

## REFLOTATION OF COAL SLURRIES FROM OKD,A.S. MINE LAZY

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### ABSTRACT

The goal of the work was verification of selective flotation on the black coal slurries sampled from slurry ponds of Mine Lazy, OKD, a.s. From the results followed that by basic flotation is possible on slurry samples to reach the marketable coal concentrates with ash content under 10% by Flotalex collector application.

Key words: Reflotation, coal slurries, analysis of variance

### INTRODUCTION

There is inhibition in coal mining at present time. Inhibition volume depends on the development of international and domestic conditions. Other factor is fulfilment of international agreement about gradational reducing of sulphur oxides emitted to the atmosphere.

Self inhibition of coal-mining is demanding from the view of coal industry. It is necessary to create financial sources for technical liquidation of collieries, social programmes as well creation of new jobs. In consequence of this inhibition is necessary to fully use mining capacity of functional collieries. General trend to use most fine coal fraction rising during black coal treatment is evoked by either effort to increase concentrate quality requested by customers or want of fine incorporated minerals processing.

Greatest losses are in sludge system. Just here is important flotation, that is supplements of other mineral processing methods and at once is most important node of own processing of coking coal (flotation of fine fractions). Today old waste dumps can be used as raw material by perfection of flotation selectivity. These dumps could not be used previously. For coal is flotation used for separation of very fine fraction that would float into tail. Coal for energetical purposes from old slurry ponds can be also recovered by the flotation.

The goal of the work was verification of black coal slurry samples reflation from locality OKD, a.s., Mine Lazy. The objective is achievement of saleable coal concentrates with ash content under 10%.

#### Chemical-technological analyses

Chemical-technological analyses were undertaken in the laboratory of Institute of Environmental Engineering on VŠB-Technical University Ostrava.

#### Water content

Determination was performed according Standard CSN 441377. Analytical sample was dried at 105°C during 30 minutes. Content of analytical water was 1% in the sample from Mine Lazy.

#### Ash content

Determination was performed according Standard CSN ISO 441378. Analytical sample was ignited in beforehand annealed crucible at 815°C during 90 minutes. Ash content was 21.2% and after recounting to water-free state of combustible was ash content 21.4%.

#### Content of volatile combustible matter

Determination was performed according Standart CSN 441351. Analytical sample was heated up in beforehand annealed crucible at 815°C during 7 minutes. Content of volatile combustible matter was 24.3% and after recounting to Vdaf is its content 31.29%.

#### Determination of swelling index

Determination was performed according Standart CSN 441373. Analytical sample was heated up at 820°C during 150 seconds. Its value was 1.

Mineralogical- petrographical analyses of samples

### Methodology

Petrographical analysis was undertaken on all samples in the laboratory of Institute of geological engineering, VSB-TU Ostrava. Sample was grinded to the granularity smaller than 1mm, and from this sample were prepared grain polished sections according Standard CSN 441343 and 441344.

Light reflectance  $R_0$  and maceral composition were measured on the microscope UMPS 30 Petro by Opton – Zeiss Jena at monochromatic light with wave length  $\lambda = 546 \text{ nm}$ , where was used immersion objective with magnification 25 times and immersion with refractive index  $n = 1,518$ .

### Petrographical analysis of sample

Vitrinite maceral group was represented by telinite and collinite. Cellular spaces of telinite were filled by fine-grained micrinite. Vitrinite formed individual grains (Fig.1) or was part of particular microlithotypes. Percentage representation of vitrinite group was 61.2%.

Maceral group of liptinite was created mainly with microsporinite, that was sporadically agglutinate, with less macrosporinite. The resinite was also observed. Macerals of this group were distinct and well distinguishable. Percentage representation of macerals of this group was 11%. Cutinite was represented sporadically. Maceral group of inertinite was substituted mainly with fine-grained micrinite, less with macrinite and fusinite. Macerals of this group occurred either apart or were part of individual microlithotypes. Percentage representation of this group was 27.8%. Durite, clarite, vitrinertite, trimacerite were mainly substituted from the microlithotypes. Liptite occurred very rarely. Inorganic admixture was created with carbonates, sporadically with clay minerals – carbargilite and pyrite. Pyrite occurred either massive (Fig.2) or framboidal form or filled rarely fusinite cellular spaces.

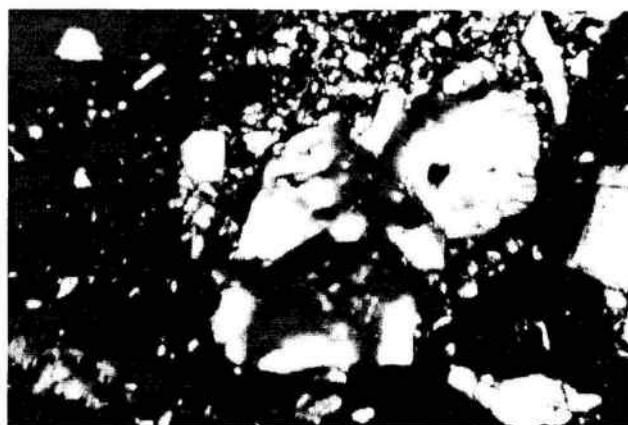


Fig.1. Vitrinite



Fig.2. Massive pyrite

### Particle size analysis

Wet particle-size analysis of coal slurry samples was performed on the screens of size 0.02, 0.063, 0.1, 0.5, 1.0 mm in the laboratory of Environmental engineering, VSB-TU Ostrava. All size fractions were filtered, dried, weighted and ash content was determined.

From the grain size analyses (Table I) followed that tested sample of black coal slurries is substantially fine-grained. Mass yield in size fraction under 0.02mm creates even cca 46% at high ash content cca 43%.

**Table I.** Particle-size analysis and ash content of coal slurry from Mine Lazy

Grain size (mm)	Yield (%)	Ash content (%)
-0,02	46,13	42,75
0,02-0,063	14,48	14,74
0,063-0,1	7,00	9,28
0,1-0,5	24,28	8,20
0,5-1,0	5,20	16,80
+1,0	2,92	53,35
Total	100,00	26,93

### Reflotation tests

Furthermore, flotation tests of coal slurries were undertaken. The characteristics and testing of individual collectors used for coal flotation followed namely by application of statistical method -analysis of variance.

### Characteristics of used flotation collectors

For selectivity testing of flotation collectors were used these collectors: Flotakol NX, Montanol, Ekofol 440, MP 125, Flotalex a  $AF_2$ . Because of chemical composition of these collectors is not mentioned by producers, collectors were analysed with IR spectroscopy in CAL VSB-TU Ostrava. From the results of these analyses follow that tested collectors have same functional groups and are greatly itself similar.

**Flotakol NX** - is currently used flotation collector of czech production. From IR analysis follows next composition: carboxylic compounds, aromatic, aliphatic, chlorinated and fluorinated hydrocarbons, alcohols, phenols, glycols, arylester of phosphoric acid, paraffines and aromates.

**Flotalex** – is currently used flotation collector also of czech production. Chemical content of this collector is very similar as collector Flotakol NX.

**Ekofol 440** - from IR analysis follows next composition: carboxylic compounds, aromatic and aliphatic hydrocarbons, alkenes, alcohols, phenols, glycols, esters, ethers and alkanes.

**Montanol** - from IR spectrum ensues similar composition as at Ekofol 440.

**MP 125** – from IR spectrum results this composition: carboxylic compounds, aromatic, aliphatic and fluorinated hydrocarbons, alcohols, alkenes, phenols, glycols, esters, ethers, aromates.

**$AF_2$**  - from IR spectrum follows next composition: carboxylic compounds, aromatic, aliphatic and fluorinated hydrocarbons, alcohols, alkenes, phenols, glycols, alkanes and paraffines.

### Flotation collectors selectivity test

Flotation achievement largely depends on the type and selective effect of selected flotation collector. That is why further work related to observing selectivity effect of tested collector into flotation results as well to the searching of its suitable dosage. Flotation tests were undertaken and evaluated by application of statistical method - analysis of variance. Flotation tests were performed in laboratory of Environmental engineering on the laboratory flotation apparatus VRF-1, product of RD Příbram thereunder:

Thickening: 150 g/l

Agitation period of sludge with collector: 1 minute

Collector dosage : 300, 500 a 700 g/t

Flotation time : 5 minutes

Basic flotation was applied, flotation concentrate and tails were filtered on pressure filter, dried at 105°C, weighted and ash content was determined.

### Testing of used collectors by means of mathematical-statistical method – analysis of variance

Method of analysis of variance results from two possibilities of data processing and hence two manners of evaluation are allowed:

1.Data of first type are get from trial with one repetition.

2.Data of second type are get by double or multiple repetition of trial.

Analysis of variance allows following of two factors effect on flotation optimalization. Individual levels of first factor represented tested collectors. Three levels of second factor formed collector dosages (300, 500 a 700 g.t<sup>-1</sup>).

**Results of flotation test**

The goal of the flotation experiments was selectivity verification of individual collectors as well verification of collector dosage effect on the flotation results. By the analysis of variance method were tested only yield and quality (ash content) of flotation concentrates. The results of analysis of variance for ash content in flotation concentrates are given in the Table II and for concentrate yield in the Table IV.

Collector dosage in all cases has not statistical significant influence on ash content in flotation concentrates, while effect of individual collectors is statistic significant. The most selective collectors are Ekofol, Flotakol NX, AF2 and Flotalex, where required quality of concentrate under 10 % was reached in all cases. From the view of yield is statistically significant dosage effect, where are high differences in the flotation concentrate yields and also is statistic significant effect of particular collectors. From the viewpoint of practice is recommended to use for operational experiments collector of czech production Flotalex, so its price is more favourable as price of imported collectors and its suitable dosage is 700g/t.

From mentioned results followed that slurries from slurry pond Lazy are very suitable for refloitation, because by one basic flotation is possible to obtain required quality of concentrates under 10% of content ash.

**Table II - Results of analysis of variance for ash content**

LAZY - ash							
	agent	dosage			$Y_i$	$Y^2_i$	$\bar{Y}$
		300 g.t <sup>-1</sup>	500 g.t <sup>-1</sup>	700 g.t <sup>-1</sup>			
t <sub>1</sub>	MP 125	11,60	12,09	12,91	36,60	1339,56	12,20
t <sub>2</sub>	Flotakol	9,15	9,77	9,57	28,49	811,6801	9,50
t <sub>3</sub>	Montanol	8,46	19,14	12,06	30,66	940,0356	10,22
t <sub>4</sub>	Flotalex	8,54	9,73	9,22	27,49	755,7001	6,50
t <sub>5</sub>	Ekofol	8,12	8,39	9,36	25,87	669,2569	8,62
t <sub>6</sub>	AF <sub>2</sub>	8,44	9,70	8,99	27,13	736,0369	9,04
$Y_i$		54,31	59,82	62,11			
$Y^2_i$		2949,576	3578,432	3857,652			
$\bar{Y}$		9,05	9,97	10,35			

**Table III - Results of analysis of variance**

source of changes (effect)	S	degrees of freedom f	variance	values of F - criterion	
				calculated	tabular $\alpha = 0,05$ $\alpha = 0,01$
factor A	$S_A$	$f_A = a - 1 = 2$	$S^2_A = \frac{S_A}{a-1} = \frac{2517}{2} = 12858$	$\frac{S^2_A}{S^2_z} = \frac{12858}{0,457} = 2754$	4,1
factor B	$S_B$	$f_B = b - 1 = 5$	$S^2_B = \frac{S_B}{b-1} = \frac{5,36}{5} = 1,072$	$\frac{S^2_B}{S^2_z} = \frac{1,072}{0,457} = 2,346$	3,33
total	$S_z$ $S_0$	$f_z = (a-1)(b-1) = 10$ $f_0 = N-1$	$S^2_z = \frac{S_z}{(a-1)(b-1)} = \frac{4,57}{10} = 0,457$		

**Verification t - test, LAZY Ash**

Lines:

$$t_6 - t_1 = 1,803$$

$$t_6 - t_2 = 0,26$$

$$t_6 - t_3 = 0,67$$

$$t_6 - t_4 = 1,45$$

$$t_6 - t_5 = 0,24$$

$$t_5 - t_1 = 2,04$$

$$t_5 - t_2 = 0,51$$

$$t_5 - t_3 = 0,91$$

$$t_5 - t_4 = 1,21$$

$$t_{tab} = 2,2281$$

- $t_4 - t_3 = 2,12$
- $t_4 - t_2 = 1,71$
- $t_4 - t_1 = 3,26$
- $t_3 - t_2 = 0,411$
- $t_3 - t_1 = 1,13$
- $t_2 - t_1 = 1,54$

Columns:

- $t_2 - t_1 = 0,75$  2,2281
- $t_3 - t_1 = 1,06$
- $t_3 - t_2 = 0,31$

**Table IV** - Results of analysis of variance for mass yield

LAZY - yield

	agent	dosage			$Y_i$	$Y_i^2$	$\bar{Y}$
		300 g.t <sup>-1</sup>	500 g.t <sup>-1</sup>	700 g.t <sup>-1</sup>			
$t_1$	MP 125	53,05	81,77	87,91	222,73	49608,85	74,24
$t_2$	Flotakol	21,70	41,97	56,94	120,61	14546,77	40,2
$t_3$	Montanol	68,91	79,49	85,33	233,73	54629,71	79,91
$t_4$	Flotalex	10,22	37,88	79,21	127,31	16207,84	42,44
$t_5$	Ekofol	56,71	78,57	79,56	214,84	46156,23	71,61
$t_6$	AF <sub>2</sub>	76,09	80,58	77,68	234,35	54919,92	78,12
$Y_i$		47,78	66,71	77,77			
$Y_i^2$		286,88	400,26	466,63			
$Y$		82185,42	160208,1	217743,6			

$$S_A = \frac{\sum Y_i^2}{3} - \frac{(Y_{...})^2}{N} = \frac{23606,92}{3} - \frac{133072,8}{18} = 4760,61$$

$$S_B = \frac{\sum Y_i^2}{6} - \frac{(Y_{...})^2}{N} = 7668,509 - 7392,9097 = 2760,41$$

$$S_0 = \sum Y_{ijk}^2 - \frac{(Y_{...})^2}{N} = 8290,432 - 7392,9097 = 897,523$$

$$S_{AB} = 145,131$$

**Table V** – Results of analysis of variance

source of changes (effect)	S	degrees of freedom f	variance	values of F – criterion	
				calculated	tabular $\alpha = 0,05$ $\alpha = 0,01$
factor A	$S_A$	$f_A = a - 1 = 2$	$\frac{4760,61}{2} = 2380,305$	16,4	4,1
factor B	$S_B$	$f_B = b - 1 = 5$	$\frac{2760,41}{5} = 552,082$	3,8	3,33
total	$S_Z$ $S_0$	$f_Z = (a - 1)(b - 1) = 10$ $f_0 = N - 1$	$\frac{1451,31}{10} = 145,131$		

**Verification t – test, LAZY Yields**

Lines:

$t_6 - t_1 = 0,12$

$t_{tab} = 2,2281$

$t_6 - t_2 = 1,22$

$t_6 - t_3 = 0,06$

$t_6 - t_4 = 1,15$

$t_6 - t_5 = 0,21$

$t_5 - t_1 = 0,08$

$t_5 - t_2 = 1,01$

$t_5 - t_3 = 0,27$

$t_5 - t_4 = 0,94$

$t_4 - t_3 = 1,20$

$t_4 - t_2 = 0,07$

$t_4 - t_1 = 1,02$

$t_3 - t_2 = 1,18$

$t_3 - t_1 = 0,18$

$t_2 - t_1 = 1,09$

Columns:

$t_2 - t_1 = 0,75$

$t_3 - t_1 = 1,06$

$t_3 - t_2 = 0,31$

Lines:  $S_z \cdot 0,8165 = 38,1$  ,  $0,8165 = 31,1$ Columns:  $S_z \cdot 0,577 = 21,98$ **CONCLUSION**

The goal of the work was selective flotation verification on the samples of black coal slurries from slurry ponds of Mine Lazy, OKD, a.s. From the results followed that by the basic flotation is possible reach at the samples by Flotalex collector application selectable coal concentrates with ash content under 10%.

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