WHY IS 'RED MARBLE' RED : COULD FE-ISOTOPES SHED LIGHT ON THIS QUESTION THROUGH THE STUDY OF THE ITALIAN AMMONITICO ROSSO AND RECENT ORGANISMS?

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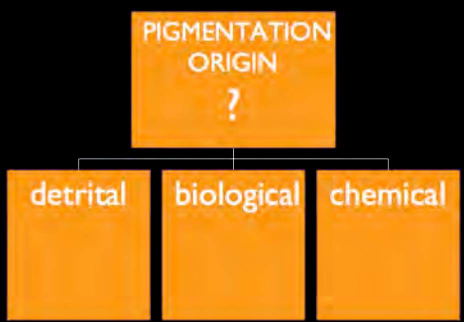
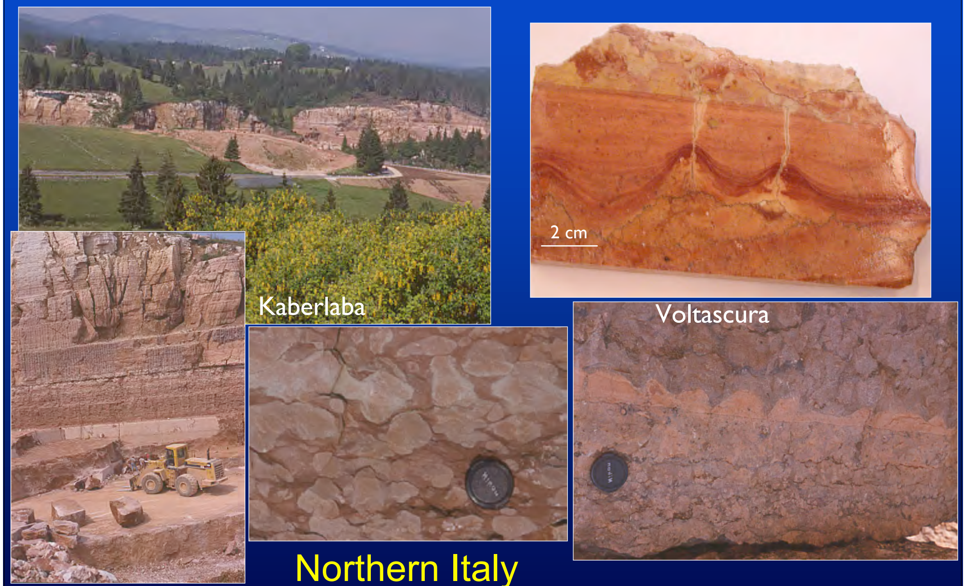
Red limestones are rare but precious …

THE PROBLEM…
is their red pigmentation



OF WHAT CONSISTS THE RED PIGMENTATION?



Northern Italy

Jurassic Ammonitico Rosso

Our studied Red limestones …

- ‘Oolite Ferrugineuse de Bayeux’ mid-Jurassic Normandy = ‘LITTORAL’
- ‘red marbles’ Devonian (Praguian), Czech Republic = INNER RAMP
- griottes Devonian S-France, Viséan N-Spain = SHALLOW PF + OUTER RAMP
- ‘red marbles’ Devonian (Frasnian), Belgium = OUTER RAMP
- red lenses in slope Carboniferous (Bashkirian), N-Spain = SLOPE
- Ammonitico Rosso Jurassic, N-Italy, S-Spain, Sicily = (HEMI)-PELAGIC
- red condensed series Devonian (F/F), Morocco = PELAGIC

shallow ↓ deep

OF WHAT CONSISTS THE RED PIGMENTATION?

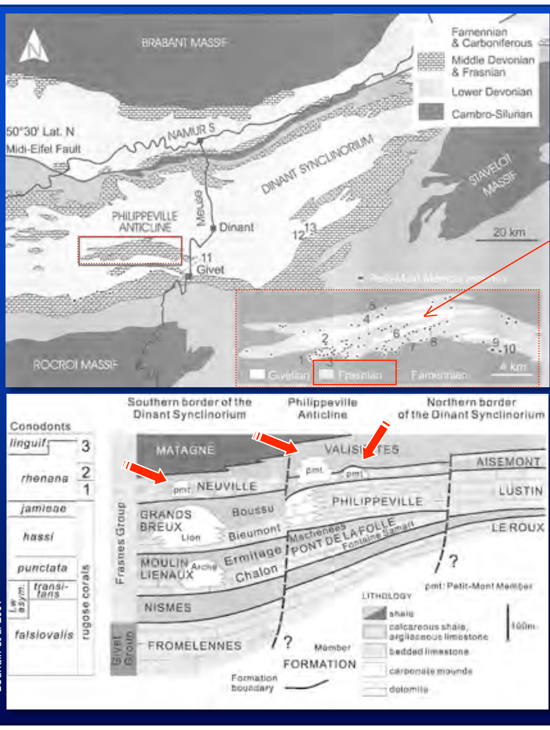
A simple solution is just to ignore the problem!

Ex: the Ammonitico Rosso Symposium (1991). The thick abstract book with its appropriate SCARLET cover deals with sedimentology, palaeontology, diagenesis ... but not a single word on the origin of the colour of the Ammonitico Rosso...

Researches in our laboratory, ULB/1989

… 'Frasnian mud mounds of the Dinant basin are bioconstructions that are built during a regressive phase that passes from an aphotic to an euphotic zone, and through the dysphotic level where the red marbles are concentrated' …

- the base of the bioconstructions is in very calm environments under the SWB, and then the mounds pass into the FWWB where sedimentation ends
- no signs of subaerial exposure as observed in modern reefs



Boulvain et al 2001

69 reported 'red' mud mounds

Severe eustatic sea level rises

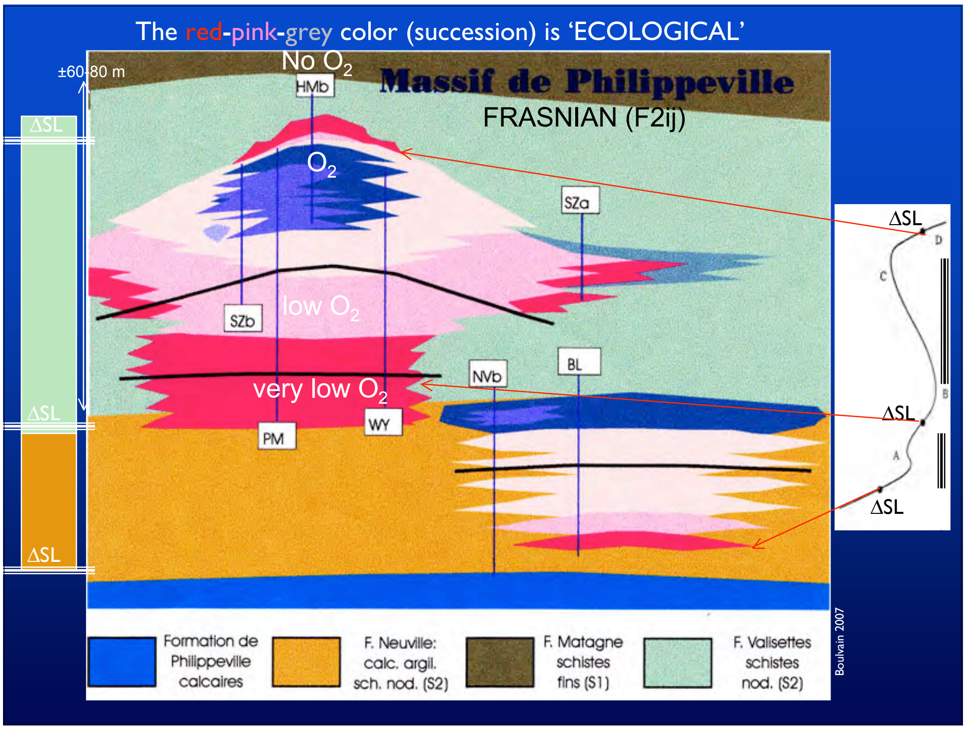
High vertical facies differenciation

High content in microaerophilitic iron bacteria (in *stromatactis* cavities)

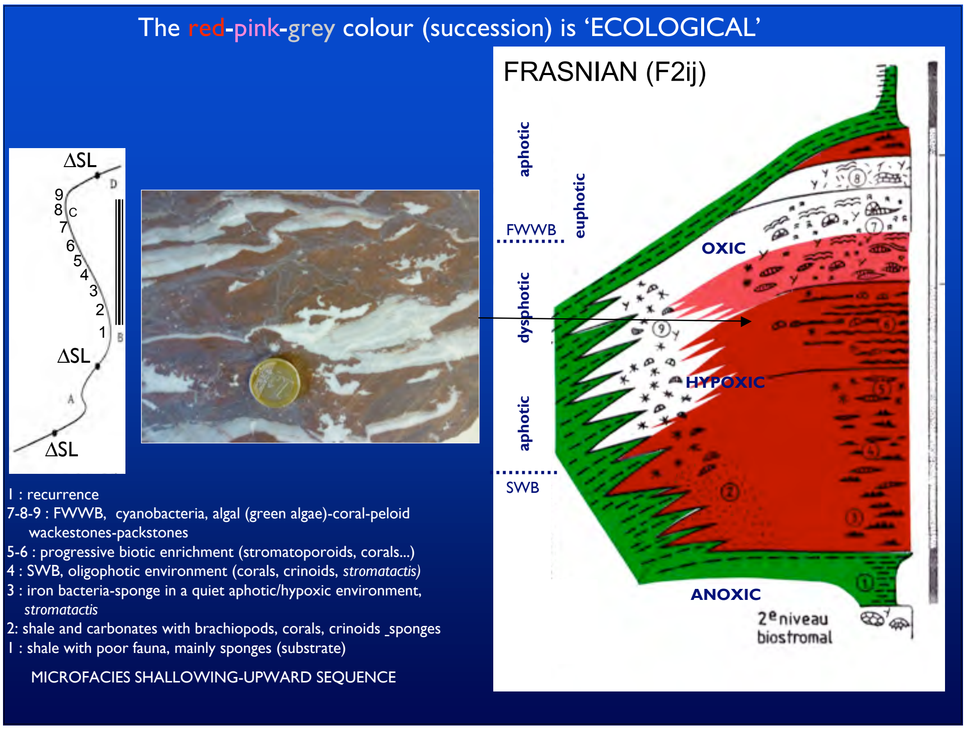
Submicronic hematite hexagonal plates dispersed in the matrix

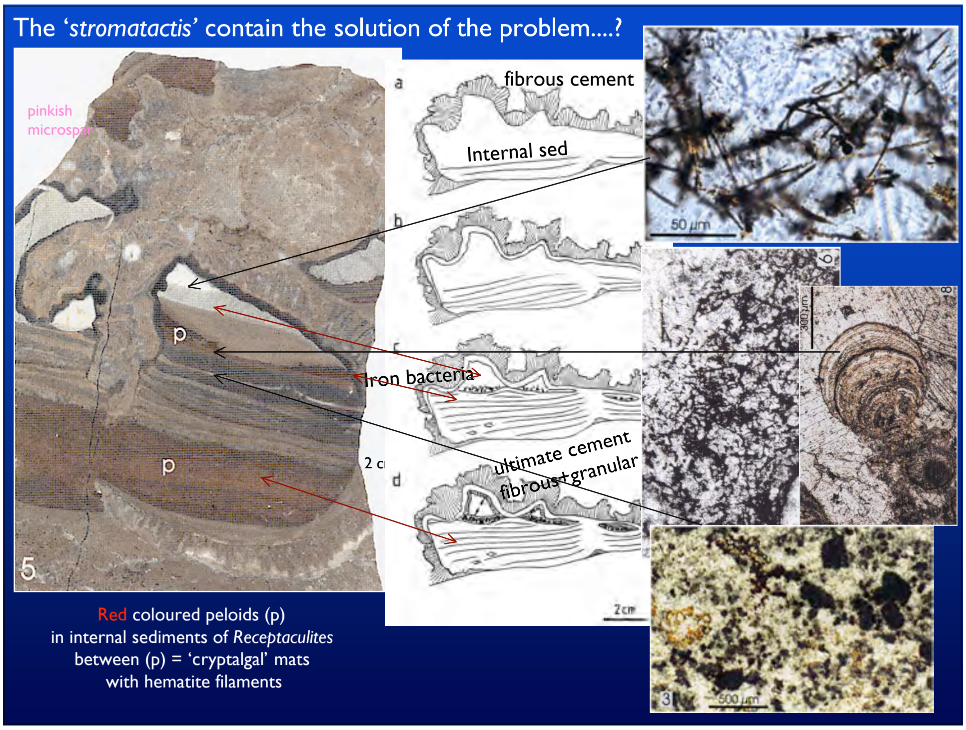
Fe_2O_3 : average 2% (max 5%)

Diam 100-250'm X H 30-80m
Only 30m (upper part) are visible on ±60m
BEAUCHATEAU QUARRY
near Senzeilles
Philippeville Massif
BELGIUM
Frasnian 'F2j'
10cm
griottes
+'stromatactis'
(1880)
Fe_2O_3 : average 1.8% (XRD)
// Mg, Si, Al i.e. // clay content
as already mentioned by Delhaye, 1908



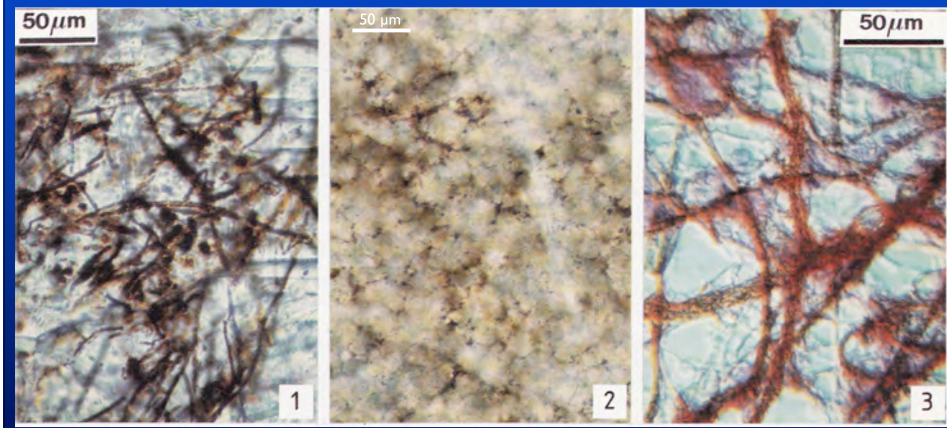
The red-pink-grey color (succession) is 'ECOLOGICAL'
±60-80 m
No O_2
Massif de Philippeville
FRASNIAN (F2ij)
HMb
O_2
SZa
SZb
low O_2
very low O_2
NVb
BL
PM
WY
ΔSL
ΔSL
ΔSL
ΔSL
ΔSL
ΔSL
Formation de Philippeville calcaires
F. Neuville: calc. argil. sch. nod. (S2)
F. Matagne schistes fins (S1)
F. Valisettes schistes nod. (S2)
Boulvain 2007


The red-pink-grey colour (succession) is 'ECOLOGICAL'
ΔSL
9
8
7
6
5
4
3
2
1
ΔSL
ΔSL
FRASNIAN (F2ij)
aphotic
euphotic
FWWB
OXIC
dysphotic
HYPOXIC
aphotic
SWB
ANOXIC
2e niveau biostromal
1 : recurrence
7-8-9 : FWWB, cyanobacteria, algal (green algae)-coral-peloid wackestones-packstones
5-6 : progressive biotic enrichment (stromatoporoids, corals...)
4 : SWB, oligophotic environment (corals, crinoids, *stromatactis)*
3 : iron bacteria-sponge in a quiet aphotic/hypoxic environment, *stromatactis*
2: shale and carbonates with brachiopods, corals, crinoids _sponges
1 : shale with poor fauna, mainly sponges (substrate)
MICROFACIES SHALLOWING-UPWARD SEQUENCE


The '*stromatactis*' contain the solution of the problem....?
pinkish
microspar
p
p
5
a
fibrous cement
Internal sed
b
c
Iron bacteria
ultimate cement
fibrous+granular
d
2cm
50 µm
500 µm
3
Red coloured peloids (p)
in internal sediments of *Receptaculites*
between (p) = 'cryptalgal' mats
with hematite filaments

GEOLOGY | BIOLOGY

Iron bacteria and iron coccoids (hematite in the sheaths) Granular sparite, internal sediment	Relicts of iron bacteria in a slitghtly microsparitized matrix (griotte)	Present day iron bacteria (*Sphaerotilus-Leptothrix* group) Small stream in Brussels



‘Perfectly’ fossilized during cementation of the cavity (*stromatactis*)	Strongly altered-broken during ‘recrystallization’ (microsparitization) of the micrite matrix	Red color due to ruthenium (interferential contrast) *Beggiatoa* sp. (microaerobic)

… how can we explain the red colour that made the stone so scarce?

XVIIIth-XIXth centuries : red = iron (Delhaye, 1908)

° the iron is detrital (Reijers, 1985), transported from the continent, then mixed with the carbonate matrix during sedimentation …

° its concentration and degree of oxidation produce colour variations (reddish)

LATER ON (1964-1988)

a relation between ferruginization/palaeogeography/climate is the fashion : washed equatorial laterite soils provide great quantities of iron oxides… thus the red limestones are used as palaeoclimatic indicators!

oxygenation degree ?
(in non clastic rocks)

° red limestones are found in oxidized facies

° green limestones indicate reducing conditions

both indicate shallow waters

but …

red silicified limestones (lydites)
indicate deep environments
with minimum amounts of iron and oxygen !
… and they are not reduced…

So what?

our first basic observation …

The Fe content of red limestones
of biotic origin is low

1 to 2 %, often < 1 %

... therefore this content is not responsible
of the coloration

our second basic observation …

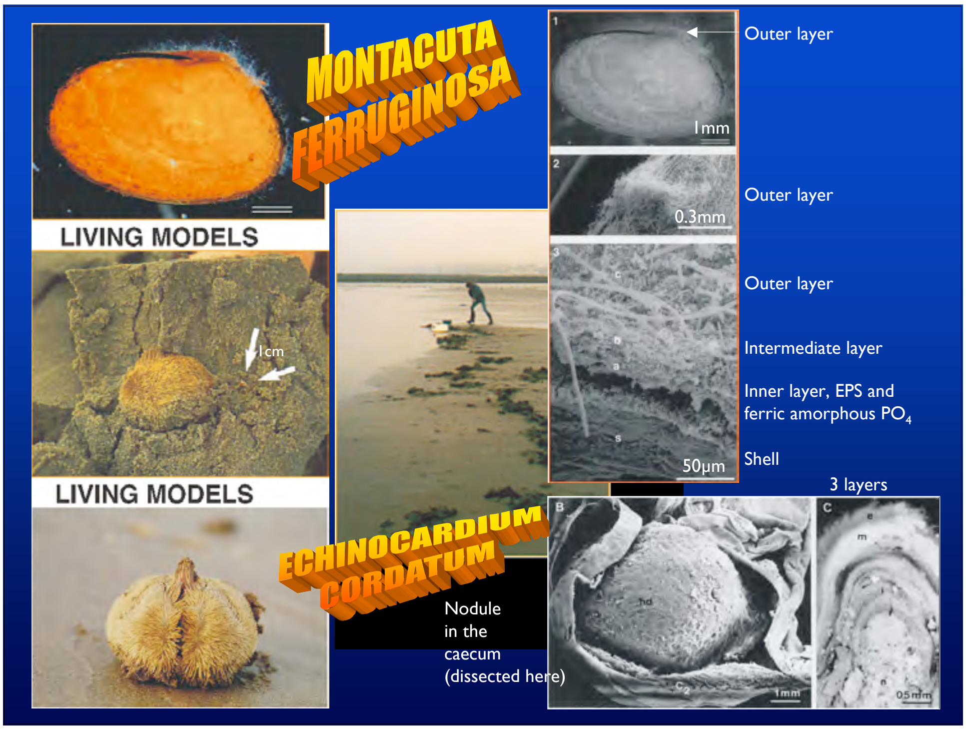
Sedimentation is in a
NORMAL OPEN MARINE
facies

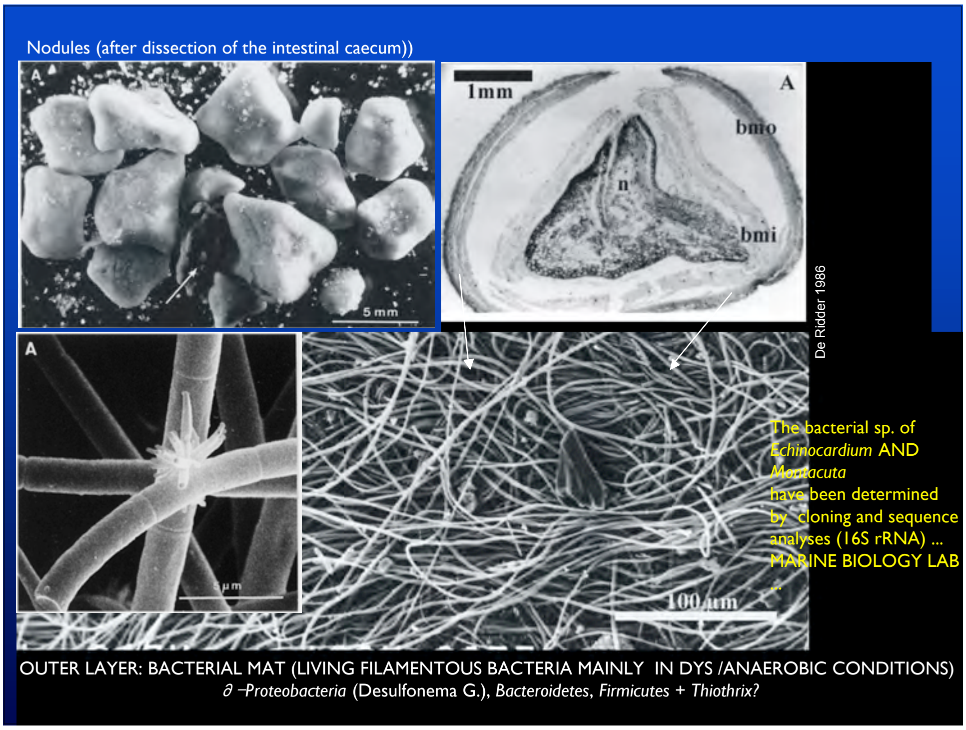
Red limestones are formed
in calm environments, with low
levels of oxygen

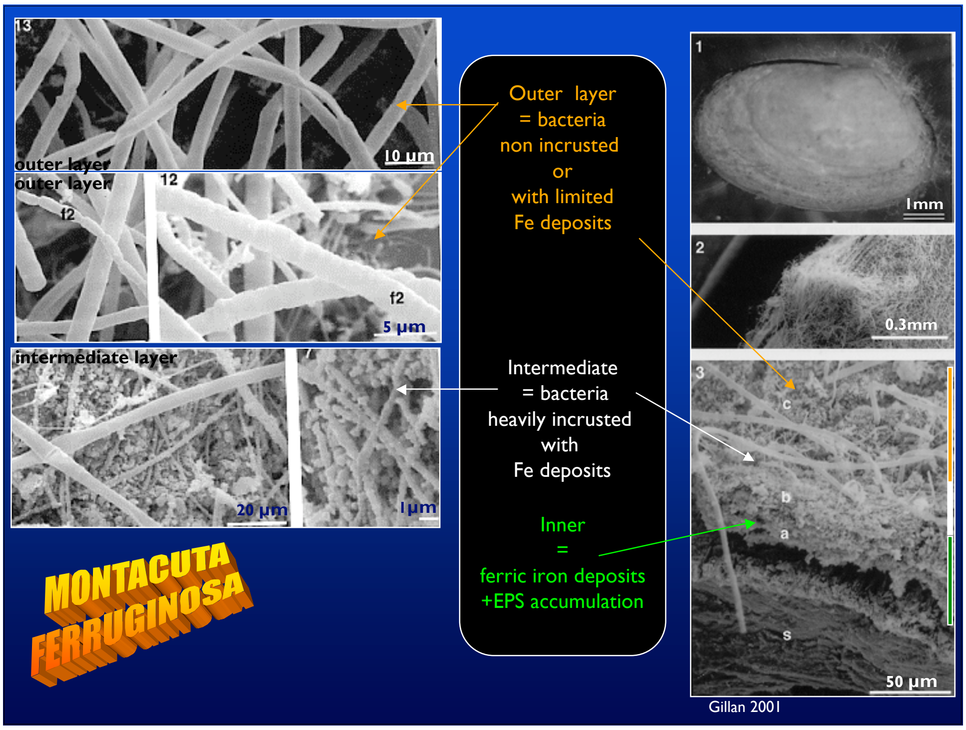
There is no direct relation between oxygen content and overall iron oxidation...
Thus the colour is not necessarily linked to shallow water marine environments where oxygen is abundant

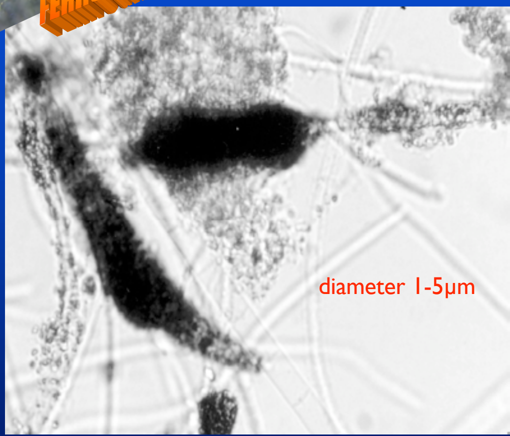
QUID ?

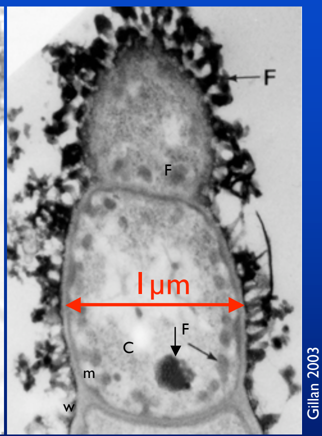

ECHINOCARDIUM CORDATUM
lives at 15 cm, and burrows the sediment (silty quartz sand)
B I O L O G Y
and its biofilms…


MONTACUTA FERRUGINOSA
LIVING MODELS
1cm
LIVING MODELS
ECHINOCARDIUM CORDATUM
Nodule in the caecum (dissected here)
1mm
Outer layer
0.3mm
Outer layer
Outer layer
Intermediate layer
Inner layer, EPS and ferric amorphous PO_4
50µm
Shell
3 layers
1mm
0.5mm


Nodules (after dissection of the intestinal caecum))
A
5 mm
1mm
A
bmo
n
bmi
De Ridder 1986
A
100 µm
The bacterial sp. of
Echinocardium AND
Montacuta
have been determined
by cloning and sequence
analyses (16S rRNA) ...
MARINE BIOLOGY LAB
...
OUTER LAYER: BACTERIAL MAT (LIVING FILAMENTOUS BACTERIA MAINLY IN DYS /ANAEROBIC CONDITIONS)
∂ -Proteobacteria (Desulfonema G.), Bacteroidetes, Firmicutes + Thiothrix?

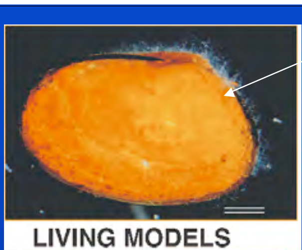

13
10 µm
outer layer
outer layer
12
f2
f2
5 µm
intermediate layer
20 µm
1µm
MONTACUTA
FERRUGINOSA
Outer layer
= bacteria
non incrusted
or
with limited
Fe deposits
Intermediate
= bacteria
heavily incrusted
with
Fe deposits
Inner
=
ferric iron deposits
+EPS accumulation
1
1mm
2
0.3mm
3
c
b
a
s
50 µm
Gillan 2001





Iron bacterial filaments
on a shell
(photonic microscopy)

Filamentous iron bacteria
(TEM)
F = Fe



Rust-coloured and ferric iron encrusted biofilm (3mm thick)

MICROBIAL MAT WITH THREE SEPARATE LAYERS

1. *Outer layer* Filamentous and coccoid bacteria, also protozoa
2. *Intermediate layer* Microbial AND mineral (heavily ferric iron-encrusted filamentous bacteria and protozoa
3. *Inner layer* Essentially mineral (ferric iron deposits), no or rare living microorganisms

GENESIS OF THE MINERAL-MICROBIAL MAT

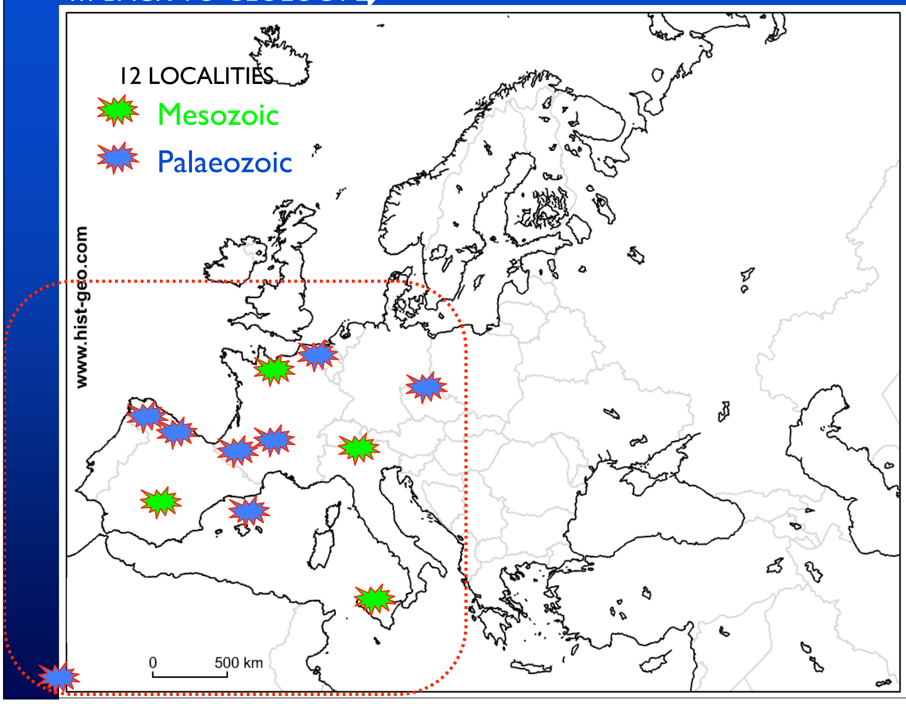
1. Ferric iron *deposition* within bacterial sheaths in the outer layer
2. *Release* and *accumulation* of heavily ferric iron-encrusted sheaths after lysis of the bacteria in the intermediary layer
3. *Degradation* of bacterial sheaths and *accumulation* of ferric iron minerals in the inner layer

ferric iron deposits = 0.05-1µm (granules, amorphous oxyhydroxide 'gel' with phosphate)

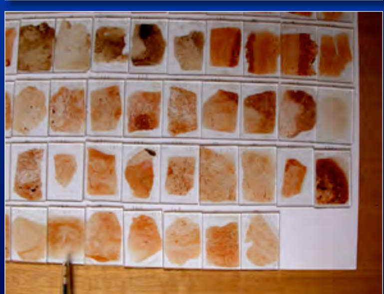
= PASSIVE PROCESS => BIOMINERALIZATION-INDUCED and BIOACCUMULATION (microbes dead or alive)

From PhD thesis David GILLAN, 1999, Marine Biology Dept, ULB (and numerous papers in Journals of Microbiology...)

… BACK TO GEOLOGY ➡ OBSERVED MICROFACIES OF DIFFERENT AGES AND LOCALITIES



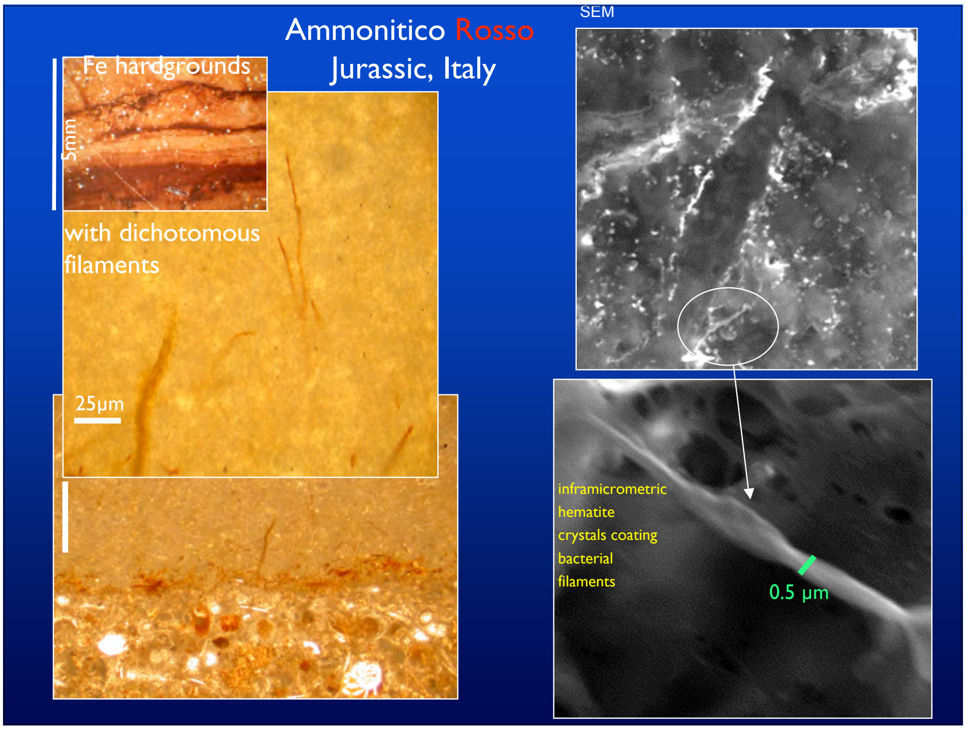
OF WHAT CONSISTS THE RED PIGMENTATION?

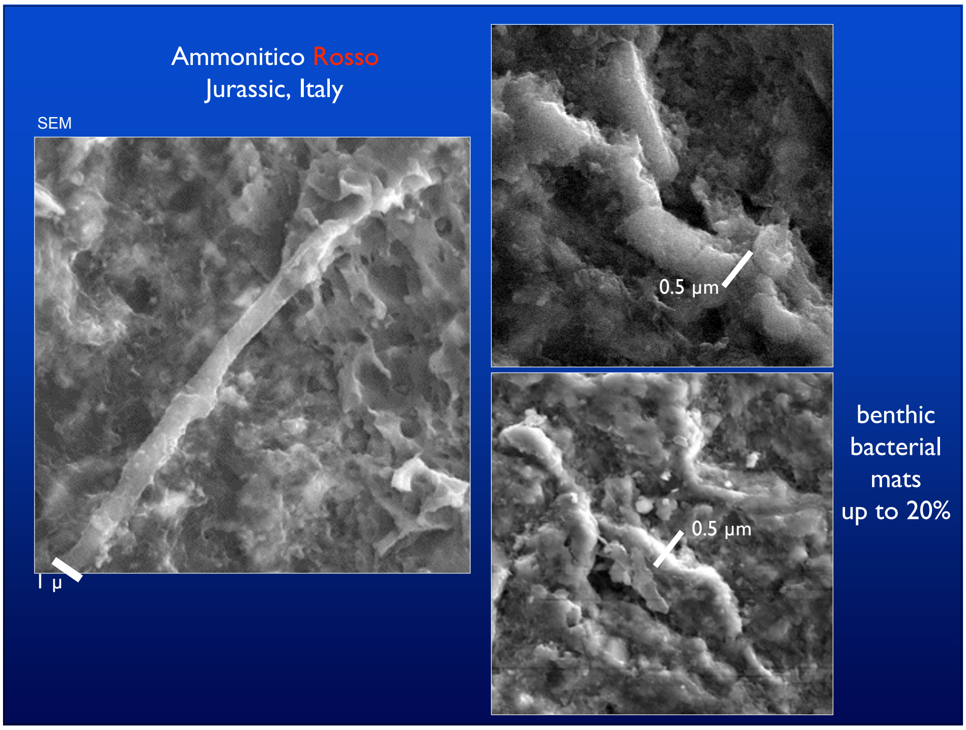


Microscopic morphologies of the iron constructions

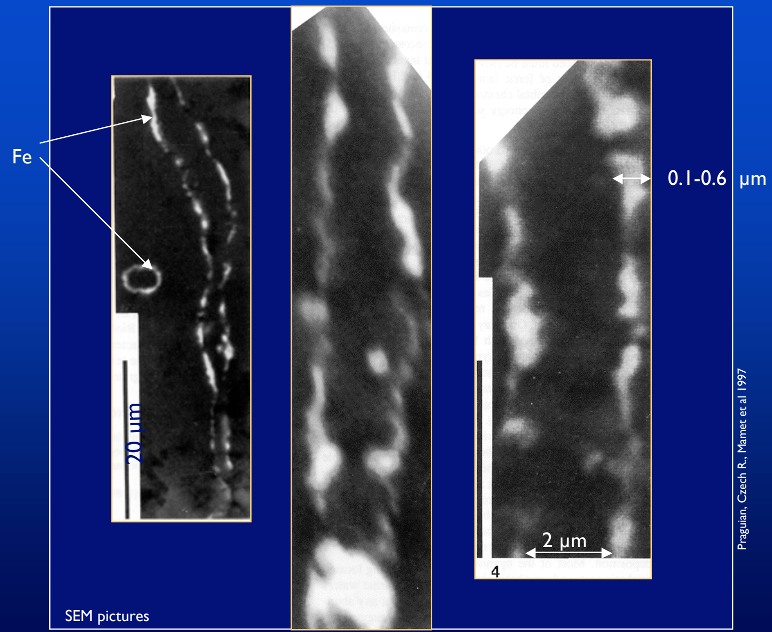
- ° infillings of original fossil cavities
- ° calcite replacement of dissolved echinoderm plates
- ° infillings of bioperforations
- ° bacterial/fungal filaments
- ° ‘hedgehogs’ and ‘erythrospheres’
- ° massive hematite/goethite coating around microfossils
- ° simple or multiple biofilms
- ° microstromatolites (exogens ou endogens, crenulated or not...)
- ° oncolites

….. *non exhaustive* ….


Ammonitico Rosso
Jurassic, Italy
SEM
Fe hardgrounds
5mm
with dichotomous
filaments
25μm
inframicrometric
hematite
crystals coating
bacterial
filaments
0.5 μm

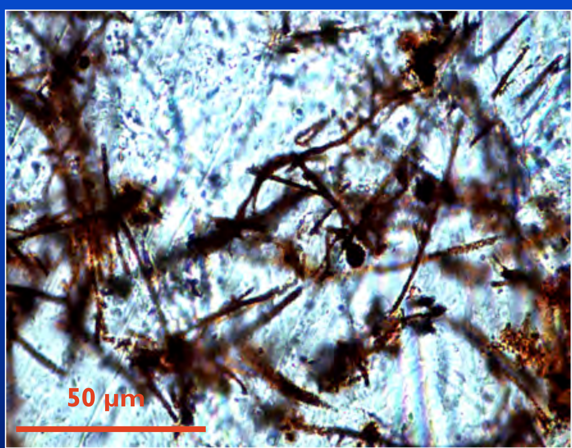

Ammonitico Rosso
Jurassic, Italy
SEM
1 μ
0.5 μm
0.5 μm
benthic
bacterial
mats
up to 20%

CZECH REPUBLIC, LOWER DEVONIAN



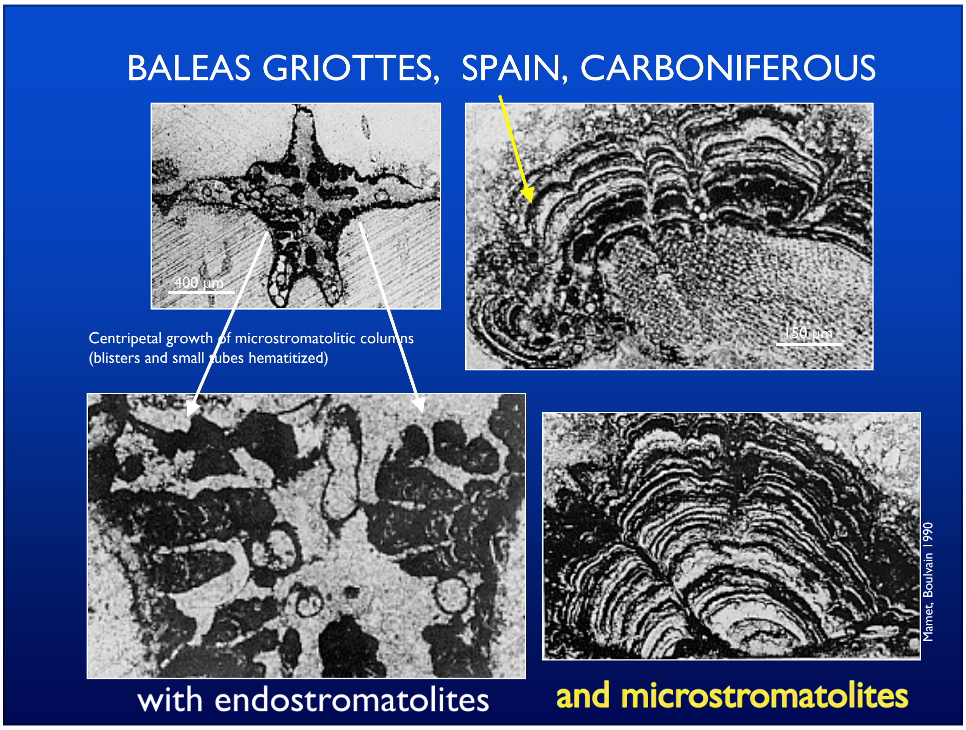
Praguian, Czech R., Mamet et al 1997

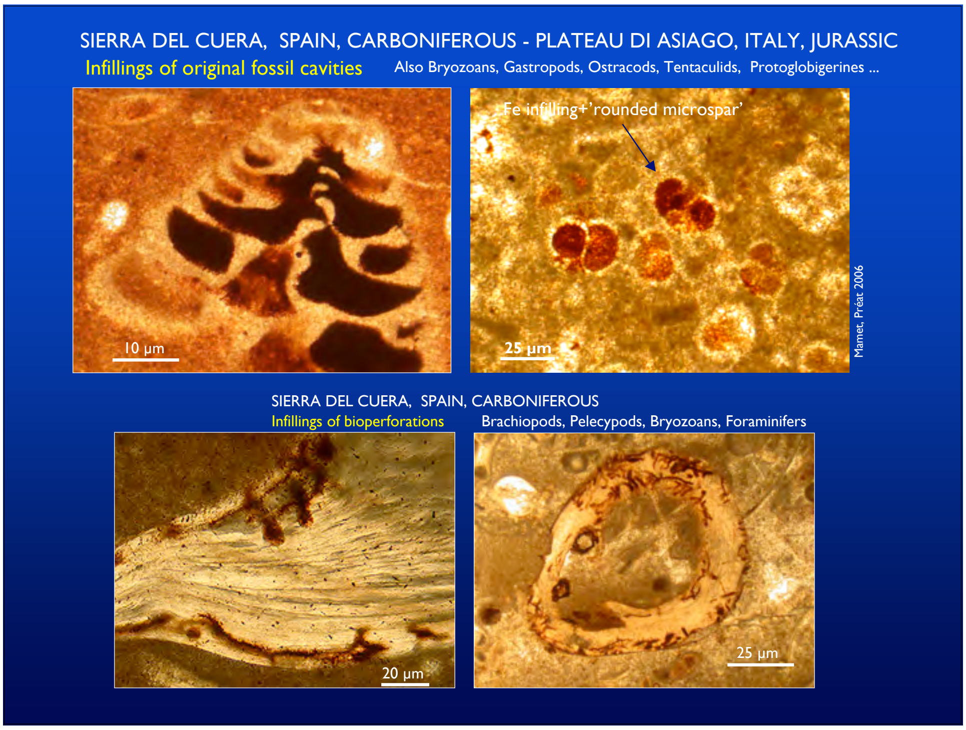
FRENCH-BELGIAN MUD MOUNDS, FRASNIAN



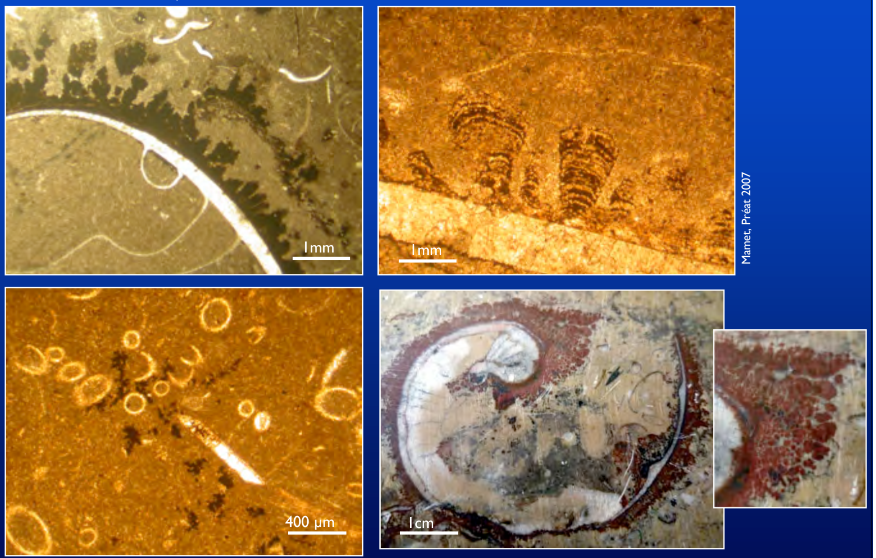
Boulvain et al. 2001

Iron-bacteria
(*Siderocapsa*-like, *Sphaerotilus-Leptothrix*-like
in the internal sediments of *Receptaculites*
Rochefontaine quarry, Franchimont, Philippeville Massif


BALEAS GRIOTTES, SPAIN, CARBONIFEROUS
400 µm
150 µm
Centripetal growth of microstromatolitic columns
(blisters and small tubes hematitized)
with endostromatolites
and microstromatolites
Mamet, Boulvain 1990


SIERRA DEL CUERA, SPAIN, CARBONIFEROUS - PLATEAU DI ASIAGO, ITALY, JURASSIC
Infillings of original fossil cavities
Also Bryozoans, Gastropods, Ostracods, Tentaculids, Protoglobigerines ...
10 µm
Fe infilling+'rounded microspar'
25 µm
Mamet, Préat 2006
SIERRA DEL CUERA, SPAIN, CARBONIFEROUS
Infillings of bioperforations
Brachiopods, Pelecypods, Bryozoans, Foraminifers
20 µm
25 µm

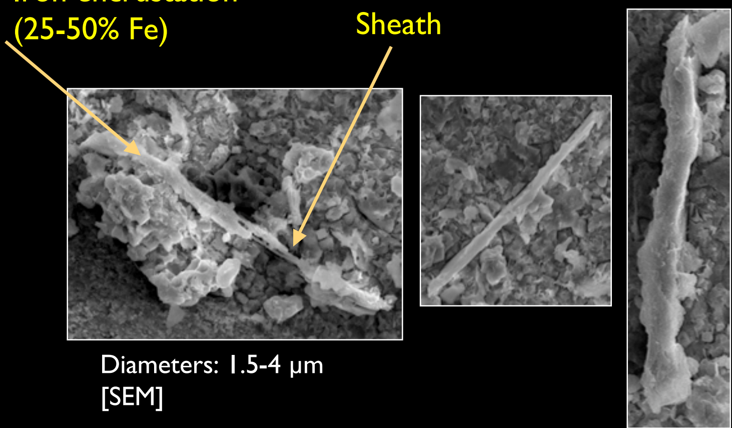
ANTI-ATLAS, MOROCCO LOWER-UPPER DEVONIAN



Mamet, Préat 2007

ANTI-ATLAS, MOROCCO LOWER-UPPER DEVONIAN

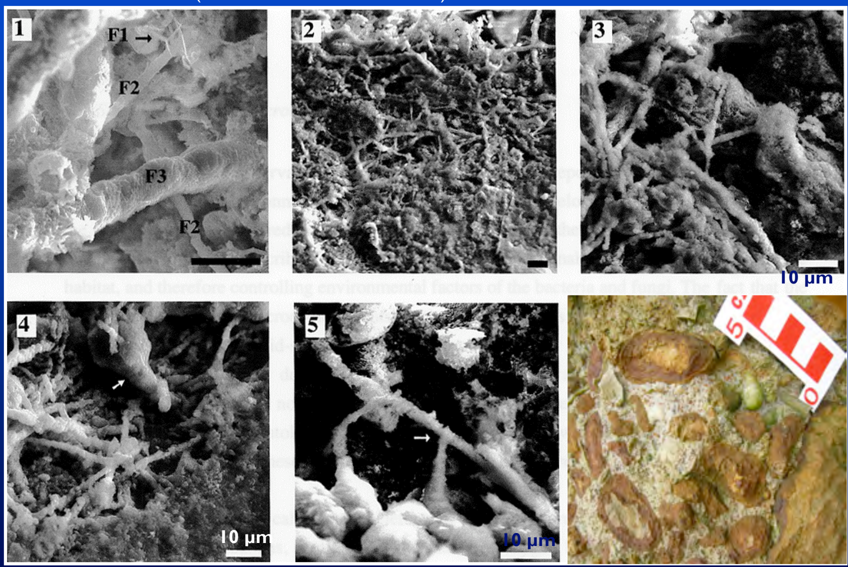
Filamentous iron bacteria



Diameters: 1.5-4 µm
[SEM]

Mamet, Préat 2007

BAJOCIAN GSSP, SAINTE-HONORINE-DES-PERTES, FRANCE
Inside a Fe-oncoid... (nucleus is a small ammonite)



Bajocian, SHP, France, Préat et al. 2000

SEM observations

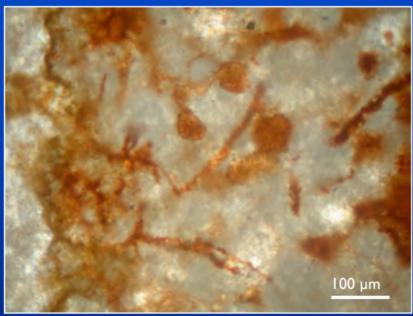
x1,000 … x35,000…

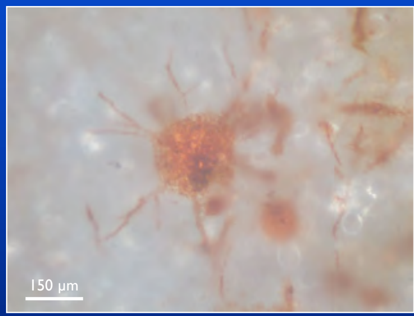
° Simple and regular filaments
° Simple filaments with regular constrictions
° Dichotomous filaments with constrictions
° Concentrations of regular sphaerules

Diameters <= 2 µm
with submicronic hematite Fe_2O_3 or goethite $FeO.OH$ in the sheath

These morphologies are suggestive of iron bacteria

Irregular filamentous forms (10’µm), sometimes forming a network and associated with spores





These morphs suggest the presence of FUNGI IMPERFECTI

First conclusion

Hematite/Goethite are not dispersed at random but follows regular patterns

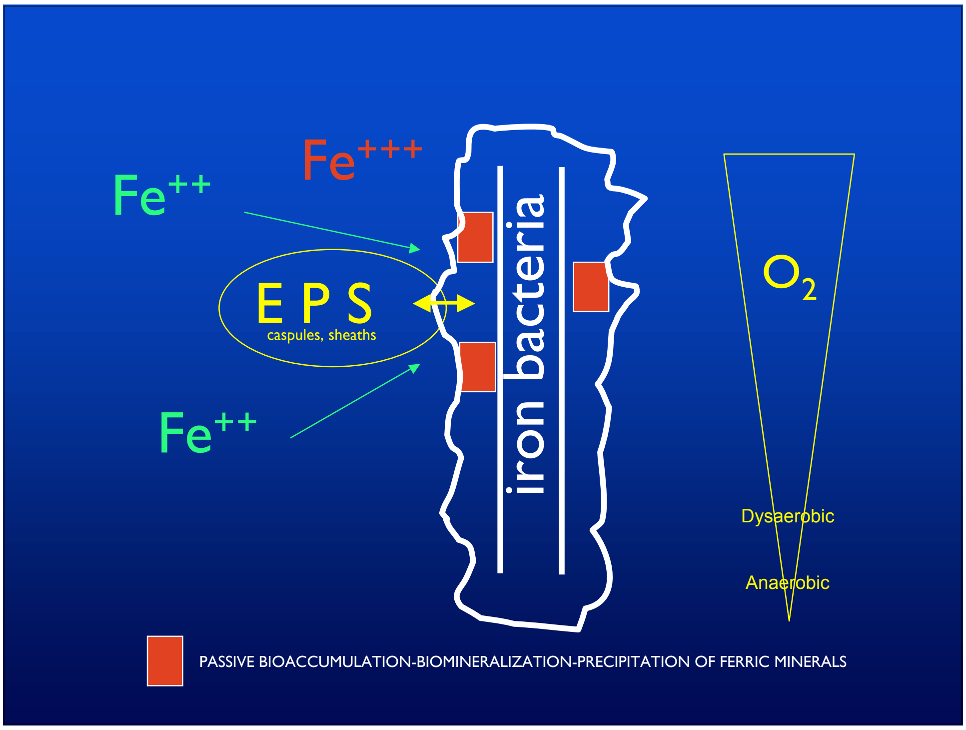
…

BIOsedimentary pathway?

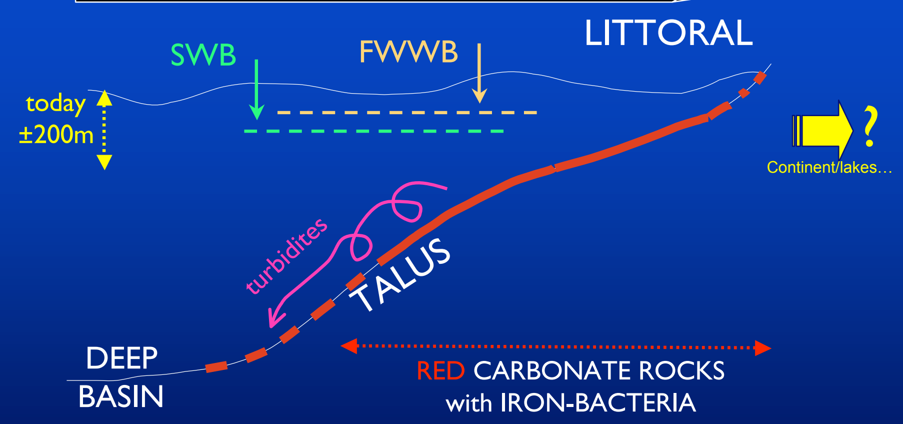
1. Today they are associated with Fe and/or Mn deposits. O_2 and pH values determine the iron solubility in aqueous solutions.

2. The neutrophile iron bacteria are associated with the oxic/anoxic interface
-Sphaerotilus, Leptothrix, Gallionella …

3. Iron biomineralization is linked to the production of EPS - exopolymeric substances = sheaths or capsules rich in polysaccharides forming the main part of the bacterial mats. The Fe^{3+} is <u>passively</u> precipitated in the EPS of the Recent bacterial films



SECOND CONCLUSION



… if iron bacteria are present, iron hydroxides are linked to oxic/anoxic gradients in poorly oxygenated waters and independent of light

CONCLUSION OF THE FIRST PART

PIGMENTATION ORIGIN
?

detrital | biological | chemical

Red pigmentation = Fe microbial activity

Microenvironmental significance
and
No paleogeographical significance

Stable isotope geochemistry of iron

- griottes Devonian S-France, Viséan N-Spain
- 'red marbles' Devonian (Frasnian), Belgium
- Ammonitico Rosso Jurassic, N-Italy, S-Spain, Sicily
- 'red marble' Devonian, Czech Republic
- red condensed series Devonian, Morocco
- red lenses in slope Carboniferous, N-Spain
- 'Oolite Ferrugineuse de Bayeux' mid-Jurassic Normandy

GEOLOGY

Echinocardium cordatum
Montacuta ferruginosa

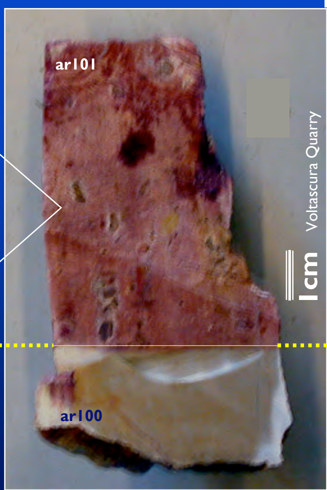
BIOLOGY

What about Fe-isotopes?

^{56}Fe 91.76%
^{54}Fe 5.84 %
^{57}Fe 2.12 %
^{58}Fe 0.28 %

4 outcrops (60 km)

Fe-hardgrounds
Filamentous Fe-microbes
Fe-microstromatolites
Fe-biofilms



RAI

U. Bajocian - L. Callovian

hiatus

Calcari Grigi

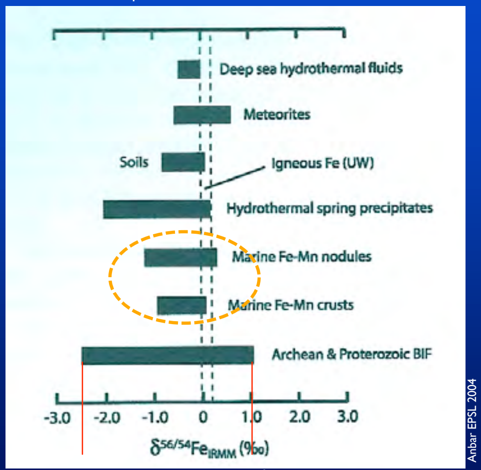
Hettangian - Domerian

$$\delta^{56}Fe = (^{56}Fe/^{54}Fe_{sample}/^{56}Fe/^{54}Fe_{IRMM014} - 1) * 10^3$$
$$\delta^{57}Fe = (^{57}Fe/^{54}Fe_{sample}/^{57}Fe/^{54}Fe_{IRMM014} - 1) * 10^3 \quad (‰)$$

What about Fe-isotopes?

$\delta^{56}Fe = (^{56}Fe/^{54}Fe_{sample}/^{56}Fe/^{54}Fe_{IRMM014} - 1) * 10^3$

Fe isotope variations in natural materials



Mass-dependent isotope variations of Fe in the rock record span a range of 4 ‰ in $^{56}Fe/^{54}Fe$ ratio.

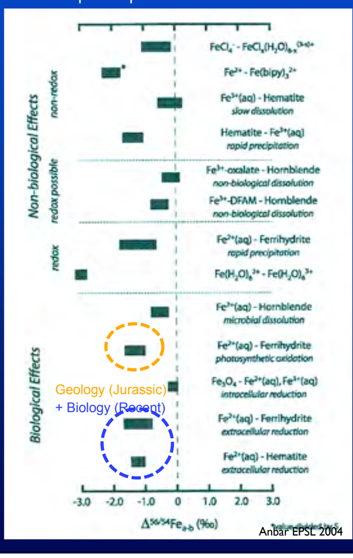
---

^{56}Fe 91.76%
^{54}Fe 5.84 %
^{57}Fe 2.12 %
^{58}Fe 0.28

What about Fe-isotopes?

$\delta^{56}Fe = (^{56}Fe/^{54}Fe_{sample}/^{56}Fe/^{54}Fe_{IRMM014} - 1) * 10^3$

Fe isotope fractionation effects
Natural mass-dependant variation in isotopic composition of Fe are small



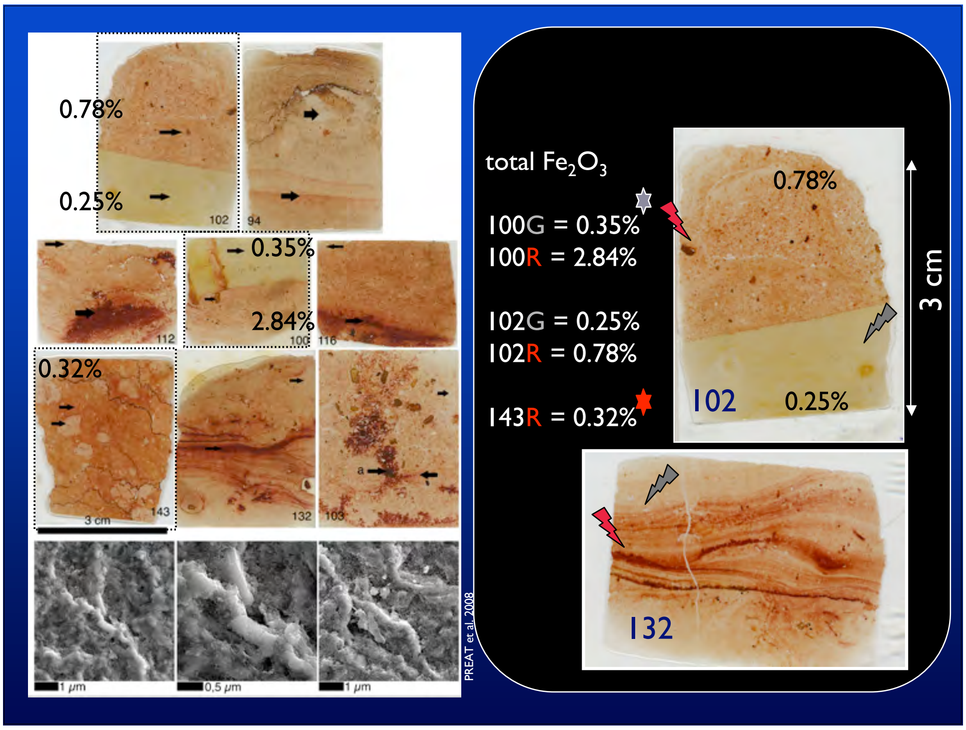
EXPERIMENTS IN BIOLOGICAL SYSTEMS

$^{56}Fe/^{54}Fe$ ratios of the (solid) ferrihydrite precipitate with an 1.5‰ increase relative to the aqueous Fe(II) source

AND

the net Fe isotope fractionation is remarkably similar (1.3% more negative) to that obtained for dissimilatory Fe(III) reducing bacteria

SO, WHAT IN NATURAL SYSTEMS (GEOLOGY AND BIOLOGY)?

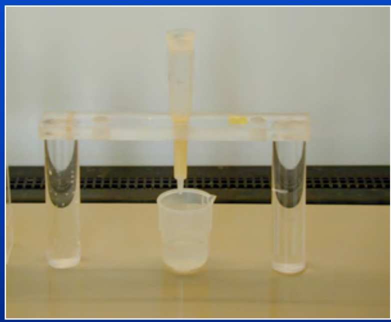


Sample preparation

A small electric drill with a titanium drill head was used to extract powder from individual grey and red layers
(25 mg for each sample)

Analytical Procedure

- Bulk sample dissolution
- Leaching (HCl 3M for 3h at 50°C) in order to remove the carbonate phases without dissolving the other phases (silicates, oxides)



Separation of Fe by one-step ion-exchange chromatography

Needs iron separation and purification...

Iron Isotopic Analyses by Nu Plasma MC-ICP-MS

Cetac Aridus

Electrostatic Analyzer

Magnet

Collector block
(12 Faraday cups)

Dry plasma: Aridus desolvating sample introduction system
Minimalizing interferences ArOH: mass 57, ArO: mass 56,ArN: mass 54

... an analysis takes about 9 minutes, and consumes about 1ml of sample or 400 ng of Fe

Analytical conditions

- $\delta^{56}Fe = (^{56}Fe/^{54}Fe_{sample}/^{56}Fe/^{54}Fe_{IRMM014} - 1) \times 10^3$
- Simultaneous external normalization (Cu-doping method in dynamic mode) and standard-sample bracketing with the IRMM014 reference material;
- Cr correction on mass 54;
- Every sample in duplicate;
- Long-term accuracy and reproducibility of 0.15 ± 0.06 ‰ for $\delta^{56}Fe$
- (1 sigma, n = 21) for a basalt relative to IRMM014.

Analysis : 160 €/sample
Per sample: 10 days (dissolution, separation) + 2 days (ICP-MS)/3 samples
Optimal timing: 15 days -1 month for 2 or 3 samples

GEOCHEMICAL STUDY

Voltascura + Forte di Campo Luserna
RAI, 25 samples

- $CaCO_3$, Mg, Fe, Mn, Sr (whole rock and selective)
- $\delta^{18}O$-$\delta^{13}C$ (selective microdrillings)

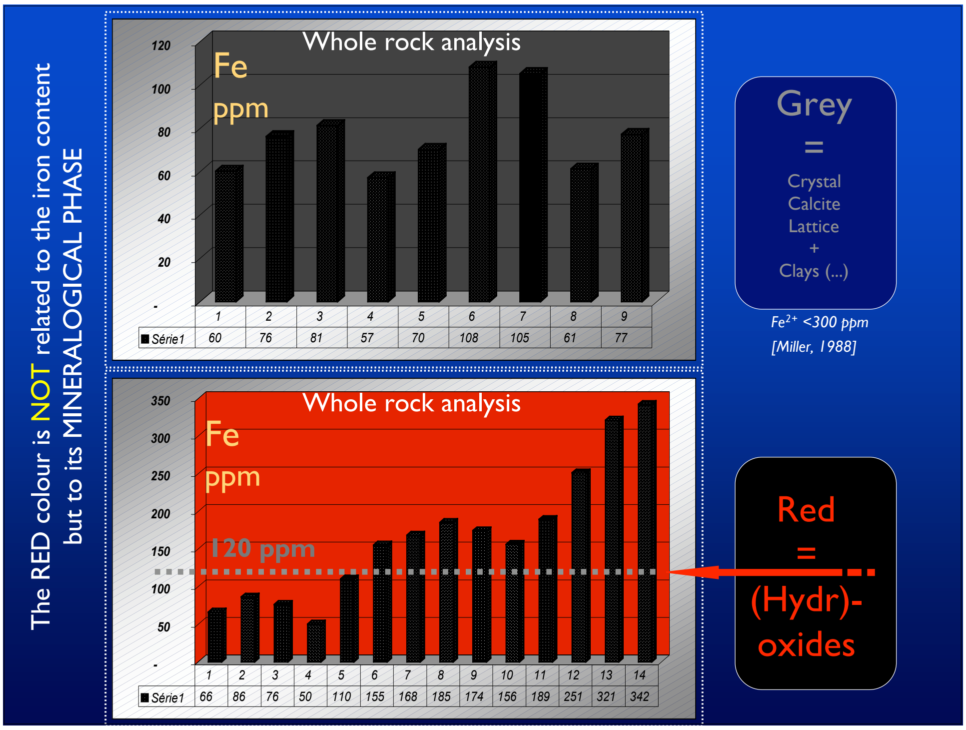
\+

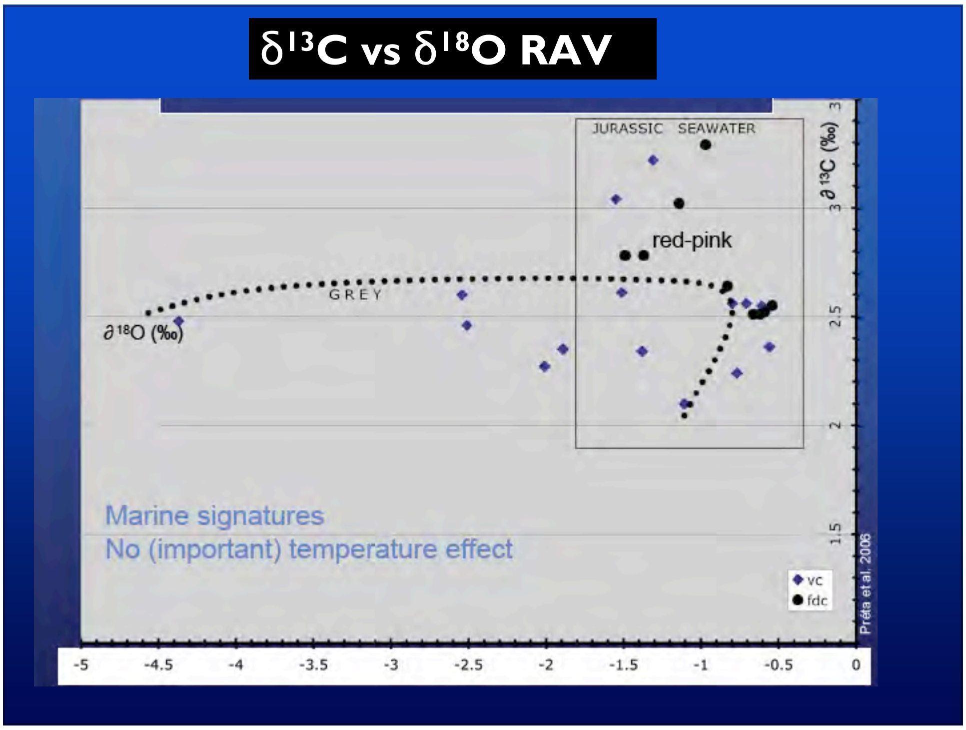
17 samples for iron isotopes
(non selective and selective)

\+

cathodoluminescence

Red
Pink
Grey


The RED colour is NOT related to the iron content
but to its MINERALOGICAL PHASE
Whole rock analysis
Fe
ppm
120
100
80
60
40
20
-
1 2 3 4 5 6 7 8 9
Série1 60 76 81 57 70 108 105 61 77
Grey
=
Crystal
Calcite
Lattice
+
Clays (...)
Fe2+ <300 ppm
[Miller, 1988]
Whole rock analysis
Fe
ppm
120 ppm
350
300
250
200
150
100
50
-
1 2 3 4 5 6 7 8 9 10 11 12 13 14
Série1 66 86 76 50 110 155 168 185 174 156 189 251 321 342
Red
=
(Hydr)-
oxides


δ13C vs δ18O RAV
JURASSIC SEAWATER
red-pink
GREY
∂18O (‰)
∂13C (‰)
Marine signatures
No (important) temperature effect
vc
fdc
Préta et al. 2006
-5 -4.5 -4 -3.5 -3 -2.5 -2 -1.5 -1 -0.5 0
3 2.5 2 1.5

FIRST CONCLUSIONS

◊ The analyzed carbonates are pure: 88-98% [$CaCO_3$]

◊ No relation between Mn and Fe contents
===> early diagenetic mobility of Mn
confirmed by cathodoluminescence analysis
(not shown here) and SEM on Mn-oncoids (Sicily) (*id.*)

◊ Very low Sr contents (50-100 ppm)
===> no aragonitic precursor?

◊ No meteoric influence

The RED colour is not related to the iron content
but to its MINERALOGICAL PHASE

First preliminary Results were discouraging!

WHOLE R-ROCK	$\delta^{56}Fe$ ‰	WHOLE G-ROCK	$\delta^{56}Fe$ ‰		Δ R-G ‰
#100/101R	-0.31	#100/101G	-0.28	=	-0.03
#102R	-0.06	#102G	-0.27	>	0.21
#160R	-0.39	#160G	-0.14	<	-0.25

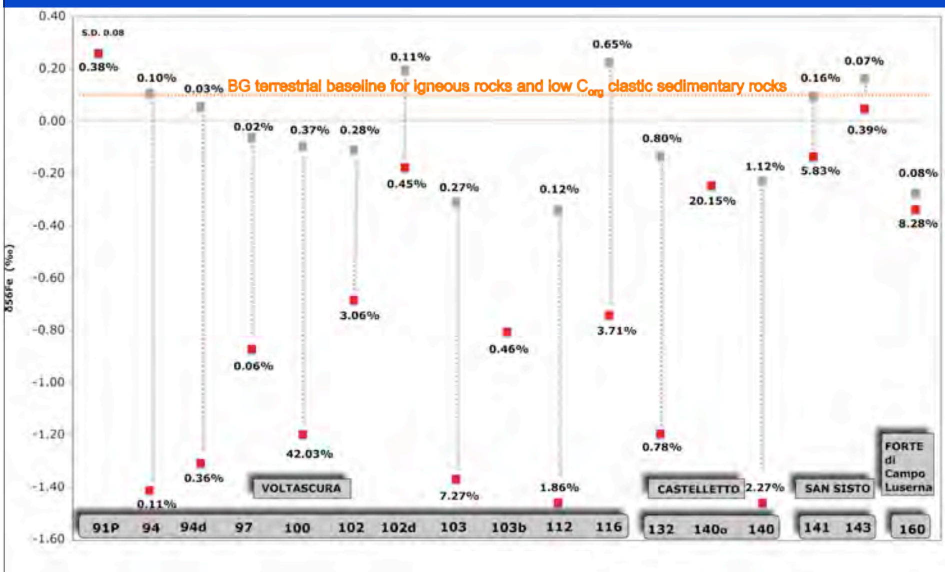
3M HCl/3h/50°C leach + total digestion residue

Hematite XRD

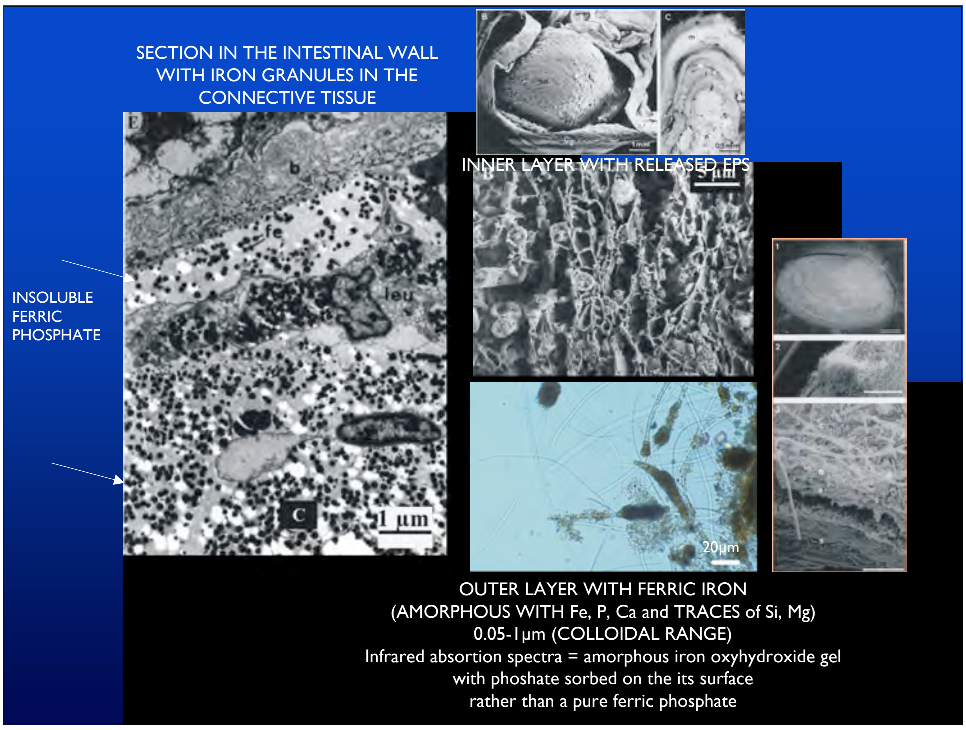
	δ56Fe ‰	Δ LEACH-RES ‰		δ56Fe ‰	Δ LEACH-RES ‰	Δ R-G ‰
#100/101R-LEACH	-1,11	0,08	#100/101G-LEACH	-0,21	-0,12	-0,90
#100/101R-RES	**-1,19**		**#100/101G-RES**	**-0,09**		**-1,10**
#102R-LEACH	-0,38	0,30	#102G-LEACH	-0,25	-0,14	-0,13
#102R-RES	**-0,68**		**#102G-RES**	**-0,11**		**-0,57**
#160R-LEACH	-0,02	0,32	#160G-LEACH	-0,39	-0,12	0,37
#160R-RES	**-0,34**		**#160G-RES**	**-0,27**		**-0,07**
Average Δ LEACH-RES		0,23			-0,13	
stdev		0,13			0,01	

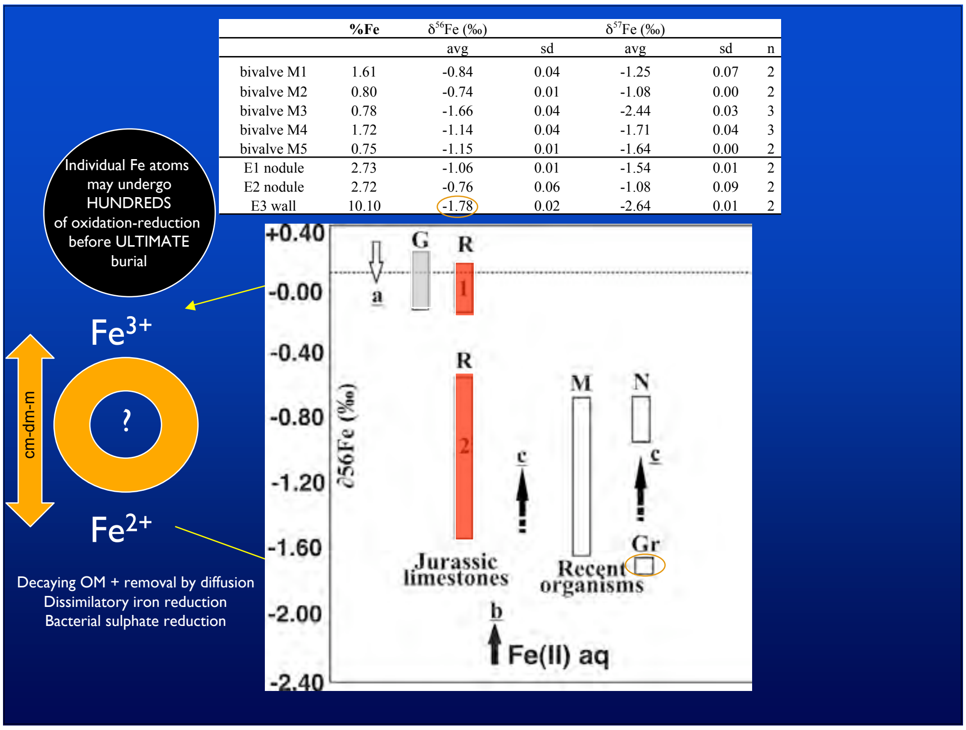
+ 0.1M HCL/18h/25°C and 3M HCL/1h/50°C and 3M HCL/2h/25°C

VITAL EFFECT? or (BIO)FRACTIONATION FROM A MORE NEGATIVE SOURCE?

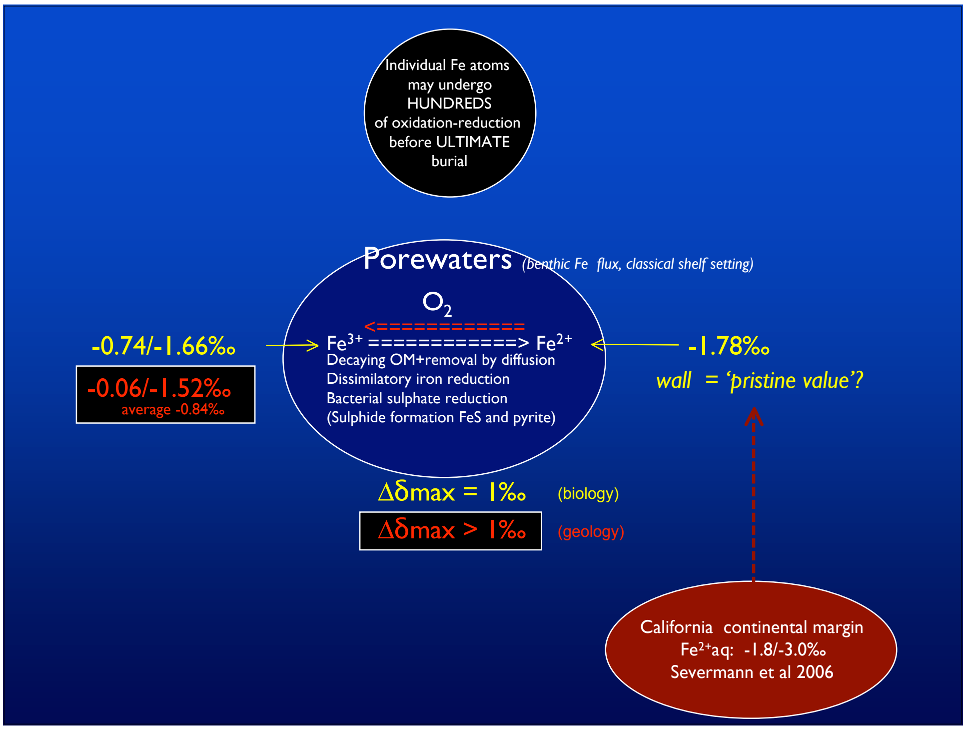



TRY TO SOLVE
FROM THE RECENT?
LIVING MODELS
Going back again to biology…


SECTION IN THE INTESTINAL WALL
WITH IRON GRANULES IN THE
CONNECTIVE TISSUE
INSOLUBLE
FERRIC
PHOSPHATE
INNER LAYER WITH RELEASED EPS
1 µm
20µm
OUTER LAYER WITH FERRIC IRON
(AMORPHOUS WITH Fe, P, Ca and TRACES of Si, Mg)
0.05-1µm (COLLOIDAL RANGE)
Infrared absorption spectra = amorphous iron oxyhydroxide gel
with phoshate sorbed on the its surface
rather than a pure ferric phosphate



	%Fe	$\delta^{56}Fe$ (‰)		$\delta^{57}Fe$ (‰)		
		avg	sd	avg	sd	n
bivalve M1	1.61	-0.84	0.04	-1.25	0.07	2
bivalve M2	0.80	-0.74	0.01	-1.08	0.00	2
bivalve M3	0.78	-1.66	0.04	-2.44	0.03	3
bivalve M4	1.72	-1.14	0.04	-1.71	0.04	3
bivalve M5	0.75	-1.15	0.01	-1.64	0.00	2
E1 nodule	2.73	-1.06	0.01	-1.54	0.01	2
E2 nodule	2.72	-0.76	0.06	-1.08	0.09	2
E3 wall	10.10	-1.78	0.02	-2.64	0.01	2



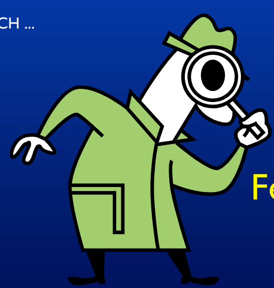
CONCLUSIONS (PRELIMINARY)

1. The red color is related to the submicronic hematite/goethite dispersed in the matrix. The hematite is a result of the activity of iron bacteria and fungi that precipitated Fe-Mn hydroxydes at dysoxic sediment-water interfaces
2. The iron contents are comparable in the red, pink and grey facies
3. The iron bacteria have passively 'fractionated' the iron isotopes at an infra-millimetric scale
4. Comparison of the Fe isotopic compositions of the 'biominerals' in the Recent organisms and the red-grey Jurassic facies suggest an isotopic biofractionation of at least +0.7 ‰, and around 1‰
5. The Recent sea-urchin and the bivalve thrive in similar microenvironmental conditions as the microorganisms of the Jurassic condensed red facies.

A biosignature is probably present….

THANK YOU!

FUTURE RESEARCH …



$Fe^{2+}aq$

This research has been carried out with

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