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FLOTATION OF FINE COKE PARTICLES FROM FLY ASH

Fine coke particles were separated from fly ash in a laboratory flotation cell. Almost 90% of particles by number were below 10 μm and one third of them was less than 2 μm. Fly ash was sampled directly from a surge bin in a power plant and from a refuse dump area. Kerosene and Diesel oil were compared as collectors, and MIBC and polyglycol – based agents – were used as frothers. The best separation coefficient of 0.8 was obtained for 18 kg/t of Diesel oil and 9 kg/t of commercial frother based on polyglycol.

LITERATURE REVIEW

Fly ash from hard coal combustion can contain 20% or even more combustible residue, usually in the form of coke. This coke with low sulphur content may be considered as valuable ecological fuel. On the other hand, commercial application of fly ash as a component in construction materials requires limited coke content. Up until recently fly ash from Gdañsk power plants was deposited at dump sites which harmed the environment. Because several million tons of this waste material have been kept in wet condition, foam flotation was selected for coke removal from the fly ash.

Literature review pertaining to fine coal and coke flotation is presented in Table 1. Besides of fly ash, fine coal flotation papers are also included because of many similarities in both flotation systems. Values in bold were calculated or estimated from the data provided by authors of the papers. First seven experimental systems, presented in Table 1, pertain to coal flotation. The coefficient of separation (CS), representing process efficiency, in the range of 31%–73% indicates that fine coal particles do not float easily. It is worth to mention that comparable process efficiency was obtained for the ASH and other flotation equipment, but the flotation time for the ASH was many times shorter. Inferior flotation results can also be attributed to the excess of collector causing increased froth viscosity and greater ash particles entrainment to the froth (Clarkson et al. 1994). Although processing results given by Babatchew are very impressive (raw concentrate grade 75–80%) no processing details were provided (Babatchew 1987).

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Table 1. Conditions and results of fine coal and coke flotation

Flotation machine	Flotation time [min]	Feed	Particle size [μm]	Collector (type, dosage) [kg/t]	Frother (type, dosage) [kg/t]	Combustible substances in feed [%]	Yield [%]	Concentrate grade [%]	Recovery [%]	CS [%]	References
2" dia. Air-sparged hydrocyclone	< 1/30	Upper Freeport Coal from Helen mine, Pennsylvania, USA	>150 (0.8%)150–38 (48.8%) <38 (50.4%)	kerosene 1.0	MIBC0.5	78.5	74.5	93.9	89.1	68.9	Gopalakrishnan et al. 1991
5 cm dia. Microcel™ column	**1**	Fine coal, USA	<150 (80%)	Diesel oil 0.35	n/a 0.35	42.4	35.5	87.5	68.7	**63.6**	Gebhardt et al. 1994
	1.5	Tailings from a refuse pond Cyclone overflow, USA	<150 (100%)	n/a 0.5	n/a 0.2–0.4	54.5	50.7	90.2	84.0	**73.2**	
	6	Micronized coal, USA	<75 (80%)	n/a 0.5	n/a 0.75	90.4	91.5	96.1	97.3	**60.5**	
Jameson cell	n/a	Coal sludge, Australia	0–45 (54%)	Diesel oil<1.0	MIBC20–60g/m^3	56.8	67.0	88.2	90.6	**52.7**	Clarkson et al. 1994
2.8 m^3 Denver type	n/a	Coal sludge, Australia	0–45 (58%)	n/a	n/a	46.5	32.5	87.2	59.3	**49.3**	
1 dm^3 laboratory cell	4	Polish steam coal, rank 31.1	150–60 (38.5%)<60 (53.5%)	Diesel oil 5.0	PG600 1.5	**~73.0**	**~75.7**	**~84**	**~81**	**~30.8**	Saleh, Iskra 1996
n/a	3	Fly ash from refuse dump area, Slovakia	<40 (73%)	Flotacol NX 9.0–11.0		~26.9	37.0	**97.9**	**71.1**	**83.2**	Michalikova et al. 1996
5 dm^3 laboratory cell	5	Fly ash, German power plant I	n/a	Montanol 551 1.6		26.2	**30.1**	77.6	**89.1**	**79.9**	Grünewald, Ottersteter 1990
		Fly ash, German power plant II	n/a			14.5	**24.3**	48.4	**81.2**	**66.5**	
		Fly ash, German power plant III	n/a			5.8	**14.7**	31.0	**78.2**	**67.4**	
n/a	n/a	Ash from two power plants,Bulgaria	200–63 (25.3%)<63 (74.7%)	n/a	n/a	30	**35.0**	80	**93.3**	**83.3**	Babatchew 1987
Jet flotation unit	n/a	Waelz furnace slug, Turkey	<500 (100%)	kerosene 1.7	isooctanol 0.3	16.5	20.8	70.2	88.1	**72.2**	Atesok et al. 1994

n/a – not available; values in bold were calculated or estimated on the basis of other data given by authors.

Regarding fly ash from Slovakia, very good results were obtained (CS > 80%), but quite significant amount of chemicals was required and the combustible content in the feed was about 30% (Michalikova et al. 1996). The fly ash from Slovakia was sampled from a dump site, where it was kept in wet condition. Results reported by Grünewald and Ottersteter (1990) can be distinguished by a low consumption of flotation agents, however, polyacryloamide flocculants were used.

The objective of the present paper was to examine the flotation response for coke particles in wet fly ash, originating from Gdañsk power plants, keeping in mind the potential utilization of the coke concentrate and the cleaned ash.

EXPERIMENTAL MATERIALS AND METHODS

The fly ash used in the tests was sampled from a dump site in Gdansk and from a storage bin in the ECII power plant in Gdansk. Ash from the dump site had 27% moisture and 14% combustible residue (on dry basis). Ash from the storage bin had 0.24% moisture and 10–18% combustible residue. The size distribution of all particles in the fly ash was determined using Sartorius sedimentation balance, see Table 2. The size distribution of coal particles was determined under optical microscope, see Figure 1. About one third of coke particles was smaller than 2 μm. The fly ash stored at the dump site was submitted to weathering for several years.

Table 2. Selected properties of fly ash used in experiments

Sample Origin	Moisture content [wt. %]	Combustible residue [wt. %]	Particle size distribution (size μm – w. %)		pH of aqueous suspension
EC II power plant	0.24	10 - 18	+ 60 – 60 +10 –10	38.8 32.9 28.3	8.5
Refuse dump area	27.0	14	+ 60 – 60 +10 –10	39.1 38.3 22.6	7.8

The following agents were used as frothers: methyl isobutylocarbinol (MIBC, 4-methyl-2-pentanol) and commercial F507, F571 and F579 frothers based on polyglycols. Emulsified Diesel oil and kerosene served as collectors.

The experiments were carried out in one-litre Denver type flotation cell for 10 minutes. The solids content in the slurry amounted 3, 5 and 10% by weight. The natural pH of ash in tap water varied from 7.8 to 8.5 depending on solids concentration. Aeration was maintained at 0.5, 1.7 and 3.3 dm^3/min. Depending on a particular run the froth was skimmed from the flotation cell from 6 times per minute to

one time per minute. The concentrates were filtered, dried at 105 °C and weighed. Combustible substances were determined by roasting in an oven at 815 °C.

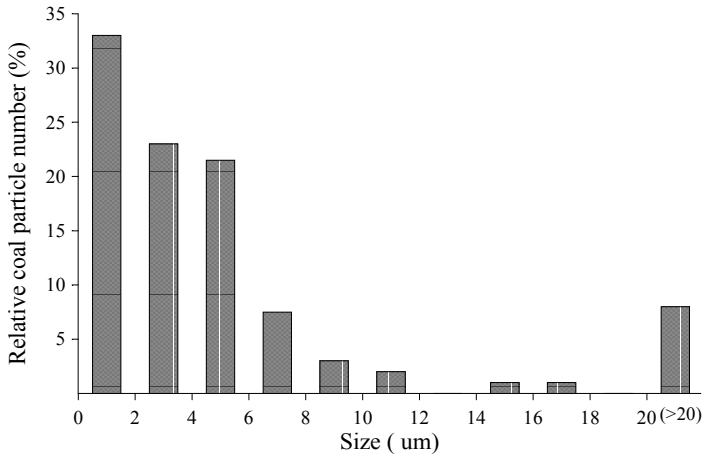

Fig. 1. Size distribution of coal particles in fly ash

Regarding terminology used, *the yield* is defined as the overall solids which reported to the concentrate, *the concentrate grade* indicates the mass fraction of coal in the dry concentrate; *the recovery* pertains to the fraction of coal which reported to the concentrate; *the coefficient of separation* (CS*)* is defined as the difference between the coke recovery and the ash recovery to the concentrate calculated for the same flotation time. The flotation agent dosage is given in kilograms per tone of dry fly ash.

RESULTS AND DISCUSSION

The flotation efficiency for Diesel oil and kerosene as collectors is presented in Table 3. Diesel oil provided higher recovery with higher combustible content in the concentrate. Diesel oil may better adsorb on coke surface than kerosene due to its higher aromaticity and greater content of heteroatoms.

The influence of kind of a frother on the flotation efficiency is given in Table 4. The best results were obtained for the F507 frother. A significant difference in yield can be noticed as frothers were changed. The F507 frother most effectively hold coke particles in the froth thus providing the best coke separation from the suspension. Much smaller difference can be noticed regarding the concentrate grades which fluctuate around 35%. The experiments, for which data is presented in Tables 3 and 4, were made with frequent froth skimming. The yield and the recovery were high, but the entrainment of mineral particles into the froth phase resulted in low separation coefficients.

Table 3. Flotation efficiency for kerosene and Diesel oil as collectors*

Collector	Yield [%]	Concentrate grade [%]	Recovery [%]	Coefficient of separation [%]
Kerosene	24.2	24.9	50.0	29.4
Diesel oil	24.0	30.1	59.5	40.4

Feed: dry fly ash, 12% combustible content, 10% solids in the slurry, 3.3 dm^3/min aeration, 2 kg/t collector, 0.6 kg/t frother (MIBC), 3 minutes flotation time.

Table 4. Influence of frother type on flotation efficiency*

Frother	Yield [%]	Concentrate grade [%]	Recovery [%]	Coefficient of separation [%]
MIBC	14.7	34.4	35.9	24.7
F507	23.1	35.0	57.4	40.0
F571	19.0	36.0	48.5	34.3
F579	23.0	33.8	55.2	37.4

Feed: wet fly ash, 14% combustible content, 3% solids in the slurry, 1.7 dm^3/min aeration, 2 kg/t collector (Diesel oil), 1.0 kg/t frother, 5 minutes flotation time.

The data presented in Table 5 reveals that the dry fly ash floats better than the wet ash at the same level of chemicals added. However, our experimental program focused on the wet ash which is more amenable for wet separation method. For the dry ash rather dry techniques are recommended for processing. Regarding the wet ash, more intense aeration gives larger separation coefficients. An explanation for the less favourable performance of the wet ash may be the porous structure of coke particles. Unlike coke particles present in the dry ash, porous coke particles from the wet ash are saturated with water, as the effect of several years of conditioning in an alkaline environment in the stockpile. Impressive increase of the separation efficiency with the increase of chemicals consumption for the wet fly ash is evident. Microscopic observations of the flotation concentrate revealed that coke particles underwent agglomeration. The size of these agglomerates was on average 30 μm, sometimes even up to 100 μm, without collector addition. After application of Diesel oil emulsion the agglomerates reached 300 μm in size and obviously contained larger number of particles. Flotation without collector of dry fly ash was ineffective similar to the flotation of the wet fly ash. It leads to the conclusion that coke particles aggregation controlled the process efficiency in the laboratory flotation cell.

Table 5. Flotation efficiency for dry and wet ash sample at different collector dosage and aeration*

Aeration [dm^3/min]	Collector dosage [kg/t]	Frother	Frother dosage [kg/t]	Yield [%]	Concentrate grade [%]	Recovery[%]	Coefficient of separation [%]	Residual coke in tailings [wt. %]
Wet fly ash								
5	2	MIBC	1	2.3	54.8	9.2	7.9	13.0
	6	F507	3	9.2	52.5	34.5	29.5	10.1
	12	MIBC	6	11.7	52.6	44.0	37.5	8.9
1.7	2	MIBC	1	5.9	43.8	18.5	14.7	12.1
	6	F507	3	12.3	48.9	42.9	35.6	9.1
	12	F507	6	17.4	48.6	60.5	50.1	6.7
	18	F507	9	22.5	56.6	90.6	79.6	1.7
Dry fly ash								
1.7	0	MIBC	1	9.9	24.6	13.5	4.4	17.3
	2	MIBC	1	20.4	50.4	57.1	44.9	9.7

*Combustible content: 14% wet ash, 18% dry ash, 5% solids in the slurry, collector: Diesel oil, 10 minutes flotation time.

Figure 2 illustrates the kinetics of wet fly ash flotation for different agents dosage and for aeration 1.7 dm^3/min. After 5 minutes of flotation 85% of recoverable coke was obtained for 18 kg/t of collector.

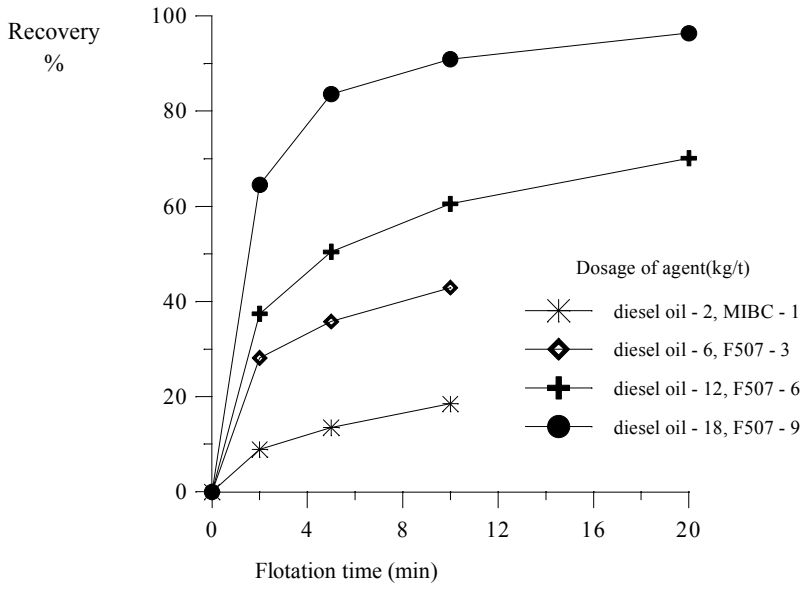

Fig. 2. Flotation kinetics of wet fly ash, aeration 1.7 dm^3/min

CONCLUSIONS

- Fresh, dry fly ash originating from a storage bin floats better than wet fly ash sampled from a dump site. However, only the wet ash was submitted to extensive investigation due to its possible processing using aqueous techniques.
- In the best case fine coke particles from the wet ash were concentrated 4 times (from 14% to 56.6%), and the recovery reached 90%. On the other hand, the coke content in tailings was reduced below 6%, which enables the fly ash addition in various industrial applications.
- The consumption of chemicals was high (18 kg/t of collector and 9 kg/t of frother), but our most recent results, which will be published later, indicate that better control of surface chemistry of the system should improve this parameter. Very fine ash particles also influence the excessive consumption of chemicals.
- The expected flotation time, during which 85% of combustible residue is recovered, should not exceed 5 minutes.

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Niewiadomski M., Hupka J., Bokotko R., Miller J.D., Flotacja koksiku z popio³ów lotnych. *Fizykochemiczne Problemy Mineralurgii*, 31, 221–228 (w jêz. angielskim)

Przeprowadzono separację koksiku z popiołów lotnych w laboratoryjnej celi flotacyjnej. Niemal 90% cząstek koksiku posiadało wymiary poniżej 10 μm, jedna trzecia zaś poniżej 2 μm. Próbki popiołu

lotnego pobrano ze zbiornika buforowego elektrociepłowni oraz z wysypiska popiołów. Jako zbieracze zastosowano naftę i olej napędowy, a jako spieniacze MIBC i odczynniki handlowe zawierające poliglikole. Najwyższy współczynnik separacji, wynoszący 80%, otrzymano dla 18 kg/t oleju napędowego
i 9 kg/t komercyjnego spieniacza opartego na poliglikolach.