Hyperfine Interact
DOI 10.1007/s10751-013-0856-z

Comparison of methods to obtain ash from coal of the Southwest of Colombia

G. Medina · J. A. Tabares · G. A. Pérez Alcazar · J. M. Barraza

Abstract The method for concentration of mineral matter at low temperature (about 250 °C), called Low Temperature Ash (LTA) was applied to a sample of coal from the mine "Las Mercedes" located in Colombia southwestern. This method provides better information about the content of mineral matter in natural coal (NC), removing the organic matter more efficiently without significant transformations of mineral phases present in that coal. These results were observed through Mössbauer spectra and X-ray patterns taken from samples of NC, (LTA) and the conventional method of High Temperature Ash (HTA). The results show that the LTA process provides more representative data of the mineral phases for natural coal than that using the conventional HTA process.

Keywords Ash · Mineral · Organic · Phases

1 Introduction

Most of the minerals found in natural coal and in residuals from its combustion known as ashes, contain Fe as its more important component, which makes Mössbauer spectroscopy a suitable technique to obtain information on the main phases present in coal such as Siderite (S), Pyrite (P), Illite (I) and Jarosite (J) [1, 2].

Proceedings of the Thirteenth Latin American Conference on the Applications of the Mössbauer Effect, (LACAME 2012), Medellín, Colombia, 11–16 November 2012.

G. Medina (✉)
Departamento de Física, Grupo de nuevos sólidos (GINSAI),
Universidad Autónoma de Occidente, A. A. 2790, Cali, Colombia
e-mail: gmedina@uao.edu.co

J. A. Tabares · G. A. Pérez Alcazar
Departamento de Física, Grupo GMTF, Universidad del Valle, A. A. 25360, Cali, Colombia

J. M. Barraza
Escuela de Ingeniería, Universidad del Valle, A. A. 25360, Cali, Colombia

Published online: 27 March 2013

The most common method for determining ash content from coal is the High Temperature Ash (HTA) [3, 4]. This method permits the removal of all organic matter in a combustion process, leaving a residue associated with coal mineral matter. However, by this method, some mineral phases present in coal are mainly transformed into oxides and some other products attach to the organic matter. Therefore, the results from applying this method could be less reliable than other methods where the losses of mineral phases present in coal [5] are reduced significantly. A more reliable method is the low temperature ash (LTA) method, which consists in eliminating organic matter at a controlled temperature of 250 °C, with a constant flow of air, in a fluidized bed [6]. At this temperature phase transformations are not present [7–9], giving a more representative result of the mineral content in natural coal (NC).

The aim of this paper is to report the results of the mineral content of coal from a southwestern region of Colombia, through HTA and LTA processes. X-ray and Mössbauer spectroscopy techniques were used to probe their effectiveness.

2 Experimental procedure

A sample of natural coal (NC) from the "The Mercedes" mine (located in the southwest section of Colombia) was selected and submitted for combustion for 4 hours. This sample has a low content of mineral matter (ash 11.7 %); according to the result obtained by the HTA process (ASTM D3174). The sample used was first powdered to particles size of $\leq$200 μm. The first hour, a sample of 1.0 g was exposed to a temperature of 550 °C at a rate of 8 °C/min, the second hour, at 750 °C at a rate of 3 °C/min, and the last two hours at a constant temperature of 750 °C. The sample was cooled to room temperature in a desiccator before being weighted and examined.

Mineral matter in a sample of the same coal was also separated by the low temperature ash (LTA) process. The setup used for the LTA method consisted of a Pyrex tube reactor of 30.0 cm long × 2.5 cm diameter with a porous frit that supported a fluidized bed of pulverized coal (90–200 μm) [4]. The temperature of the reactor was monitored by a thermocouple in the exterior wall, while the coal temperature was monitored by a thermocouple located in the centre of the fluidized bed. Oxidation of the coal samples was accomplished by heating the fluidized coal at ≈10 °C/min up to 250 °C and maintaining this temperature for a designated period of time. Fluidization was achieved with dry air flowing at ≈500 cm^3/min. Mössbauer measurements at room temperature were performed using a conventional multichannel spectrometer with a ^{57}Co source. Spectra were fitted using the VARFIT program and the α-Fe as the calibration sample. The X-ray powder diffraction patterns were obtained using a conventional X-ray spectrometer, using Ni-filtered Cu K_α radiation. Typical scan speeds were 0.02° 2θ min^{-1}, and patterns were fitted using the MAUD program.

3 Results and discussion

Results from LTA and HTA processes, gave residual mineral matter. In order to establish the efficiency of the LTA process in identifying l the mineral matter

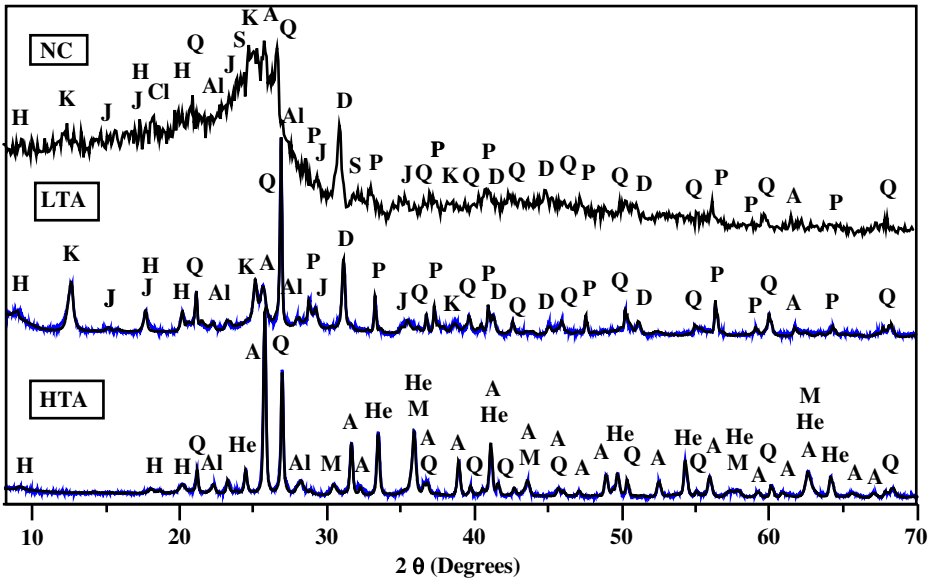

Fig. 1 X-ray diffractions results obtained on the natural coal (NC), low temperature ash (LTA) and high temperature ash (HTA), respectively. The main minerals identified by NC - LTA are: K, kaolinite; Q, quartz; D, dolomite; A, anhydrite; J jarosite; Al, albite; H, halloysite; and P, pyrite. Minerals identified by HTA are: A, anhydrite; He, hematite; Q, quartz; Al, albite; H, halloysite; and M, magnetite

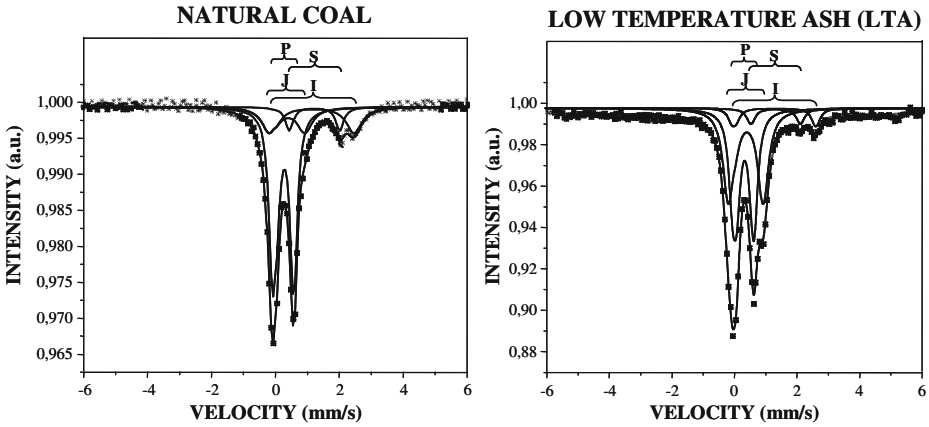

Fig. 2 Mössbauer spectroscopy results obtained on natural coal (NC) and low temperature ash (LTA) samples, respectively. Phases shown: siderite (S), pyrite (P), illite (I) and jarosite (J)

without significant phases changes, tests were carried out by x-ray diffraction and Mössbauer spectroscopy on NC, LTA and HTA samples. These results are shown in Figs. 1 and 2, respectively.

Figure 1 shows the results obtained by X-ray diffraction for the NC, LTA and HTA samples, respectively. In the diffractogram of the NC sample a background

Table 1 Mössbauer parameters and spectral areas of the phases found in the CN and CBT samples.

Mineral phases	Mössbauer parameters	Sample	
		NC	LTA
Pyrite	δ (mm/s) + 0.01	0.35	0.36
	Δ (mm/s) + 0.01	0.59	0.58
	% Spectral Area	57	56
Illite	δ (mm/s) + 0.01	1.24	1.25
	Δ (mm/s) + 0.01	2.58	3.02
	% Spectral Area	17	20
Siderite	δ (mm/s) + 0.01	1.30	1.32
	Δ (mm/s) + 0.01	1.58	1.45
	% Spectral Area	8	10
Jarosite	δ (mm/s) + 0.01	0.47	0.55
	Δ (mm/s) + 0.01	1.04	1.15
	% Spectral Area	16	13

line is observed with a widening trace in the 2theta angle region of 15–30°, which could be associated with the presence of organic and amorphous material [5].

It is also possible to identify in the LTA produced samples the major picks corresponding to the main crystalline phases, such as: kaolinite (K), quartz (Q), dolomite (D), anhydrite (A), jarosite (J), albite (Al), halloysite (H), and pyrite (P); while in the HTA sample some other mineral phases can be identified: anhydrite (A), hematite (He), quartz (Q), albite (Al), halloysite (H), and magnetite M.

In the diffraction patterns of the LTA and HTA samples it is possible to note a very flat horizontal background line, with an appreciable decrease in the widening trace respect to that observed in the X-ray diffraction pattern of the NC sample. These results demonstrate that there is a better removal of organic matter by these processes. Furthermore, it is observed that the major mineral phases identified in the natural coal, are also present in the LTA sample without significant changes. These results also show that the LTA process removes organic matter more efficiently, without significant changes in mineral phases.

However, unlike the results obtained for the LTA sample, in the HTA sample new mineral phases such as hematite (He) and magnetite (M) can be identified. The disappearance of phases for kaolinite (K), jarosite (J), pyrite (P) and dolomite (D) is notorious. A decrease of the intensity peak for halloysite (H) and an increased of the intensity peak for anhydrite (A) are observed. These results show that the HTA process removes organic matter too, but transforms the mineral phases present in coal.

Figure 2 shows Mössbauer spectra for the NC and LTA samples, respectively. These spectra show that the Fe-containing mineral phases present in the NC sample persist after a CBT process. In these spectra it is possible to observe the following phases: siderite (S), pyrite (P), illite (I) and jarosite (J). In the Table 1 the Mössbauer parameters and spectral areas of the phases found in the NC and LTA samples are shown and it is possible to observe a decrease in the spectral area of the P and J, and an increase for the spectral area of the S and I phases. These behaviors may be associated with the influence of oxygen on these phases during the LTA process.

The results found by the techniques mentioned above confirm the effectiveness of the process of LTA to remove the organic matter having a higher concentration of mineral matter, without considerable changes of their original phases.

4 Conclusions

Results obtained by X-ray diffraction and Mössbauer spectroscopy on CN, HTA and LTA samples, show that the low temperature ash (LTA) process is very reliable to eliminate organic matter and condense the mineral matter without a big loss or significant transformations of the mineral phases present in coal. This occurs because the sample is exposed to 250 °C, which is below the temperature at which transitions occur in the respective minerals phases [3].

References

1. Fajardo, M., Mojica, J., Barraza, J., Pérez Alcázar, G.A., Tabares, J.A.: Mineral identification in Colombian coals using Mössbauer spectroscopy and X-ray diffraction. Hyperfine Interact. **122**, 129–138 (1999)
2. Gracia, M., Marco, J.F., Gancedo, J.R.: Uses and perspectives of Mössbauer spectroscopic studies of iron minerals in coal. Hyperfine Interact. **122**, 97–114 (1999)
3. Vassilev, S.V., Vassileva, C.G.: Occurrence, abundance and origin of minerals in coals and coal ashes. Fuel Process. Technol. **48**, 85–106 (1996)
4. Martínez-Tarazona, M.R., Martínez-Alonso, A., Tascón, J.M.D.: Mineral matter in Spanish bituminous and brown coals. Part. 2. Mineral matter quantification. Erdol Kohle Erdgas Petrochem. **46**, 202–209 (1993)
5. Stanislav, V., Vassilev, Tascón, J.M.D.: Methods for characterization of inorganic and mineral matter in coal: a critical overview. Energy Fuels **17**, 271–281 (2003)
6. Herman, R.G., Simmons, G.W., Cole, D.A., Kuzmicz, V., Klier, K.: Catalytic action of minerals in the low temperature oxidation of coal. Fuel **63**, 673–677 (1984)
7. Martínez-Tarazona, M.R., Spears, D.A., Palacios, J.M., Martínez-Alonso, A., Tascón, J.M.D.: Mineral matter in coals of different rank from Asturian Central basin. Fuel **71**, 367–372 (1992)
8. Sentorun, C., Schobert, H.H.: Determination of the mineral matter contents of some Turkish lignites by low-temperature ashing. In: Proc. 9th Intern. Conf. Coal Science, vol. 1, pp. 401–404. Germany (1997)
9. Helle, S., Alfaro, G., Tascón, J.M.D.: Mineralogical and chemical characterisation of coals from Southern Chile. Int. J. Coal Geol. **44**, 85–94 (2000)