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AGING EFFECT ON MÖSSBAUER SPECTRUM OF COPROLITE

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Résumé.- La technique de l'effet Mössbauer a été utilisée pour l'étude d'échantillons de coprolite (bouse fossile), provenant d'horizons géologiques différents du désert nord-est de l'Egypte. Des phénomènes de relaxation sont observés dans les spectres, en relation systématique avec la profondeur de l'horizon d'origine, c.a.d. l'âge de l'échantillon. Ceci confirme l'existence de relation entre la taille des particules et l'effet de vieillissement.

Abstract.- The Mössbauer Effect (ME) technique was applied to study coprolite (fossil dung) samples from different geological horizons in the North-Eastern Desert of Egypt. Relaxation phenomena were observed in the spectra which is in a systematic relation with the depth of the horizon, i.e., the age of the sample. This supported the existence of a relation between the particle size of the iron oxide and the aging effect.

1. <u>Introduction</u>.- In studying "coprolite" which is fossil dung, i.e. petrified excrements of animals living during the time a rock formation was deposited, the results showed the presence of the same iron forms in all samples, but with a remarkable difference in the intensity of the same iron form from type to type of coprolite. This led to suggest that these differences may be due to either a relation between the original nutritive organic matter and the replacing material in the different coprolites, or the difference in the age of the samples which can aid in transforming some iron form to the other. So it was decided to study coprolite samples from different geological horizons to detect the aging effect, if it is exist.

2. Experimental details. - Three different types of coprolite samples were collected from different localities and from the first type /1/, other samples were collected from the same locality but from different geological horizons at lower depths (see table I). The samples were studied using ME technique at both room and liquid nitrogen temperatures. DTA measurements were carried also for some samples. The ME absorbers were prepared from 200 mg/4.5 $\rm cm^2$ of the fine powdered material using a constant acceleration driving system coupled to a 512 multichannel analyzer. The source was 20 mCi 57Co in Cr matrix. 3. Results and discussion. - Figure 1 shows the room temperature ME spectra of a representative sample from each of the three coprolite types. The ME results are summarized in table I. These results showed the presence of α -Fe₂O₃ in all the samples, but with varying amounts. This result is identical with that

obtained for the fossils of the proper animal or plant from the same region /1/.



Fig I : The R.T. ME spectra of a representative sample from each of the three coprolite types.

The ME results showed also the presence of two paramagnetic components beside α -Fe₂O₃. One component has the room temperature ME parameters characteristic

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Sample No.	: Shape	Locality	: :Temperature :	: Component:	I.S. mm/s	Q.S. mm/s	H. k0e	: % of : total : iron
l a	: Rolled bodies : : :	: : Phosphatic : region at : Safaga : (ground : surface)	: R.T. :	: : : A :	0.44	0.48	: : 498	: : 25
				в	0.32	0.64	zero	68
			: :	C :	0.40	1.20	zero	; 7 ;
2	Ellipsoidal : :	Nubban Sand-stone at Aswan	R.T.	A	0.44	0.40	500	58
			:	в :	0.29	0.49	zero	: 31
			:	c ;	0.37	1.14	zero	: 11
3	: Spiral : : (small size) : :	: : Safaga : :	: : R.T.	: : : A :	0.42	0.40	: : 505	: : 59,5
			:	: c	0.33	1.06	zero	29.5
				. D :	0.56	0.56	393	: 11
l _b		30m depth	R.T.	A		0.40	500	:
			:	: D :	:	0.36	340	:
l a			L.N.	A	0.44	0.40	525	: : 25
			:	в:	0.32	0.56	zero	: : 68
		:	:	c :	0.36	1.13	zero	: 7
1 _b			: : L.N.	: A :	0.50	0.48	492	: 68
			:	D	0.72	0.56	467	32
1 c		60m depth	: : L.N.	: A :	0.44	0.48	480	: 74
			:	Е.	0.32	zero	zero	26

Table I : The ME parameters of the different coprolite samples

I.S. is relative to Fe in Cr. Errors in velocity scale \pm 0.08 mm/s. Errors in magnetic fields \pm 5 kOe.

of either pyrite, glauconite, $Fe(OH)_3$ or γ -FeOOH where these compounds have nearly the same room temperature ME parameters, and all these compounds were identified in other fossils /1,2/. The DTA measurements of sample l_a showed an endothermic peak at 200°C and an exothermic one at 550°C. These thermal behaviours together with the values of the ME parameters of the main paramagnetic component in the spectra of this sample at both room and liquid nitrogen temperatures (table I) are characteristic of lipedocrocite (γ -FeOOH) /3,4/.

So this component which constitutes 68% of the total iron present in coprolite type I_a may be due to the presence of γ -FeOOH. The pattern due to γ -FeOOH disappeared in the spectrum of the sample representing type 3 (Fig. 1) with the appearance of a magnetic pattern characteristic of α -FeOOH /5/. The second paramagnetic component has IS = 0.40 and QS = 1.12 mm/s which are characteristic of Fe³⁺ ion in octahedral site /6/.

It is seen that samples 1 and 3 which are from the same locality, but belong to two different types of animals showed differences in their results indicating that there may be a relation between the sort of the original nutritive organic matter and the replacing material. The age of the coprolite sample can also be a factor which aids in transforming one iron form to the other.

Figure 2 shows the room temperature ME spectra of three samples of type 1 from different three depths. The results of sample I_a which is from ground surface showed the stability of the state and percentage of each iron component after cooling to the liquid nitrogen temperature (table I).

On the other hand sample l_b which is from a lower horizon (30m depth), i.e. older than the first sample showed a relaxed spectrum with broad lines and slope in its baseline. This is due to superparamagnetic behaviour resulting from small particle sizes /7/.



Fig. 2 : The ME spectra of three samples from different depths, I_a from the ground surface, l_b from 30m depth and I_c from 60m depth.

The analysis of this spectrum (table I) showed the presence of two magnetic patterns (A and D). The values of the parameters of these two patterns at room and liquid nitrogen temperatures are characteristic of finally divided particles of α -Fe00H (for the small field pattern) and α -Fe₂O₃. The same values of parameters and the shape of the spectrum were obtained previously /5/ for natural goethite (α -Fe00H) of particle diameter ~200 Å.

The spectrum of sample l_c which is from the lowest horizon (60m depth), is rather broad and the field began to collapse. It has the appearance of a spectrum taken just below the magnetic ordering temperature. The spectrum of the same sample at liquid nitrogen temperature (table I) showed mainly a magnetic pattern typical to α -Fe₂O₃. So it is seen that the older sample has the smallest particle size and the less number of iron forms, where as time elapsed one iron form transformed to the other till all reach to the final iron form α -Fe₂O₃.

The shape of the spectra of these three samples indicates /8/ also that : for the first sample the spin relaxation time (of the iron oxide) $\tau_s >> \tau_L$, while for sample l_b , $\tau_s \approx \tau_L$ which equals $4x10^{-8}s$ and for sample l_c $\tau_s \leqslant \tau_L$.

The observed correlation between the particle size and the depth, which is an indication of the time elapsed between the petrification process and today, confirms the existence of the aging effect /9,10/. This effect has been proposed recently by Danon et al. /9/ and Gangas et al. /10/ from their ME measurements on ancient pottery. They explained the time dependence of the particle size observed by the ME spectra of ancient artifacts to be due to a slow disintegration process of the particle size of the iron oxide. This fact indicates that the originally large size iron oxides particles in coprolites suffered an erosion reduction to smaller particles.

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