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A STUDY OF THE EFFECT OF OPERATING PARAMETERS IN COLUMN FLOTATION USING EXPERIMENTAL DESIGN

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Abstract: The effect of air flow rate, pulp density and particle size was studied using central composite design for coal samples from the Lazy mine. Evaluation of column flotation tests was based on two dependant variables such as ash content and combustible matter recovery in the concentrate. The ash content in the concentrate was from 4.61 to 9.62% with the recovery of combustible matter from 17.43 to 81.98%. The ANOVA statistical analysis showed that the main effect of air flow rate has a significant impact on the combustible matter recovery and ash content in the concentrate. The main effect of pulp density on the combustible matter recovery is significant, whereas for the ash content it is not seen. There is a strong effect of the particle size on the ash content and combustible matter recovery in the concentrate. The interaction of the effect of the pulp density and particle size has a significant impact on the ash content in the concentrate.

Keywords: froth flotation, column flotation, coal, experimental design, operating parameters

Introduction

Froth flotation is a separation process based on the difference in hydrophobic/hydrophilic surface properties of mineral particles. It is conducted in a three-phase system (gas-liquid-solid) in which hydrophobic particles attach to a gas bubble and travel upwards to the pulp surface into the froth layer, while hydrophilic particles remain in the pulp. Particle surface properties can be changed by flotation reagents, therefore flotation is considered to be a universal method for mineral processing. In addition to mining engineering, where flotation is used for separation of minerals, it is also used for removal of oil from water, wastewater treatment and de-inking of recycling paper (Uribe-Salas et al., 2007). Although the process was introduced in the beginning of the 20th century, it is still not entirely understood and relatively inefficient (Shean and Cilliers, 2011). One of the reason is the fact that

flotation is influenced by a large number of various variables. Attachment of a particle to a bubble is an elementary act in flotation (Ralston et al., 1999, Shahbazi et al., 2010) that lasts for a very short period. It is influenced by a number of parameters, ranging from physical properties of particles and gas bubbles to chemical properties of a pulp (Albjanic et al., 2010). It has been confirmed that recovery of floating particles highly depends on the period of a particle adhering to a gas bubble, which is related to other parameters that characterise the flotation process (Albjanic et al., 2010).

Flotation is used for fine coal beneficiation, where operating parameters must be optimised for individual coal (Kaylani et al., 2005). A flotation column is used in the cleaning stages of flotation circuits due to a better selectivity in relation to conventional mechanical flotation cells. The design of flotation column was patented in early 1960s (Finch and Dobby, 1990), and since then several advanced flotation technologies have been commercialised (Mohanty and Honaker, 1998). During 1980s development and commercialization of column flotations were one of the most important achievements in fine coal cleaning (Honaker and Mohanty, 1996).

The aim of this work is to study the impact of operating parameters on coal flotation, with the special emphasis on the impact of particle size, air flow rate and pulp density on the recovery of combustible matter and ash content in the concentrate. The particle size has an important role in bubble-particle collision. Fine particles have a larger specific surface that influences reagent adsorption, i.e. the chemical activity of a particle. In the case of coarse particles buoyancy of a bubble-particle aggregate may decrease, and increasing particle sizes may result in longer induction time and commensurate deterioration in floatability (Feng and Aldrich, 1999). The maximum particle size in overflow depends on its density, hydrophobicity, process dynamics and other parameters (Kowalczyk et al., 2011; Watanabe et al., 2011). The air flow rate has a significant role in determining the flotation rate constant and also in creation of a froth layer. As the particle size increases, time needed for the particle to attach to the bubble increases significantly (Ye et al., 1989; Yoon and Yordan, 1991). Despite testing conducted on the impact of a particle size on flotation kinetics (Hernainz and Calero, 2001; Polat et al., 2003, Ucurum and Bayat, 2007; Humeres and Debacher, 2002; Abkhoshf et al., 2010), and despite the fact that many physical and chemical factors related to the particle size have been identified, it is difficult to predict the real effects of those factors. The recently conducted tests demonstrated that the ash content in tailings and the recovery in the concentrate decreased as the particle size increased and the ash content in the concentrate increased as the particle size decreased (Brozek and Mlynarczykowska, 2013). The tests carried out on the particle size of 0.5–0.25, 0.25–0.075 and –0.075 mm showed lower ash content and higher recovery for coarse particles 0.5–0.25 mm and opposite results for fine particles –0.075 mm at the beginning of flotation. As the process continued the cumulative recovery of combustible material increased to approximately 95% (Li et al., 2013).

The pulp density influences the bubble size and mixing characteristics (Goodal and O'Connor, 2003). The increase in the solid phase content in the pulp influences the

pulp viscosity and the bubble–particle aggregate may be overloaded attaining high densities. As the consequence a number of particles will sink and not float. With the increased content of mineralized bubbles the friction between them increases leading to coarse particle detachment that influences a flotation process (Perez Garibay et al., 2002; Akdemir and Sonmez, 2003).

Separation results can be estimated by grade-recovery plot also called the Halbich upgrading curve (Drzymala, 2006). The Halbich upgrading curve uses two parameters that are often used both in the laboratory tests and practical use (Drzymala et al., 2013; Sahbaz, 2013). On the other hand, factorial design offers many advantages during estimation of parameters that control a certain process, e.g. fewer numbers of necessary tests. Due to that less material and time is needed for tests, possibility of error is reduced and it can be also used to determine the optimum conditions for an individual process (Box et al., 1978).

Materials and methods

Applied material

Samples used in the tests were obtained from the Lazy hard coal mine near Ostrava in the Czech Republic. The Ostrava-Karviná coal basin, where Lazy coal mine is situated, makes approximately 1/6 of the total surface of the Upper-Silesian basin, the largest part of which is situated in Poland, west of Krakow (Katowice and Rybnik).

Sampling was conducted at the run-of-mine feed to the preparation plant. The chemical analysis of the coal sample is: C = 77.15%; H₂ = 18.32%; N₂ = 0.89%; O₂ = 3.92%; S = 0.05%; moisture W = 8.06%; volatile matter V_{daf} = 7.53% and ash content A = 8.21%. The petrographic analysis established that coal contains 63.2% of vitrinite, 7.6% of liptinite and 29.2% of inertinite and inorganic admixture consisted mainly of carbonates, with occasional clay minerals and pyrite (Bedeckovic et al., 2003). The tests carried out with the aim of quantifying hydrophobicity of individual macerals on a variety of US coal samples showed that for the majority of samples the contact angle for liptinite varied from 90 to 130°, for vitrinite from 60 to 70° and inertinite from 25 to 40° (Honaker et al., 1995).

Sample preparation and experimental procedure

The samples were homogenized and sieved for appropriate size needed for testing. The coal was dispersed in the appropriate quantity of water and a collector necessary for achieving the appropriate concentration was added to the suspension. The agitation time of coal sample with the collector was one minute. Flotalex (containing carboxylic compounds, aromatic, aliphatic, chlorinated and fluorinated hydrocarbons, alcohols, phenols, glycols, arylester of phosphoric acid, paraffines and aromates) was used as a collecting-frothmaking reagent. The collector dosage used in all experiments was equivalent to the consumption of 700 g of collector per 1000 kg of coal. After that the

pulp was gradually introduced in the column, special attention was given to regulate the air flow rate. The flotation products were taken in certain time intervals (1, 3, 5, 10, 15 minutes) with the total flotation time of 15 minutes. The obtained flotation products (concentrates and tailings) were filtered with a vacuum pump on previously weighed filter papers and dried at the temperature of 105 °C. The mass of individual products was determined by weighing. Further analysis included determination of the ash content in products by torrefying at 815 °C to a permanent mass.

Testing equipment

The tests were conducted in a laboratory flotation column (Fig. 1) produced in the laboratory of Faculty of Mining, Geology and Petroleum Engineering, University of Zagreb. The column was made of glass approximately 3.5 mm thick. The pulp was prepared in a conditioning tank (4) with a number of revolutions that can be regulated. After agitation the pulp was transferred to a flotation column through valve (6) and column feed port (5). The concentrate overflowed into a concentric launder (1) from which it was continuously drained (2) by the gravity. A compressor (11) of a capacity of 190 dm³/min with a container of 24 dm³ and pressure of up to 0.8 MPa was used as an air source. The air flow rate was controlled by a valve (10) and measured by a flow meter (9) with measurement scope of 0.05–0.5 dm³ per minute, i.e. 3 to 30 dm³ per hour. Air was introduced into the column through a sparger (8) made of sintered glass with an opening of 10 µm. A total height (H) of the flotation column was 705 mm, reaction zone height (h) was 550 mm, pulp feed height (h_p) was 360 mm, column diameter (D) was 65.6 mm and internal column diameter (d) was 58 mm.

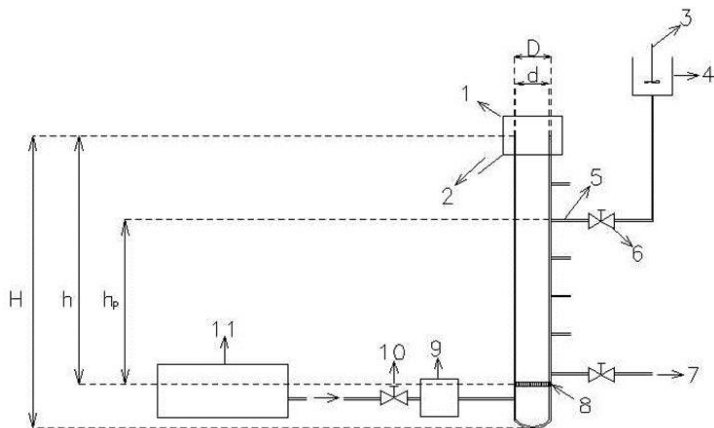


Fig. 1. Scheme of laboratory flotation column

Experimental design

A testing plan for flotation tests was composed in accordance with a central composite design (CCD). The central composite design enabled the collection of data adequate

for the statistical analysis based on which it was possible to make objective and reliable conclusion on the effect of independent on dependent variables of a certain system. The first step was to determine dependent and independent variables (or factors). The changes of independent variables affected the changes of dependent variables. The air flow rate, pulp density and particle size were independent variables (or factors) in the tests. The central composite design with three independent variables consisted of 17 tests divided in two blocks. The first block consisted of 10 tests representing 2-level full factorial experiment upgraded with a central point. The second block contained so-called star points that provided five levels for each factor and an additional central point. The efficiency of flotation separation was evaluated based on the value of two dependent variables, that is the combustible matter recovery and ash content in the concentrate. The combustible matter recovery was calculated by equation (Vapur et al., 2010):

$$CR = 100 \cdot \left[\frac{M_c (100 - A_c)}{M_f (100 - A_f)} \right]$$

where CR is combustible recovery in percentage, M_c is the mass of the concentrate in grams, M_f is the mass of the feed in grams, A_c is the ash content of the concentrate in percentage and A_f is the ash content of feed. The concentrate grade (G) represents the percentage of valuable component (combustible matter) in the concentrate as the final product, and it can be expressed by equation:

$$G = 100 \cdot \frac{m_M}{m_C}$$

where m_M is the mass of combustible matter in the concentrate in grams and m_C is the mass of the concentrate in grams. However, in this paper the concentrate grade is monitored indirectly on the basis of ash content in the concentrate, where higher quality concentrate contains lower ash content and vice versa.

Results and discussion

Table 1 shows the results of conducted tests, such as values of dependent variables (ash content and combustible recovery), as well as conditions under which tests were conducted, i.e. values of independent variables. The air flow rate was changed with the interval from 11.59 to 28.41 dm³/h, pulp density from 66.36 to 133.64 g/dm³. The tests were conducted on different particle sizes: 0.08 (0.063–0.1 mm), 0.15 (0.1–0.2 mm), 0.25 (0.2–0.3 mm), 0.35 (0.3–0.4 mm) and 0.42 mm (0.4–0.45 mm). The obtained concentrates contained from 4.61 to 9.62% of ash with the combustible matter recovery from 17.43 to 81.98%.

Figures 2–7 graphically show the combustible matter recovery and ash content in the concentrate. We can observe the scope of values of independent variables in which

the minimum and maximum values of dependent variables are achieved, that is the highest quality concentrates (the lowest ash content) and combustible recovery.

Table 1. Test conditions and results of flotation tests

Run	Independent variables			Dependant variables	
	Air flow rate dm ³ /h	Pulp density g/dm ³	Particle size mm	Ash content in concentrate %	Combustible matter recovery %
1	15.00	80.00	0.15	5.40	49.03
2	25.00	80.00	0.15	7.23	72.55
3	15.00	120.00	0.15	6.61	17.43
4	25.00	120.00	0.15	6.99	33.29
5	15.00	80.00	0.35	6.46	65.04
6	25.00	80.00	0.35	8.95	70.14
7	15.00	120.00	0.35	5.34	30.82
8	25.00	120.00	0.35	7.43	64.65
9	20.00	100.00	0.25	5.66	33.00
10	20.00	100.00	0.25	6.56	45.93
11	11.59	100.00	0.25	4.61	29.52
12	28.41	100.00	0.25	8.14	68.75
13	20.00	66.36	0.25	7.29	81.98
14	20.00	133.64	0.25	6.52	27.80
15	20.00	100.00	0.08	9.62	35.66
16	20.00	100.00	0.42	5.95	66.33
17	20.00	100.00	0.25	6.95	51.21

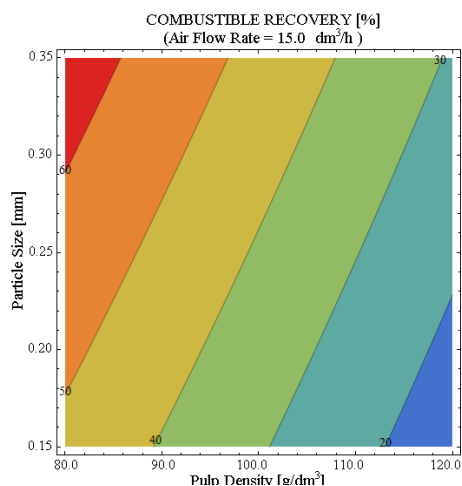
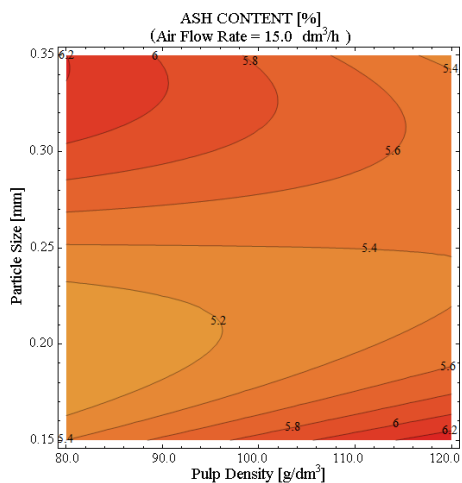


Fig. 2. Conture plot of ash content at air flow rate of 15 dm³/h as a function of particle size and pulp density

Fig. 3. Conture plot of combustible matter recovery at air flow rate of 15 dm³/h as a function of particle size and pulp density

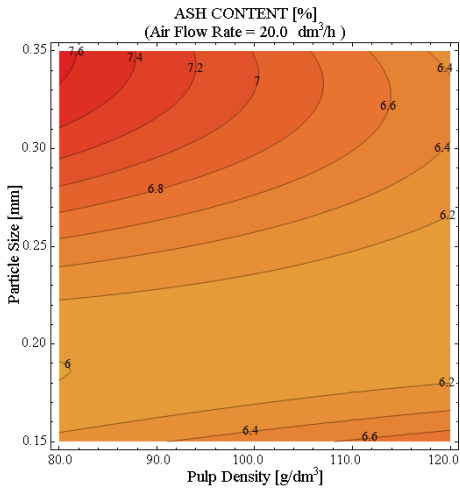


Fig. 4. Conture plot of ash content at air flow rate of 20 dm³/h as a function of particle size and pulp density

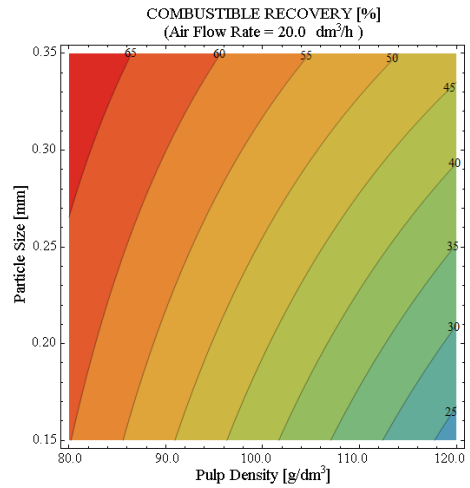


Fig. 5. Conture plot of combustible recovery at air flow rate of 20 dm³/h as a function of particle size and pulp density

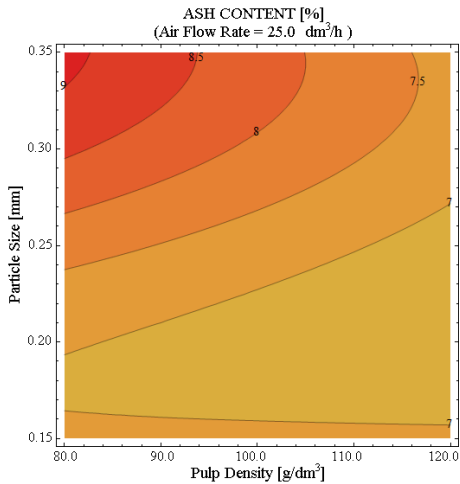


Fig. 6. Conture plot of ash content at air flow rate of 25 dm³/h as a function of particle size and pulp density

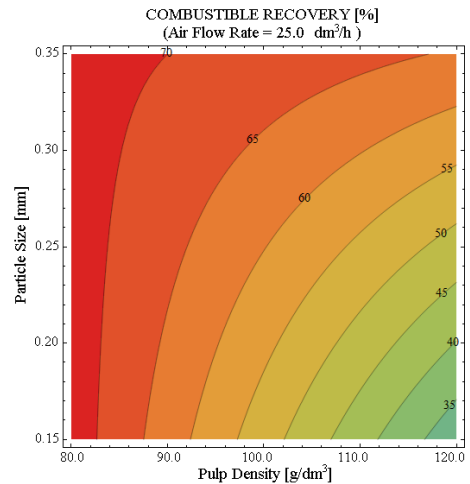


Fig. 7. Conture plot of combustible recovery at air flow rate of 25 dm³/h as a function of particle size and pulp density

Tables 2 and 3 show the results of a statistical analysis carried out by reduced cubic model in SAS JMP software, version 11. The first column shows tested variables (factors), that is their cubic, square, linear effect and linear interactions of variables. The second column shows estimated effect values. The third column shows standard errors in the estimated effect values and the fourth column shows the value of test

statistic in *t*-test (*t* ratio). The level of importance of the conducted test (*p*-level) is presented in the fifth column, while the sixth column presents variable inflation factors (VIF). Since variable inflation factors (VIF) are close to 1, it can be concluded that the assumptions about independent factors in the multiple linear regression model are not violated. The VIFs for particle size and cubic influence of particle size are almost 5, because they are highly correlated. The lack of fit tests for the model proposed in Table 2 is not significant (*F* Ratio = 13.8545 and *p*-value = 0.0011), hence the lack of fit variations is not significantly greater than the pure error variation, which means that the model fits data well.

Table 2. Analysis of variance for ash content in the concentrate

Coefficient of determination $R^2 = 0.946845$; ANOVA <i>F</i> Ratio = 13.8545, <i>p</i> -value = 0.0011					
	Estimate	Std. Err.	<i>t</i> Ratio	Prob. > <i>t</i>	VIF
Intercept	6.3264909	0.193874	32.63	<0.0001	
Air Flow Rate (15, 25)	0.9318931	0.121267	7.48	0.0001	1
Pulp Density (80, 120)	-0.217106	0.121267	-1.79	0.1165	1
Particle Size (0.15, 0.35)	0.9738024	0.265881	3.66	0.0080	4.8071694
Interaction of Air Flow Rate & Pulp Density	-0.23125	0.158443	-1.46	0.1878	1
Interaction of Air Flow Rate & Particle Size	0.29625	0.158443	1.87	0.1037	1
Interaction of Pulp Density & Particle Size	-0.45125	0.158443	-2.85	0.0248	1
Quadratic effect of Pulp Density	0.143175	0.127515	1.12	0.2985	1.0548571
Quadratic effect of Particle Size	0.454302	0.127515	3.56	0.0092	1.0548571
Cubic effect of Particle Size	-0.730052	0.134643	-5.42	0.0010	4.8071694

The model (for coded values) of the dependence of ash content in the concentrate (AC) on the air flow rate (x_1), pulp density (x_2), and particle size (x_3) is:

$$AC = 6.32 + 0.93x_1 - 0.22x_2 + 0.97x_3 - 0.23x_1x_2 + 0.30x_1x_3 - 0.45x_2x_3 + 0.14x_2^2 + 0.45x_3^2 - 0.73x_3^3$$

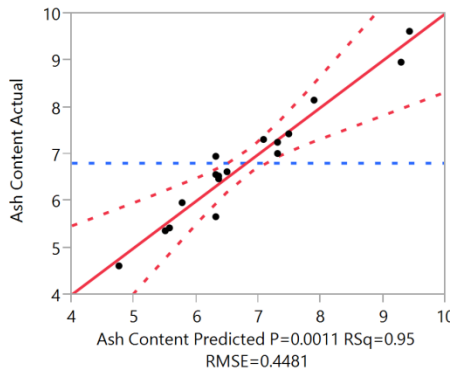


Fig. 8. Comparison of measured and predicted ash content in concentrate

Comparison of the measured values and the values that gives the model for the ash content in the concentrate and combustible recovery are presented in Figs. 8 and 9, respectively.

Table 3. Analysis of variance for combustible recovery

Coefficient of determination $R^2 = 0.935263$, ANOVA F Ratio = 18.5747, p -value = 0.0001					
	Estimate	Std. Err.	t Ratio	Prob > t	VIF
Intercept	49.595882	1.610098	30.80	<.0001	.
Air Flow Rate (15, 25 dm ³ /h)	10.565151	1.796393	5.88	0.0002	1
Pulp Density (80, 120 g/dm ³)	-14.76837	1.796393	-8.22	<.0001	1
Particle Size (0.15, 0.35 mm)	8.0494808	1.796393	4.48	0.0015	1
Interaction of Air Flow Rate & Pulp Density	2.63375	2.3471	1.12	0.2909	1
Interaction of Air Flow Rate & Particle Size	-0.05625	2.3471	-0.02	0.9814	1
Interaction of Pulp Density & Particle Size	3.89375	2.3471	1.66	0.1315	1
Interaction of Air Flow Rate & Pulp Density & Particle Size	4.54875	2.3471	1.94	0.0846	1

The model (for coded values) of the dependence of combustible matter recovery (CR) on the air flow rate (x_1), pulp density (x_2), and particle size (x_3) is:

$$CR = 49.60 + 10.56x_1 - 14.77x_2 + 8.05x_3 - 2.63x_1x_2 - 0.06x_1x_3 + 3.89x_2x_3 + 4.55x_1x_2x_3 .$$

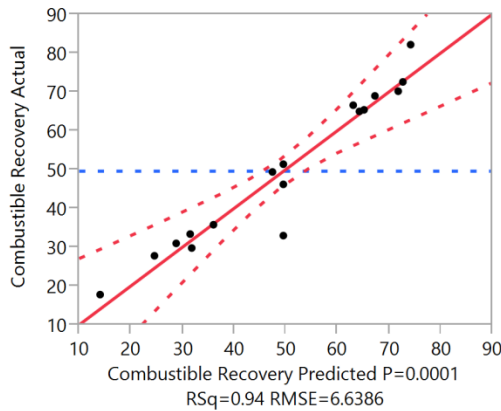


Fig. 9. Comparison of measured and predicted combustible recovery

The pulp aeration depends on the quantity of air introduced into the process and it significantly affects the flotation response. It also plays an important role in the establishment of froth (Tao et al., 2000). On the basis of conducted analyses (Table 2, ANOVA) it can be concluded that the air flow rate has a significant effect on the ash content in the concentrate ($p = 0.0001$) and the combustible matter recovery (Table 3, $p = 0.0002$). According to the models, the increase in the air flow will result in the increase in the ash content in the concentrate and the combustible recovery matter.

Also, the same can be seen by comparing Figs. 2, 4 and 6. At low air flow (15 dm³/h, Fig. 2) the ash content varies approximately from 5 to 6%, at medium air flow (20 dm³/h, Fig. 4) from 6 to 7.6% and at high air flow (25 dm³/h, Fig. 6) varies from 7 to 9%. Similarly, at low air flow (15 dm³/h, Fig. 3) most of the diagram area shows the combustible matter recovery below 50%. Conversely, at high air flow (25 dm³/h, Fig. 7) most of the diagram area shows the combustible matter recovery above 50%. The research of air flow rate effect on column flotation of coal particles (Fujimoto et al., 1999) proved the increase of the concentrate grade at lower gas velocity. The increase of ash content at higher gas velocity was explained by a bubble flow that causes favourable circumstances for hydrophilic particles to travel upwards to the pulp surface. Tao et al. (2000) showed that the increase in the gas flow rate stabilises the froth and improves the combustible matter recovery in the column flotation of fine coal particles. The testing of fine coal by the column flotation confirmed that the air flow rate is the most important and critical parameter (Bayrak et al., 2002). The testing of copper ore floatability in the column (Cilek and Yilmazer, 2003) confirmed the significant effect of the air quantity and flotation time on the recovery.

The particle size plays a very important role in flotation because it affects gas bubble mineralization, stability of a formed aggregate, reagent adsorption and floating kinetics. All these are reasons why the role of particle size in flotation has been widely studied. In this study the particle size showed the significant effect on the ash content in the concentrate (Table 2, $p = 0.008$) as well as quadratic (Table 2, $p=0.092$) and cubic effect (Table 2, $p = 0.001$). Also, the particle size showed the significant effect on the combustible matter recovery (Table 3, $p = 0.0015$). Figures 3, 5 and 7 show that the combustible matter recovery is higher for the coarse particles. Li et al. (2013) also concluded that the combustible matter recovery increased with the particle size. The fine particles have low collision efficiencies due to their low mass and inertial force, while the coarse particles have a higher degree of heterogeneity. Figures 2, 4 and 6 show that the ash content in the concentrate is higher for the coarse particle size. It can be explained that the coarse particles with low ash content are prone to detach from the bubbles (Rahman et al., 2012). The effect of particle size on formation of a bubble-particle aggregate results from the fact that the surface tension is the function of contacts of two phases of different polarity, and the contact surface is the function of diameter of particle and gas bubble. The particle size affects the probability of bubble-particle collision in such a manner that the increase in the particle size diameter increases the probability of collision but simultaneously decreases the probability of adhesion to a gas bubble. Also, the high densities of heavy-mineralized air bubbles may cause difficulties in their rising along the collection zone. Brozek and Mlynarczykowska (2013) also showed the increase in the ash content in the concentrate with decrease in the particle size. They showed that the intensity of entrainment of gangue particles to the concentrate increased with the decrease in the floating particle size as well as with increase in the flotation rate.

The statistical analyses showed that the impact of pulp density on the ash content in the concentrate is not significant (Table 2, $p = 0.1165$). Figures 2, 4 and 6 show that the increase in the pulp density results in decrease of the ash content in the concentrate in the case of coarse particles. The pulp density has the significant impact on the combustible matter recovery (Table 3, $p = 0.0001$). According to the proposed models, the increase in the pulp density will result in the decrease of combustible matter recovery, which is also clearly visible in Figs. 3, 5 and 7. It is caused by the fact that the increase in the pulp density results in the growing number of particles in the pulp, and consequently in the growing resistance when the bubble-particle aggregates travel upwards to the froth. Under such conditions detachment of particles from the bubbles is easier. The flotation experiments in the column on samples of sphalerite ore showed that higher pulp density (20 wt. %) achieved better results than lower density (7.5 wt.%) for Zn in terms of recovery and grade and opposite for Fe (Ucurum and Bayat, 2007). The flotation experiments in mechanical Denver cell on samples of bituminous coal of particle size $-500 \mu\text{m}$ showed that the pulp density influenced both entrainment and recovery of coal and associated gangue (Akdemir and Sonmez, 2003). In these tests, the most selective results and the lowest entrainment were obtained at the lowest solid concentration (5%) due to low amount water that was transferred to the froth.

When interaction of variables is taken into consideration only interaction of the pulp density and particle size (Table 2, $p = 0.0248$) has a significant impact on the ash content in the concentrate. Interactions of air flow rate and particle size is strong ($p = 0.1037$) but insignificant and interaction of air flow rate and pulp density is also insignificant ($p = 0.1878$). In relation to the combustible matter recovery, the impact of interactions of air flow rate and pulp density and particle size is very important ($p = 0.0846$). Interaction of pulp density and particle size is also important ($p = 0.1315$). None of interactions on the combustible matter recovery is not statistically significant ($p < 0.05$).

Conclusion

A study was carried out to determine the impact of air flow rate, pulp density and particle size on the ash content and combustible matter recovery in the concentrate in a laboratory flotation column. The main (linear) effects of air flow rate on the ash content in the concentrate and combustible matter recovery are significant. The results showed that the increase in the air flow rate increased the combustible matter recovery and ash content in the concentrate. The main effect of pulp density on combustible recovery was significant, whereas for the ash content it was not seen ($p=0.1165$). The increase in the pulp density decreased the combustible matter recovery. The effects (linear, quadratic and cubic) of the particle size on the ash content in the concentrate were significant and only the main effect of particle size on the combustible recovery was significant. The increase in the particle size increased the combustible matter recovery. The interaction effect of pulp density and particle size had a significant impact on the ash content in the concentrate, while its effect on the combustible

recovery was strong ($p = 0.1315$) but not significant. The interaction effect of air flow rate and particle size on the ash content in the concentrate was strong ($p = 0.1037$) but insignificant. The interaction effect of air flow rate and pulp density on the ash content in the concentrate was not significant. The triple interaction effect of air flow rate, pulp density and particle size on the combustible matter recovery was statistically very important ($p = 0.0846$).

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