# Preparation of low-ash products from Slovak sub-bituminous coals – a material balance

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The fines of sub-bituminous coals from the Cígel and the Handlová Collieries in use as steam coal in coal-fired power plant were subjected to washing in a water-only cyclone (WOC) with the aim to obtain suitable input material for organic compounds extraction. The WOC with a diameter of 150 mm and a cone part consisting of three angle sections  $135^{\circ}-75^{\circ}-20^{\circ}$  was applied. Vortex finder (overflow) and spigot (underflow) diameters were of 68 mm and 14.6 mm, respectively. Two basic products overflow and underflow were obtained. The third one, slurry or circulating charge was also considered into total material balance of washing. Thus, the contents of ash, combustible matter, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, MgO, Fe<sub>TOTAL</sub>, S<sub>TOTAL</sub>, S<sub>SULPHIDIC</sub> and As were determined in the products of washing. Subsequently, on the basis of analyses the recoveries of individual components into products of washing were calculated. The washing resulted in the obtaining of significantly deashed coal at the overflow of WOC. In such way a washed coal with ash content in dry basis of 6.99 % at a mass yield of 20.74 % was won in the case of coal from the Cígel Colliery. Similarly, in the case of coal from the Handlová Colliery and its eastern field the products with ash content in dry basis of 7.70 % and 9.01 % at mass yield of 29.37 % and 29.50 % were obtained. Finally, the washing resulted in over 90 % ash rejection and about 70 % total sulphur rejection.

#### Introduction

Low rank coals, namely lignite and sub-bituminous coal are the only significant domestic energy source in Slovakia (Hredzák, 2010). In 2009 a total production of raw coal attained 2,327,852 ton (SMA, 2010). So, domestic production of low-rank coals covers 70 % of Slovak consumption (Baláž, 2009). Studied coal samples come from the Cígel' and Handlová Collieries, which are divisions of the Hornonitrianské Bane Prievidza, Inc. (abbrev. HBP – Upper Nitra Coal Mines Prievidza – Central Slovakia). The HPB, Inc. is the biggest producer of brown coal in Slovakia. An detailed description of its deposits geology was performed by Machajová et al. (2000, 2002) and Fazekaš (2009). The production of the company achieved 2,031,650 ton in 2009 (SMA, 2010), what is an 87.28 % of total raw coal production in Slovakia. Recently, only a 3 % of the HBP total production was delivered to retail consumers as washed coal (HBP, 2010). The washing of coarse coal is performed using the Drewboy dense medium separator.

It is well known that lignitic and sub-bituminous coals often contain interesting substances such as humine acids, polycyclic aromatic hydrocarbons (naphtalene and derivates, fluorene, phenantrene and its methyl derivates, fluorantene, benzoantracene, chrysene), alkyl derivates with lower content of aromatic terpenoidic biomarkers, heterocyclic compounds of nitrogen, fullerens etc. An extraction of these substances from coal requires deashed feed material. Thus, a water only cyclone (WOC) was applied for its preparation. The samples of coal fines from Cígel' and Handlová Collieries were washed.

### **Selected WOC applications**

The WOCs were developed in the early 1959s in the Central Testing Station of Dutch State of Mines at Treebeck. The WOC creates itself own heavy medium by accumulation of grains in its cone part, thereby eliminating the usage of external medium as required in heavy media separation process. Generally, three types of WOCs are known, namely the DSM cyclone with constant cone angle about 80°, the Var-a-Wall cyclone, the cone of which consists of two distinct angles in two stages, and finally the Compound cyclone. The cone of last named cyclone consists of three different cone angles. The angle in the first stage is wide ranging from 80° to 140°, subsequently dropping to 60° and 30° in the second and the third stage, respectively. The WOCs are suitable for coal preparation as well as for beneficiation of Pb-Zn, Sn and Au (Suresh et al., 1990). As to coal preparation the WOCs are applied in washing of fines, namely a grain size of 0,25-2 mm, middlings and also in scavenging stage to prevent the combustible matter losses in tailings. Thus, several examples of research results and applications of WOCs may be introduced.

So, for instance 62 WOCs of 35.56 mm diameter are being operated in the washery of the Balmer Mine in British Columbia (Canada) with the production of 6 million tons per year. The WOCs process 20 % of feed to washery with a grain size of 0.25-0.6 mm. Ash content in washed coal for metallurgy attains 8.9 % (Kennedy, 1991).

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The preparation of west Canadian coal in WOC is also reported by Bustin (1982). Thus, from the feed with a grain size of 0.15-0.6 mm and ash content of 18.7 % the clean coal with 8.3 % of ash at a mass yield of 69 % was received.

An influence of WOC cone angle on yield and quality of clean coal was studied by Suresh et al. (1996). The cones with angles 80°, 100°, 120° and 136° were tested. An enlargement of cone angle resulted in a mass yield increase, but also in higher ash content in washed coal obtained in overflow of WOC. A mass yield of clean coal and ash content gradually increased from 14.2 % to 69.6 % and from 13.3 % to 25 %, respectively.

The washing of run-of-mine (ROM) coal in WOC ground to grain size under 0.2 mm is referred by Rubiera et al. (1997). Thus, washed coal with 26.5 % of ash and 2.17 % of sulphur at a mass yield of 83.6 % from the feed with 31.8 % of ash and 3.02 % of sulphur was obtained. The 59.1 % of ash and 7.36 % of sulphur were assayed in reject.

Majumder and Barnwal (2008) have reported about fines preparation in Indian coking coal washeries. They have compared results of preparation obtained using various gravity concentrators. So, as to washing of higherash coals in WOC the clean coals with ash content of 21.56% and 15.84% at mass yield of 50.75%and 65.11% from the feeds with 36.61% and 30.50% of ash were won. Authors conclude that water-onlycyclone will be very effective in processing coal fines where the ash contents of the ultra fines are less than the feed ash contents.

An intensive research on physical preparation methods application in Slovak coal cleaning has begun in 1996 (Jakabský et al., 1997). These activities were induced by environment pollution caused by low-rank Slovak coal combustion. The investigation was focused on identification of mineral impurities in coal (Hredzák et al., 1997; 1999; 2002; 2009; Hredzák and Zubrik, 2008; Machajová et al., 2000; 2002;) as well as on deashing and desulphurisation of coal fines, which are used as a steam coal in coal-fired power plant (Jakabský et al., 1997; 1998; Hredzák, 1999; 2001; Hredzák et al., 1998; 2000a; 2000b). Recently, the coal washing in bench-scale condition is performed with the aim to receive low ash products, which are suitable for chemical processing and in such way the recovering of noble organic compounds from deashed coal (Hredzák et al., 2006; 2007; Zubrik et al, 2009; 2010).

The WOC performance in comparison with the heavy media cyclone (HMC) is typical by its lower separation sharpness. The écart probable ( $E_p$ ) for HMC and WOC usually attains the values in the range 0.03÷0.06 (Kozák and Cagaš, 1965) and 0.22÷0.40 (Schlepp and Schmidt, 1988), respectively. So, the basic parameters such as density cut ( $d_{50}$ ), écart probable ( $E_p = (d_{75} - d_{25})/2$ ), imperfection (I =  $E_p/(d_{50} - 1)$ ) and Grumbrecht's efficiency (W – calculated on the basis of the determination of mean relative erroneous yields) attained at the washing of Slovak coals from Cigel' and Handlová collieries are introduced in Table 1.

grain size [mm]		Cí	gel'		Handlová				
	d <sub>50</sub>	E <sub>P</sub>	Ι	W [%]	d <sub>50</sub>	E <sub>P</sub>	Ι	W [%]	
0.5 - 5	1.52	0.135	0.260	78.98	1.46	0.120	0.261	78.68	
0.25-0.5	1.92	0.365	0.397	54.62	2.11	0.230	0.207	53.92	

Tab. 1. Coal separation sharpness and efficiency in the WO cyclone.

### Material and method

The sub-bituminous coal fines from the Cígel and Handlová collieries were subjected to washing in WOC. Their basic parameters are introduced in Table 2 (HBP, 2010b). Preparation of feed into WOC consisted in two-stage crushing and dry screening. The two types of jaw crushers, namely PS D-160 and VČM-3 as well as laboratory screen with a mesh size of 5 mm were applied. In such way the feed into WOC with a grain size of 0–5 mm from original steam coals with a grain size of 0–20 mm was prepared.

Colliery	Product	Grain size [mm]	W <sup>r</sup> <sub>t</sub> [%]	A <sup>d</sup> [%]	S <sup>d</sup> [%]	$\mathbf{Q}^{\mathbf{r}_{i}}$ [MJ.kg <sup>-1</sup> ]
Cígeľ	fines 1		24.0 - 27.0	27.63 - 32.88	1.84 - 2.19	11.0 - 12.5
Handlová	Thics I	0-20	13.0 - 16.0	42.53 - 47.62	1.61 – 1.90	12.0 - 13.5
Cígeľ	с <u>э</u>		24.0 - 26.0	39.47 - 43.24	1.84 - 2.16	9.0 - 10.5
Handlová	fines 3		13.0 - 16.0	40.23 - 46.43	1.49 - 1.90	11.0 - 14.0

Tab. 2. Quality of coal fines (after to HBP, 2010b, modified).

 $W_t^r$  – water content as received,  $A^d$ ,  $S^d$  – ash and total sulphur content in dry matter,  $Q_i^r$  – net heating value as received

The coal washing realised by WOC, which was installed in a semi-plant station with hydrocyclone and reverse circuits as it is illustrated in Fig. 1. Real view on the station is in Fig. 2. The WOC with a diameter of 150 mm and a cone part consisting of three angle sections  $135^{\circ}-75^{\circ}-20^{\circ}$  was applied. Vortex finder (overflow) and spigot (underflow) diameters were of 68 mm and 14.6 mm, respectively. The washing was performed at an input pressure of 100 kPa and a solids concentration of 100 g.l<sup>-1</sup> (10 kg of coal per 100 l of water). Such relatively low solids concentration was selected owing to expectant higher sharpness of washing according to previously obtained experiences in this field.

The products of washing were caught on the overflow and the underflow of WOC by screens with a mesh size of 0.5 mm. All finer grains were considered as slurry or more precisely said circulating feed. The products were dried, weighed and subjected to chemical analyses.

Ash content was determined by ignition in muffle oven at 815 °C to constant value of weight (after 3 h) (ISO, 1997). The SiO<sub>2</sub> content was assayed gravimetrically. Other elements have been determined by atomic absorption spectroscopy using the device VARIAN with accessories: Fast Sequential AAS AA240FS, Zeeman AAS AA240Z with Programmable Sample Dispenser PSD120, Graphite Tube Atomizer GTA120 and Vapor Generation Accessory VGA-77. Total sulphur and its forms were analysed using ESCHKA method (ISO, 1992) and the procedure described in (ISO, 1996), respectively. C, H and N were determined in the Geoanalytical laboratories of the State Geological Institute of Dionýz Štúr in Spišská Nová Ves using an elementary analysis with thermal conducting detector.

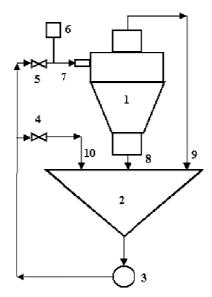


Fig. 1. Scheme of bench-scale hydrocyclone station.



Fig. 2. Hydrocyclone station - real view.

Description to Fig. 1.: 1 - hydrocyclone, 2 - agitating tank, 3 - pump, 4 - valve of reverse circuit, 5 - valve for regulation of input press to hydrocyclone, <math>6 - pressure gauge, 7 - hydrocyclone input, 8 - heavy product - underflow, 9 - light product - overflow, 10 - circulating charge.

The analytical recalculation were performed using equations (1-3), where W – water content, A – ash content, CM – combustible matter content, superscripts a, d and daf mean analytical sample, dry and dry ash free matter.

$$W^a + A^a + CM^a = 100\tag{1}$$

$$A^d + CM^d = 100 \tag{2}$$

$$A^{d} = \frac{100}{(100 - W^{a})} A^{a}, \quad C^{d} = \frac{100}{(100 - W^{a})} C^{a}, \quad C^{daf} = \frac{100}{(100 - A^{d})} C^{d}$$
(3)

The assessment of washing was carried out using the basic balance equations:

$$Q_{feed} \cdot A^d_{feed} = Q_{overflow} \cdot A^d_{overflow} + Q_{underflow} \cdot A^d_{underflow} + Q_{slurry} \cdot A^d_{slurry}$$
(4)

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$$Q_{feed} \cdot CM_{feed}^{d} = Q_{overflow} \cdot CM_{overflow}^{d} + Q_{underflow} \cdot CM_{underflow}^{d} + Q_{slurry} \cdot CM_{slurry}^{d}$$
(5)

$$Q_{feed} = 100 = Q_{overflow} + Q_{underflow} + Q_{slurry}$$
 (6)

where Q with given subscripts are mass amounts (mass yield) of obtained products. If mass amount are expressed in percentage, it can be written 100 instead of Q.

The basic assessment of separation was carried out by calculation of recovery/distribution values  $\varepsilon$ . This value expresses distribution of observed component in given products in percentage. On the basis of material balance, it is valid:

$$100 = \varepsilon_{overflow} + \varepsilon_{underflow} + \varepsilon_{slurry} \tag{7}$$

Coming out of equation (4), the recovery/distribution of ash into overflow will be as follows:

$$\varepsilon_{overflow} = \frac{Q_{overflow} \cdot A_{overflow}^{d}}{Q_{feed} \cdot A_{feed}^{d}} \cdot 100 = \frac{Q_{overflow} \cdot A_{overflow}^{d}}{100 \cdot A_{feed}^{d}} \cdot 100 = \frac{Q_{overflow} \cdot A_{overflow}^{d}}{A_{feed}^{d}}, \quad (8)$$

and the recovery/distribution of ash into underflow and slurry is given:

$$\varepsilon_{underflow} = \frac{Q_{underflow} \cdot A^d_{underflow}}{A^d_{feed}}, \quad \varepsilon_{slurry} = \frac{Q_{slurry} \cdot A^d_{slurry}}{A^d_{feed}}, \quad (9)$$

# **Results and discussion**

The results of coal washing are introduced in Tables 3-11. The content of basic organic elements in washing product is performed in Tables 12-13.

Tab. 3. Content and recovery of ash and combustible matter in products of washing – the Cigel Colliery.

Product	Mass yield		Conte	Recovery [%]			
rrouuci	[%]	$\mathbf{W}^{\mathbf{a}}$	$\mathbf{A}^{\mathbf{a}}$	A <sup>d</sup>	CM <sup>d</sup>	$\mathbf{A}^{\mathbf{d}}$	CM <sup>d</sup>
Overflow	20.74	10.68	6.24	6.99	93.01	3.16	35.61
Underflow	24.62	8.99	22.58	24.81	75.19	13.33	34.16
Slurry	54.64	5.88	65.91	70.03	29.97	83.51	30.23
Feed	100.00	6.44	42.87	45.82	54.18	100.00	100.00

Tab. 4. Content of basic chemical components in products of washing – the Cígel' Colliery.

Product	SiO <sub>2</sub>	$Al_2O_3$	CaO	MgO	Fetotal	STOTAL	S <sub>SULPHIDIC</sub>	As
riouuci	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[ppm]
Overflow	2.04	1.25	0.560	0.298	0.50	1.22	1.02	76.50
Underflow	11.21	5.61	0.504	0.431	1.37	1.32	1.01	87.90
Slurry	39.40	15.49	0.868	1.111	3.11	0.74	0.60	70.20
Feed	24.71	10.11	0.714	0.779	2.14	0.98	0.79	75.86

Product	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fetotal	STOTAL	S <sub>SULPHIDIC</sub>	As
Overflow	1.71	2.56	16.26	7.99	4.85	25.76	26.85	20.92
Underflow	11.17	13.67	17.36	13.69	15.76	33.08	31.55	28.52
Slurry	87.12	83.77	66.38	78.32	79.40	41.16	41.60	50.56
Feed	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Tab. 6. Content and recovery of ash and combustible matter in products of washing – the Handlová Colliery.

Product	Mass yield		Conte	Recovery [%]			
Froduct	[%]	$\mathbf{W}^{\mathbf{a}}$	A <sup>a</sup>	A <sup>d</sup>	CM <sup>d</sup>	$\mathbf{A}^{\mathbf{d}}$	CM <sup>d</sup>
Overflow	29.37	11.95	6.78	7.70	92.30	5.70	44.91
Underflow	21.91	10.43	16.74	18.69	81.31	10.33	29.53
Slurry	48.72	7.51	63.21	68.34	31.66	83.97	25.56
Feed	100.00	8.02	36.47	39.65	60.35	100.00	100.00

Product	SiO <sub>2</sub> [%]	Al <sub>2</sub> O <sub>3</sub>	<b>CaO</b> [%]	MgO [%]	Fe <sub>TOTAL</sub>	S <sub>TOTAL</sub>	S <sub>SULPHIDIC</sub>	As [ppm]
Overflow	3.33	1.50	0.371	0.108	0.65	1.18	0.92	73.40
Underflow	10.01	3.36	0.478	0.202	1.63	1.44	1.24	136.30
Slurry	37.10	15.34	0.784	1.194	3.82	0.80	0.78	79.40
Feed	21.25	8.65	0.602	0.658	2.41	1.05	0.92	90.11

Ta	ab. 7. Conte	nt oʻ	f basic chemica	l com	ponents in	products of	f was	shing	- the Hand	dlová Collie	ery.

Tab. 8. Recovery of basic chemical components into products of washing – the Handlová Colliery [%]										
Product	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fetotal	STOTAL	S <sub>SULPHIDIC</sub>	As		
Overflow	4.60	5.08	18.28	4.82	7.96	32.94	29.23	23.92		
Underflow	10.32	8.52	17.60	6.73	14.82	30.02	29.57	33.15		
Slurry	85.08	86.40	64.12	88.45	77.22	37.04	41.20	42.93		
Feed	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00		

9. Content and recovery of ash and combustible matter in products of washing - the Handlová Colliery (Eastern field).

Product	Mass yield		Conte	Recovery [%]			
Frouuci	[%]	W <sup>a</sup>	A <sup>a</sup>	Ad	CM <sup>d</sup>	A <sup>d</sup>	CM <sup>d</sup>
Overflow	29.50	7.55	8.33	9.01	90.99	8.23	39.65
Underflow	35.30	7.59	19.44	21.04	78.96	22.99	41.18
Slurry	35.20	7.65	58.31	63.14	36.86	68.79	19.17
Feed	100.00	7.60	29.85	32.31	67.69	100.00	100.00

Tab. 10. Content of basic chemical components in products of washing – the Handlová Colliery (Eastern field).											
Product	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fetotal	STOTAL	SSULPHIDIC	As			
Product	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[ppm]			
Overflow	4.14	1.91	0.434	0.199	0.59	1.73	0.75	66.00			
Underflow	10.80	4.23	0.644	0.398	1.25	2.35	1.57	73.00			
Slurry	36.80	13.76	0.923	1.441	1.94	1.19	0.78	40.00			
Feed	17.99	6 90	0.686	0.712	1 30	1 76	1.05	59 32			

Tab. 11. Recovery of basic chemical components into products of washing – the Handlová Colliery (Eastern field)[%].									
Product	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fetotal	STOTAL	S <sub>SULPHIDIC</sub>	As	
Overflow	6.79	8.16	18.81	8.30	13.41	29.02	21.07	32.82	
Underflow	21.19	21.66	33.40	19.87	33.99	47.17	52.78	43.44	
Slurry	72.02	70.18	47.79	71.83	52.60	23.82	26.15	23.74	
Feed	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	

Tab. 12. Content of basic organic elements in dry							matter [%].		
Colliery	Cigel'			Handlová			Handlová (Eastern field)		
Product	Cd	H <sup>d</sup>	N <sup>d</sup>	Cd	H <sup>d</sup>	N <sup>d</sup>	Cd	Hd	N <sup>d</sup>
Overflow	63.35	4.89	0.64	61.33	5.57	0.68	68.12	4.49	0.62
Underflow	52.72	4.15	0.58	53.59	4.13	0.67	59.44	4.43	0.60
Slurry	19.12	2.34	0.29	20.03	2.70	0.32	23.92	1.93	0.30
Feed	36.56	3.31	0.43	39.51	3.86	0.50	49.50	3.57	0.50

	Tab. 13. Content of basic organic elements in dry ash free matter [%]								matter [%].
Colliery	Cigel'			Handlová			Handlová (Eastern field)		
Product	C <sup>daf</sup>	$\mathbf{H}^{daf}$	N <sup>daf</sup>	Cdaf	$\mathbf{H}^{daf}$	N <sup>daf</sup>	C <sup>daf</sup>	$\mathbf{H}^{daf}$	N <sup>daf</sup>
Overflow	68.11	5.26	0.69	66.45	6.03	0.74	74.86	4.93	0.68
Underflow	70.12	5.52	0.77	65.91	5.08	0.82	75.62	5.63	0.76
Slurry	63.80	7.81	0.97	63.27	8.54	1.02	64.88	5.23	0.81
Feed	67.48	6.11	0.79	65.47	7.04	0.90	71.62	5.28	0.74

Thus, in all three cases a clean coal with ash content under 10 % was obtained. Recovery of ash to overflow products also does not exceed 10 %. But on the other hand combustible matter losses in underflow and slurry are relatively high. The ash content in underflow products attains 18-25 % and for this reason these products may be considered as the middlings. The recovery of ash to slurry was of 68-84 %. The content of total sulphur in washed coal is slightly higher in two cases in comparison with feed. The recovery of total sulphur to washed coal achieves 25-33 %. The highest contents of sulphur and arsenic were assayed in underflow products. The arsenic recovery to washed coal reaches the values about 20-33%. Thus, the washing was successful from viewpoint of ash content reduction and ash rejection. The carbon content in dry matter of washed coal is over 60 %. The highest carbon contents in combustible matter were determined in underflow products in two cases.

#### Conclusion

The behaviour of sub-bituminous coal fines in WOC from three Slovak mine fields, namely Cigel', Handlová and Handlová-east was studied. The washed coals with ash content under 10 % at a mass yield of 20-30 % have been obtained. The washing resulted in over 90 % ash rejection and about 70 % total sulphur rejection. So, these products are suitable input material for organic compounds extraction.

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