

STABLE ISOTOPE GEOCHEMISTRY AND MINERALOGY OF SOME MESSINIAN EVAPORITIC CARBONATE OUTCROPS IN NORTH EASTERN SICILY (ITALY)

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Abstract: Stable isotope ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) and SEM analyses were performed on 40 samples collected in the Upper Miocene (Messinian) evaporitic carbonate sequences (locally named *Calcare di Base*) of North Eastern Sicily. To recognize the carbonate parageneses less affected by post-depositional exchange phenomena that are representative of the paleoenvironment, the isotopic informations were integrated with SEM observations. The samples with extensive dissolution - reprecipitation evidences showed the most negative $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values, while the samples having a relatively preserved micritic framework showed more positive values. In the literature concerning the *Calcare di Base*, the wide dispersion of the isotopic values has been interpreted as due to random syngenetic inputs of meteoric water in the depositional environment. Our results put forward the hypothesis that exchange processes in diagenetic or vadose-phreatic environment might also have happened.

Key words: isotope exchange process, evaporitic carbonate, Messinian, Mediterranean area.

Introduction

In recent years, in order to establish the depositional paleoenvironment of the different lithotypes, $^{18}\text{O}/^{16}\text{O}$ and $^{13}\text{C}/^{12}\text{C}$ isotopic ratios of the evaporitic sequences cropping out in the Mediterranean area have been extensively investigated (Dessau et al. 1959, 1962; Pierre 1974; Pierre & Catalano 1975; Cita et al. 1978; Longinelli 1979; Censi et al. 1980; Mc Kenzie 1985; Belanca et al. 1986).

Decima et al. (1988) examined in detail the origin of the evaporitic limestones belonging to the *Calcare di Base* member of the Messinian *Gessoso-Solfifera Formation*. They collected 95 samples from 51 different localities distributed over the Central Sicily. Because of their fine-grained feature, these authors performed stable isotope studies on bulk samples. Their data show a great variability in both the $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values that also seem to be related to the aragonite content of each sample. However, Bellia & Censi (1989) found a wide scattering of the isotopic ratios even in the samples with holocalcitic composition. These results may be interpreted as an effect of a post-depositional isotope exchange process, but also as random differences in the depositional environment (e.g. fresh water input).

The aim of this study is to supply new isotopic data on the peripheral *Calcare di Base* outcrops and to identify (by SEM observations) the samples that might have maintained the isotopic memory of their own paleoenvironment.

Geological outline

During the Upper Miocene (Messinian) the Mediterranean basin progressively evolved to an evaporitic environment. In such condition the *Gessoso Solfifera Formation* (Ogniben 1957; Richter-

Bernburg 1973; Catalano 1986) was formed. In Central Sicily, Decima & Wezel (1971) recognized two different sequences.

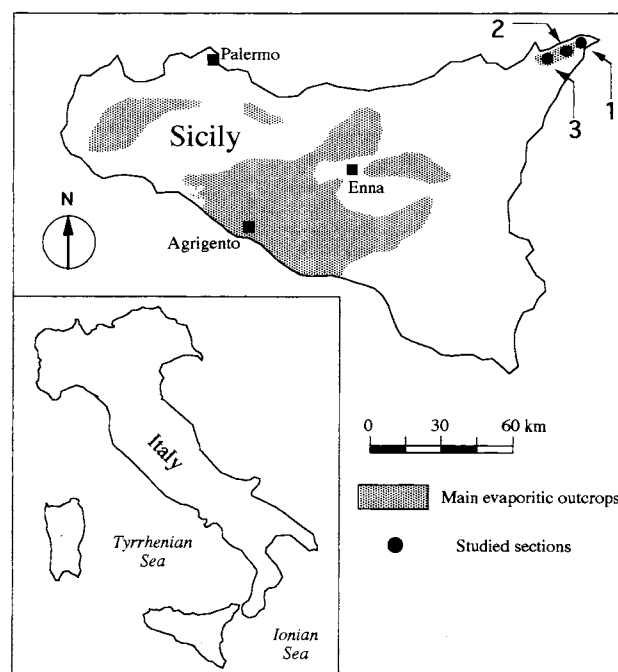


Fig. 1. Schematic map showing the locations of the "Gessoso-Solfifera" Formation in Sicily (shaded pattern). Solid dots represent the sampled "Calcare di Base" outcrops: 1 = Castanea (CST); 2 = Venetico (VNT); 3 = Pace del Mela (PDM).

The first, called *Lower Evaporitic Complex* includes: 1) 5 to 50 m of diatomite deposits; 2) up to 60 m of evaporitic limestone (*Calcare di Base*), sometimes with embedded marl levels; 3) selenite and laminar microcrystalline gypsum, often with embedded marl levels; 4) halite and potassic salts deposits or gypsum arenites.

The second, called *Upper Evaporitic Complex* only consists of gypsum and terminates with a transgressive conglomerate. Both the complexes are followed by calcareous marls, related to the restoring of pelagic conditions.

The area concerned in this work is located in the NE Sicily. Only beds of gypsum and evaporitic limestone with limited thickness crop out in this part of Sicily. They lie unconformably on the Paleozoic metamorphic rocks, schists and gneisses.

Materials and methods

Fourty samples were collected from three different sites of the North - Eastern Sicily (Fig. 1): 1 - Castanea (CST) 38° 15' 05" Lat. N, 15° 31' 07" Long. E; 2 - Venetico (VNT) 38° 11' 58" Lat. N, 15° 22' 44" Long. E; 3 - Pace del Mela (PDM) 38° 10' 16" Lat. N, 15° 18' 11" Long. E. The outcrops vary from 6 m (Venetico) to 20 m (Castanea) in thickness, hence in each section a dense specimens collection was performed. The three sections are composed of beds of whitish-yellowish massive limestone, that are fine to very fine-grained and weakly to strongly lithified. They are covered by an unconsolidated soil, generally less than 60 cm in thickness, consisting of weathered rock fragments in clay matrix. The fresh rock is moderately to highly cracked and commonly shows a vuggy feature. In some places it contains thinly bedded marly limestone layers, and a sandy level was also found.

X-ray diffraction (Philips 1400/10) was performed by $\text{CuK}\alpha$ (Ni). Photomicrographs in polarized light were carried out by a Zeiss Axiomat microscope. The secondary electron images as well as the X-ray elemental maps were obtained using a Cambridge Instruments Stereoscan 360 SEM, fitted with a Link Analytical 10/55S energy dispersive unit (accelerating voltage: 20 KV). Samples were sputter-coated with carbon.

For the isotopic analyses the powdered calcite samples were heated under vacuum at 400 °C for 20'; then they were reacted with 100 % H_3PO_4 at 25 °C for 24 h in a on-line gas extraction system. Strontianite samples were prepared according to O'Neil et al. (1969). Isotopic analyses were carried out by a Varian Mat 250 mass spectrometer. Results are expressed in δ versus PDB-1 standard. The standard deviation of the measurements was ± 0.05 ‰ (1σ) for oxygen and ± 0.02 ‰ (1σ) for carbon isotopes.

Results

Lithology and isotopic compositions are summarized in Tab. 1 - XRD indicates that the carbonate fraction is completely constituted of calcite. Its MgCO_3 content is within 3 %. It was obtained by measuring the 2θ value of 104 with respect the 111 reflection of a fluorite internal standard (Griffin 1971). Most of the samples are characterized by notably variable amounts of clay minerals, quartz and feldspar. Only two samples show the presence of strontianite (VNT-7 and VNT-8).

Trends of the isotopic composition along the studied sequences are shown in Fig. 2. In general, $\delta^{18}\text{O}$ values range from -7 ‰ and +5 ‰, while $\delta^{13}\text{C}$ ranges between -7 ‰ and +3 ‰. In the sections n° 1 and n° 2 the most negative values were found in the marly

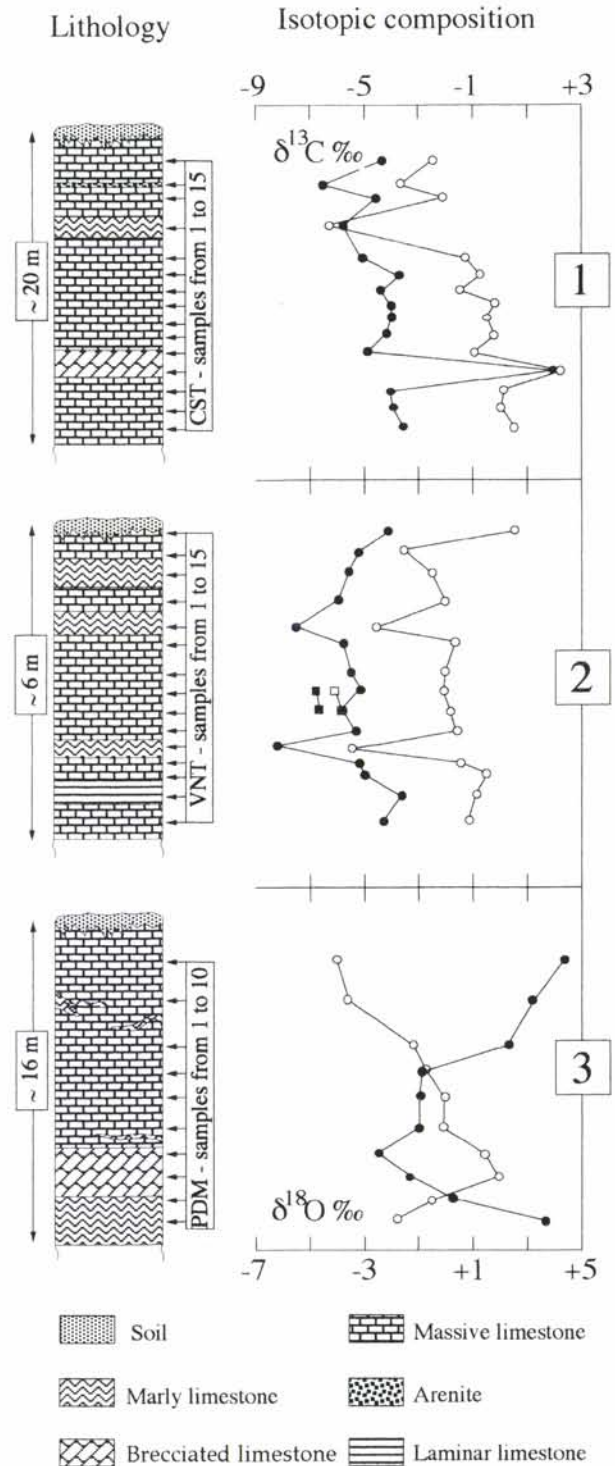


Fig. 2. Isotopic data plotted against generalized stratigraphic column for the studied sections.

Arrows localize the collected samples. 1 = Castanea; 2 = Venetico; 3 = Pace del Mela. Squares: strontianite; circles: calcite. Open symbols: $\delta^{13}\text{C}$; full symbols $\delta^{18}\text{O}$.

limestone layers. In these sections a positive correlation between $^{18}\text{O}/^{16}\text{O}$ and $^{13}\text{C}/^{12}\text{C}$ ratios was also observed. On the contrary, samples from section 3 showed an inverse evolution of $\delta^{18}\text{O}$ with respect to $\delta^{13}\text{C}$ and any relation between isotopic values and the presence of marly limestone levels was not found.

Optical and SEM observations were suitable for showing off the main structural and textural aspects of the studied limestones. The calcitic matrix has a micritic structure with diffuse micro-sparite recrystallization. An aggrading neomorphism is mainly developed close to the dissolution cavities. In some cases regular shaped vugs, from rectangular to cubic, were recognized (Fig. 3A). According to Ogniben (1957) they were interpreted as ghosts of halite crystals. Decima et al. (1988) think these voids were originally filled by individual displacive halite crystals and an early lithification of the carbonate matrix permitted the preservation of the original morphology. The presence of peloidal cryptocrystalline allochems, circular to elliptic in shape is also characteristic (Fig. 3B). They might have had an organic origin (Tamajo 1961; Tamajo - pers. commun.). On the basis of crystal size and morphology SEM images allowed us to recognize three generations of calcite: *a* - anhedral microcrystalline calcite (Fig. 4A); *b* - subhedral and euhedral microsparite and sparite, grown as rhombohedral crystals (Fig. 4B) or as rhombohedral and scalenohedral composite forms (Figs. 4C and 4D); *c* - euhedral well developed calcite ($\geq 50\mu\text{m}$) filling the largest fractures (Fig. 4E). As will be seen below, the outstanding morphology of the constituent calcite is significantly related to the isotope composition of each sample, reflecting the intensity of more or less recent exchange phenomena. Stro nianite, when detected, grows as fibrous aggregates of prismatic crystals in the void spaces between calcite granules (Fig. 5).

Table 1: Summary of the lithological and isotopic characteristics.

Sample	Lithology	Isotopic composition in ‰ vs. PDB-1			
		CaCO ₃		SrCO ₃	
		$\delta^{18}\text{O}$	$\delta^{13}\text{C}$	$\delta^{18}\text{O}$	$\delta^{13}\text{C}$
<i>Section n° 1, Castanea</i>					
CST 1	massive limestone	-1.57	+0.47		
CST 2	"	-1.90	+0.10		
CST 3	"	-2.02	+0.14		
CST 4	brecciated limestone	+4.01	+2.44		
CST 5	"	-2.81	-0.93		
CST 6	massive limestone	-2.08	-0.31		
CST 7	"	-1.90	-0.39		
CST 8	"	-2.04	-0.18		
CST 9	"	-2.37	-1.43		
CST 10	"	-1.82	-0.70		
CST 11	"	-3.07	-1.19		
CST 12	marly limestone	-3.71	-6.32		
CST 13	massive limestone	-2.68	-2.06		
CST 14	arenite	-4.46	-3.79		
CST 15	massive limestone	-2.36	-2.76		
<i>Section n° 2, Venetico</i>					
VNT 1	massive limestone	-2.44	-1.16		
VNT 2	laminar limestone	-1.65	-0.94		
VNT 3	massive limestone	-3.01	-0.71		
VNT 4	"	-3.21	-1.40		
VNT 5	marly limestone	-6.31	-5.44		
VNT 6	massive limestone	-3.24	-1.53		
VNT 7	"	-3.68	-1.87	-4.76	-5.81
VNT 8	"	-3.10	-2.03	-4.91	-6.02
VNT 9	"	-3.28	-1.96		
VNT 10	"	-3.36	-1.69		
VNT 11	marly limestone	-5.51	-4.76		
VNT 12	massive limestone	-4.01	-1.89		
VNT 13	marly limestone	-3.72	-2.42		
VNT 14	massive limestone	-3.35	-3.59		
VNT 15	"	-2.33	+0.70		
<i>Section n° 3, Pace del Mela</i>					
PDM 1	marly limestone	+3.76	-3.91		
PDM 2	"	+0.45	-2.46		
PDM 3	brecciated limestone	-1.07	-0.01		
PDM 4	"	-2.35	-0.68		
PDM 5	massive limestone	-0.84	-2.14		
PDM 6	"	-0.77	-1.96		
PDM 7	"	-0.73	-2.85		
PDM 8	"	+2.49	-3.18		
PDM 9	"	+3.37	-5.79		
PDM 10	"	+4.59	-6.11		

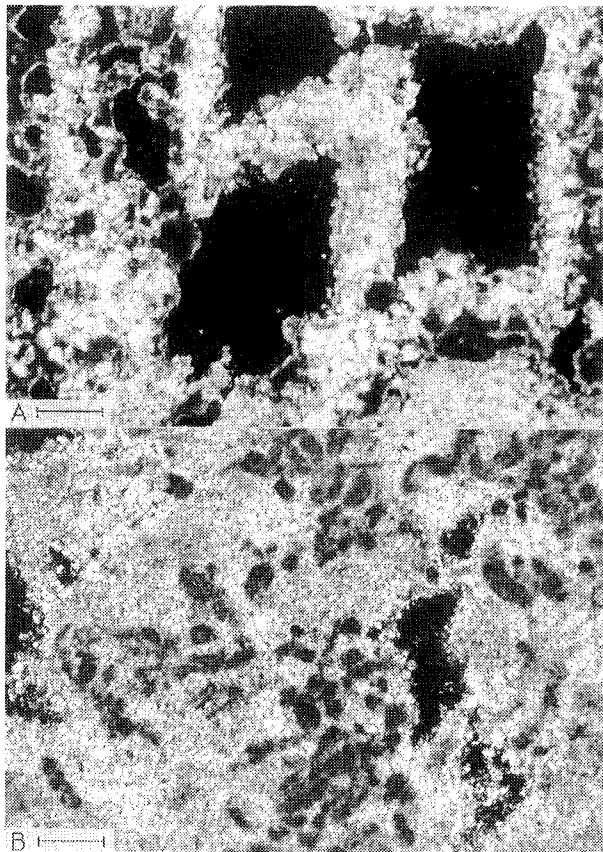


Fig. 3A. Photomicrograph showing sub-regular vugs (secondary porosity) in a micritic matrix partially altered to coarser calcite (sample CST-7). Scale bar: 100 μm ; crossed nicols. **B** - Photomicrograph of micritic allochems lacking internal structure (peloids) and irregular vugs scattered in a fine grained calcite matrix which shows aggrading neomorphism (sample CST-8). Scale bar: 100 μm ; crossed nicols.

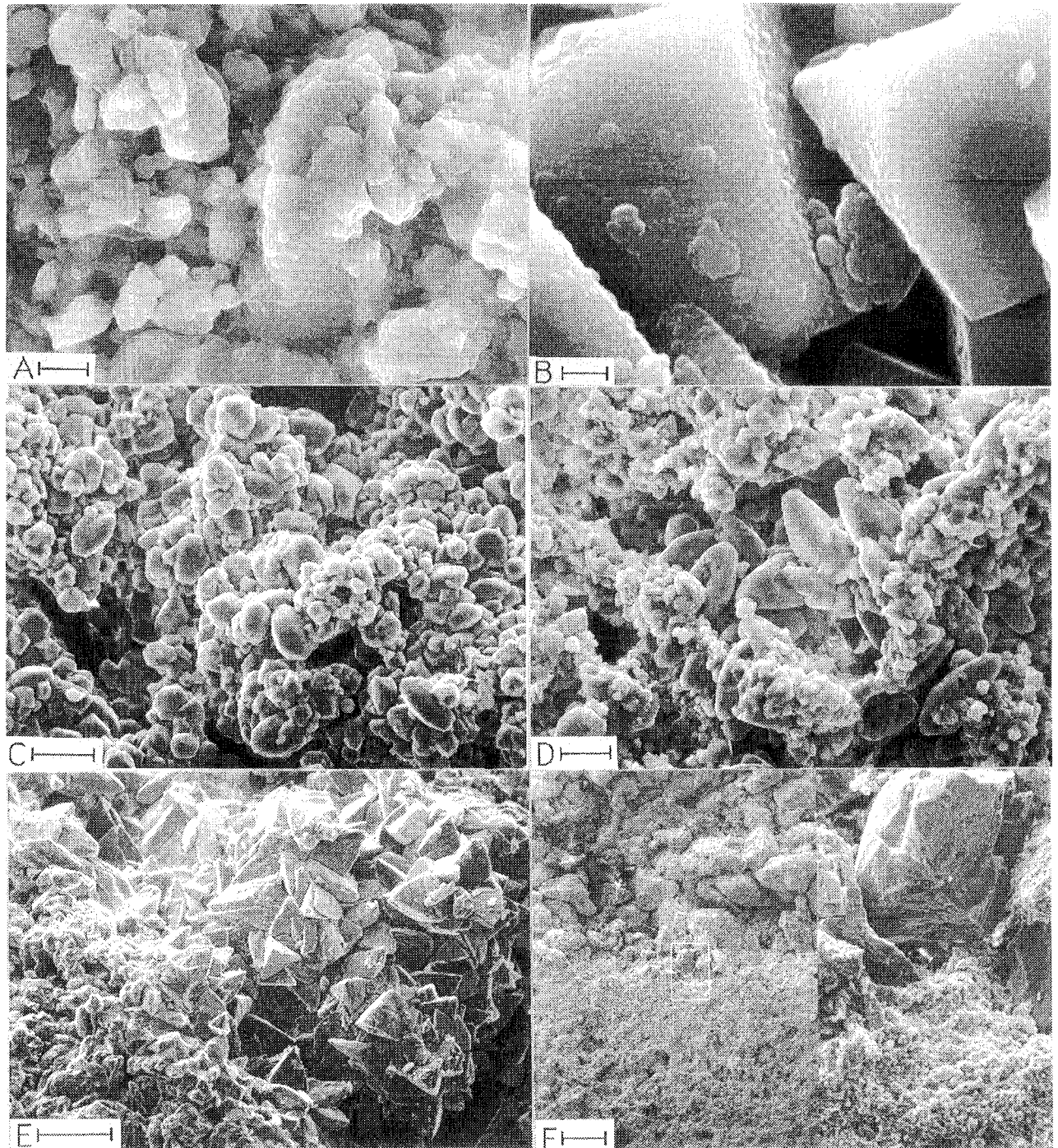


Fig. 4. Secondary electron images showing calcite fabric and morphology. **A** - Unaltered micritic material showing the primary fabric (sample PDM-10). Scale bar: $2\mu\text{m}$. **B** - Euhedral rhomb-shaped crystals of calcite on which overgrowths are developed (sample PDM-4). Scale bar: $1\mu\text{m}$. **C** - Euhedral and subhedral microspar and micrite relicts (sample PDM-4). Scale bar: $10\mu\text{m}$. **D** - Incomplete replacement of the primary micritic matrix (samples CST-4). Scale bar: $10\mu\text{m}$. **E** - Fracture completely filled with a euhedral calcite mosaic which shows the characteristic habit of a cement precipitated in the meteoric phreatic zone (sample CST-5). Scale bar: $100\mu\text{m}$. **F** - Cross section between samples CST 13 (lower part) and CST 14 (upper part) showing a terrigenous apport. The right part of the photograph is a detailed view of the area marked with white rims in the left part (zoom = $8\times$). Scale bar: $500\mu\text{m}$.

Discussion

Carbon and oxygen isotope data for the studied carbonate samples define two distinct trends. In the scatter plot shown in Fig. 6 two groups may be clearly distinguished.

The first group (A) includes all the samples from outcrops 1 (CST) and 2 (VNT). They show $\delta^{18}\text{O}$ values ranging from -1‰ to -7‰ and $\delta^{13}\text{C}$ from 1‰ to -7‰ . Only a specimen of this group has an isotopic composition falling outside this range (CST-4). It shows the most positive ratios:

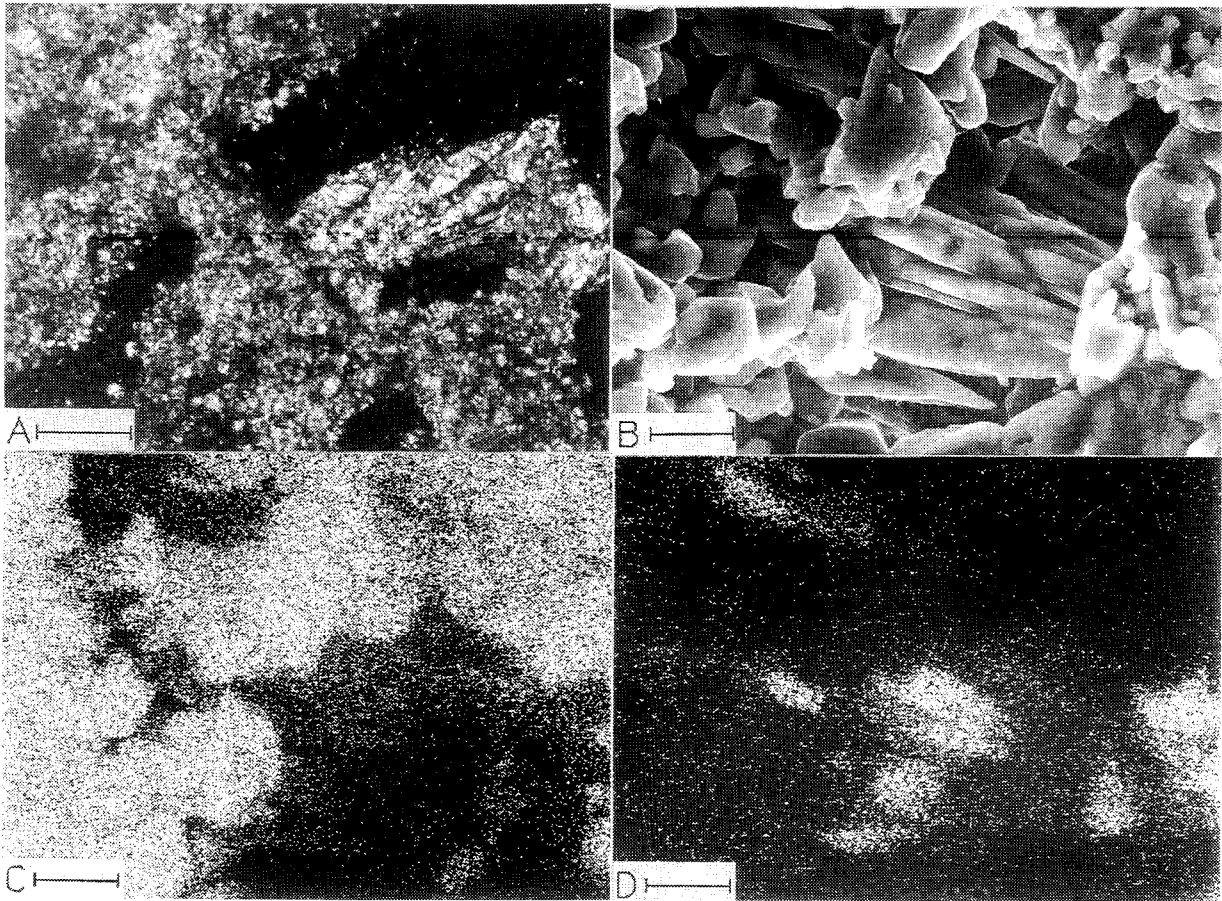


Fig. 5A. Photomicrograph of radial fibrous strontianite partially filling the secondary porosity (VNT-8). Scale bar: 90 μm ; crossed nicols. **B** - Secondary electron image of strontianite prismatic crystals in the primary matrix. Scale bar: 10 μm . **C** and **D** are calcium and strontium X-ray images of B respectively.

$\delta^{18}\text{O} = 4.01 \text{‰}$ and $\delta^{13}\text{C} = 2.44 \text{‰}$. SEM observations established that it is also characterized by the best preserved micritic structure with only very limited recrystallized areas (Fig. 4D). These considerations indicate the sample CST-4 as the only one that still maintains the isotopic memory of a precipitation from evaporating sea water. All the others samples show more negative oxygen and carbon isotope ratios as a consequence of the more or less intense and recent solubilization-recrystallization phenomena, which are proved by SEM images (Fig. 4E) and which involved isotope exchange between carbonate rock and circulating fluids. These sample might have had the initial $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values similar to the CST-4. The trend of figure 6 may be also obtained through the isotope exchange model of Sverjensky (1981) which describe the amount of isotopic alteration of a carbonate rock as a function of progressively increasing water/rock ratio at a given temperature.

For the studied samples, we can write the Sverjensky's equations as:

$$\delta^{18}\text{O}_f = \frac{\chi_{\text{HCO}_3^-} (\Delta^{13}\text{C}_{wr} + \delta^{13}\text{C}_{\text{HCO}_3^-}^i - \delta^{13}\text{C}_f^i) (\Delta^{18}\text{O}_{wr} + \delta^{18}\text{O}_w^i) + n_o \delta^{18}\text{O}_f^i}{n_c + \frac{(\delta^{13}\text{C}_f^i - \delta^{13}\text{C}_r^i)}{\chi_{\text{HCO}_3^-} (\Delta^{13}\text{C}_{wr} + \delta^{13}\text{C}_{\text{HCO}_3^-}^i - \delta^{13}\text{C}_f^i)}}$$

The parameters we need to define the system are: $\delta^{18}\text{O}$ of the circulating water ($\delta^{18}\text{O}_w^i$), $\delta^{13}\text{C}$ of the dissolved HCO_3^- ($\delta^{13}\text{C}_{\text{HCO}_3^-}^i$), the initial isotope composition of the rock ($\delta^{18}\text{O}_r^i$ and $\delta^{13}\text{C}_r^i$), the isotope fractionation factors between rock and fluids ($\Delta^{18}\text{O}_{wr}$ and $\Delta^{13}\text{C}_{wr}$), the amount of dissolved HCO_3^- (κHCO_3^-) and the number moles of oxygen and carbon per mole of carbonate mineral (n_o and n_c). The model was applied estimating a temperature of 20 $^\circ\text{C}$ and calculating the isotope fractionation according to O'Neil et al. (1969) and Emrich et al. (1970). The curve reported in Fig. 6, which clearly fits the group "A" samples, represents the variation of the isotopic composition of the carbonate rock as a function of the increasing water/rock ratio, starting from the fixed initial conditions ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ of CST-4).

The group "B" of Fig. 6 includes only the samples from outcrop 3 (PDM) showing an inverse linear relationship between $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$. This behaviour might be explained with a con-

tinuous precipitation of calcium carbonate under closed system conditions (isolated basin) which produces a decrease of the inorganic HCO_3^- , promoting the hydrolysis of the dissolved biogenic CO_2 , according to the equilibrium:



The result is a progressive enrichment in ^{18}O and ^{12}C , respectively related to the increasing of the evaporation rate and to the amount of biogenic component in the balance of the total dissolved carbon species in the evaporating body.

The observed discrimination between the examined samples is confirmed by SEM observations: samples enriched in light oxygen show evidence of diagenetic transformations. Often euhedral sparite more or less extensively replaces the original micritic framework (Fig. 4C). Sometimes relatively recent dissolution-precipitation phenomena in a vadose/phreatic environment are shown (Fig. 4E). Rarely the depletion in ^{18}O and ^{13}C is clearly related to a sudden fresh water input in the evaporative basin (Fig. 4F). Finally, the samples with a relatively well preserved micritic assemblage (Fig. 4A) show higher $^{18}\text{O}/^{16}\text{O}$ ratios.

The presence of secondary strontianite (VNT-7 and VNT-8) may be attributed to a Sr-rich aragonitic precursor. This is in agreement with Decima et al. (1988) who considered the transformation of pre-existent evaporative aragonite under early diagenetic conditions, in presence of fresher meteoric water. The solubilization of aragonite releases Sr^{2+} in excess with respect to the amount that is incorporated in the new-formed calcite (Mucci & Morse 1983; Zhong & Mucci 1989) and the Sr^{2+} is removed from the interstitial solutions and precipitated as strontianite (in SO_4^{2-} poor environment). This is supported by the textural context (Fig. 5) but also by the ^{12}C enrichment respect to the coexisting calcite (Fig. 2) that might be due to the increase of biogenic CO_2 and to the HCO_3^- removal from the solution during the precipitation of calcite as well.

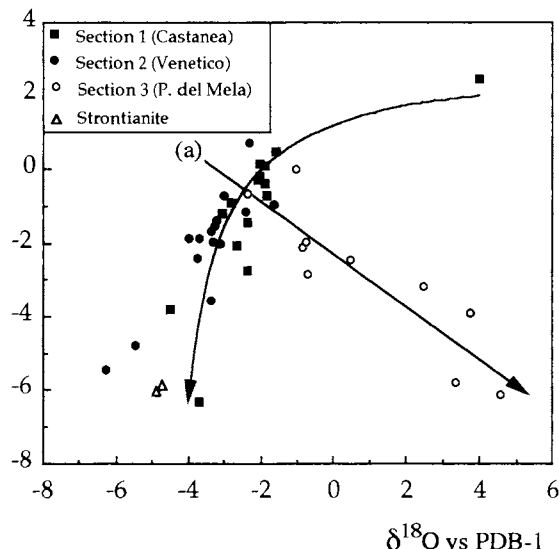


Fig. 6. Plot of $\delta^{18}\text{O}$ vs. $\delta^{13}\text{C}$ in which two different isotopic variation trends are shown (solid lines). The isotopic exchange path represented by the curve (group A) was calculated assuming ($\delta^{13}\text{C}_{\text{HCO}_3^-} = -12\text{‰ (PDB)}$; $\delta^{18}\text{O}_{\text{ri}} = 34.9\text{‰ (SMOW)}$; $\delta^{13}\text{C} = 2\text{‰ (PDB)}$; $\text{HCO}_3^- > 0.003$; $\delta^{18}\text{O}_{\text{wi}} = -8\text{‰ (SMOW)}$).

Conclusive remarks

Oxygen and carbon isotopic ratios integrated with SEM observations suggest that the formation of the studied *Calcarea di Base* outcrops should have occurred in a shallow seawater environment which progressively evolved to an isolated evaporitic basin. The observed ^{16}O and ^{12}C enrichments seem to be related to more or less recent isotope exchange phenomena which have occurred in diagenetic and/or vadose/phreatic environment. Therefore the isotope ratios of most of the collected samples are not representative of the depositional paleoenvironment any more. The dispersion of the oxygen and carbon isotopic values of calcium carbonate, often hastily interpreted as evidence of a meteoric water input in similar basins of the world, should be checked by mineralogical and petrographic observations.

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UNESCO IGCP Projekt 362: TETHYAN AND BOREAL CRETACEOUS (TBC) in 1993



This Project, approved early in this year by the IGCP Board, UNESCO Department of Earth Sciences, is guided by **Dr. Han Leereveld** (University of Utrecht, The Netherlands) and by **Dr. Jozef Michalík** (Geological Institute, Slovak Academy of Sciences, Bratislava, Slovakia). Its full title is "Stratigraphical correlation and definition of geoevents in Cretaceous sedimentary sequences of the Tethyan- and Boreal Regions".

The Plenary Meeting of this Project, with participation of 55 specialists from fifteen European countries, has been held in Coimbra (Portugal) during October 25 - 30th, 1993. Twenty papers have been given, dealing with Cretaceous biostratigraphy and facies, Cretaceous bio- and lithoevents, correlation of the Tethyan and Boreal sequences, new methods in stratigraphy, as well as by paleobiogeography and paleobiology of the Cretaceous faunas and floras. Attention has been laid on penetrations of Tethyan organisms in French, English, German and Polish Boreal basins and, vice versa, Boreal biofacies influences in Iberia, Alpine and Carpathian countries of the Mediterranean area. Territory of Slovakia occupies an important position on the boundary between the European Boreal- and the Mediterranean Tethyan bioprovince.

Four working groups have been established during work meeting, namely integrated stratigraphy, Pre-Aptian event, Aptian - Albian "greenhouse period", and Late Cretaceous event groups.

The field trip guided by the Portuguese organizers was oriented to the results of the Cretaceous research in the Lusitanian Basin between Coimbra, Nazaré, and the westernmost point of the Portugal Atlantic coast. The sequences in this region recorded the Cretaceous development of the basins adjacent to the opening of Atlantic.

Next general meeting planned during *October 3 - 6th, 1994 in Smolenice (Slovakia)* will be devoted to the inauguration of working groups, as well as to several special questions of the Mediterranean and Boreal correlations.

RNDr. Jozef Michalík, CSc.
co-leader of the Project 362