Journal of the Serbian Chemical Society
JSCS@tmf.bg.ac.rs • www.shd.org.rs/JSCS

J. Serb. Chem. Soc. 90 (0) 1–10 (2025)
JSCS–13179

Original scientific paper
Published 23 Dec 2025

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

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(Received 27 December 2024, revised 4 February, accepted 11 April 2025)

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 (C_{20}–C_{24}TT, C_{26}TT, C_{24}TeT) 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, the application of specific organic-geochemical parameters based on the distri-

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https://doi.org/10.2298/JSC241227026J

bution 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, the 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).[1,2] Only a few proxies are available for assessing depositional environments, and they rarely allow unambiguous interpretations. Among them, the tricyclic terpanes seem to be the most useful.[2]

The tricyclic terpanes are the organic compounds ubiquitous in sediments and oils spanning the geological record.[1,3] 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 the oil-to-oil correlations,[4] the oil-to-source correlations,[5,6] the thermal maturity,[7] as well as the depositional environment characteristics[8] and the OM inputs.[9–11] In the identifying depositional settings, the specific tricyclic terpanes (*e.g.*, C_{19} + C_{20}, C_{21}, C_{23}TT) are key for determining of the origin of OM: C_{23} TTs are often associated with marine environments and can indicate the presence of planktonic organic matter,[1,3] C_{21}TT is typically derived from the degradation of higher plant material, such as terrigenous plants,[9] while C_{22}TTs are similar to C_{21}TT also derived from higher plant material, but can show variations based on the maturity and depositional environment.[5]

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, the sediments that were exposed to prolonged chemical weathering, and almost all of them contain over 50 % SiO_2,were considered in this study.

EXPERIMENTAL

Geological settings

Upper Cretaceous–Eocene carbonate and siliciclastic sediments (Fig. 1) were formed as sedimentary cover 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 while Jadar unit consist of Paleozoic clastics and carbonates. These rocks are overlaid and are overlaid by the Lower Triassic continental to shallow marine meta (clastics) and the Middle to Upper Triassic shallow marine to basinal formations.[14,15] The Jadar-Kopaonik and the Drina-Ivanjica thrust sheets are bonded by ophiolites and ophiolitic mélange of western Vardar tectonic unit or thrust sheet.[12,16]

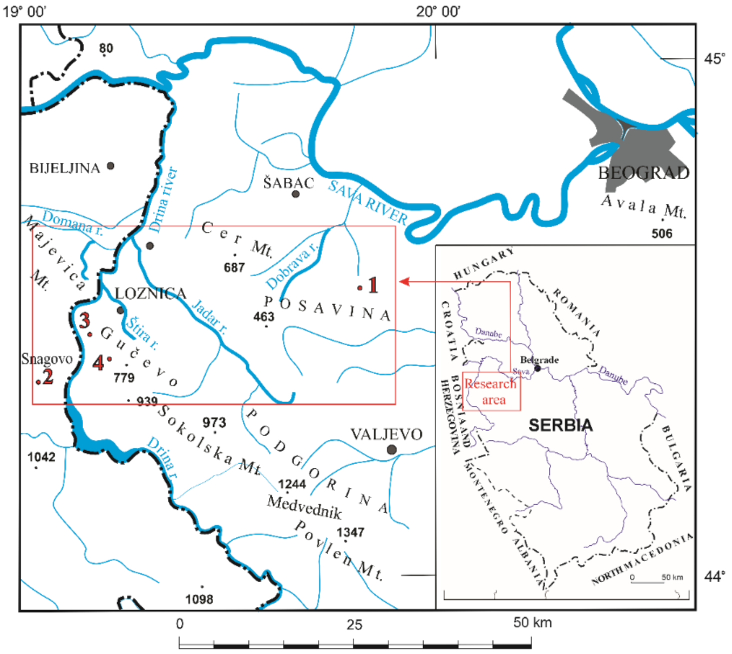

Fig. 1. Map of the investigated area, with localities that have been sampled. Legend: 1. Jazovnik; 2. Snagovo; 3. Drina River; 4. Gučevo Mt.

Methods

All analysed samples were crushed using a jaw crusher, pulverised in a mill, and later sieved through a 63 µm sieve.

The Soxhlet extraction method was used to isolate the soluble organic matter (bitumen). An azeotropic mixture of dichloromethane and methanol (88:12 volume ratio) served as the solvent. Elemental sulphur was removed by adding copper to the mixture. Afterwards, the saturated hydrocarbon fraction was isolated from bitumen using column chromatography, using the silica gel and aluminium oxide (in a 2:1 mass 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 cm^3/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 the 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).

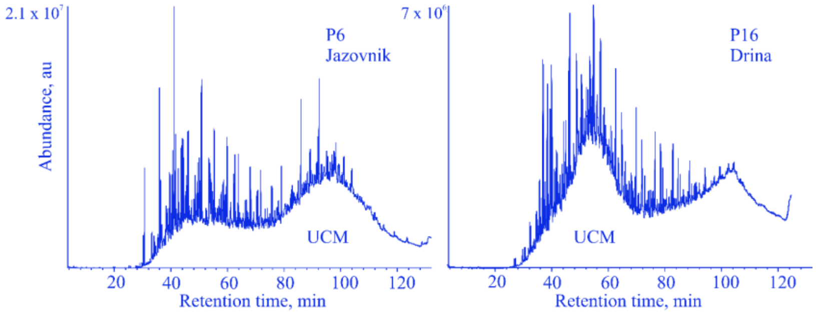

Fig. 2. Total ion currents (TICs) of investigated samples.

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*-C_{18}alkane (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] The highly abundant branched alkanes in a large group of studied sediments suggest the presence of microbiologically reworked OM.

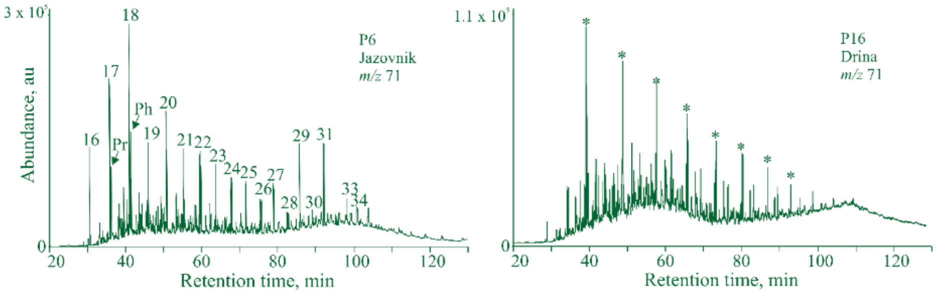

Fig. 3. Fragmentograms of *m*/*z* 71 for *n*-alkanes and isoprenoids. *n*-Alkanes are labelled according to their carbon number; Legend: Pr – pristane; Ph – phytane; * – 5,5-diethylalkanes.

However, the 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 C_{20}TT, C_{21}TT, C_{22}TT, C_{23}TT, C_{24}TT and C_{26}TT, as well as C_{24}TeT and C_{26}TeT. The representative chromatograms of the *m*/*z* 191 ion are shown in Fig. 4, while the geochemical parameters based on the abundance of certain tri-

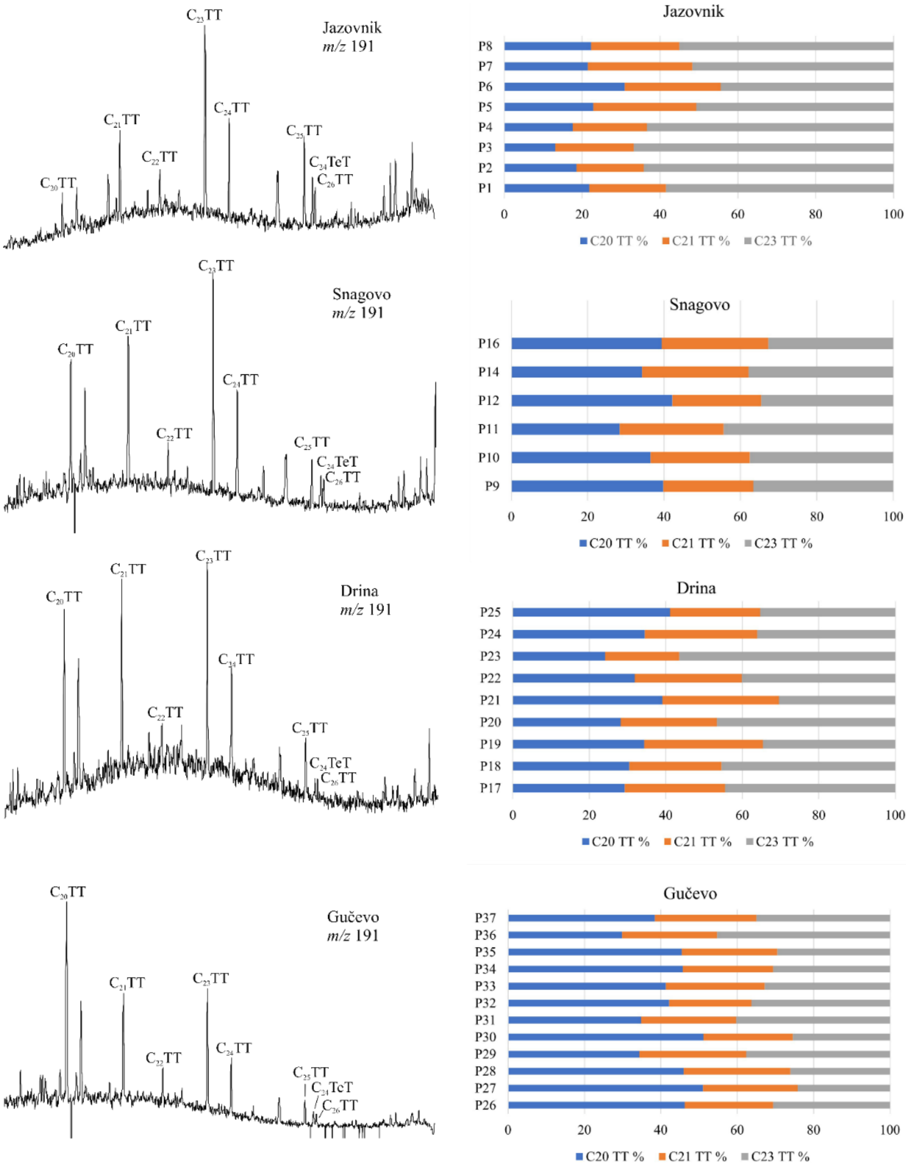

Fig. 4. C_{19}–C_{26} TT distribution in *m*/*z* 191 mass chromatograms for representative samples.

cyclic and tetracyclic terpanes are summarised in Table I. The C_{23} tricyclic terpane is typically the dominant homolog among C_{20}–C_{23} tricyclic terpanes in Jazovnik samples (P1–P8), where C_{23}TT > 50 % (Table I, 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 C_{20} and C_{21}TT (Table I, Figs. 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 I, Figs. 4–6).

TABLE I. Organic geochemical parameters based on the abundance of tricyclic and tetracyclic terpanes; Legend: TTs – tricyclic terpanes, TeTs – tetracyclic terpanes, $C_{20} + C_{22}$ (%) = 100($C_{20} + C_{22}$TTs)/($C_{20} + C_{21} + C_{22} + C_{23}$TTs), C_{21} (%) = 100C_{21}TT/($C_{20} + C_{21} + C_{22} + C_{23}$TTs), C_{23} (%) = 100C_{23}TT/($C_{20} + C_{21} + C_{22} + C_{23}$TTs)

Sam.	Formation	C_{23}/C_{21}TTs	C_{21}/C_{20}TTs	C_{20}/C_{23}TTs	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	Snagovo	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		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		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	Gučevo	1.32	0.59	1.287	0.874	46.24	23.13	30.63

TABLE I. Continued

Sam.	Gučevo	C_{23}/C_{21}TTs	C_{21}/C_{20}TTs	C_{20}/C_{23}TTs	C_{24}/C_{26}TeTs	C_{20}+C_{22} (%)	C_{21} (%)	C_{23} (%)
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

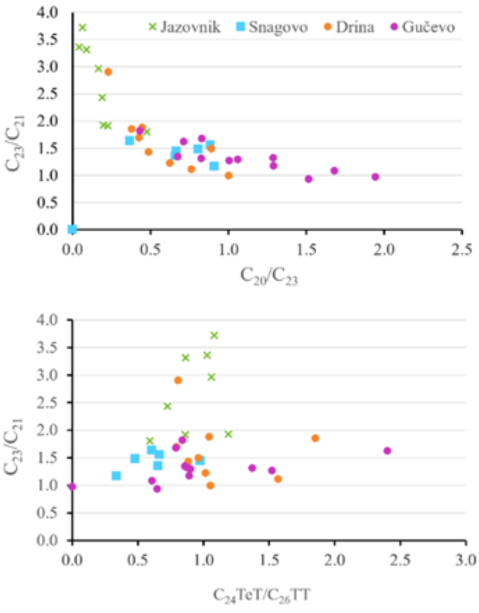

Fig. 5. The cross plots of C_{23}/C_{21} *vs.* C_{20}/C_{23} TTs and C_{23}/C_{21} *vs.* C_{20}/C_{23} TTs for investigated samples.

A recent study introduced a ternary diagram that enables the differentiation of depositional environments of sediments based on the analysis of C_{19}–C_{23} TTs.[4,11,26] In general, the rocks with abundant terrigenous organic matter input exhibit higher concentrations of C_{19}TT and C_{20}TT, while a predominance of C_{21}TT characterises sediments deposited in freshwater lacustrine environments.[6] A slightly modified ternary plot (C_{22}TT +C_{20}TT, C_{21}TT and C_{23}TT, Fig. 6) suggests that Jazovnik sediments fall within zone IV, indicating a brackish/marine depositional environment. Some Drina samples belong to the same zone, while others are within zone III, indicating a mixed floodplain/freshwater lacustrine depositional environment. In contrast, the Gučevo samples fall within zone II, indicating the fluvial-deltaic depositional settings. Besides, the most of Snagovo samples overlap between the Zone III (floodplain/freshwater lacustrine environment) and the Zone IV (brackish/marine environment).

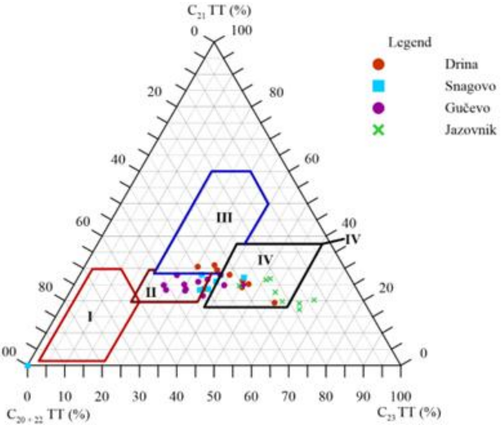

Fig. 6. A triangular diagram of C20+C22TT, C21TT and C23TT depicting distinct depositional settings. Legend: I – Swamp facies, II – Fluvial deltaic, III – Floodplain/freshwater lacustrine, IV – Brackish/marine (modified and adapted from Xiao et al., 2019c, Bao et al., 2023, and Xiao et al., 2024)[10,11,26]

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 brackish/marine depositiional environments, while the sediments from Gučevo were deposited under continental fluvial-deltaic settings. On the other hand, the 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.

Moreover, 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 (Fig. 5) and the ternary diagram C_{19}–C_{23} TTs (Fig. 6) more reliably depicted paleoenvironmental settings under which investigated sediments were formed.

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 % SiO_2 and are characterized by the absence of preserved diagnostic 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 the brackish/marine lacustrine depositional environments, while the sediments from Gučevo were deposited under continental fluvial-deltaic settings. On the other hand, the transitions in sedimentary conditions were observed during the formation of the Drina and Snagovo sediments, which resulted from the salt enrichment within the alluvial system. The conditions were changing between the floodplain/freshwater lacustrine and brackish/marine. Once again, this approach clearly demonstrates the differences in depositional settings of the Jazovnik, Snagovo, Drina and Gučevo sediments, offering some valuable insights into the geological history of the studied area.

Acknowledgments. The study was partly financed by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia (Contract numbers 451-03-136/2025-03/200026 and 451-03-136/2025-03/200168).

ИЗВОД

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

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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-делтне, 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.

(Примљено 27. децембра 2024, ревидирано 4. Фебруара, прихваћено 11. априла 2025)

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