

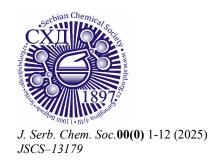


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Tri- and tetracyclic terpanes as proxies for depositional environment reconstruction for weathered siliciclasticsof Internal Dinarides

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Abstract: This study examines the potential of using tri- and tetracyclic terpanes to determine the depositional environment of sediments lacking preserved fossils and whose biomarker distribution has been disrupted by biodegradation, weathering or other alteration processes. In the case of sediments from Jazovnik, Snagovo, Drina and Gučevo (Internal Dinarides), tricyclic terpanes have demonstrated easy applicability and strong predictive power for distinguishing depositional settings. This study analysed siliciclastic sediments exposed to prolonged chemical weathering, influencing the distribution of hydrocarbons, and resulting in highly abundant branched hydrocarbons and the presence of unresolved complex mixtures (UCMs). The abundances and interrelationships of tri- and tetracyclic terpanes (C20-C24TT, C24TeT) showed valuable results. The triangular diagram incorporating $C_{20} + C_{22}\%$, $C_{21}\%$, and $C_{23}\%$ has proven particularly effective in distinguishing between swamp, fluvial-deltaic, freshwater lacustrine, and marine-saltwater lacustrine depositional environments. Once again, this method clearly depicts different sedimentary environments, providing valuable insights into the geological history of the studied area.

Keywords: chemical weathering; biodegradation, organic matter; branched alkanes; unresolved complex mixture; paleoenvironmental settings.

INTRODUCTION

Reconstructing the conditions of paleoenvironmental deposition represents one of the most difficult challenges in organic geochemistry. Traces of changes in paleoenvironmental settings are preserved in molecular structures incorporated into the organic matter (OM) of sedimentary rocks, representing the "guardians" of important information about biogeochemical transformations of OM. Therefore,

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the application of specific organic-geochemical parameters based on the distribution and abundance of individual classes of saturated and aromatic hydrocarbons is one of the most widely used approaches in interpreting paleoredox conditions, paleoclimate, and paleosalinity of the environment, which is most often in connection with other scientific disciplines such as stratigraphy, sedimentology, mineralogy, petrography, etc. However, reconstructions of depositional environments based on organic proxies are often ambiguous or even impossible due to the intense and prolonged chemical weathering that affects the distribution of hydrocarbons, resulting in high relative abundances of branched hydrocarbons and the presence of unresolved complex mixtures (UCMs). Only a few proxies are available for assessing depositional environments, and they rarely allow unambiguous interpretations. Among them, tricyclic terpanes seem to be the most useful.²

Tricyclic terpanes are organic compounds ubiquitous in sediments and oils spanning the geological record. Their ubiquities and ability to participate in solving a wide range of different geochemical and geological problems. Abundance, clear origin and resistance to biodegradation (robustness) allow them to have been widely applied in geochemical analyses to determine oil-to-oil correlations, oil-to-source correlations, thermal maturity, as well as depositional environment characteristics, and OM inputs. In identifying depositional settings, specific tricyclic terpanes (e.g., C₁₉ + C₂₀, C₂₁, C₂₃TT) are key to determining the origin of OM: C₂₃ TTs are often associated with marine environments and can indicate the presence of planktonic organic matter, C₂₁TT is typically derived from the degradation of higher plant material, such as terrigenous plants, while C₂₂TTs are similar to C₂₁TT also derived from higher plant material but can show variations based on the maturity and depositional environment.

With all said, the idea behind this work is to reconstruct the depositional settings using tri- and tetracyclic terpanes in sediments of Jazovnik, Snagovo, Drina and Gučevo (Internal Dinarides) that have participated in three sedimentary cycles and characterise the absence of preserved fossils. For this reason, sediments that were exposed to prolonged chemical weathering, and almost all of them contain over 50% SiO₂, were considered in this study.

EXPERIMENTAL

Geological settings

Upper Cretaceous–Eocene carbonate and siliciclastic sediments (Fig. 1) were formed in two Internal Dinaridic domains: Jadar-Kopaonik and Drina-Ivanjica, ^{12,13} close to the Sava zone. The Neoproterozoic-Carboniferous basement metamorphics predominantly belonging to green schist facies comprise the Drina-Ivanjica domain and are overlaid by Lower Triassic continental to shallow marine (meta) clastics and Middle to Upper Triassic shallow marine to basinal

formations. 14,15 Jadar-Kopaonik and Drina-Ivanjica thrust sheets are bonded by ophiolites and ophiolitic mélange of western Vardar tectonic unit or thrust sheet. 12,16

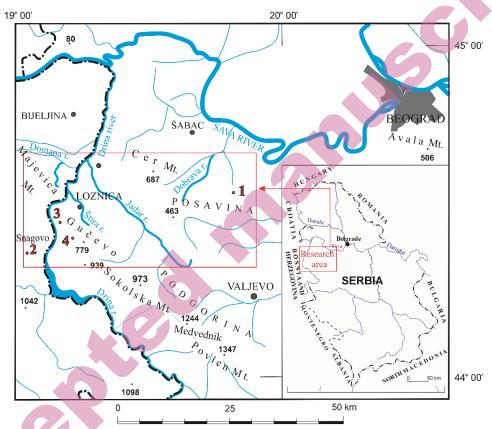


Fig.1. Map of the investigated area, with localities that have been sampled. Legend: 1. Jazovnik; 2. Snagovo; 3. Drina River; 4. Gučevo Mt.

Methods

All analysed samples were crushed using a jaw crusher, pulverised in a mill, and later sieved through a $63~\mu m$ sieve.

The Soxhlet extraction method was used to isolate the soluble organic matter (bitumen). An azeotropic mixture of dichloromethane and methanol (88:12, v:v) served as the solvent. Elemental sulfur was removed by adding copper to the mixture. Afterwards, the saturated hydrocarbon fraction was isolated from bitumen using column chromatography, utilising a silica gel and aluminium oxide (in a 2:1 ratio) as the adsorbent, while *n*-hexane was employed as the eluent. Subsequently, the total saturated fraction was analysed using Gas Chromatography-Mass Spectrometry (GC-MS) in Total Ion Chromatogram (TIC) mode. This analysis was conducted with an Agilent 7890A gas chromatograph equipped with an HP-5MS column (30 m × 0.25 mm, 0.25 μm film thickness, with helium as the carrier gas at a flow rate of 1.5 cm³/min), coupled to an Agilent 5975C Mass Selective Detector. For a detailed analysis

of alkanes, branched alkanes and tricyclic terpanes, the typical mass fragments were analysed: m/z 71, 127, and 191, respectively.

RESULTS AND DISCUSSION

The total ion current (TIC) chromatograms of an aliphatic fraction for most investigated samples are characterised by the presence of an unresolved complex mixture (UCM). The unresolved complex mixture often indicates matured organic matter or a significant contribution of degraded or reworked organic matter. ^{1,3}The TIC chromatograms of two representative samples are shown in Fig.2. This distribution reflects prolonged chemical weathering to which the sediments from Drina, Gučevo, and Snagovo were exposed. The mentioned sediments passed through three cycles of sedimentation (see Geological settings), which influence hydrocarbon distributions, resulting in highly abundant branched hydrocarbons and the presence of UCMs (Fig.2).

Further, n-alkanes are absent or present in low abundance in most samples, except for P1–P8 (Jazovnik). In these samples, even homologs predominate with a maximum at n-C₁₈alkane (Fig.3). At the same time, branched alkanes, monitored using ion m/z 127, are present in all samples. These hydrocarbons have been identified in cyanobacterial cultures,¹⁷ as well as modern and ancient sediments associated with cyanobacterial mat assemblages. ^{18–21}Highly abundant branched alkanes in a large group of studied sediments suggest the presence of microbiologically reworked OM.

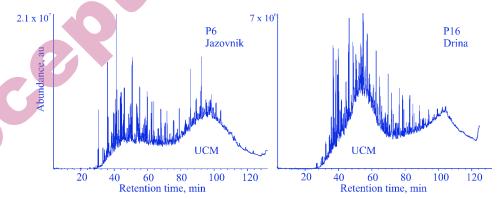


Fig 2. Total ion currents (TICs) of investigated samples.

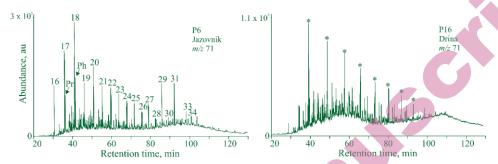


Fig. 3.Fragmentograms of *m/z* 71 for *n*-alkanes and isoprenoids.*n*-Alkanes are labelled according to their carbon number; Legend: Pr—pristane; Ph—phytane; *—represents 5,5-diethylalkanes.

However, literature data indicate that tricyclic terpanes (TT) are significantly more resistant to chemical changes and can be a reliable tool for determining the depositional environment.^{1,3} For example, some studies show that the difference in the distributions of tricyclic and tetracyclic terpane (TeT) ratios can help distinguish sediments and crude oils derived from different sedimentary environments. 4,6,9 In the aliphatic fraction, a series of tricyclic terpanes were identified, including C₂₀TT, C₂₁TT, C₂₂TT, C₂₃TT, C₂₄TT and C₂₆TT, as well as C₂₄TeT and C₂₆TeT. Representative chromatograms of the m/z 191 ion are shown in Fig.4, while the geochemical parameters based on the abundance of certain tricyclic and tetracyclic terpanes are summarised in Table 1. The C23 tricyclic terpane is typically the dominant homolog among C₂₀–C₂₃ tricyclic terpanes in Jazovnik samples (P 1–8), where $C_{23}TT > 60\%$ (Table 1, Figs.4–6). The $C_{23}TT$ domination is characteristic of the marine, reducing marine carbonate setting or saline lacustrine depositional environment. ^{22–24} Since Jazovnik sediments are from the Cretaceous-Paleogene boundary, it can be assumed that they were deposited in an environment that is the remnant of a former sea or in a lake environment that often resembles a marine sedimentation environment in its characteristics. Opposite to the Jazovnik, most samples from Gučevo are characterised by a predominance of C₂₀ and C₂₁TT (Table 1, Fig.4-6). Domination of these tricyclic terpanes is characteristic of terrigenous input. 1,25 The other examined samples from Drina and Snagovo are formed in a mixed depositional environment between continental and marine depositional settings (Table 1, Fig. 4–6).

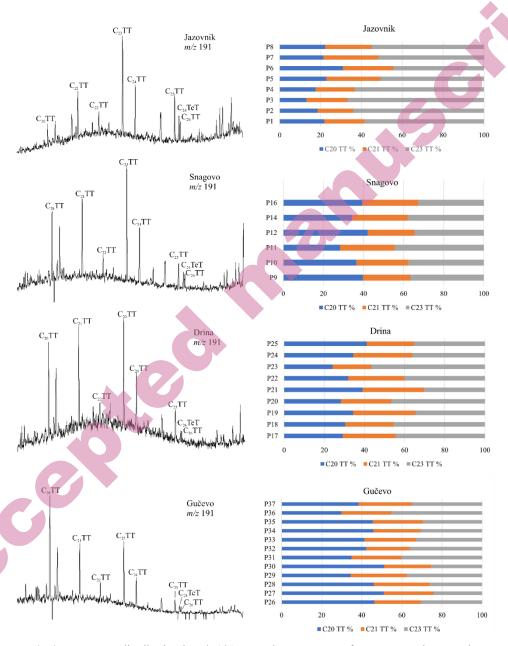


Fig 4.C₁₉–C₂₆ TT distribution in m/z 191 mass chromatograms for representative samples.

Table 1. Organic geochemical parameters based on the abundance of tricyclic and tetracyclic terpanes

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Sam.	Formation	C23/C21TTs	C21/C20TTs	C20/C23TTs	C ₂₄ /C ₂₆ TeTs	$C_{20}+C_{22}$	C ₂₁	C ₂₃
						(%)	(%)	(%)
P1	Jazovnik	2.96	2.03	0.166	1.061	21.85	19.71	58.44
P2		3.72	4.22	0.064	1.082	18.55	17.25	64.20
P3		3.36	7.14	0.042	1.028	13.12	20.16	66.73
P4		3.31	3.39	0.089	0.863	17.58	19.11	63.32
P5		1.92	2.30	0.226	0.862	22.86	26.45	50.69
P6		1.80	1.16	0.477	0.591	30.96	24.62	44.43
P7		1.93	2.61	0.198	1.190	21.48	26.82	51.70
P8		2.43	2.17	0.189	0.727	22.31	22.62	55.07
P9		1.55	0.73	0.885	0.667	39.78	23.58	36.64
P10		1.44	1.04	0.667	0.976	36.48	26.00	37.52
P11	G.	1.64	1.66	0.368	0.606	28.39	27.17	44.44
P12		1.48	0.84	0.807	0.480	42.14	23.33	34.54
P13	Snagovo	0.00	0.00	0.000	0.000	0.00	0.00	0.00
P14		1.36	1.12	0.659	0.654	34.24	27.91	37.85
P15		0.00	0.00	0.000	0.000	0.00	0.00	0.00
P16		1.17	0.55	0.910	0.338	39.37	27.96	32.67
P17	Drina	1.70	1.38	0.427	0.795	29.23	26.22	44.55
P18		1.88	1.19	0.445	1.044	30.45	24.13	45.42
P19		1.12	1.18	0.762	1.568	34.38	31.01	34.61
P20		1.86	1.42	0.379	1.852	28.20	25.13	46.67
P21		1.00	1.00	1.001	1.052	39.07	30.49	30.43
P22		1.43	1.44	0.487	0.884	31.83	28.05	40.11
P23		2.91	1.51	0.228	0.805	24.11	19.42	56.47
P24		1.23	1.31	0.623	1.014	34.46	29.44	36.10
P25		1.50	0.75	0.890	0.962	41.06	23.61	35.32
P26		1.32	0.59	1.287	0.874	46.24	23.13	30.63
P27		0.97	0.53	1.942	0.000	51.05	24.80	24.15
P28		0.93	0.71	1.515	0.647	46.00	27.91	26.09
P29		1.35	1.10	0.674	0.857	34.37	27.98	37.65
P30		1.09	0.55	1.680	0.607	51.14	23.39	25.47
P31		1.63	0.86	0.712	2.400	34.91	24.79	40.30
P32		1.68	0.72	0.830	0.789	42.19	21.56	36.25
P33		1.27	0.78	1.006	1.522	41.31	25.83	32.86
P34		1.30	0.73	1.059	0.900	45.79	23.60	30.62
P35		1.18	0.66	1.289	0.890	45.48	25.04	29.47
P36		1.82	1.27	0.431	0.838	29.89	24.85	45.26
P37		1.32	0.92	0.826	1.371	38.45	26.58	34.97
								/

Legend: TTs—tricyclic terpanes, TeTs—tetracyclic terpanes, $C_{20} + C_{22}$ (%) = (($C_{20} + C_{22}$ TTs) × 100) / ($C_{20} + C_{21} + C_{22} + C_{23}$ TTs), C_{21} (%) = $C_{21}TT \times 100$ / ($C_{20} + C_{21} + C_{22} + C_{23}$ TTs), C_{23} (%) = $C_{23}TT \times 100$ / ($C_{20} + C_{21} + C_{22} + C_{23}$ TTs).

Moreover, cross-plots of C_{23}/C_{21} vs. C_{20}/C_{23} TTs and C_{23}/C_{21} vs. C_{20}/C_{23} TTs (Fig. 5) and the ternary diagram C_{19} – C_{23} TTs (Fig. 6) more reliably depicted paleoenvironmental settings under which investigated sediments were formed.

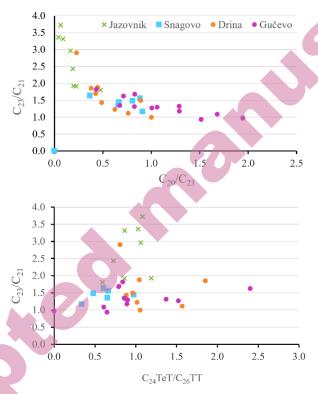


Fig 5. The cross plots of C_{23}/C_{21} vs. C_{20}/C_{23} TTs and C_{23}/C_{21} vs. C_{20}/C_{23} TTs for investigated samples.

A recent study introduced a ternary diagram that enables the differentiation of depositional environments of sediments based on the analysis of C₁₉–C₂₃ TTs.^{4,11,26} In general, rocks with abundant terrigenous organic matter input exhibit higher concentrations of C₁₉TT and C₂₀TT, while a predominance of C₂₁TT characterises sediments deposited in freshwater lacustrine environments.⁶A slightly modified ternary plot (C₂₂TT +C₂₀TT, C₂₁TT, and C₂₃TT, Fig. 6)suggests that Jazovnik sediments fall within zone IV, indicating a marine/saline lacustrine depositional environment. Some Drina samples fall in the same zone, while others are within zone III, indicating a mixed freshwater lacustrine and/or marine/saline lacustrine depositional environment. In contrast, Gučevo samples fall within zone II, indicating fluvial-deltaic depositional settings. Besides, most Snagovo samples

overlap between Zone III (freshwater lacustrine environment) and Zone IV (marine/saline lacustrine environment).

The distribution of tri- and tetracyclic terpanes indicates that the examined sediments were deposited in distinct depositional environments. The sediments from Jazovnik were formed in marine-saltwater lacustrine depositional environments, while sediments from Gučevo were deposited under continental fluvial-deltaic settings. On the other hand, transitions in sedimentary conditions were observed during the formation of the Drina and Snagovo sediments. For some sediments, a salt enrichment within the alluvial system was noticed.

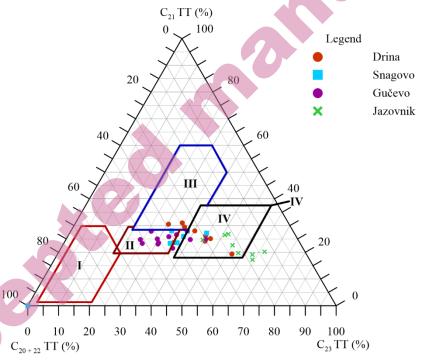


Fig 6. A triangular diagram of C₂₀+C₂₂TT, C₂₁TT, and C₂₃TT depicting distinct depositional settings. **Legend:** I—Swamp facies, II—Fluvial deltaic, III—Freshwater lacustrine, IV—Marine-saltwater lacustrine(modified and adapted from Bao *et al.*, 2023).²⁶

CONCLUSION

The study focused on tracking changes in hydrocarbon distributions of siliciclastic sediments from Jazovnik, Snagovo, Drina and Gučevo (Internal Dinarides) exposed to prolonged chemical weathering. A large group of studied sediments participated in three sedimentary cycles, containing over 50% SiO₂and are characterized by the absence of preserved fossils. Biodegradation, chemical weathering and/or other alteration processes influenced the distribution of

hydrocarbons, resulting in highly abundant branched hydrocarbons and UCMs. In such cases, tri- and tetracyclic terpanes proved susceptible to transitions in paleoenvironmental settings since they are well-known for their resistance to the above-mentioned processes.

The results showed that tri- and tetracyclic terpanes effectively determined the changes in the depositional environments of examined sediments. The sediments from Jazovnik were formed in marine-saltwater lacustrine depositional environments, while sediments from Gučevo were deposited under continental fluvial-deltaic settings. On the other hand, transitions in sedimentary conditions were observed during the formation of the Drina and Snagovo sediments, which resulted from salt enrichment within the alluvial system. The conditions were changing between the freshwater lacustrine and marine/saline lacustrine. Once again, this approach clearly demonstrates differences in depositional settings of the Jazovnik, Snagovo, Drina and Gučevo sediments, offering valuable insights into the geological history of the studied area.

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ИЗВОД

РЕКОНСТРУКЦИЈА СРЕДИНЕ ТАЛОЖЕЊА ЕРОДОВАНИХ СИЛИЦИКЛАСТИЧНИХ СЕДИМЕНАТА ИНТЕРНИХ ДИНАРИДА НА ОСНОВУ РАСПОДЕЛЕ И ОБИЛНОСТИ ТРИ-И ТЕТРАЦИКЛИЧНИХ ТЕРПАНА

ИВАНА ЈОВАНИЋ 1 , АЛЕКСАНДРА ШАЈНОВИЋ 2 , САЊА СТОЈАДИНОВИЋ 2 , НИКОЛА БУРАЗЕР 2 , БОЈАН ГЛАВАШТРБИЋ 1 , БРАНИМИР ЈОВАНЧИЧЕВИЋ 3

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Ова студија испитује могућност коришћења три- и тетрацикличних терпана за карактеризацију средине таложења седимената који немају очуване фосиле и чија је расподела биомаркера нарушена биоразградњом или другим процесима. Анализирани су силицикластични седименти који су били изложени продуженом хемијском и атмосферском деловању на стене, које је утицало на расподелу угљоводоника. То је резултирало високом обилношћу разгранатих угљоводоника и присуством нераздвојене комплексне смеше једињења (UCM). Из тог разлога је релативна обилност и расподела три- и тетрацикличних терпана (C_{20} — C_{24} TT, C_{26} TT, C_{24} TeT), као резистентних биомаркера, коришћена за процену услова таложења. Триангуларни дијаграм који укључује C_{20} + C_{22} %, C_{21} % и C_{23} % показао се посебно ефикасним за разликовање мочварне, флувијално-делтске, слатководне и морско-слане језерске средине седиментације. Овај приступ је јасно разграничио депозиционе средине седимената из Јазовника, Снагова,

Дрине и Гучева (унутрашњи Динариди), пружајући вредан увид у геолошку историју проучаваног подручја.

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