

LOW-TEMPERATURE PROCESSING OF COLOMBIAN COAL IN EXPERIMENTAL RETORT

V. YEFIMOV
I. PULEMYOTOV
S. DOILOV

Institute of Oil Shale
Tallinn Technical University
12 Järveküla St., Kohtla-Järve
30198 Estonia

Processing Colombian coal in experimental retort demonstrated a number of specific features of the process:

- *Normal downward passage of the initial material through the retort was possible only by dry extraction of the solid residue (semicoke) from the retort*
- *Low-temperature retorting proceeds smoothly, the residual content of volatiles is readily reduced from 36 % to as low as 2 % producing paraffinic oil and high calorific low ash low sulfur semicoke*
- *The Colombian low ash coal may be regarded as an attractive feed for retorting in low-temperature zero-waste technology*

Colombia has the largest coal deposits in South America. The coal reserves of the 35 deposits found on her territory are estimated to amount to 21 billion tons [1].

A trial sample of Colombian coal was delivered to *Kiviter AS*, Kohtla-Järve, Estonia, by one foreign company for testing as feed for low-temperature processing (semicoking). Thermal processing tests were performed in a 600-700 kg-per-day experimental retort in November 1995 [2]. The objective of testing was to evaluate the low-temperature retorting process as suitable for yielding useful retort products from Colombian coal in a practically zero-waste technology complying with increased environmental protection requirements. In these tests both oil and gas produced were studied concerning their further use. The solid retorting char residue (semicoke) with a very low volatiles content was assessed for possible use in metallurgy for special purposes.

Properties of the initial coal tested (Table 1) confirm a high heating value of the coal, its low sulfur content and a relatively high Fischer assay oil yield. The mineral part of the coal sample was represented predominantly by compounds of silica and aluminum.

The composition of Fischer assay gas was represented mostly by saturated hydrocarbons (Table 2).

Table 1. Properties of Colombian Coal

Indices	Value
Moisture of coal sample tested in experimental retort, %	7.2
Content (dry basis), %:	
Carbon dioxide (CO ₂) ^{d_M}	1.10
Ash <i>A^d</i>	1.45
Organic matter*	97.45
Total sulfur <i>S^{d_t}</i>	0.36
Volatile matter (dry basis), %	35.7
Fischer assay product yield (standard retort), %:	
Oil	15.02
Pyrogenic water	4.61
Semicoke	75.94
Gas and losses (by difference)	4.43
Heating value (bomb calorimeter):	
MJ/kg	33.75
kcal/kg	8060
Ash composition, %:	
SiO ₂	53.73
Fe ₂ O ₃	6.98
Al ₂ O ₃	24.67
CaO	4.00
MgO	4.58
SO ₃	2.62
Total	96.58

* Here and later on the organic matter content is equal to: $100 - (\text{CO}_2)^{d_M} - A^d$.

Processing tests in experimental retort were conducted under three different modes of operation (Table 3). Modes 1 and 2, especially mode 2, were characterized by severe thermal conditions aimed at identifying the minimum attainable level of volatiles of the discharged semicoke. According to mode 3, the coal sample was processed under selectively mild thermal conditions, fairly close to those maintained in commercial retorts on low-temperature processing of oil shale.

The results of retorting tests showed that normal downward passage and discharge of the retorted material was possible only by the method of dry extraction of the semicoke from the retort. Inspection of the retort inner surface after testing revealed practically no traces of coke depositions. Even the metal grate on the "cool" side of the retorting chamber remained clean.

Processing of coal in experimental retort resulted in an average oil yield of 10 % (10.77 %, dry basis) or 71.7 % of the Fischer assay oil. The properties of the oil produced (Table 4) indicate that the oil is of paraffinic nature, has a low sulfur content and a high heating value. It may be seen in Table 5 that the solid char residue (semicoke) has a low ash content and a low volatiles content. In all retorting modes the semicoke was characterized by a low sulfur content and a high heating value. It may also be seen that the use of severe retorting conditions (modes 1 and 2) is practically not required.

Table 2. Yield and Composition of Fischer Assay Gas* from Colombian Coal

Indices	Value
Specific gas yield (dry basis), m ³ /t	51.8
Content of components, vol. %:	
CO ₂	15.2
H ₂ S	3.3
H ₂	6.5
CO	8.1
C _n H _{2n + 2}	61.9
Including:	
CH ₄	46.4
C ₂ H ₆	11.6
C ₃ H ₈	2.7
C ₄ H ₁₀ :	
<i>n</i> -butane	0.7
<i>iso</i> -butane	0.2
C ₅ H ₁₂ - <i>n</i> -pentane	0.3
C _n H _m	5.0
Including:	
C ₂ H ₄	1.8
C ₃ H ₆	1.8
C ₄ H ₈ :	
butene-1	0.8
<i>trans</i> -butene-2	0.2
<i>cis</i> -butene-2	0.1
C ₅ H ₁₀ :	
pentene-1	0.1
<i>trans</i> -pentene-2	0.1
C ₆ H ₁₂ - hexene-1	0.1
Total	100.0
Calculated heating value, $\frac{\text{MJ/m}^3}{\text{kcal/m}^3}$:	
gross	<u>35.88</u> 8570
net	<u>32.74</u> 7820
Density, kg/m ³	1.052

* Here and later on all characteristics of gas are given at 20 °C and 760 mm Hg.

The yield of retort gas produced by processing coal samples averaged 1000-1200 m³/t (incl. recycle gas from commercial oil shale retorts used in the burner of the experimental retort). The heating value of the gas was somewhat lower than that of the gas from commercial oil shale retorts (Table 6).

Table 3. Operating Conditions of Processing Colombian Coal in the Experimental Retort

Indices	Operational modes		
	1	2	3
Feed coal throughput rate, kg/day	480	480	960
Residence time of coal in the retorting zone in contact with heat carrier gas, h	6,8	6,8	3,4
Temperature, °C:			
Heat carrier into retorting zone	835	865	860
Oil vapours from retorting zone	215	215	175
Specific combustion air, m ³ /t		400-450	

Table 4. Properties of Oil Obtained in the Experimental Retort from Coal

Indices	Value
Density at 20 °C, g/cm ³	0.9534
Water, %	0.3
Viscosity, 1·10 ⁻⁶ , m ² /s:	
at 75 °C	2.7
at 80 °C	5.8
Entrained fines, %	0.08
Ash, %	0.03
Flash point, °C	118
Pour point, °C	29
Coking value, % (Conradson Carbon Residue)	4.1
Phenolic compounds, %	13.0
Heating value (bomb calorimeter):	
MJ/kg	42.20
kcal/kg	10080
Initial boiling point, °C	214
Distillation, vol.%, at:	
220 °C	1
240 °C	6
250 °C	7
260 °C	9
280 °C	16
300 °C	24
320 °C	33
340 °C	55
350 °C	62
Elemental composition (dry basis), %:	
C	86.1
H	10.2
S	0.4
N	0.4
O (by difference)	3.9

Table 5. Properties of Semicoke Obtained in the Experimental Retort from Coal

Indices	Operational modes		
	1	2	3
Moisture content, %	7.58	2.05	6.34
Content (dry basis), %:			
Carbon dioxide (CO ₂) ^{d_M}	1.2	1.5	0.9
Ash A ^d	3.9	3.7	2.6
Organic matter	94.9	94.8	96.5
Total sulfur S ^{d_t}	0.85	0.89	0.80
Volatile matter (dry basis), %	2.16	1.45	2.24
Heating value (bomb calorimeter):			
MJ/kg	<u>31.94</u>	<u>33.37</u>	<u>32.11</u>
kcal/kg	7630	7970	7670

Table 6. Gas Obtained in the Experimental Retort from Coal

Indices	Value
Content of components, vol. %:	
CO ₂	13.3
H ₂ S	0.1
H ₂	6.9
O ₂	1.5
CO	3.7
CH ₄	2.7
C _n H _{2n+2}	0.26
C _n H _{2n}	0.10
N ₂	71.44
Calculated heating value, $\frac{\text{MJ/m}^3}{\text{kcal/m}^3}$:	
gross	<u>2.64</u> 630
net	<u>2.39</u> 570
Density, kg/m ³	1.172

The yield of retort water from low-temperature processing of Colombian coal amounted to 160 l/t. It may be seen in Table 7 that the retort water is of alkaline character caused by relatively high concentrations of volatile bases. The water-soluble phenols are characterized by a relatively high content of monohydric phenols and pyrocatechols (Table 8).

Table 7. Retort Water Obtained in the Experimental Retort from Coal

Indices	Value
pH	8.47
Volatile phenols, mg/l	3416
Total phenols, mg/l	5950
Neutral oils, mg/l	39.60
Chloride (Cl ⁻), mg/l	612.50
Total sulfur, mg/l	6020
Volatile ammonia, mg/l	4872
Volatile acetic acid, mg/l	2182

Table 8. Composition of Water-Soluble Phenols Obtained in the Experimental Retort from Coal, % by weight

Indices	Value
Monohydric phenols, total	30.9
Including:	
Phenol	16.3
<i>o</i> -Cresol	3.6
<i>m</i> -Cresol	4.0
<i>p</i> -Cresol	3.7
Xylenols	3.3
Dihydric phenols, total	55.2
Including:	
Resorcinol	12.8
2-Methylresorcinol	5.5
4-Methylresorcinol	6.8
5-Methylresorcinol	4.3
Pyrocatechol	12.4
Methylpyrocatechols	13.4
Not identified	13.9
Total	100.0

Conclusions

Testing of Colombian coal samples in experimental retort revealed special features of the process.

Normal downward passage of coal by gravity through the retort could be secured only by using dry extraction of the semicoke. The oil yielded from Colombian coal was of paraffinic nature (congealed at summertime ambient temperatures). On some sections of the condensation system steam heated piping had to be used.

The experimental retort proved highly sensitive to ingress of air through the charging device. Therefore, retorting of Colombian coal was conducted at very low underpressure values (not exceeding 10-15 mm H₂O) in the gas collector.

The volatile products evolved from Colombian coal possess a sharp specific smell. Therefore, escape of the volatile products into workrooms with service personnel present should be strictly avoided.

The retort water from Colombian coal is characterized by a high pH value (8.5). The water-soluble phenols are represented with 30 % of monohydric phenols (hydroxybenzene, *o*-, *m*-, *p*-cresols, etc.) and 55 % of dihydric compounds (resorcinol, pyrocatechols, etc.).

On low-temperature processing of Colombian coal volatile products are quite readily separated resulting in a retort residue characterized by a volatiles content as low as around 2 %. Because of that, and also due to a relatively high heating value and a low sulfur content, the residual coal char (semicoke) may be considered as a high quality target product for use as fuel and in metallurgy. It also evidences of a possibility of developing low-temperature retorting of Colombian coal as zero-waste technology.

Acknowledgments

The research was financially supported by Estonian Science Foundation and *Kiviter AS*.

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Received January 4, 1999