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W. A. Pacheco Serrano · D. Quintão Lima · J. D. Fabris

Abstract Three samples of coke produced from coal from a mine in the municipality of Samacá, department of Boyacá, Colombia, were studied essentially with Mössbauer spectroscopy. The samples were treated with NaOH 5 mol L^{-1} in order to increase the proportion of iron oxides, by selectively dissolving silicate minerals and any remaining gibbsite, before the physical analysis. Room temperature Mössbauer data revealed that all samples do contain major proportions (>50 % of the relative subspectral area) of hematite along with (super) paramagnetic species as iron-bearing chemical compounds. The superparamagnetic contribution may be due to very fine grains of iron oxides, including nanometric hematite.

Keywords Iron in coal · Coke · Hematite · Chemical dissolution

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Fig. 1 Mössbauer spectrum of coal coke sample G 01 without chemical treatment at RT
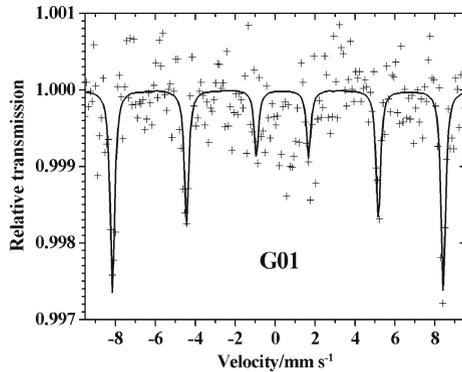
1 Introduction

Coal production is experiencing a growing economical importance in Colombia. A significant part of the coal production is direct to supply the industrial demand in the country; part is exported after being processed to form coke. In Boyacá, especially in Samacá (5° 29′ 60.00″ N 73° 30′ 0″ E), the coal activity has been taking a particular economical importance. The extraction of coal mineral and its industrial processing to produce coke have been playing a very significant role in the whole economy of Boyacá. However, many concerns on any potential threats to the natural environment are permanent issues regarding national policies to mining and other industrial activities. Knowing more about the chemical and mineralogical nature of such an ore requires efforts of ample scientific researches in order to allow planning for its rational exploitation, while preventing uncontrolled harm to the natural environment.

The relationship between coal and iron oxides is a fundamental matter in the various application processes in industrial scale, especially if the coke is to be used for energy generation.

Mössbauer spectroscopy is a nuclear physical technique that allows assessing the intrinsic properties of the iron-bearing mineral phases in coal as well as following the subsequent stages of industrial processing, such as in the coking process. An example of this state of affairs is the correlation between the coal aromaticity and chemical state of iron in the ore [1].

In order to improve the Mössbauer analysis, these three coal coke samples were treated with NaOH 5 mol L^{-1}, following the method originally proposed by Norrish and Taylor [2, 3], to selectively remove silicates and, eventually, any remaining gibbsite.

2 Experimental

Prior to coking the coal production, as a first industrial procedure, in the Samacá industrial plant, the material is usually subjected to a cleaning process with water; in some cases magnetite is added intending to remove iron phase inclusions of the mineral coal. Magnetite is mixed in water at different densities and during gravimetric

**Fig. 2** RT - Mössbauer spectra for samples after chemical treatment
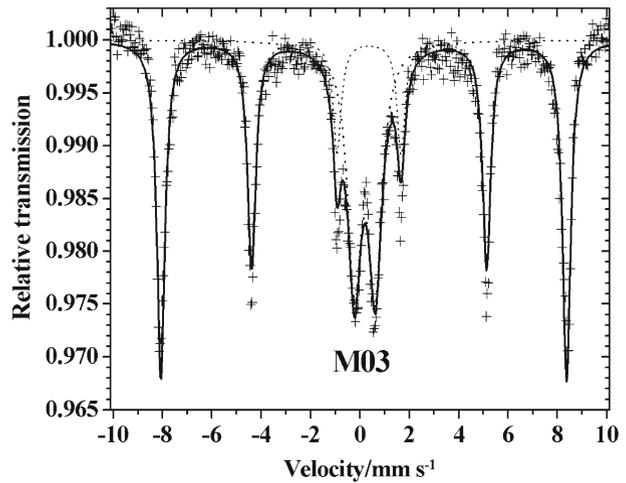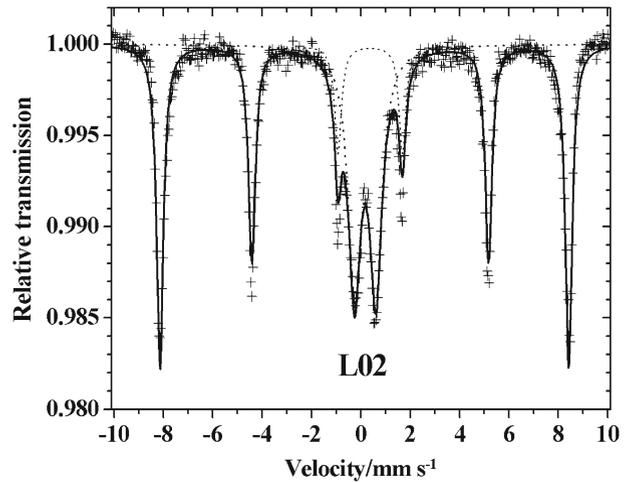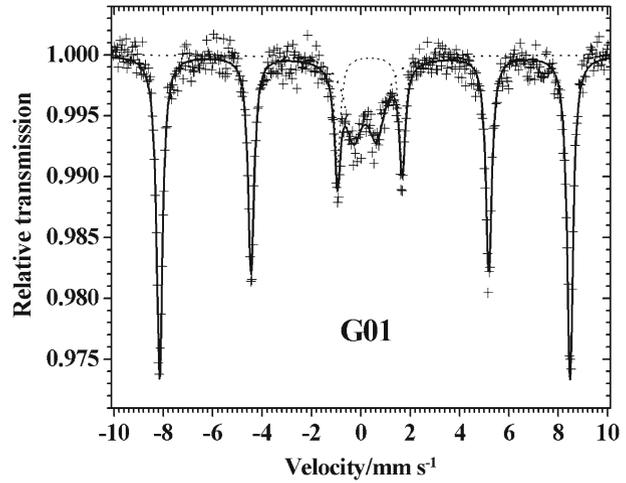
Table 1 Hyperfine parameters measured by ^{57}Fe Mössbauer spectroscopy at room temperature for coal coke samples after chemical treatment with NaOH 5 mol L^{-1}

Sample	δ/mms^{-1}	ε/mms^{-1}	Δ/mms^{-1}	B_{hf}/T	RA/%	Assignment
G01	0.371(2)	–		51.57(1)	76(1)	Hm
	0.28(2)	0.208(3)	0.92(3)		24(1)	
L02	0.368(1)	–		51.349(7)	60(1)	Hm
	0.283(3)	0.237(2)	0.867(4)		40(1)	
M03	0.369(2)	–		50.98(1)	63(1)	Hm
	0,306(4)	0.216(3)	0.847(7)		37(1)	

δ = isomer shift relative to αFe; Δ = quadrupole splitting, ε = quadrupolar shift; B_{hf} = hyperfine magnetic field; RA/% = relative subspectral area; Hm = hematite. Numbers in parentheses are uncertainties over the last significant digit corresponding to the value, as outputted by the least squares-fitting computer program

steps some minerals like pyrite, marcasite, gibbsite and others are removed from raw coal mineral. But the iron oxides are difficult to be removed from the raw coal.

Three samples from different parts of the region were subjected to oven heating to 900 °C in separate stages. Mössbauer measurement were performed on samples without any chemical treatment, and, depending on the observed results, with chemical treatment with NaOH 5 mol L^{-1} at 90 °C for 2 hours; such a treatment made it possible to concentrate the magnetic phases in other mineralogical components of the coke [3–5]. Mössbauer measurements were performed at RT on a conventional spectrometer in a transmission configuration, using a ^{57}Co/Rh source with a nominal activity of ∼20 mCi. Chemical formulae were allocated through balance of charges and masses based on chemical composition data, following the algorithm described elsewhere [6].

3 Results and discussions

Figure 1 shows the Mössbauer spectrum for G01 ore sample without chemical treatment. The absorption is relatively low; ∼ 0.3 %. The magnetic hyperfine structure is characteristic of that for hematite. Despite of the scattered points on the pattern, fitting spectrum with just a sextet leads to parameters in Table 1.

For the other two samples, namely L02 and M03, Mössbauer spectra (not shown) also show very low absorption (∼ 0.2 %), again due to the low concentration of iron in the coal. In both cases, a larger data scattering made it more difficult to fit and assign parameters to spectra.

Figure 2 shows corresponding Mössbauer spectra obtained after chemical treatment with NaOH 5 mol L^{-1}.

Table 1 presents the Mössbauer corresponding hyperfine parameters for samples G01, L02 and M03 after chemical treatment with NaOH 5 mol L^{-}1.

All samples were found to contain hematite (ideal formula, αFe$_2$O$_3$) and Fe^{3+} (super) paramagnetic component. The iron oxides obtained through the coking process at 900 °C may be actually regarded as being a relatively complex mixture of decomposition product of hydrated iron(III) oxide – oxides in varying proportions . Parameters for the sextets are characteristic for hematite. The doublets are generally assignable to (super) paramagnetic ferric chemical species. Any superparamagnetic

contribution would be due to iron oxides in very fine particles, including nanosized hematite. To confirm this hypothesis further Mössbauer measurements at low temperatures down to 4.2 K are still needed.

The chemical treatment allowed us to clearly observe the six lines of the hyperfine structure due to hematite at room temperature, along with few details of the whole doublet on spectra for these three samples.

4 Conclusion

Hematite and (super) paramagnetic Fe^{3+} species form the iron-containing fractions in this coal, the starting material to obtain coke in Samacá, Colombia.

The current steps to clean the raw coal in the industry, to remove iron oxides, is relatively optimized, but still far from ideal. The coke product from this coal may be more safely used in industrial metallurgical furnaces but this is less evident for applications in energy generation as the remaining iron forming hematite and other Fe^{3+} chemical species can produce outcomes that may be harmful to the natural environment.

The selective chemical treatment of the raw coal samples with NaOH 5 mol L^{-1} at 90 °C for 2 hours has proved to be effective enough to allow a clear Mössbauer identification of hematite, the dominant iron oxide in these coal samples.

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