Analysis of flotation of unburned carbon from bottom ashes

Oktay Şahbaz¹, Mustafa Çınar² and Şadan Kelebek³

Bottom ash from power plants contains unburned carbon (UC) as a waste, which is a potential contaminant for the environment unless recycled as a source of energy. In the present study, separation of the UC (3.1%) from the bottom ash of a power plant has been studied. The overall grade of combustible matter for the concentrates ranged from 20.6% to 28.8 while the recovery levels varied from 45.7 % to 84.0%. A statistical analysis of the combustible recoveries according to a 3-variable 2-level (2^3) factorial experimental design, examined the influence of dosages collector (kerosene) and frother (AF65) and also impeller speed. The main effects of all three factors on the recovery are positive. The most important factor is collector dosage, while the frother has a secondary significance. The effect of impeller (conditioning) speed is also statistically significant. Two of the 2-way interaction effects involving kerosene consistently point out the selectivity of the floation process for carbonaceus particles, which have been discussed in relation to dispersion and attachment of collector droplets to the particles, interfacial area and probable collecting properties of the frother used.

Key words: Bottom ash, flotation, unburned carbon, factorial design

1. Introduction

Currently, most of the energy requirement in many developing countries is supplied from conventional power plants using coals. Trends of statistical data on exploration projects show the continuity of importance of lignite as a source of energy in Turkey. Thus, investigations on separation of combustible matter from raw coal and associated waste from the combustion process are also important. A great deal of waste is generated as a result of the lignite combustion process, which ends up in disposal sites with potential negative effects on the environment. As the environmental legislations have become stricter there are increasing obligations to minimize or eliminate potential damage to the environment.

Out of millions of tons of ash discharged from power stations annually, only a small amount (about 1 %) is evaluated by the national cement industries. Specifically bottom ashes have limited usage (Şahbaz et al., 2008). The main reason for this is the unburned carbon (UC) content. The ash material containing high levels of UC promotes defects, unwanted color (black) development, depletion of chemicals in the process environment through adsorption, and carries undesirable chemicals such as ammonia into processes. The organic nature of UC and its reactivity increases the risk of introduction and build-up of organic contaminants such as chlorobenzenes, dioxins, polychlorinated biphenyls, chlorophenols and polycyclic aromatic hydrocarbons, etc. (Huang et al., 2003; Asokan et al., 2005; Şahbaz et al., 2008). These organic contaminants have relatively high toxicity with potential negative impact related to groundwater pollution and contamination of sub-soils in land filling areas. Therefore, it is an economical and environmental obligation to remove the carbon from bottom ash and fly ash.

Recently, some studies have been reported on the topic. Demir et al. (2008) demonstrated a reduction of ash content in bottom ash from 77 % to 42 % at a recovery of 53 % by using a laboratory flotation column. Şahbaz et al. (2008) used a laboratory Jameson cell and reported that the ash content can be decreased from 90 % to 44 % at a recovery of 68 %. Additional studies on the bottom ashes involved environmental aspects and use of other separation techniques (Bayat and Toraman, 1996; Ozdemir et al., 2001; Huang et al., 2003; Asokan et al., 2005; Kurama et al., 2007). A review of low rank coal and bottom ash processing literature indicates a lack of statistically based studies on application and/or interaction of reagents with ashes. Kelebek et al. (2008) studied the effects of dodecylamine (DDA), kerosene and pH on the recovery of combustible matter and ash content of a lignitic coal from Tuncbilek (Turkey) using a 23 factorial design. Uçurum (2009) reported results on separation of carbonaceous residue from combustion of petroleum coke using a 23 full factorial design approach for assessment of the effect of types and dosages of frother (pine oil, MIBC), of collector (diesel oil, kerosene) and pulp density.

In the present study, separation characteristics between the carbonaceous matter and ash components in a bottom ash sample from Tuncbilek Power Plant were investigated through statistical analysis. A three-variable and two-level (23) factorial experimental design utilized recovery of carbonaceous matter and ash content as

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dependant variables as a function of the kerosene dosage and Aeroforth 65 (AF65) together with the effect of conditioning speed.

2. Material and Methods

2.1 Samples and reagents

The sample used in the present study was obtained from the bottom ashes of Tuncbilek Power Plant, which is 50 km away from Kutahya, Turkey. The carbon content of the sample was 3.1 % (i.e., 96.9 % ash content) and the particle size (d_{80}) was nearly 6 cm. Based on a previous experience with the Tuncbilek coal (Kelebek et al., 2008), the sample was ground to decrease the top particle size to about 75 µm. Representative charges that were prepared using standard laboratory techniques were kept in a freezer until required for testing.

The collector was kerosene used as a mixture of neutral hydrocarbons. The frother was AF65 (Cytec Aerofroth 65), which is a polypropylene glycol based frother used in its commercial quality form "as received".

2.2 Flotation tests

Twelve flotation tests were required for this analysis. The tests were carried out in a Denver cell of 21 capacity using slurry at about 7.5 % solids. As a standard reference, distilled water was used throughout the tests. The impeller speed of the cell for conditioning was varied from 1300 to 2100 rpm. The airflow rate was fixed at 4 l/min. The temperature during experimentation was 20 ± 1 °C. The natural pH of the pulp was about 8.5, which was maintained for all tests. Since the flotation feed was produced using dry grinding in a shutter box, each sample was conditioned in water for 5 minutes following grinding to ensure complete wetting at required pH before any reagent addition was made. The conditioning period for each of kerosene addition was 2 minutes while it was 30 seconds for frother. Kerosene and frother were added in their full strengths in terms of droplets. Five concentrates were collected each for one minute corresponding to a total period of five minutes. At the end, the flotation products were filtered, dried, weighed and analyzed for ash to determine recovery of combustible matter.

2.3 Experimental design

The statistical design of experiments is useful in that the simultaneous assessment of several factors can be made by determining the main and interaction effects. A three variable and two level (2^3) design was used in this study with four replicates carried out at the center-points. Each value of flotation variables was converted into the following three coding levels: -1, +1 and 0 for low, high and center point, respectively. The center point is the arithmetic mean of the high and low levels. The formulas used for the coding are given below:

Kerosene, A = (Kerosene dosage $- 272.5$) / 102.5	(1)
AF 65, B = (AF 65 dosage - 170) / 68	(2)

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		P	e on are on one	5 00000/, 0				1,00,	,	(2)	

Operating conditions of the tests performed (i.e., variables and their levels) for this particular experimental design are shown Table 1.

	C	oded variable leve	ls	Actual variable levels			
Run Order	Collector X1	Frother X2	Mixing X3	Kerosene [g/tonne]	AF65 [g/tonne]	Impeller (RPM)	
1	-1	1	-1	170	238	1300	
2	-1	-1	1	170	102	2100	
3	1	1	-1	375	238	1300	
4	1	-1	1	375	102	2100	
5	1	-1	-1	375	102	1300	
6	-1	1	1	170	238	2100	
7	-1	-1	-1	170	102	1300	
8	1	1	1	375	238	2100	
9	0	0	0	272.5	170	1700	
10	0	0	0	272.5	170	1700	
11	0	0	0	272.5	170	1700	
12	0	0	0	272.5	170	1700	

Tab. 1. Selected operating conditions of the tests according to the 2^3 *factorial design used.*

Combustible matter recovery and ash content have been treated as "responses". Design Expert software (www.statease.com) was used for the analysis of experimental data from the randomized tests with experimental conditions, according to the format of the statistical program, which yielded the main and interaction effects that are specific to the flotation system under investigation. The main effect of a factor is given as the change in a response produced by the change between the upper and lower level of that factor. A confidence interval of 95 % is commonly chosen for determination of the significance of the model, main and interaction effects. In the current analysis with k=3 factors, 4 center points have been used to estimate the experimental error and the variance, σ^2 . The variance of main and interaction effects are given by Equation 4 and 5. (4)

Variance (Effects) = $4\sigma^2/2k$

Calculated main or interaction effect / (Variance (Effects))
$$0.5 \ge t_{3,0.025}$$
 (5)

The value of $t_{3,0,025}$ is 3.182, which can be obtained from the t – distribution table and if the estimated main and interaction effects are significant at 95 % confidence level, then they will satisfy the above criteria. A general expression representing the main and interaction effects of the 2^3 factorial design is given below (Montgomery and Runger, 2011):

Response = $\beta_0 + \beta_a X_1 + \beta_b X_2 + \beta_c X_3 + \beta_{ab} X_1 X_2 + \beta_{bc} X_2 X_3 + \beta_{ab} C X_1 X_3 + \beta_{ab} C X_1 X_2 X_3$ (6)

Where, β_0 , β_a , β_b , β_c , β_{ab} , β_{bc} , β_{ac} , and β_{abc} represent the coefficients. The main and interaction effects can be calculated by using matrices and details of such calculations can be found in the general source referred to above.

3. Results and Discussion

3.1 Flotation kinetics

Results of flotation tests are given in Figure 1, which shows recovery of combustible matter as a function of time. Highest recoveries were obtained using the high levels of the variables used (kerosene and mixing level, Test No.8 in Table 1). Two experiments which utilized the lowest levels of reagents (kerosene and AF65, respectively) resulted in the lowest recoveries (Tests no.1 and 5), which make sense from the point of enhaced probability of collisions (higher surface area of bubbles and mixing rate) and adhesion (larger coverage by collector at higher dosage).



Fig. 1. Flotation recovery of combustible matter as a function of time.

The rest of the data are scattered in between these lines. In general, these kinetics curves are characterized by a time delay, which has been occasionally observed in the first cell of coal flotation circuits (Aplan and Arnold, 1991). This time-lag can be attributable to poor conditions involving reagent interactions leading to froth destability as well as lack of slurry mixing. The feed characteristics of the sample used in the current study (i.e., lack of hydrophobicity and low grade of the combustible matter and extremely slimy nature combustion products in the slurry) are also believed to contribute to the time-lag. Further details on flotation kinetics are under examination, which may be available later.

3.2 Grade-recovery and flotation selectivity

The overall concentrate grades from all twelve tests carried out are shown together with corresponding recoveries of combustible matter in Figure 2. The amounts of both carbonaceous matter and ash are plotted. The concentrate carbon contents vary between 20.6 % and 28.8 %, corresponding to an upgrading ratio of 6.4 and 9.0, respectively. The ash content is reduced from 96.8 % to values below 80 %. Although these levels are typical of a single stage flotation for a complex material, there is clearly room for improvement in rejection of ash constituents, which are mostly in a highly fine state due to disintegration during the combustion process experienced. Thus, the full potential of this material for upgrading requires cleaning stage(s) that can be carried out as part of locked cycle tests.



Fig. 2. Concentrate grade as a function of recovery.

As can be noted, the recovery varies practically independent of the concentrate grade. Compared to the case with recovery, a variation of the concentrate grade occurs in a narrow range regardless of experimental conditions.



Fig. 3. Recovery of combustible matter versus recovery of ash.

Selectivity in separation of unburned carbonaceous matter from ash components can also be examined by plotting their recoveries against each other. Figure 3 shows combustible recovery as a function of ash recovery. It can be noted that boundary of trends observed in figure 1 is the same in figure 3. The lower curve representing Test No.1 has low collector dosage and low mixing rate compared to Test No. 8 with all three variables at their high levels. The fact that highest level of collector, frother and mixing (Test No.8, Tab. 1) representing highest flotation rate (Tab. 2) also represent the highest selectivity against ash recovery, which can mainly be attributed to effectiveness of kerosene on carbonaceous matter as a selective collector. On the other hand, the poorest selectivity experienced at the lowest collector dosage (Test No.1) is attributable to a relatively greater amount of entrainment of extreme fines, which is expected due to slimy nature of ash components in the slurry.

3.3 Statistical analysis

A total of 12 experiments were carried out according to the 23 factorial design for statistical analysis. Four tests represented the mid point repeat tests to account for assessment of experimental error. Analysis of variance (ANOVA) for the combustible recovery is given in table 5, which represents two cases- one with the center point information (curvature) separated from the model (i.e., the mean is adjusted for curvature). The term ABC was included in the analysis to check for a three-way interaction. As can be noted, the model and all coefficients with the exception of BC and ABC are significant at 95 % confidence level since their corresponding p-values all less than 0.05. The term "A", the kerosene having the highest F-value is the most significant parameter contributing to the recovery. The second contributor to the recovery is the frother (B). Effect of the other parameter (C) is much smaller. From the two-way interaction effects, AC (Collector-impeller speed) has a greater significance compared to AB (Collector-frother).

The other case shown in table 2 represents no adjustment for the curvature (i.e., center points are included in statistical treatment). The regression model, in this case, is not adequate at 95 % significance. It is valid, however, at 90 % confidence since its p-value is less than 0.1, but higher than 0.05. Two of the three coefficients for main effects (A, the kerosene, and B, the frother) are significant. All other coefficients are insignificant at 95 % or 90 % confidence level. The correlation coefficient, R^2 as an indication of the fit of the model is 0.89. It means that 89 % of the variability in the recovery can be accounted for the model, and the rest is attributed to errors. When the three-way interaction term ABC is removed, the model is greatly improved as its p-value is reduced to 0.027. Thus, the model becomes significant at 95 % confidence with no change in the significance of other terms.

Sources	Curvat	ure adjusted	Unadjusted		
Sources	F Value	p-value Prob > F	F Value	p-value Prob > F	
Model	158.5	0.0008	4.58	0.0802	
A-Kerosene, g/t	592.6	0.0002	17.11	0.0144	
B-AF65, ppm	447.6	0.0002	12.92	0.0229	
C-Impeller, rpm	21.6	0.0188	0.62	0.4742	
AB	14.0	0.0334	0.40	0.5600	
AC	23.7	0.0165	0.69	0.4542	
BC	9.7	0.0524	0.28	0.6238	
ABC	0.0	0.9186	0.0004	0.9858	
Curvature	135.5	0.0014			

Tab. 2. Analysis of variance for combustible recovery.

The regression equation for the adjusted curvature model of the combustible recovery is given in Eq. (7), which includes all terms. For the unadjusted model, the only difference is in the intercept value; it becomes 68.20.

$Y_CRec = 65.63 + 9.32A + 8.10B + 1.78C - 1.43AB + 1.87AC - 1.19BC - 0.043ABC$ (7)

Analysis of variance for the ash content is shown in table 3. According to the results, the ash content model is statistically insignificant whether an adjustment for curvature is applied or not. None of the effects of flotation variables on ash content are statistically significant at 95 % or 90 % confidence level. Considering that the variation of ash content occurs in a narrow range (Fig. 2), the insignificance of the model and model coefficients is not unexpected. The scatter observed is attributable to experimental noise.

Sources	Curvat	ure adjusted	Unadjusted		
	F Value	p-value Prob > F	F Value	p-value Prob > F	
Model	0.81	0.6112	0.99	0.5147	
A-Kerosene, g/t	0.62	0.4742	0.76	0.4229	
B-AF65, ppm	0.84	0.4105	1.03	0.3567	
C-Impeller, rpm	1.56	0.2799	1.91	0.2259	
AB	0.33	0.5967	0.40	0.5536	
AC	0.18	0.6920	0.22	0.6574	
BC	1.33	0.3135	1.62	0.2588	
ABC	0.09	0.7789			

Tab. 3. Analysis of variance for ash content.

The value of R^2 for the ash content model is very low at about 0.55, which means the variability in the ash content and the rest 46% is attributed to errors, which is quite high. This low value of R^2 for Eq. 8 practically means that the ash content model does not satisfactorily represent the relation between the selected parameters and this response.

 $Y_{ash} = 75.90 - 0.73A + 0.85B - 1.16C + 0.53AB - 0.4AC + 1.07BC - 0.084ABC$ (8)

3.3.1 Effects of variables and their interactions

According to results obtained from variance and regression analyzes, the effect of kerosene is positive as the most significant contributor to the combustible recovery, corresponding to about 47.5 % contribution according to standardized effects table (not shown). However, its effect on the ash content is negative and relatively small, although this model is not valid at 95 % confidence level. A negative effect of the collector on ash content as well as its positive effect on recovery is compatible with a selectivity of kerosene towards unburned carbon, i.e., genuine recovery of carbonaceous material, which is linked to adsorption of kerosene on the coal particles.

As a polyglycol type of frother, the AF65 is generally used for the flotation of hard to float coal particles. The increase in its dosage is expected to reduce the bubble size by decreasing the frequency of coalescence, an event which promotes the gas hold-up and total interfacial area, thus resulting in the selective recovery of carbonaceous material in a given time. Another reason, why an increase in AF65 dosage can be increasing the recovery may be related to its possible collecting ability of the coal particles. However, this seems to occur somewhat non-selectively since it also has a positive effect on a collection of ash minerals. These effects on grade and recovery can probably be isolated, but this is not attempted since the ash content model is not as good as the recovery model.

The impact of impeller speed on the recovery and ash content is similar in sign to that of kerosene. The main difference is in their magnitude, which is less in recovery effect and more in grade. A greater impeller speed is expected to promote a better agitation in flotation slurry necessary for breaking air into bubbles. It also provides a better dispersion of bubbles in the cell while suspending solids, long enough for bubble-particle interactions, hence resulting in improved collision efficiency and a higher flotation rate. The frequency of collisions between the bubbles and the relatively fine particle is enhanced by an increase in the impeller speed, especially in the regions away from the impeller (Ahmed and Jameson, 1985).

According to statistical analysis, the two-way interaction effect between kerosene and frother (AB) has a negative contribution to recovery. This can occur by a negative effect of oil on frothability and froth stability. Oil is known to decrease dynamic froth stability (Niewiadomski et al., 2001). The total interfacial area provided by frother dosage is probably somewhat smaller due to the same reason.

The two-way interaction between kerosene and impeller speed (AC) has a positive effect on the recovery. As an insoluble collector, the action of kerosene occurs through attachment of distinct droplets to mineral particles (MacKenzie, 1970). A greater impeller speed is believed to produce a higher number of oil droplets with finer diameters due to a greater energy input, which enhances the probability of attachment oil droplets to coal particles. This is, in turn, helpful for improved recovery of such particles. There is a parallelism between the effect of oil and impeller speed on combustible recovery when these effects are compared with those on ash content. Again, these results are consistent with the previous remark made on the selectivity of the flotation process for carbonaceous particles.

3.3.2 3D representation of main trends

Simultaneous effects of frother and collector dosage on combustible recovery are shown in figure 4. Recovery of combustible matter reaches its greatest level at the highest dosage of these reagents. The dependence of the combustible recovery on frother at the highest collector dosage is somewhat less than at its lowest dosage. Dependence of the combustible recovery on kerosene at the highest frother dosage is also

somewhat less than at its lowest dosage. In other words, the flotation recovery benefits relatively more from the reagent that is available in a greater dosage. The recovery suffers most from a simultaneous lack of these two reagents.

The 3D variation of recovery as a function of both impeller speed and frother dosage is illustrated in figure 5, which shows that in the range studied, the dependence of combustible recovery on frother dosage is greater than that of impeller speed. As long as frother is at its highest dosage, impeller speed has a little positive effect on recovery. Maximum recovery is obtained when both collector and impeller speed are at their highest level.



Fig. 4. 3D variation of combustible recovery with respect to frother and collector dosage.



Fig. 5. 3D variation of recovery with respect to impeller speed and frother dosage.

This maximum level occurs at about 77 %, which is lower than the case involving collector (Fig. 1). However, a contradictory statement can be made for the lowest recovery level, which is higher than the case involving collector. The reason for the lowest recovery involving the collector is attributable to an interaction between frother and collector, as explained above.

The 3D variation of recovery with respect to impeller speed and kerosene dosage is shown in figure 6. In the range tested, the impeller speed has almost no influence on the recovery when the collector level is at its lowest dosage. The effect of impeller becomes most definitive at the highest collector dosage, at which the recovery increase by about 9 % when the impeller speed is increased to its highest level (i.e., 2100 rpm).



Fig. 6. 3D variation of recovery with respect to impeller speed and kerosene.

The collector level clearly has a greater influence at both the low and high speed of collector. Despite the use of 375 g/ton of kerosene as the maximum, the recovery is only about 84 %. This is related to the substantially oxidized nature of this sample. A further study can explore possibilities of increasing the recovery levels with an attempt to optimize process conditions. Particle size can be studied as an additional variable. The dosage of the collector as well as the impeller speed for conditioning can increase to attain higher recovery levels.

4. Conclusions

In the present study, bench-scale flotation of bottom ash of Tuncbilek Power Plant was studied through a 2^3 factorial experimental design. The variables used were dosages of kerosene (collector) and AF65 (frother) as well as the impeller speed (conditioning). The following conclusions can be drawn from this study:

- 1. For the curvature adjusted case, the statistical model developed for recovery of the combustible matter is significant at 95 % confidence level. The main effects of all three factors in the recovery, as well as two of the three two-way interactions (i.e., the collector-frother and collector-impeller speed), are also significant at the same confidence level.
- 2. For the unadjusted case, the model for recovery is significant at 90 % confidence level. When the three-way interaction term is removed from the analysis, the model is improved to 95 % confidence level. In this case, only A, the kerosene, and B, the frother, as the main effects are significant at the same confidence level.
- 3. The regression equation of the model obtained for the ash content is not significant at 95 % confidence level, at which none of the parameters were significant either.
- 4. The main effects of all three factors on the recovery are positive. The most important contributor to the recovery is collector dosage while the frother dosage is the second contributor. The impeller speed (for conditioning) has a lesser impact compared to these two.
- 5. The recovery process is mainly determined by the selective attachment of kerosene droplets on the unburned coal particles. Frother effect is primarily related to bubble production and enhancement of interfacial area available to carbonaceous particles. The contribution of frother to hydrophobicity (hence recovery) by adsorption on carbonaceous particles is not ruled out.
- 6. As a two-way interaction between kerosene and impeller speed, better dispersion of oil droplets is facilitated by greater speed of the impeller, which results in a positive effect on the recovery.
- 7. The two-way interaction effect between kerosene and frother (AB) has a negative contribution to recovery. This is explainable by a negative effect of oil phase on frothability and froth stability. The total interfacial area provided by frother dosage is probably somewhat smaller due to the same reason.

The further study can be performed to optimize unburned carbon flotation by using the different statistical methods such as Taguchi method, and various upgrading curves (i.e., Fuerstenau upgrading curve).

Acknowledgements: Funding of this research work provided internally by the Dumlupinar University (DPU) is greatly appreciated. The authors thank Tuncbilek Power Plant management for the bottom ashes sample used in this study and Mr. A. Cantas for his experi help.

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