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# Mineral and Chemical Composition of Rudist Valves from Upper Cretaceous Limestones of Southern Istria, Croatia

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**Key words:** Rudist valves, Mineral composition, Trace element content, Turonian to Upper Santonian, Istria, Croatia.

## Abstract

Turonian to late Santonian limestones of southern Istria are rich in rudist remains. The main aim of this study was to determine the mineral and chemical composition of 22 samples of rudist valves belonging to the genera *Durania, Praeradiolites*, *Radiolites*, *Gorjanovicia* and *Vaccinites*, and to explain any observed differences. X-ray diffraction and chemical analyses showed that in all the analysed samples, the primary constituents of rudist shells, aragonite and low-Mg calcite, were transformed into diagenetic low-Mg calcite. The strontium concentrations of all the analysed shells correspond to pelagic bulk rock samples altered by diagenesis and are significantly lower than those for biological calcite. Observed differences in the chemical composition of diagenetically altered rudist shells belonging to different rudist genera, namely a slightly higher content of Sr in specimens of *Gorjanovicia* and *Durania* and lower concentrations of Mg in shells of *Vaccinites* are most probably the result of different shell structures and original mineral constituents.

## 1. INTRODUCTION

Turonian to Upper Santonian limestones rich in rudist remains crop out in the southernmost part of Istria, at the Mrlera and Premantura peninsulas (Fig. 1). According to the latest stratigraphic and palaeoenvironmental investigations of the rudists from these rocks (MORO, 1997; MORO & ĆOSOVIĆ, 2000) specimens of the genera *Durania*, *Praeradiolites* and *Radiolites* thrived in the shallower parts of the subtidal zone in an inner shelf environment, while specimens of the genera *Gorjanovicia* and *Vaccinites* lived in the deeper subtidal environment with pelagic influxes (outer shelf).

The main aim here was to determine the mineral (i.e. phase) and chemical composition of the valves of several rudist genera from pure carbonates without the influence of terrigenous material, and to explain any observed differences.

## 2. MATERIALS AND METHODS

During the study, 22 samples of rudist valves (five of the genus *Durania*, samples D1–D5; four of the genus *Gorjanovicia*, G1–G4; four of the genus *Praeradiolites*, P1–P4; five of the genus *Radiolites*, R1–R5, and four of the genus *Vaccinites*, V1–V4) were analysed. According to the biostratigraphy, the rudist genera *Durania*, *Radiolites* and *Praeradiolites* lived during the Middle to Upper Turonian, *Gorjanovicia* lived in the time span from Upper Turonian to Santonian while *Vaccinites* specimens are of Santonian age (MORO, 1997; MORO & ĆOSOVIĆ, 2000). Zonation of shells was absent in observed hand specimens, so complete shell material, comprising the outer and inner layers, was taken for analysis. Micro- and macro-photographs of some analysed rudists from the investigated area are given in POLŠAK (1967b – plates 18–21, 69, 70) and MORO (1997 – plate 1: figs. 1, 2, 3, 5 & 6; plate 2: figs. 1 & 3).

The mineral composition was determined by the X-ray powder diffraction method (XRD) using a Philips instrument equipped with vertical goniometer PW 1050, Cu tube, a graphite monochromator, and a proportional counter. Unit cell dimensions of calcite were calculated by the UnitCell software (HOLLAND & REDFERN, 1997) with the aim of determining the Mg-content of the calcite on the basis of diagrams constructed by MACKENZIE et al. (1983) that show the relationship between unit cell dimensions and magnesium content of Mg-calcites.

Concentration of Fe, Mn, Mg and Na in the valves was determined by the commercial ACME Analytical Laboratories, Canada by the ICP-ES method. The 0.5 g of the sample was leached for one hour with 3 ml of hot (95°C) aqua regia and diluted to 10 ml. Content of other trace elements (Cu, Nb, Pb, Rb, Sr, U, Zn, Zr, Y) was determined on an ARL 8410 X-ray fluorescence (XRF) spectrometer equipped with Rh-tube on pressed powder pellets, using a slightly modified procedure from NISBET et al. (1979). Mass absorption coefficients for the wavelengths of measured lines were calculated assuming, in accordance with the results of performed XRD analyses, that all of the samples have practically the

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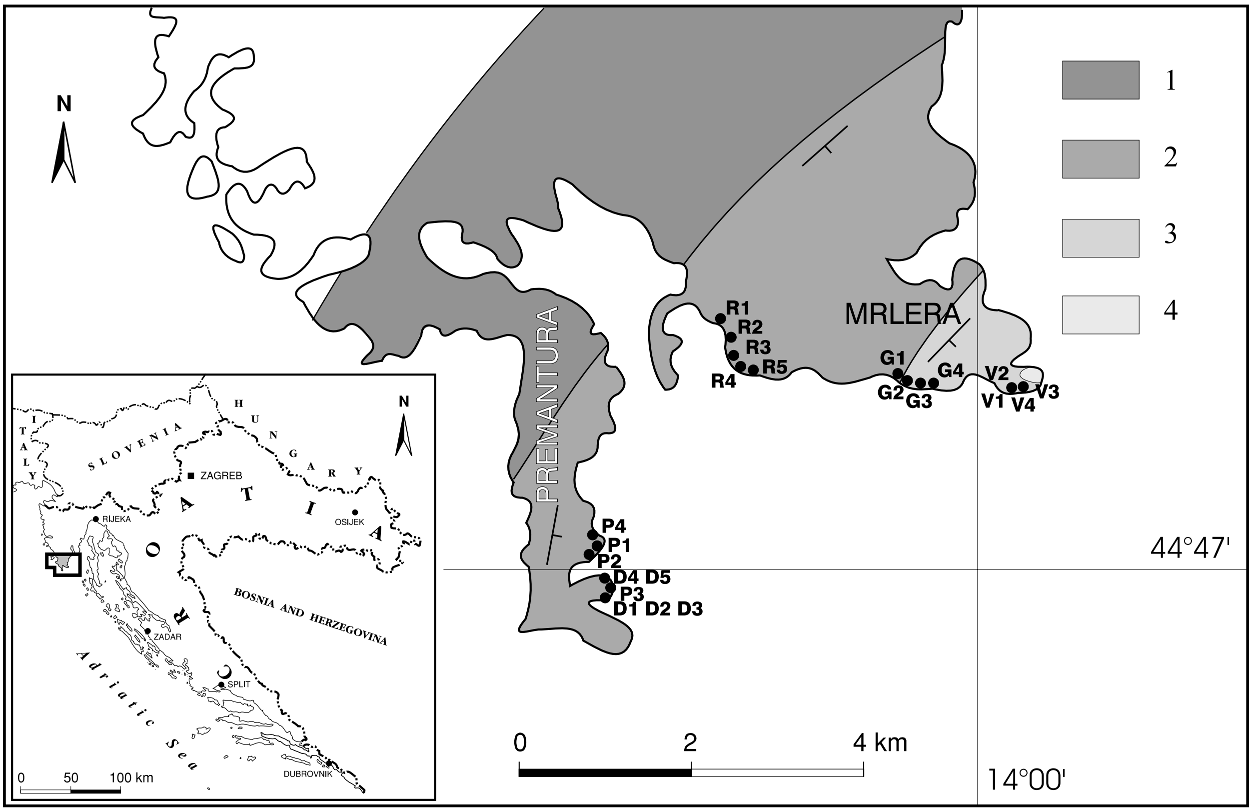

Fig. 1 Map with sampling locations (D1 – *Durania* sample 1 etc., G = *Gorjanovicia*, P = *Praeradiolites*, R= *Radiolites*, V = *Vaccinites*; abbreviations are also used on Figs. 2–5). Simplified geology after POLŠAK (1967a), MORO (1997) and MORO & ĆOSOVIĆ (2000): 1 – Cenomanian, 2 – Turonian, 3 – Coniacian–Santonian, 4 – Palaeogene.

same composition that correspond to the $CaCO_3$. Average accuracy of the XRF measurements, as compared to recommended values (GOVINDARAJU, 1994) for USGS (GXR–1 to 6), GSJ (JB–2, JB–3, JF–2, JG–2, JG–3, JR–2) and IGGE (GSD–1 to 12, GSR–1 to 6) standard rocks was 4% for Sr, 20% for Pb, 12% for Ga, 8% for Zn, 14% for Cu and 11% for Ni. The precision of XRF and ICP-ES measurements was within several %, except when values were very close to the limit of detection (LOD).

The results of chemical analysis have been statistically processed by the Statistica computer software (STATSOFT, 1995). Besides descriptive statistical methods, discriminant analysis was used as it is a powerful tool for classifying individual cases (samples) into previously defined groups on the basis of multiple variables, with wide application in solving problems in geosciences (DAVIS, 1986). For statistical analysis Mn concentrations bellow LOD were replaced with 70% of LOD (MIESCH, 1976).

## 3. RESULTS

Phase analysis has shown that all the samples consist of just one mineral phase, almost pure calcite. No aragonite was registered. Comparison of calculated unit cell dimensions of calcite with diagrams of MACKENZIE et al. (1983) revealed a very low magnesium content (0 to 2 mol %) (Table 1).

The results of chemical analyses of valves of selected rudist genera are given in Table 2 while in Table 3 relevant basic statistical parameters are presented. Concentrations of niobium, zirconium, yttrium, uranium, and rubidium are not shown because in all analysed samples they were below the LOD (app. 1 ppm for all of these elements except U for which the LOD was 4 ppm). The results showed some differences in chemical composition of valves belonging to different rudist genera, although the content of most analysed trace elements is not influenced by rudist genus. The strontium content of valves of different genera varies significantly (Fig. 2). *Praeradiolites*, *Radiolites* and *Vaccinites* have very similar concentrations of strontium while the genera *Durania* and *Gorjanovicia* are differentiated by their remarkably higher concentration of strontium. Generally, the low content of magnesium, already inferred by X-ray investigations, is especially low in *Vaccinites* (Fig. 3). Minor differences were also observed in the sodium content, rudists of the genera *Durania* and *Vaccinites* are characterised by lower concentrations of Na (Fig. 4).

These differences in chemical composition were confirmed by discriminant function analysis. The results of this analysis, though limited due to the number of samples on which analysis has been based, are represented by Fig. 5 and Tables 4 and 5. These results suggest that each rudist genus has a moderately distinct chemical composition, and samples of different rudist

| Sample | a Å | c Å | c/a Å | V $Å^3$ | Mg mol.% |
|---|---|---|---|---|---|
| D1 | 4.9788 | 17.0303 | 3.421 | 365.5904 | 1 |
| D2 | 4.9861 | 17.046 | 3.419 | 367.0039 | 1 |
| D3 | 4.9912 | 17.0459 | 3.415 | 367.7535 | 1 |
| D4 | 4.9847 | 17.0357 | 3.418 | 366.5737 | 1 |
| D5 | 4.9872 | 17.0248 | 3.414 | 366.7116 | 2 |
| G1 | 4.9934 | 17.0473 | 3.414 | 368.1167 | 1 |
| G2 | 4.9890 | 17.0695 | 3.421 | 367.9478 | 0 |
| G3 | 4.9809 | 17.0424 | 3.422 | 366.1645 | 1 |
| G4 | 4.9843 | 17.0279 | 3.416 | 366.3588 | 2 |
| P1 | 4.9878 | 17.0509 | 3.419 | 367.3691 | 1 |
| P2 | 4.9918 | 17.0564 | 3.417 | 368.0699 | 1 |
| P3 | 4.9921 | 17.0426 | 3.414 | 367.8125 | 2 |
| P4 | 4.9840 | 17.0441 | 3.420 | 366.6538 | 1 |
| R1 | 4.9922 | 17.0452 | 3.414 | 367.8906 | 1 |
| R2 | 4.9870 | 17.0497 | 3.419 | 367.2159 | 1 |
| R3 | 4.9936 | 17.0687 | 3.418 | 368.6063 | 0 |
| R4 | 4.9763 | 17.0387 | 3.424 | 365.4098 | 1 |
| R5 | 4.9865 | 17.0344 | 3.416 | 366.8188 | 2 |
| V1 | 4.9843 | 17.0611 | 3.423 | 367.0628 | 0 |
| V2 | 4.9915 | 17.0709 | 3.420 | 368.3358 | 0 |
| V3 | 4.9840 | 17.0487 | 3.421 | 366.7595 | 1 |
| V4 | 4.9913 | 17.0467 | 3.415 | 367.7886 | 1 |

Table 1 Unit cell dimensions and inferred (after MACKENZIE et al., 1983) Mg-content of calcite in valves of different rudist genera. Legend: R = *Radiolites*, P = *Praeradiolites*, D = *Durania*, V = *Vaccinites*, G = *Gorjanovicia*.

| Sample | Cu ppm | Fe % | Ga ppm | Mg % | Mn ppm | Na % | Ni ppm | Pb ppm | Sr ppm | Zn ppm |
|---|---|---|---|---|---|---|---|---|---|---|
| D1 | 18 | <0.01 | 3 | 0.20 | <2 | 0.03 | 4 | 5 | 295 | 19 |
| D2 | 2 | <0.01 | 4 | 0.19 | 3 | 0.03 | 3 | 7 | 289 | 8 |
| D3 | 9 | <0.01 | 4 | 0.18 | <2 | 0.04 | 5 | 4 | 256 | 12 |
| D4 | 9 | <0.01 | 4 | 0.22 | 9 | 0.03 | 4 | 6 | 442 | 13 |
| D5 | 6 | <0.01 | 4 | 0.23 | <2 | 0.03 | 5 | 7 | 492 | 12 |
| G1 | 9 | <0.01 | 3 | 0.23 | 3 | 0.06 | 5 | 9 | 630 | 17 |
| G2 | 11 | <0.01 | 3 | 0.16 | <2 | 0.07 | 3 | 6 | 540 | 16 |
| G3 | 3 | <0.01 | 4 | 0.05 | 7 | 0.01 | 4 | 5 | 220 | 10 |
| G4 | 2 | <0.01 | 4 | 0.12 | 11 | 0.07 | 3 | 4 | 465 | 10 |
| P1 | 14 | <0.01 | 3 | 0.16 | <2 | 0.10 | 4 | 5 | 169 | 15 |
| P2 | 12 | <0.01 | 3 | 0.20 | 6 | 0.12 | 4 | 8 | 169 | 15 |
| P3 | 2 | <0.01 | 4 | 0.21 | <2 | 0.04 | 4 | 4 | 239 | 9 |
| P4 | 2 | <0.01 | 4 | 0.17 | <2 | 0.06 | 4 | 6 | 175 | 8 |
| R1 | 14 | <0.01 | 4 | 0.18 | 6 | 0.06 | 4 | 7 | 209 | 16 |
| R2 | 17 | <0.01 | 4 | 0.18 | 4 | 0.04 | 2 | 5 | 255 | 19 |
| R3 | 9 | <0.01 | 4 | 0.18 | 3 | 0.05 | 5 | 7 | 248 | 13 |
| R4 | 7 | <0.01 | 3 | 0.18 | 9 | 0.05 | 3 | 7 | 226 | 14 |
| R5 | 1 | <0.01 | 2 | 0.17 | 15 | 0.08 | 2 | 3 | 213 | 10 |
| V1 | 2 | <0.01 | 3 | 0.06 | <2 | 0.02 | 4 | 7 | 158 | 9 |
| V2 | 23 | 0.03 | 4 | 0.08 | 8 | 0.01 | 4 | 10 | 164 | 20 |
| V3 | 12 | 0.02 | 4 | 0.07 | 3 | 0.01 | 7 | 6 | 182 | 15 |
| V4 | 16 | <0.01 | 4 | 0.06 | 5 | 0.02 | 5 | 5 | 258 | 16 |
| LOD | 0.7 | 0.01 | 1.1 | 0.01 | 2 | 0.01 | 0.7 | 1.6 | 0.9 | 0.9 |

Table 2 Elemental concentrations in valves of different rudist genera. Legend: R = *Radiolites*, P = *Praeradiolites*, D = *Durania*, V = *Vaccinites*, G = *Gorjanovicia,* LOD = limit of detection.

| Rudist genera | Cu ppm | Ga ppm | Mg % | Mn ppm | Na % | Ni ppm | Pb ppm | Sr ppm | Zn ppm |
|---|---|---|---|---|---|---|---|---|---|
| ***Durania*** | | | | | | | | | |
| *Min.* | 2 | 3 | 0.18 | <2 | 0.03 | 3 | 4 | 256 | 8 |
| *Max.* | 18 | 4 | 0.23 | 9 | 0.04 | 5 | 7 | 492 | 19 |
| *Mean* | 9 (6) | 4 (0) | 0.20 (0.02) | 3 (3) | 0.03 (0) | 4 (1) | 6 (1) | 355 (105) | 13 (4) |
| ***Gorjanovicia*** | | | | | | | | | |
| *Min.* | 2 | 3 | 0.05 | <2 | 0.01 | 3 | 4 | 220 | 10 |
| *Max.* | 11 | 4 | 0.23 | 11 | 0.07 | 5 | 9 | 630 | 17 |
| *Mean* | 6 (4) | 4 (1) | 0.14 (0.08) | 6 (4) | 0.05 (0.03) | 4 (1) | 6 (2) | 464 (176) | 13 (4) |
| ***Praeradiolites*** | | | | | | | | | |
| *Min.* | 2 | 3 | 0.16 | <2 | 0.04 | 4 | 4 | 169 | 8 |
| *Max.* | 14 | 4 | 0.21 | 6 | 0.12 | 4 | 8 | 239 | 15 |
| *Mean* | 8(6) | 3 (1) | 0.19 (0.02) | 3 (2) | 0.08 (0.04) | 4 (0) | 6 (2) | 188 (34) | 12 (4) |
| ***Radiolites*** | | | | | | | | | |
| *Min.* | 1 | 2 | 0.17 | 3 | 0.04 | 2 | 3 | 209 | 10 |
| *Max.* | 17 | 4 | 0.18 | 15 | 0.08 | 5 | 7 | 255 | 19 |
| *Mean* | 10 (6) | 3 (1) | 0.18 (0) | 7 (5) | 0.06 (0.02) | 3 (1) | 6 (2) | 230(21) | 14 (3) |
| **Vaccinites** | | | | | | | | | |
| *Min.* | 2 | 3 | 0.06 | <2 | 0.01 | 4 | 5 | 158 | 9 |
| *Max.* | 23 | 4 | 0.08 | 8 | 0.02 | 7 | 10 | 258 | 20 |
| *Mean* | 13 (9) | 4 (1) | 0.07 (0.01) | 4 (3) | 0.02 (0.01) | 5 (1) | 7 (2) | 190 (46) | 15 (5) |
| **All samples** | **9 (6)** | **4 (1)** | **0.16 (0.06)** | **5 (4)** | **0.05 (0.03)** | **4 (1)** | **6 (2)** | **286 (136)** | **13 (4)** |

Table 3 The minimum, maximum and mean concentrations of trace and minor elements in valves of different rudist genera. Standard deviations calculated from measured concentrations for samples of each genus are given in parentheses. Data for Fe are not given because most of the measured values are below LOD. For calculation purposes concentrations of Mn bellow LOD were replaced with 70% of LOD (MIESCH, 1976).

genera can be distinguished in discriminant space fairly well (Fig. 5).

## 4. DISCUSSION

According to XRD analyses, no remnants of bimineralic valves, which are otherwise characteristic for rudist shells (STEUBER, 2002) were found. This indicates diagenetic alteration of the studied valves and the transformation of aragonite to diagenetic low-Mg calcite (dLMC).

The grade of diagenetic alteration of calcareous fossils is often inferred from their chemical composition. As a result of meteoritic diagenesis Mn and Fe concentrations typically increase while Sr and Na concentrations decrease (BRAND & VEZIER, 1980; AL-AASM & VEZIER, 1986). Measured concentrations of Mn in the investigated rudist valves is quite low (Table 2, Fig. 6), well within the range of concentrations of

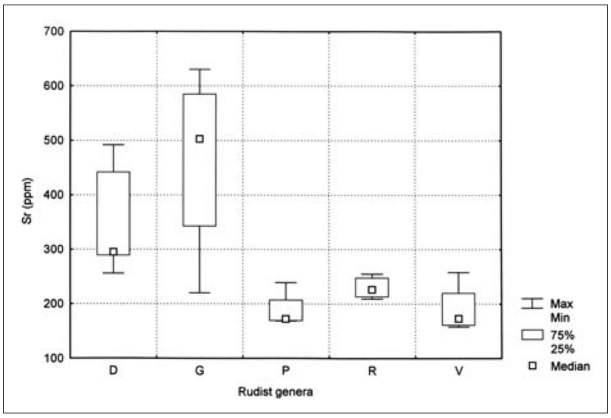

Fig. 2 Strontium concentration in shells of different rudist genera.

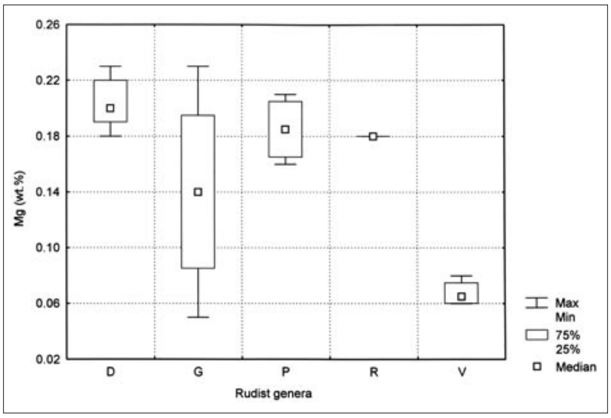

Fig. 3 Magnesium concentration in shells of different rudist genera.

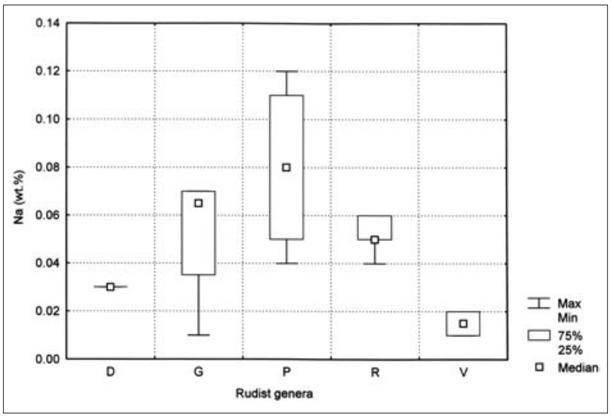

Fig. 4 Sodium concentration in shells of different rudist genera.

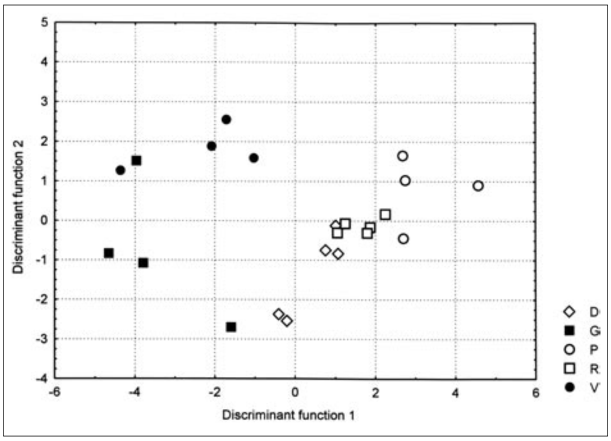

Fig. 5 Plot of discriminant scores along Function 1 vs. Function 2, to discriminate rudist valves of different genera.

recent marine bivalve calcite shells (STEUBER, 1999 and references therein), and diagenetic alteration is indicated only by concentrations of Sr which are much lower than in recent shells. Observed Sr concentrations are significantly lower than those presented by several authors (McARTHUR et al., 1994, 2000; PODLAHA et al., 1998; STEUBER, 2002) for biological calcite, but in good agreement with data for diagenetically altered pelagic carbonate rocks (RENARD, 1986). This is also visible in Fig. 7, where the measured Mg and Sr concentrations cluster close to the regression line showing compositions of abiotic calcite. Similar diagenetic trends, with Mn and Fe that are not elevated and low Sr, were already observed by STEUBER (2002) in some rudist shells. Therefore, the same author considered that Mg/Sr ratios were the best indicator for diagenetic alteration of shells. The chemical pattern indicates that diagenesis ended with transformation of both primary constituents of the shells, aragonite and low-Mg calcite (LMC), into dLMC, despite the fact that LMC is considered to be relatively stable in a meteoric environment. According to the measured low Mn and Fe contents there was no precipitation of late ferroan calcitic cement. Transformation processes were most probably accomplished in the vadose or phreatic–meteoric zone (AL-AASM & VEZIER, 1986).

The results of chemical and discriminant analyses point out that the chemical content of shells of different rudist genera is rather distinct. Due to the fact that analysed shells are diagenetically altered, chemical composition of the shells should be primarily the result of diagenetic processes. These processes are controlled by a

| | Discriminant function | | | |
|---|---|---|---|---|
| | 1 | 2 | 3 | 4 |
| log Sr | -1.48068 | -0.568105 | 0.373568 | 0.082307 |
| Mg | 1.53415 | -0.542829 | -0.489472 | -0.036865 |
| Mn | 0.02954 | -0.266035 | -0.307657 | -0.992138 |
| Na | 0.10066 | 0.209920 | 1.068870 | -0.010185 |
| Eigenvalue | 7.36468 | 1.586070 | 0.949597 | 0.285667 |
| % of variance – relative | 72.30 | 15.57 | 9.33 | 2.80 |
| % of variance – cumulative | 72.30 | 87.87 | 97.20 | 100.00 |

Table 4 Standardised discriminant function coefficients and related statistics.

| | Percent correct | *Durania* | *Gorjanovicia* | *Praeradiolites* | *Radiolites* | *Vaccinites* |
|---|---|---|---|---|---|---|
| *Durania* | 100.00 | 5 | 0 | 0 | 0 | 0 |
| *Gorjanovicia* | 75.00 | 0 | 3 | 0 | 0 | 1 |
| *Praeradiolites* | 75.00 | 0 | 0 | 3 | 1 | 0 |
| *Radiolites* | 100.00 | 0 | 0 | 0 | 5 | 0 |
| *Vaccinites* | 100.00 | 0 | 0 | 0 | 0 | 4 |
| Total | 90.91 | 5 | 3 | 3 | 6 | 5 |

Table 5 Classification matrix; rows contain number of samples in each observed group while in columns the number of samples in a predicted group are given.

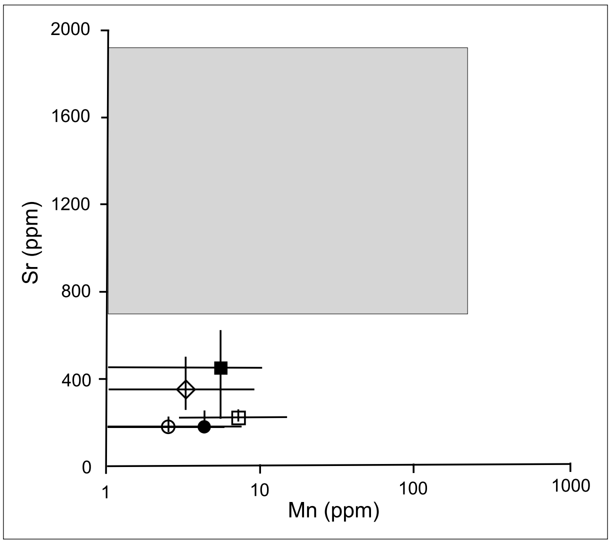

Fig. 6 Ranges and means of Mn and Sr concentrations in rudist valves from southern Istria compared with concentrations of modern bivalve calcite shells represented by the shaded area (STEUBER, 1999 and references therein).

Symbols: ◇ = *Durania*, ■ = *Gorjanovicia*, ○ = *Praeradiolites*, □ = *Radiolites*, ● = *Vaccinites*.

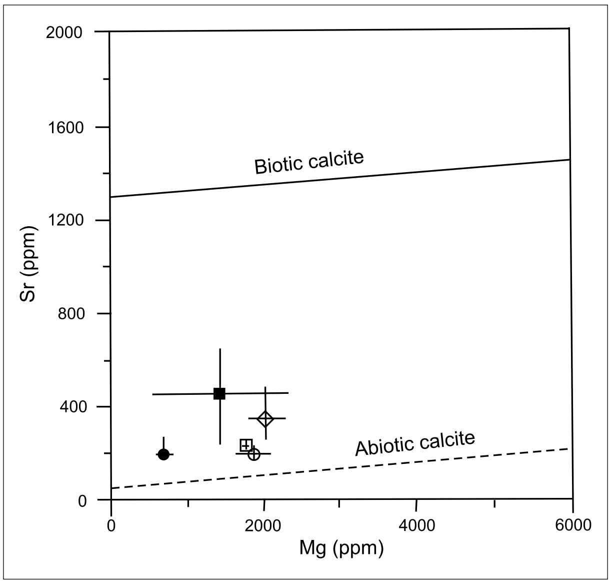

Fig. 7 Ranges and means of Mg and Sr concentrations in rudist valves from southern Istria compared with mean compositions of modern biotically (solid line) and abiotically (broken line) precipitated calcite (CARPENTER & LOHMANN, 1992). Symbols as in Fig. 6.

variety of factors, such as their original mineralogy and chemistry and shell structure, and the physicochemical properties of diagenetic fluids and their position in the sedimentary column. It can be reasonably presumed that the latter factors were practically the same for all the analysed samples, since their sampling positions were very close, and Cretaceous limestones are only slightly tectonically deformed (POLŠAK, 1967a).

Strontium concentrations measured in valves of different rudist genera was slightly variable. A possible explanation for different Sr contents lies in the fact that some analysed shell material most probably contained a considerable portion of diagenetic cement (AL-AASM & VEZIER, 1986; STEUBER, 1999), which frequently has lower Sr concentrations than a biogenic one, so consequently the measured concentrations were lower. The presence of diagenetic cements is due to the cellular structure of the outer shell of most radiolitids which allows entrance of diagenetic fluids and cement formation (AL-AASM & VEZIER, 1986; STEUBER, 1999; REGIDOR-HIGUERA et al., 2002). The highest measured Sr contents were observed in *Gorjanovicia*, a radiolitid rudist with a compact, fibrous prismatic structure, similar to that of most hippuritids, in which the proportion of diagenetic cements was probably the lowest. Relatively high Sr concentrations were also observed in *Durania*. Although it has an open cellular structure, the wall structures (funnel plates and muri) are relatively thick which increases the proportion of biogenic calcite. Low Sr concentrations measured in *Radiolites* and *Praeradiolites* could be explained by a higher amount of diagenetic calcite in the analysed material due to their very thin shells with typical radiolitid microstructure. However, the low Sr concentration in the hippuritid cannot be explained by shell structure alone, because its compact structure inhibits access of diagenetic fluids, and it is probably the result of the primary mineral composition. Hippuritid shells have a greater proportion of aragonite (STEUBER, 2002) which is less stable then LMC in the diagenetic environment, and consequently they will be more diagenetically altered. The observed low Mg content of *Vaccinites* is most probably also influenced by their structure, primary mineral and chemical composition. Unaltered shells of *Vaccinites* contained less Mg because a significant part of them was composed of aragonite, and the more compact structure inhibited sub-marine cementation by high-Mg cements which could result in the lower Mg content of altered shells.

## 5. CONCLUSIONS

Mineral composition and trace element contents were obtained in order to find possible differences in rudist genera from specimens that were taken from pure carbonates without the influence of terrigenous material.

In all analysed samples, the primary constituents of rudist shells, aragonite and low-Mg calcite, are transformed into diagenetic low-Mg calcite. Measured strontium concentrations of all analysed shells correspond to pelagic bulk rock samples altered by diagenesis and are significantly lower than those for biological calcite. Observed differences in the chemical composition of diagenetically altered rudist shells belonging to different rudist genera, namely the higher strontium content in *Gorjanovicia* and *Durania*, and lower magnesium content in *Vaccinites*, are most probably the result of different shell structures, i.e. openness of the structure for diagenetic fluids and cement precipitation, and different primary mineral and chemical compositions.

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