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### Full Length Article

# Application of central composite design method to coal flotation: Modelling, optimization and verification

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HIGHLIGHTS

• Investigation of flotation parameters by using central composite design method.

• Collector and frother dosage effects were found to be the most significant.

• The optimum values of response variables were predicted from the models.

• Verification tests proved the validity and adequacy of the predicted models.

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#### 1. Introduction

#### ABSTRACT

In this study, collector dosage, frother dosage, solid ratio and air flow rate that can be effective parameters on the combustible recovery and ash content of clean coal and their interactions in flotation are described with a mathematical model by using statistical design method. The test results have shown that both models obtained for ash content and combustible recovery were found to be statistically significant, and also the effects of collector and frother dosage on the response variables are found to be higher than those of other parameters. Additionally, verification experiments at optimum conditions proposed by the models were done to determine the validity and adequacy of the predicted models. © 2016 Elsevier Ltd. All rights reserved.

According to the estimations, while 20–40% of coal produced globally are undergoing physical cleaning processes like heavy media or jigging, 40% of produced coal including fine coal are cleaning by flotation [1,2]. Coal flotation is widely applied in countries such as China, USA, Australia, Canada and India [3]. However, fine coals are generally utilized in coal fired power plants for electricity generation in Turkey without any concentration.

Heavy medium separation methods are mostly preferred in coarse coal cleaning. In this method, heavy medium is prepared generally with magnetite. During the separation, coal can be easily broken into fine particles due to its brittle nature. This causes fine coal particles to blend with fine magnetite particles, which makes recycling of magnetite particles difficult. In a study, the application of floatation to this fine coal and magnetite particle mixture before magnetite recycling was increased the recovery of magnetite particles [4].

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Different coals have different floatability characteristics according to their rank, oxygen content, and amount and type of impurities. It is generally known that low rank coals, especially lignites, are among the difficult to clean by flotation. Their poor floatability has been mainly attributed to a high oxygen contents [5]. Oily collectors will not be spread on the surface of low rank coals that contain greater amounts of oxygen, which leads to poor flotation performance and large reagent requirements even to obtain moderate recovery [6]. When literature related to coal flotation is examined, the studies can be classified in three main subjects: flotation of oxidized/low floatability coals [2,5-11], parameter optimization [12-16] and comparison of conventional and column flotation techniques [17,18]. In the parameter optimization studies, statistical design of experiment (DOE) methods were mostly employed. There are many available techniques to be used in DOE. One of these techniques is called Response Surface Methodology (RSM) that enables multi-variable optimization. This multi-variable optimization makes this technique more advantageous in areas like mineral processing where there are two responses such as grade and recovery.







In this study, collector dosage, frother dosage, solid ratio and air flow rate and their interactions which are considered to be effective parameters on the ash content and combustible recovery of clean coal were described with mathematical models by using Central Composite Design (CCD) method in the flotation of low quality lignites. The interactions of these parameters were also studied. The optimum conditions for three different requirements such as lowest ash content, highest combustible recovery and possible lowest ash content with highest combustible recovery were determined from these models. Verification tests were also performed according to the conditions proposed by models for these three purposes. Finally, test results and results predicted by models were compared.

#### 1.1. Statistical design of experiment

Statistical design of experiments has several advantages over classical optimization methods where one parameter is optimized at each time [19]. In order to obtain the required data in statistical designs, all experiments are carried out in planned way and the results can be analyzed systematically by means of variance analysis. The obtained data also can be assessed for optimization goals [20]. Full factorial design is one of the methods used to evaluate the effects of parameters and their interactions [21]. However, the increase in the number of parameters in full factorial designs causes the number of experiments to increase parabolically. This fact limits the usage of this method in the studies where the number of parameters is high. On the other hand, RSM are the sum of statistical and mathematical methods enabling more data collection with lower number of experiments. In RSM, every parameters should have at least three levels so that parabolic effects can be determined. After variance analysis, generally, a second order regression equation of response variable (y) is obtained in the form of dependent variables  $(X_{ii})$  and their coefficients  $(\beta_{ii})$  as given in Eq. (1). In this equation, squared terms represent the quadratic effects [19,22,23]. The estimation of optimum conditions can be estimated by using the model equations obtained from RSM together with desirability functions developed by Derringer and Suich [24]. Besides, multiple optimization for multiple response variables can be done simultaneously [22].

$$y = \beta_0 + \sum_{j=1}^k \beta_j x_j + \sum_{j=1}^k \beta_{jj} x_j^2 + \sum_{i < j = 2}^k \beta_{ij} x_i x_j + \varepsilon$$
(1)

In this study, CCD method was used in order to optimize simultaneously two different response variable; the ash content and combustible recovery of clean coal in coal flotation.

#### Table 1

Characterization of feed sample.

#### 1.2. Central composite design

CCD is an experimental design method and widely used to establish a second-order response surface model in process optimization studies [25]. CCD as an effective alternative to full factorial design enables to gather more data with lower number of experiments [23]. The discriminating property of CCD from the other methods is the usage of axial points defined as  $\alpha$  value [26]. Axial points require two more experiments for each parameter at lowest and highest levels. In this case, experimenting each factors at 5 level causes the quadratic effect mentioned earlier to be defined precisely.  $\alpha$  takes different values depending on rotatability and orthogonality of the design and number of experiments [27,28].

#### 2. Material and methods

#### 2.1. Materials

The samples used in the experiments were taken from Cayirhan lignite region underground pits. Cayirhan region lignite is located in middle part of Turkey. Samples were dried and crushed with jaw crusher to -1 mm and sieved into five size fractions. Flotation experiments were performed at the size fraction of  $-0.1 \pm 0.038 \text{ mm}$ . Proximate, ultimate and chemical analysis of flotation feed are given in Table 1.

#### 2.2. Methods

Experimental studies were carried out by using laboratory type floatation machine with 1.5 L cell at the Mineral Processing Laboratory of Eskisehir Osmangazi University.

In the experiments, pulp temperature, collector conditioning time and stirring speed were kept constant at 21 °C, 5 min and 1200 rpm, respectively. pH level was chosen as natural pH and measured as 8–8.5.

In the preliminary tests, collector type, dispersant/depressant amount, collector/toluene ratio and retention time were tested and the most convenient conditions were determined. Different collector types such as fuel-oil, kerosene and Philflo (supplied by Chevron Philips Co. [14]) were tested and fuel oil was chosen as optimum.

Generally, both in laboratory and large scale flotation processes, for the purpose of homogeneous dispersion of collectors in pulp, emulsifier reagents that helps forming of small oil drops are used

Proximate analysis		Ultimate analysis						
Combustible matter (%)	Ash content (%)	C <sup>daf</sup> (%)	H <sup>daf</sup> (%)	N <sup>daf</sup> (%)	O <sup>daf</sup> (%)	S <sup>d</sup> (%)	Q <sub>gross</sub> (kcal/kg)	Q <sub>net</sub> (kcal/kg
13.92	86.08	46.05	4.54	0.97	36.34	2.59	475.00	435.00
Chemical analysis of ash <sup>(c</sup>	<sup>1)</sup> (%)							
Al <sub>2</sub> O <sub>3</sub>			12.00					
CaO			4.60					
Fe <sub>2</sub> O <sub>3</sub>			7.60					
K <sub>2</sub> O			1.60					
MgO			6.10					
MnO			0.10					
Na <sub>2</sub> O			2.90					
P <sub>2</sub> O <sub>5</sub>			0.20					
SO <sub>3</sub>			4.40					
SiO <sub>2</sub>			53.00					
TiO <sub>2</sub>			0.30					

<sup>(d)</sup> dry basis; <sup>(daf)</sup> dry, ash free basis.

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