction effects are given in Eqs 5 and 6 (Kelebek et al., 2008):

$$Variance\ (Effects) = \frac{4\sigma^2}{2^k}, \tag{5}$$

$$\text{Calculated main or interaction effects} / [\textit{Variance (Effects)}]^{0.5} \geq t_{3,0.025}, \tag{6}$$

The model (regression equation) with the main and interactive terms can be written as following formula:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC + \beta_{123} ABC \tag{7}$$

where Y is response (recovery or ash content of lignite), β is the coefficients for the main and interaction terms.

Results and discussion

The 2^3 design matrix, which includes the factors and levels, and responses (recovery and ash content) are presented in Table 2. The regression model and its analysis by using the variance of analysis were used to analyses the results of the experimental study.

Table 2. 2^3 factorial design matrix and responses

Run	Coded factors			Response		
	A	B	C	Recovery	Ash	Grade (100 – Ash)
1	–1	–1	–1	39	10.11	89.89
2	1	–1	–1	36	7.98	92.02
3	–1	1	–1	53	7.20	92.80
4	1	1	–1	71	8.91	91.09
5	–1	–1	1	15	7.32	92.68
6	1	–1	1	48	9.30	90.70
7	–1	1	1	37	6.94	93.06
8	1	1	1	61	9.18	90.82
9	0	0	0	57	8.19	91.81
10	0	0	0	49	8.07	91.93
11	0	0	0	48	7.71	92.29
12	0	0	0	53	7.77	92.23

The Pareto Charts for the combustible matter recovery and the ash content of the products are shown in Fig. 1. The charts give the relative importance of the main and interaction effects of AF65, kerosene, and aeration for the flotation recovery. The vertical lines in the charts show the minimum statistically important effect magnitude at the 95% confidence level. If any main or interacting effects exceed the reference line, the effect of parameter will be important for the response. In this case, as seen in Fig.1a, AF65 (B), kerosene (A), aeration (C) and interaction of kerosene-aeration (AC) show an important effect on the combustible matter recovery. In addition, Fig. 1b shows the significant factors of the ash removal. According to Fig. 1b, the most effective factor is the interaction of kerosene-aeration in the removal of ash (ash content), and it is followed by kerosene-AF65 interaction and triple interaction of the three considered parameters.

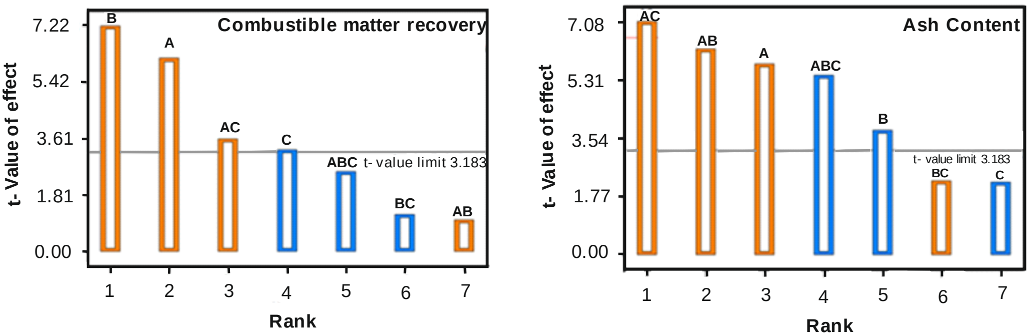

Fig. 1.The Pareto Charts for the parameters affecting recovery and ash content

On the other hand, Fig. 2 is the normal probability plot which is plotted to identify the most effective factors. The significance of the parameters is detected from Fig. 2 by plotting the calculated effects of all factors against a line representing normal distribution. Significance of factors is determined according to their distance from the distribution line in Fig. 2. The effects on the efficiency of the factors on the right side of the line can be described as positive whereas the ones on the left are negative. Accordingly, AF65 (B) is the farthest factor to the normal distribution line with the greatest impact on combustible matter recovery. AF65 is followed by kerosene (A factor), interaction of kerosene-aeration (AC factor), aeration (C factor), and interaction ABC factor for all factors, respectively. The interactions of kerosene-AF65 and AF65-aeration are very close to the normal distribution line. Similar inferences can be made for the ash content as seen in Fig. 2. While the positive effects on the ash content is performed by AC, AB, A and BC, negative effects of the factors can be obtained as ABC, B and C.

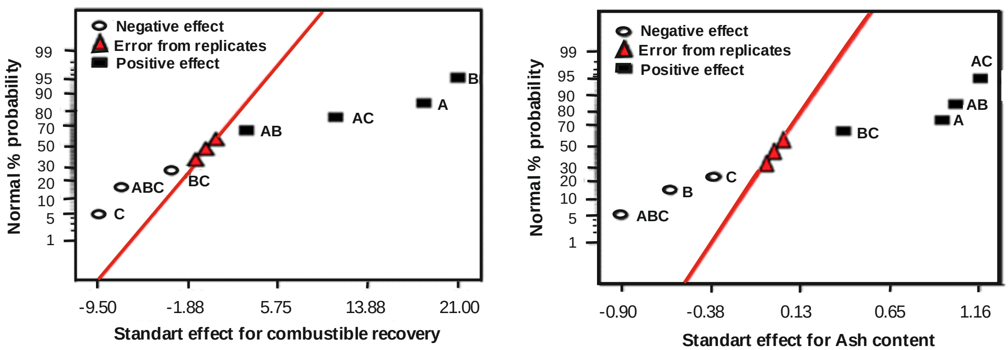

Fig. 2. Normal probability plot for combustible matter recovery and the ash content

Analyses of results

The effects of factors on the combustible matter recovery and the ash content of the lignite sample were determined quantitatively by the use of the F-test at the 95% confidence interval and regression model. The F-test indicates that all parameters significantly affect the combustible matter recovery of the Tuncbilek lignite while only AF65 and kerosene affect the ash content of the lignite (Table 3).

Table 3 includes both *F*-value of the Fisher-test and *p*-value for the combustible matter recovery. The model *F*-value of 17.62 implies that the model is statistically significant. Therefore, the model can be used to navigate the design space. In addition, "Curvature *F*-value" of 7.18 implies that there is a significant curvature, as measured by the difference between average of the center points and average of the factorial points in the design space. There is only a 7.51% chance that a "curvature *F*-value" this large could occur due to noise effect. The *p*-value of curvature in the factorial experiment for the recovery is 0.0751, which is greater than 0.05, indicating an insignificant curvature measured by the difference between the average of the centre points and the factorial points in the design space. An insignificant curvature suggests that the relation between the variables and the response has a linear form.

The main effect of all these factors on the recovery is significant at 95% confidence level (Table 3). According to the variance analyses (Table 3) and the regression equation (Eq. 8) factor A and B, that is kerosene and AF65, is the most effective factors in flotation of Tuncbilek lignite because of the highest *F*-values (Table 3). The order of main effect is AF65 > kerosene > aeration rate for the recovery and kerosene > AF65 > aeration rate for the ash removal. As Kelebek et al. (2008) suggested AF65 has a great importance for lignite flotation because it increases coal cleaning selectivity by the increase of froth depth and improves the stability of bubbles.

Table 3. Variance analysis for combustible matter recovery and ash content

Source	Combustible matter recovery		Ash content	
	F value	*p*-value prob > F	*F* value	*p*-value prob > F
Model	17.6	0.0192	25.30	0.0114
A-Kerosene	38.3	0.0085	33.61	0.0102
B-AF65	52.1	0.0055	14.32	0.0324
C-Aeration	10.7	0.0469	4.96	0.1123
A B	1.1	0.3782	39.13	0.0082
A C	13	0.0365	50.12	0.0058
B C	1.5	0.3151	5.1	0.1091
A B C	6.7	0.0819	29.83	0.0121
Curvature	7.2	0.0751	9.29	0.0555

The main influences of kerosene and AF65 on ash content are significant at the 95% confidence level. Positive coefficients of kerosene indicate an increase in the ash content, while negative coefficient of AF65 indicates a decrease in the ash content. According to the variance analyses (Table 3) kerosene is the most effective factor for the ash content of the floated product because of the highest F-value (33.61). On the other hand, the interactions of kerosene-AF65 and kerosene-aeration are also significant for the ash content (Table 3). In addition, the interaction between kerosene-AF65-aeration influences significantly the ash content (Table 3). The only insignificant effect is the interaction of AF65-aeration in the given range of parameters. The only statically significant interaction at 95% confidence level is kerosene-aeration for the recoveries while the AF65-kerosene, kerosene-aeration and interactions of all parameters (kerosene-AF65-aeration) significantly affect the ash content of Tuncbilek lignite (Table 3).

The regression equation for the combustible matter recovery (Y_{Cr}) and ash content (Y_{ac}) can be derived from Eq. 7 and has the following forms:

$$Y_{\mathrm{Cr}} = 45 + 9A + 10.50B - 4.75C + 1.5AB + 5.25AC - 1.75BC - 3.75ABC \quad (8)$$

$$Y_{\mathrm{ac}} = 8.37 + 0.47A - 0.31B - 0.18C + 0.51AB + 0.58AC + 0.19BC - 0.55ABC. \quad (9)$$

The regression analyses can be used not only for evaluating the main and interaction effects of factors, but also for evaluating the fitness of Eqs. 8 and 9. In addition, Eqs.8 and 9 can be utilized to determine the combustible matter recovery and ash content of the coal within the experimental conditions limits. In this case, a coefficient of determination, R^2, should be calculated. R^2 is a measure of the amount of variation around the mean value. When the R^2 value is close to 1, it means that predicted values can be appropriately referred to the experimental values. Recovery model (Eq. 8) has $R^2 = 0.9762$ meaning that 97.62% variability in the recovery and the rest 2.38% is attributed to errors. When the differing number of observations in analysis is relatively small, it decreases the R^2 value. Likewise, a smaller number of independent variables increases the R^2 value and vice-versa (Kelebek et al., 2008). Therefore, the modified new value is expressed as an adjusted R^2. The adjusted R^2 of 0.9208 is also satisfactory and verifies the significance of the model. On the other hand, ash content model (Eq. 9) has R^2= 0.9833. It means that 98.73% of the variability is caused by the ash content and the rest 1.27% is attributable to errors. The adjusted R^2= 0.9445 also implies the model significance.

According to the regression models the highest possible recovery (Eq. 8) was obtained as 70.99% at the high level of kerosene (3 kg/Mg), high level of AF65 (40 ppm) and low level of aeration (0.60 cm/s), while the lowest ash content (or highest combustible matter grade) (Eq. 9) was obtained as 6.94% at the high level of AF65 (40 ppm) and aeration (0.26 cm/s) and low level of kerosene (500 g/Mg). These results, taken together are unrealistic as will be shown in the next section of this paper.

This is so because recovery and ash content must be considered together for a given separation process.

Determining the optimal conditions

Statistical software was used to determine the optimal conditions by taking into consideration data and calculations obtained from the factorial design. Combustible matter recovery and grade together were taken as criteria and possible highest points selected for numerical determination of the optimal condition. All levels of factors were chosen in the range of –1, 0 or +1 for the calculations. According to the numerical optimization study, 53% recovery and 92.80% grade were obtained at the low level of kerosene (500 g/Mg), high level of AF65 (40 ppm) and low level of aeration (0.60 cm/s). It should noticed that this kind of statistical approach should be verified by using mineral processing approaches. Therefore, the result must be confirmed and evaluated from the mineral processing point of view.

It is known the best separation results for a series of experiments can be determined by using the so-called upgrading curves, including the grade-recovery plot known as the Halbich curve (Drzymala, 2006). The reason for this is that the Halbich curve has some advantages over other upgrading curves because it considers two essential parameters of separation results which are recovery and grade. These two parameters are widely used in industrial, kinetic and theoretical studies (Drzymala et al., 2012).

The Halbich plot for all 12 experimental points presented in Table 2 is given in Fig. 3. From the point of view of selectivity of the process (Drzymala et al., 2012) the best results are those forming an upgrading line which is the closest to the ideal separation line. This occur for the a1 line which represents flotation results obtained for runs 3 and 4 conducted at increasing amount of collector and constant (higher) level of frother and constant (lower) level of the aeration rate. Other lines denoted as a2, a3 and a4 were plotted by using the experimental data points of runs of 7-8, 5-6 and 1-2. The line for base points was drawn by using the experimental values repeated at centre points of factors (9-10-11-12) (level 0).

According to Kelly and Spottiswood (1982) for a given upgrading line the best result is the one which provides the greatest value of the mathematical product of grade and recovery for the combustible matter. Taking this criterion into account, the best result obtained in this study for the investigated range of parameters was for +1 levels of collector and frother and –1 level of the aeration rate. Thus, the best (optimal) flotation recovery was equal to 91.10% providing 71% of combustible matter in the concentrate at the 3 kg/Mg kerosene, 40 ppm AF65 and 0.16 cm/s aeration rates. It should be noticed the optimum point of separation depends on the criterion imposed on data. Here the criterion is the maximum value of multiplication of grade and recovery values. Thus, the optimal flotation point is not a result as calculated from the factorial design by considering recovery and grade independently (recovery: 70.99% at the high level of kerosene (3000 g/Mg), high level of AF65 (40 ppm) and low level of aeration (0.60 cm/s), ash content or highest combustible matter grade 6.94% at the high level of AF65 (40 ppm) and aeration (0.26 cm/s) and low level of kerosene (500 g/Mg)).

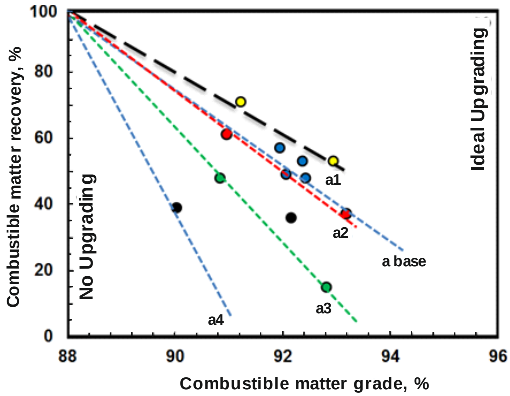

Fig. 3. The Halbich upgrading curve with the results of all experiments. Each straight line connects data points obtained for a constant level of both frother dose and aeration rate and increasing amount of collector. The line denoted as "a base" approximates the data points obtained for experiments conducted four times at the zero level of all parameters

The same conclusion regarding the optimum level of the investigated parameters for Tuncbilek lignite can be obtained using no directly the flotation results but the data points generated with Eqs. 8 and 9 and plotted on the Halbich curve. It means that the 3-variable 2-level (2^3) full factorial experimental design with four base point replicates can be useful not only for determination, expressed in numbers, interactions between parameters, but also optimum flotation conditions for the investigated coal provided that a proper criterion for the optimum is applied. The optimum result obtained from the numerical optimization is also one of the points on line a1 in the Halbich curve.

Conclusions

This study was performed in order to determine, expressed in numbers, interactions between parameters and best conditions for the flotation of Tuncbilek lignite by using both statistical approach and upgrading curves. The flotation experiments were carried out using the 3-variable 2-level (2^3) full factorial experimental design with four base point replicates, and the results were analyzed by using the regression model, F-test and Halbich's upgrading curve for the responses of ash content (or combustible matter grade) and combustible matter recovery. The following findings are obtained from the present study:

- results obtained from this study (Pareto charts and normal probability plot) indicated that the most effective parameters on the combustible matter recovery and the ash content were frother (AF65) and collector (kerosene), respectively.

- the regression and F-test qualitatively showed the main and interaction effect of factors. All parameters investigated in the study indicated a significant influence on the recovery. However, the only significant interaction occurred between kerosene and aeration. On the other hand, except of AF65–aeration interaction, all interactions showed a significant effect on the ash content while only the main significant effects were kerosene and AF65 on the ash content.
- the optimal conditions for the flotation can be determined by taking into account both recovery and grade simultaneously using either the results of the factorial design or the Halbich plot and using criterion of the maximum value of mathematical product of grade and recovery. According to the plot, the best results for the investigated coal can be obtained with the ash content of about 9% and the recovery of 71% at the higher amount of kerosene (3 kg/Mg) and AF65 (40 ppm) with lower aeration rate (0.16 cm/s).

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