




# Mössbauer study of iron ore from El Hobo, Huila, Colombia

W. A. Pacheco Serrano<sup>1</sup>  · A. J. Chacón Velasco<sup>1</sup> ·  
F. E. Wagner<sup>2</sup>

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**Abstract** Iron ore from the El Hobo municipality located in the Huila department in Colombia ( $2^{\circ}35'7''$  N  $75^{\circ}13'27''$  E) was studied by Mössbauer spectroscopy. The material was quite inhomogeneous, consisting of light brown and dark brown concretions of up to about a centimeter in size, and a sandy fraction. Mössbauer spectra at ambient temperature and at 4.2 K were taken of the two kinds of concretions and the sandy fraction. The concretions contain iron mainly as goethite with only a few percent of hematite. The sandy fraction contains goethite and hematite in roughly equal amounts and a minor fraction of divalent iron that splits magnetically at 4.2 K and may be an impure kind of siderite.

**Keywords** Mössbauer spectroscopy · Iron ore · El Hobo Colombia

## 1 Introduction

Iron ore occupies only a modest place in Colombia's GDP although it is significant in the dynamics of regional economies. They suffer from the effects of globalization by compet-

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This article is part of the Topical Collection on *Proceedings of the 15th Latin American Conference on the Applications of the Mössbauer Effect (LACAME 2016), 13–18 November 2016, Panama City, Panama*

Edited by Juan A. Jaén

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✉ W. A. Pacheco Serrano  
william.pacheco@uptc.edu.co

A. J. Chacón Velasco  
angel.chacon@uptc.edu.co

F. E. Wagner  
fwagner@tum.de

<sup>1</sup> Escuela de Física, Universidad Pedagógica y Tecnológica de Colombia, UPTC, Tunja, Boyacá, Colombia

<sup>2</sup> Physics Department E15, Technical University of Munich, 85747 Garching, Germany

ing with international prices for raw materials and finished products. Given this fact, it is desirable to deepen the knowledge on local raw materials with the aim of achieving the best use of such resources, making a successful impact on the creation of the country's wealth. In this context we have studied samples of an iron ore from a deposit near the El Hobo municipality in the Huila department in Colombia ( $2^{\circ}35'7''$  N  $75^{\circ}13'27''$  E) using  $^{57}\text{Fe}$  Mössbauer spectroscopy at ambient temperature and 4.2 K, which allows one to determine the kind of iron oxides present in the ore and to obtain an estimate of the iron content of the ore. The ore specimen under study consisted of light brown and dark brown concretions and a sandy fraction, which were studied separately.

## 2 Experimental

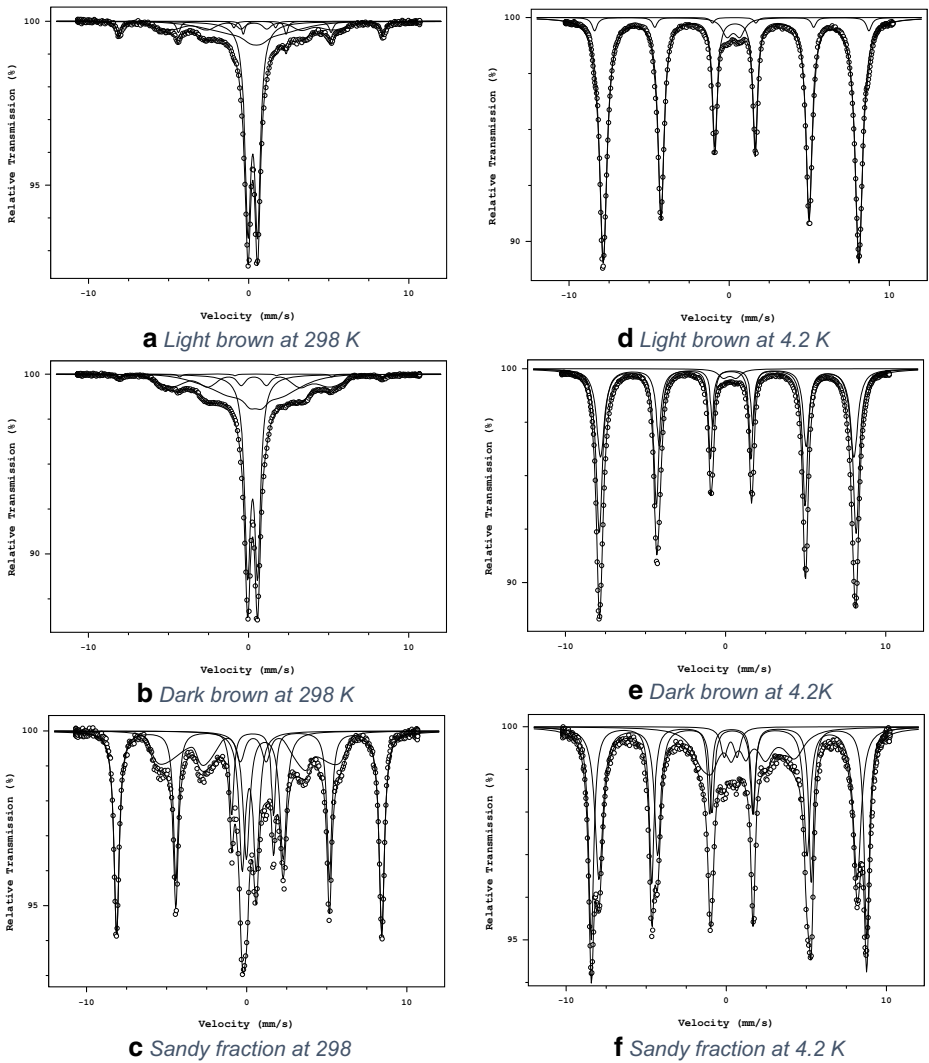
The concretions were crushed in a mortar, while the sandy fraction was used for Mössbauer spectroscopy without further treatment. The samples were filled into Lucite capsules to make absorbers containing 35, 37, and 166  $\text{mg}/\text{cm}^2$  thickness of the light brown, dark brown and sandy fraction, respectively.

The Mössbauer spectra were recorded with a source of  $^{57}\text{Co}$  in Rh in transmission geometry. The loudspeaker type velocity drive was operated with a sinusoidal velocity waveform. For the measurements at 4.2 K a liquid helium bath cryostat was used, in which the source was also cooled to 4.2 K. The spectra were fitted with appropriate superposition of Lorentzian lines grouped into quadrupole doublets, magnetic sextets or magnetic sextets. The broad hyperfine patterns of goethite observed in the room temperature spectra of all three samples were fitted with patterns corresponding to Gaussian distributions of magnetic hyperfine fields. The iron content of the samples was estimated from the total areas of the 4.2 K spectra using the spectrum of an absorber of 9.1  $\text{mg}/\text{cm}^2$  of hematite measured under the same conditions as a reference.

## 3 Results

The Mössbauer spectra are shown in Fig. 1; the results of the least squares fits are given in Table 1 for the room temperature spectra and in Table 2 for the 4.2 K spectra. The total iron content given as  $\text{Fe}_2\text{O}_3$  determined from total areas under the respective 4.2 K spectra, is 34 wt.% for both the light and brown concretions, while the sandy fraction, with only 6 wt.%, contains much less iron. The relative uncertainty in these numbers is estimated to be about 10%.

The spectra of the concretions are very similar, both at room temperature and at 4.2 K. The former consist of a strong quadrupole doublet of ferric iron and broad magnetic patterns that were fitted with a superposition of two Gaussian distributions of hyperfine fields. Such broad magnetic patterns are typical of goethite with small particle sizes possibly also containing impurities; nanosized goethite may even exhibit no magnetic hyperfine splitting at all at room temperature [1–4]. Very small amount of hematite is also visible in the RT spectra. The 4.2 K spectra of both concretions show that practically all the iron visible in the RT spectra, including the quadrupole doublets, is goethite that splits magnetically at 4.2 K, albeit with somewhat broadened lines due to its small particle or impure nature. A minor fraction still yielding a quadrupole doublet at 4.2 K presumably is a clay fraction. The hematite that is clearly visible in the room temperature spectra is hardly visible at 4.2 K. In the spectrum of the light brown concretion it is visible as weak shoulders on the high-field



**Fig. 1** Mössbauer spectra of three fractions of the El Hoho iron ore measured at ambient temperature and at 4.2 K

side of the goethite pattern, in the spectrum of the dark concretion the goethite pattern is slightly broader and the hematite, which is very weak in the room temperature spectra is practically invisible and no effort was made to fit it to the 4.2 K spectrum.

The sandy fraction presents more complicated spectra, both at room temperature and a 4.2 K. The room temperature spectrum exhibits a dominant hematite component as well as the broad magnetically split component of goethite and a ferric doublet. In addition to these, however, a ferrous doublet occurs with a quadrupole splitting (Table 1) that is too big for siderite or ankerite, which at room temperature have a quadrupole splitting of only 1.8 mm/s [5] or 1.5 mm/s [6, 7], respectively. It might be a clay mineral or a perovskite in which the M1 and M2 lattice site cannot be distinguished or are very unequally populated

**Table 1** Hyperfine parameters measured by  $^{57}\text{Fe}$  Mössbauer spectroscopy at room temperature for light brown (LB), dark brown (DB) and sandy fraction (SF) at 298 K

$\delta$  = isomer shift relative to  $\alpha\text{Fe}$ ;  
 $\Delta$  = quadrupole splitting,  $\varepsilon$  =  
quadrupolar shift;  $B_{\text{hf}}$  =  
hyperfine Magnetic field; RA/%  
= relative subspectral area

Samples		$\delta / \text{mm}^{-1}$	$\Delta, \varepsilon / \text{mm}^{-1}$	$B_{\text{hf}} / \text{T}$	RA%
LB	[Fe $^{3+}$ ]	0,36	0,60		52
	[Fe $^{2+}$ ]	1,11	2,67		2
	Hematite	0,37	-0,22	51,0	7
DB	[Fe $^{3+}$ ]	0,36	0,61		49
	Hematite	0,39	-0,26	50,5	4
SF		0,40	0,62		17
		1,11	2,55		16
		0,38	-0,28	36,8	25

**Table 2** Hyperfine parameters measured by  $^{57}\text{Fe}$  Mössbauer spectroscopy at 4.2 K for the light brown (LB), dark brown (DB) and sandy fraction (SF) of the iron ore

$\delta$  = isomer shift relative to  $\alpha\text{Fe}$ ;  
 $\Delta$  = quadrupole splitting,  $\varepsilon$  =  
quadrupolar shift;  $B_{\text{hf}}$  =  
hyperfine Magnetic field; RA/%  
= relative subspectral area

Samples		$\delta / \text{mm}^{-1}$	$\Delta, \varepsilon / \text{mm}^{-1}$	$B_{\text{hf}} / \text{T}$	RA%
LB		0,50	0,8		4
		0,48	-0,24	49,5	92
		0,48	-0,14	52,9	3
DB		0,50	0,8		2
		0,48	-0,19	49,5	98
SF		0,48	0,74		1,3
		1,30	0,59	17,9	19
		0,49	-0,27	50,0	40
		0,49	-0,16	53,4	37

by iron [8, 9]. In hedenbergite, for instance, only the M1 sites are occupied and therefore only a single quadrupole doublet is observed [10]. The 4.2 K spectrum confirms the results from the room temperature spectrum and shows that the ferric doublet observed at room temperature is nearly all goethite that splits magnetically at 4.2 K. The weak ferric doublet remaining at 4.2 K may be iron in a clay mineral. The ferrous iron splits magnetically at 4.2 K with rather broad lines. It was fitted with a simple sextet, whose parameters are given in Table 2. Pyroxenes use to split magnetically at low temperatures if they contain enough iron. Hedenbergite, for instance, exhibits a single octet pattern with a hyperfine field of about 18 T at 4.2 K [11].

## 4 Conclusions

The concretions in El Hobo iron mineral are rather rich in iron, which occurs mainly as goethite, with hardly any difference between the light brown and the dark brown color of the concretions. The Mössbauer spectra observed at ambient temperature show that the goethite occurs as small particles that exhibit strongly broadened magnetic patterns or even quadrupole doublets. The sandy fraction contains much less iron in the form of both goethite and hematite in roughly equal amounts. The hematite does not undergo the Morin transition and thus seems to be either impure or occur as rather small particles. The sandy fraction contains about 20% of the iron in the ferrous state. This fraction splits magnetically at 4.2 K but the mineral phase in which it is contained could not be identified unambiguously.

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