

Middle Miocene marine flooding: new $^{40}\text{Ar}/^{39}\text{Ar}$ age constraints with integrated biostratigraphy on tuffs from the North Croatian Basin

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Abstract

In the North Croatian Basin which is located in the southwestern part of the Pannonian Basin System, Miocene tuff deposits have been observed at several localities in the area of Banovina, Medvednica Mt. and Slavonia. Here we present new $^{40}\text{Ar}/^{39}\text{Ar}$ age results obtained from volcanic glass from the Laz tuff (15.42 ± 0.15 Ma) intercalated with lacustrine freshwater/brackish deposits, the Jovac tuff (15.10 ± 0.06 Ma) intercalated with lacustrine freshwater deposits, the Čučerje tuff (14.81 ± 0.08 Ma) and the Nježić tuff (14.40 ± 0.03 Ma) both deposited in a marine environment. Fossil data (calcareous nannofossils/foraminifera) from the underlying and overlying beds of the tuffs from Čučerje and Nježić match the geochronological data i.e. NN5 zone and M6 zone were determined. Integration of biostratigraphic and geochronological data enable a better understanding of the NCB sedimentary evolution and constrain the Middle Miocene marine flooding event in the marginal areas of the western part of the NCB at ~15 Ma i.e. early/middle Badenian boundary. These results together with the existence of lower Badenian marine sediments in the Sava depression (in the southern part of NCB) suggest it is possible to conclude that during the early Badenian in the NCB, freshwater lacustrine and marine environments coexisted.

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1. INTRODUCTION

The Neogene Pannonian Basin System (PBS) (Fig. 1A) was part of the Central Paratethys Sea (RÖGL & STEININGER, 1983; RÖGL, 1999; PAVELIĆ, 2001; POPOV et al., 2004; HARZHAUSER & PILLER, 2007, KOVÁČ et al., 2017a). From the Oligocene to the late early or early middle Miocene however, the sea covered only the NW part of the PBS, whereas its other parts were subaerially exposed or characterized by continental deposition. Marine sedimentation became widespread in the entire PBS only during the middle Miocene, Badenian age.

The North Croatian Basin (NCB, Fig. 1B) represents the south-western part of the PBS where specific lacustrine depositional environments developed prior to the marine flooding. The freshwater and brackish sediments contain endemic fauna, complicating biostratigraphic correlation and dating. The exact timing of the initial marine flooding in the NCB is thus currently unknown. Estimates of the timing of the marine flooding event in the southern part of the Pannonian Basin range from 17 Ma to 15 Ma (HAJEK-TADESSE et al. 2009; PREMEC FUČEK et al., 2017; HERNITZ KUČENJAK et al., 2018; PAVELIĆ & KOVAČIĆ, 2018; SANT et al., 2018, 2019; MANDIĆ et al 2019a, BRLEK et al., 2020) and are a matter of debate.

The main objective of this paper is to provide new radio-isotopic data using the $^{40}\text{Ar}/^{39}\text{Ar}$ method for tuffs from several localities in the NCB. In combination with micropaleontological analysis (calcareous nannofossils, foraminifera, ostracods and palynomorphs) these ages should enable better understanding of the NCB sedimentary evolution and constrain the Middle Miocene marine flooding event in the NCB.

2. GEOLOGICAL SETTING

The North Croatian Basin (NCB) geographically occupies the southwestern part of the Pannonian Basin System (PBS) (Fig. 1A). The PBS is located between the mountain ranges of the Carpathians, Alps and Dinarides. It is composed of a series of smaller, deep depressions or basins, separated by relatively shallow pre-Neogene basement (SCHMIDT et al., 2008; USTASZEWSKI et al., 2014). Palaeogeographically it belongs to the Central Paratethys area (CP) (RÖGL & STEININGER, 1983; POPOV et al., 2004; HARZHAUSER & PILLER, 2007). The origin of the PBS is considered to be the time when lithospheric fragments of ALCAPA and Tisza Dacia were placed in their more or less current position, creating a domain for the initial stage of rifting (e.g. FODOR et al., 1989; KOVÁČ et al., 2018b). The evolution of the PBS has been subdivided into the syn-rift and post-rift phases (HORVÁTH & TARI 1999; HORVÁTH et al., 2006). The duration of these phases was diachronous across the basin (MATTENCO & RADIVOJEVIĆ, 2012; BALÁZS et al., 2016).

In the southwestern part of the PBS, i.e. in the NCB, the syn-rift phase lasted from the Oligocene to the middle Badenian (PAVELIĆ, 2001). It was characterized by tectonic thinning of the crust, isostatic subsidence, increased volcanic activity (PAMIĆ, 1997; LUKÁCS et al., 2015, 2018; SZAKÁCS et al., 2018) and a transition from continental to marine sedimentary environments (PAVELIĆ & KOVAČIĆ, 2018). This change was caused by the interplay of extensional tectonics, climate change and eustatic sea level changes.

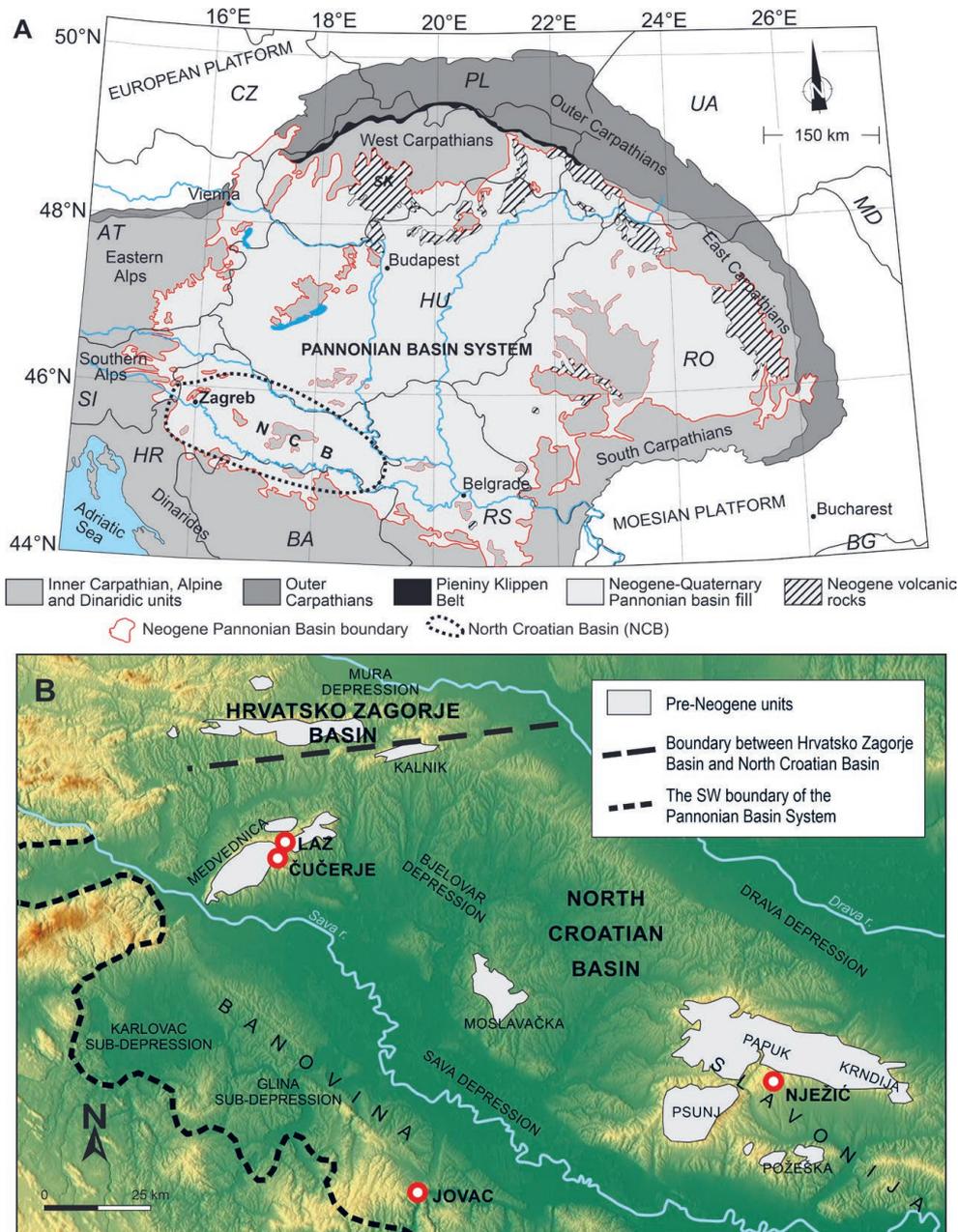


Figure 1. The geographic position of the North Croatian Basin (NCB) within the Pannonian Basin System (simplified after CVETKOVIĆ et al., 2019) (A). Geographic position of the localities: Jovac, Laz, Čučerje and Nježić within the NCB (B).

The post-rift phase started during the middle Badenian and extended into the Quaternary. During this phase, rifting stopped and subsidence continued due to cooling of the lithosphere (ROYDEN, 1988; PAVELIĆ, 2001; PAVELIĆ & KOVAČIĆ, 2018). The post-rift phase is characterized by the transition from marine to continental sedimentary environments, a halt in volcanic activity and two compressional phases that caused the inversion of the basin and the uplift of individual blocks (JAMIČIĆ, 1995; MÁRTON et al., 1999, 2002; PAVELIĆ, 2001; TOMLJENOVIĆ & CSONTOS, 2001; PAVELIĆ et al., 2003; USTASZEWSKI et al., 2014).

Although connections with the Mediterranean and the Indo-Pacific Ocean were established occasionally during the Oligocene and the Middle Miocene (NAGYMAROSY & MÜLLER, 1988; STEININGER et al., 1988; RÖGL, 1996a; HARZHAUSER & PILLER, 2007; KOVÁČ et al., 2007, 2017; PILLER et al., 2007; SANT et al., 2017), most of the time the PBS was an isolated sedi-

mentary environment. Isolation has led to the development of endemic fauna. Correlation of PBS Miocene sediments to Mediterranean sediments is therefore not straightforward (RÖGL & STEININGER, 1984; NAGYMAROSY & MÜLLER, 1988; STEININGER et al., 1988). Biostratigraphy based on endemic fauna only allows the establishment of regional stages for the Central Paratethys area (STEININGER et al., 1976; modified by HOHENEGGER et al., 2014; MANDIĆ et al., 2015; NEUBAUER et al., 2015).

The stratigraphic position of the oldest terrestrial deposits of the PBS in the NCB was problematic in the past due to either endemic fossil species or the complete lack of fossils. Age data (ca. 18 Ma) obtained from pyroclastics intercalated with alluvial deposits from Kalnik Mt. (MANDIĆ et al., 2012; BRLEK et al. 2020) confirmed their Otnangian age and marked the initial time of deposition. Early Miocene environments with alluvial deposits (JAMIČIĆ et al., 1987) and a salina type lake (ŠČAVNIČAR et al.,

1983) lasted up to the end of the Karpatian (PAVELIĆ & KOVAČIĆ, 2018). In the early Badenian a fresh-water lacustrine environment was formed (ĆORIĆ et al., 2009). This lake covered either the whole area of the NCB (PAVELIĆ, 2001) or only the syn-rift depocentres (SAFTIĆ et al., 2003) forming a system of lakes (MANDIĆ et al., 2019a). The lacustrine environment enabled an emergence of fresh-water mollusc and ostracod fauna. Sedimentation was accompanied by the deposition of tuffs and tuffites and was influenced by a significant change to a humid climate and water level changes (MUTIĆ, 1969, 1973, 1980; JAMIČIĆ et al., 1987; PAVELIĆ et al., 2000; PAVELIĆ, 2001). In older studies, the lacustrine deposits were correlated by their freshwater fauna and flora (ŠIKIĆ, 1968; KOCHANSKY-DEVIDÉ, 1979; SOKAČ, 1987; ŽAGAR-SAKAČ, 2004), and they were attributed to the Ottungian–Karpatian period. Palynological study (KRIZMANIĆ, 1995) suggested an age between the Karpatian and early Badenian. Radio-isotope ages of tuff layers of ~16Ma within lacustrine sediments defined an important chronostratigraphic marker (MANDIĆ et al., 2012). The transition from lacustrine to marine conditions was gradual in many localities in the NCB (PAVELIĆ & KOVAČIĆ, 2018) except at Medvednica Mt. (BRLEK et al., 2016) and the Nježić locality (KOVAČIĆ et al., 2015a). Recent biostratigraphic studies (ĆORIĆ et al., 2009; MARTINUŠ et al., 2013; BRLEK et al., 2016) placed the initial marine sedimentation in the early middle Badenian. The marine deposition regime persisted until the end of the Middle Miocene (11.6 Ma) when the basin was finally isolated (RÖGL, 1996a, b; MAGYAR et al. 1999; PILLER & HARZHAUSER, 2005).

3. METHODS

3.1. Fieldwork

Four outcrops were studied in the NCB, ranging from 1 – 140m in thickness, that include tuff layers: Jovac, Laz, Čučerje, and Nježić. GPS coordinates are provided in the results section. The sections were logged in detail, including full lithostratigraphic and sedimentologic descriptions. In addition to recording observations in a field diary, all localities were documented with photos. The immediate underlying and overlying beds of the main tuff layers were sampled for micropaleontological analyses (calcareous nannofossils, foraminifera, ostracods and palynomorphs). The main tuff itself (or the main layers) was sampled at each site for mineralogical analysis i.e. for mineral separation of sanidine for $^{40}\text{Ar}/^{39}\text{Ar}$ dating. For thicker layers of tuff, two samples were obtained, one at the bottom and one at the top of the layer. Sample codes are given according to the nearest toponym.

3.2. $^{40}\text{Ar}/^{39}\text{Ar}$ dating

Bulk tuff samples were crushed, washed, sieved and processed using standard heavy liquid and magnetic separation techniques. Volcanic glass was irradiated for 12h at the Oregon State CLICIT facility, together with Fish Canyon tuff sanidine (FCs). Sample and standards were fused and released gas was analyzed on a Helix MC noble gas mass spectrometer. For data reduction, the ArArCalc software (KOPPERS, 2002) was used, with FCs of 28.201 Ma (KUIPER et al., 2008), MIN et al. (2000) decay constants and the atmospheric air value of LEE et al. (2006). Analytical details are provided in the Supplementary file 1 (6.1. & 7.1.)

3.3. Palaeontological analysis

3.3.1. Calcareous nannofossil analyses

Six samples from the Nježić locality and two samples from the Čučerje locality were prepared for calcareous nannofossil analy-

ses. Standard preparation techniques for calcareous nannofossil analyses were used as described in PERCH-NIELSEN, 1985. Before smear slide preparation, a small amount of the sample was dispersed in an ultrasonic bath. The dispersed sample was mounted on the slide and fixed with *Eukit* mounting medium. Microscopic analysis was performed using a LEICA DMLP light microscope with 1000x magnification (cross and parallel nicols). Biostratigraphic zones were defined using standard zonation after MARTINI, 1971.

3.3.2. Palynological analyses

Palynological analyses were carried out for samples from Laz, Čučerje and Nježić. Standard palynological processing techniques were used to extract the organic matter (e.g. MOORE et al., 1991; WOOD et al., 1996). To remove the carbonates and silica, samples were treated with cold HCl (15%) and HF (40%). Separation of the organic matter from undissolved inorganic components was carried out using a heavy liquid (ZnCl_2 , density $>2.1 \text{ g/cm}^3$). The organic residue was sieved through a 10 μm mesh sieve. For palynomorph analysis, slides were mounted in silicon oil. Microscopic analysis was performed using an Olympus BH-2 and Leica DM2500 microscope (HGI-CGS). Photomicrographs were taken using an AmScopeTM camera adapter connected to the AmScope v.3.7 camera software and a Leica MC190 HD camera connected to the Leica LAS EZ software.

3.3.3. Foraminiferal and ostracod analyses

All available samples from both underlying and overlying beds of the tuff samples (12 samples in total) were prepared for ostracod and foraminiferal analyses. Approximately 200 g of sediment per sample was disintegrated in diluted hydrogen peroxide for 48 hours. Samples were then sieved (0.5; 0.25; 0.2; 0.125; 0.09; 0.063 mm) and dried at room temperature. Some samples required additional cleaning, so the treatment with hydrogen peroxide was repeated, and then the samples were immersed in an ultrasonic bath for 20 seconds. All microfossil remains (ostracods, foraminifera, fish teeth, claws of freshwater crabs, algal thalli and rhizoliths) were examined under a Zeiss Stemi 2000 microscope (HGI-CGS), Zeiss Stemi SV 11 (University of Zagreb) and Olympus SZX16 stereo microscope (INA).

Determination of the planktonic foraminifera genera and species was based on CICHA et al. (1998); WADE et al. (2018) and YOUNG et al. (2020). Biostratigraphic interpretation was based on ĆORIĆ et al. (2009), IACCARINO et al. (2011), WADE et al. (2011), HOHENEGGER et al. (2014), KOVAČ et al. (2017a), KOVAČ et al. (2018a) and LIRER et al. (2019). Photomicrographs of the planktonic foraminifera were taken by Scanning electron microscope (SEM) JEOL JSM-6510 LV (INA).

For the analysis of benthic foraminifera the dried material (four samples from Nježić locality and one sample from Čučerje) was repeatedly split by a Retsch microsplits to obtain standard samples with 300 randomly chosen foraminiferal specimens. Then the plankton/benthos (P/B) ratio was calculated only for samples rich in individuals (Njež-A-b, Njež-B-t, Njež-C-b and Njež-C-t). Furthermore, 300 individual benthic foraminifera were picked for further analyses. Taxonomic identification of benthic foraminifera followed PAPP et al. (1978), PAPP & SCHMID (1985), CICHA et al. (1998), and LOEBLICH & TAPPAN (1988a, b). Ecological/palaeoecological requirements of benthic foraminifera species were taken from PEZELJ et al., 2013 (including all references therein). Middle Miocene local small benthic foraminifera ecozones for the North Croatian Basin were

defined after PEZELJ & DROBNJAK, 2019 and correlated to the regional and standard stages.

Ostracods were picked but not analyzed statistically. Species identifications were based on VAN MORKHOVEN (1963), MEISCH (2000), FRENZEL et al. (2010) and FUHRMANN (2012). SEM photomicrographs of ostracods, fish teeth and claws of freshwater crab were taken using gold-coated samples in an SEM JEOL JSM-35CF system at the Croatian Geological Survey.

4. RESULTS

4.1. Materials – sample location and outcrop description

4.1.1. Laz

The outcrop is located on Medvednica Mt. along the road at the Laz pass (N45° 57' 28.36"; E16° 05' 26.03") (Fig. 1B). The sequence begins with a layer of gray-yellow marl, which is ex-

remely rich in the fossil remains of leaves and ostracods (Fig. 2). The thickness of the layer is at least 120 cm, (based on the exposed part of the layer). In the middle of the sequence a thin layer (2-3 cm thick), is prominent being dark green in colour (Fig. 2C), which represents only the upper part of the tuff layer. The tuff layer consists of two parts. The lower part is very weathered ochre-brown in colour and relatively coarse-grained (Fig. 2C). The upper part is dark green and completely fine-grained (clayey). It can be traced laterally over a length of 10 m. The transition to marls in the overlying bed is gradual. This tuff layer doesn't look reworked i.e. there are no signs of reworking. The overlying beds are > 2 m thick, and the rest of the sequence is covered with vegetation. Above the tuff layer itself, remnants of very delicate ostracod shells were observed. Layers 1 to 4 cm thick and fossil leaf remains have been observed in the marl. Five samples were taken (Fig. 2A).

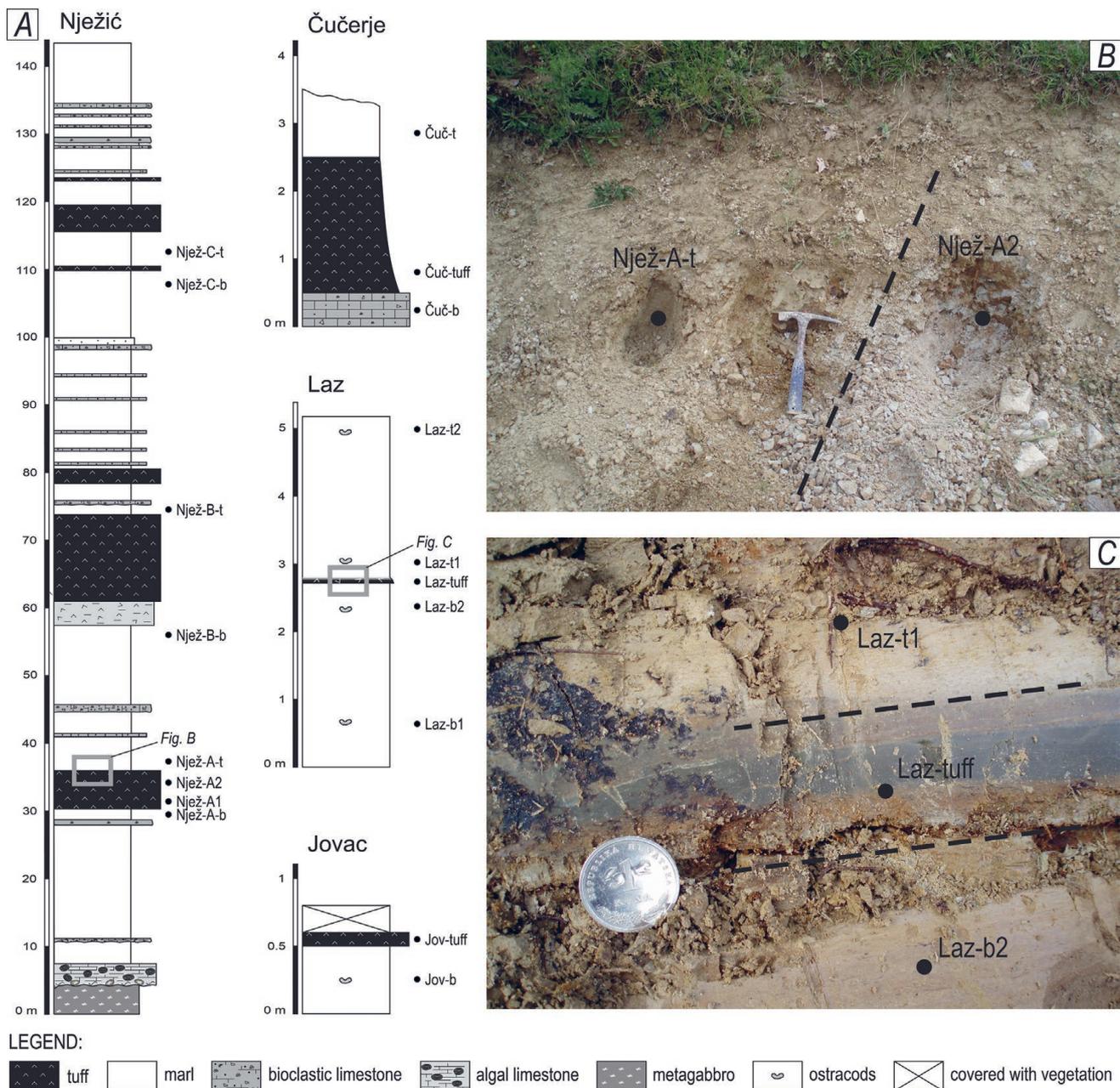


Figure 2. Stratigraphic columns from localities: Jovac, Laz, Čučerje and Nježić with marked sample locations (A); contact between Njež-A-t (marl) and Njež-A2 (tuff) (B); Layer of tuff from Laz locality (in between the dashed lines, marked Laz-tuff) (C) Coin diameter = 2.2 cm.

4.1.2. Jovac

In the area of Banovina, on the right side of the road leading to the village of Jovac, on the saddle before the descent towards the bridge on the stream, a layer of tuff about ten centimetres thick was discovered in the outcrop (N45° 10' 33.95"; E16° 28' 03.82"). The tuff layer is gray, clayey and contains large crystals of biotite (up to 1.5 mm), bronze in colour, only in its lower part. This tuff layer doesn't look reworked i.e. there are no signs of reworking. The underlying layer is composed of a clay rich marl at least 50 cm in thickness. Two samples were taken (Fig. 2A).

4.1.3. Nježić

Nježić is located in the eastern part of the NCB in the Slavonia Region near the village of Nježić on the southwestern slopes of Papuk Mt. (N45° 26' 28.76"; E17° 31' 52.73") (Fig. 1B). The outcrop is described in (KRKALO & MUTIĆ, 1978) and is located along a macadam road leading from the village to the northwest, along which a 140-metre-thick sequence can be traced. Within this sequence, five lithofacies were distinguished: metagabbro facies, algal limestone facies, marl facies, bioclastic limestone facies and tuff facies (Fig. 2).

The metagabbro facies occupies the lower 4 metres of the sequence and represents PBS basement rocks and corresponds to the metagabbro of the Psunj metamorphic complex (JAMIČIĆ, 1988).

The algal limestones facies transgressively overlies the metagabbro, and is present only in the lower part of the sequence as a 3 metre thick layer. It consists of massive Lithothamnium limestone in which there are rhodoliths preserved in some places.

Marl facies appear along the entire sequence and comprise about 70% of the deposits in the sequence. The marl is most often massive, weakly siltose, rich in microfossil fauna (benthic and planktonic foraminifera), as well as calcareous nanoplankton and fragments of echinoderms and bryozoans.

The facies of bioclastic limestones comprises up to 5% of the sequence and appears along the entire sequence in the form of layers of centimetre-decimetres thicknesses. Bioclastic limestones are composed of redeposited fragments of algae, foraminifera, molluscs, echinoderms and bryozoans.

The tuff facies occupies about 25% of the sequence and is most abundant in its central part. The thickness of the tuff layers ranges from >10 cm to almost 20 m. Tuff is fine-grained, light gray to greenish-gray in colour. Tuff layers do not show signs of reworking. Samples from three layers of tuff (at the bottom and at the top of the tuff layer) and marls from their immediate underlying and overlying beds were taken for analyses Fig. 2B. The oldest in the tuff sequence is marked (A), the slightly younger (B) and the youngest (C).

4.1.4. Čučerje

On the southern slopes of Medvednica Mt., near the centre of Čučerje village (N45° 53' 45.44"; E16° 03' 17.80") (Fig. 1B) along the stream, there is a steep outcrop described in the literature as the Plaz or Podplaz locality. A sequence of 42 m in thickness can be observed. The first to describe this outcrop was GORJANOVIĆ-KRAMBERGER (1923) followed by other studies: KOCHAN-SKY-DEVIDÉ (1944, 1956), ŠIKIĆ (1968), AVANIĆ (1995b, 1997) and ČORIĆ et al. (2009). The first 15 metres of the sequence consist of marls and silts intercalated with thin layers of sand. The middle part consists of partially stratified sands and sandstones, while the upper part of the sequence contains a layer of tuff (~36m) and marl. The outcrop marked "Čučerje" is located in a very steep

cut in the forest about twenty metres east of the outcrop described as the Podplaz locality. The sequence (Fig. 2A) begins with a layer of limestone on top of which there is a 2m thick layer of gray tuff. The beds overlying the tuff consist of marl. The tuff layer is coarse-grained in the lower part with relatively large biotite crystals (up to 1.5 mm), while the upper part of the tuff layer is fine-grained. The tuff layer doesn't show signs of reworking. Limestone from bottom deposits marked Čuč-b, coarse-grained tuff marked Čuč-tuff and marl from the overlying beds Čuč-t were sampled (Fig. 2A). The underlying and overlying beds of the tuff described in the Podplaz outcrop are siltstones and limestone. These are not present in our section, so these two tuffs cannot be correlated based on lithology. Based on the small distance (20 m) from the Podplaz outcrop and the close position (altitude) of the layers, the sequence on the Čučerje outcrop is very likely a continuation of the Podplaz outcrop, so the tuff from the Čučerje sequence is younger than the one exposed at the Podplaz outcrop.

4.2 $^{40}\text{Ar}/^{39}\text{Ar}$ dating results and age interpretation

An overview of the results is displayed in Tab. 1 and Fig. 3. All the relevant analytical data is presented in supplementary tables for both samples and standards: Jovac, Čučerje, Laz and Nježić, Fish Canyon sanidines with sample codes F7, F8 and F9 (Supplementary file 1: 1.1.–5.4. & 8.1). The standard data yield a K/Ca ratio between ~11–100 with radiogenic ^{40}Ar contents of ~99%. The weighted mean J value or neutron flux parameter/monitor of the three standards is reproducible and yields 0.0030906 ± 0.0000010 ($n=20/21$; MSWD 1.37).

Ten fusion experiments with ~5 grains per hole for the 250–500 μm Jovac glass fraction (VU101-R2) were performed. The samples have low K/Ca ratios. Glass can have issues with Ar retention or excess argon MCDOUGALL & HARRISON (1999). However, the radiogenic argon content is around 90% which is good for a low K sample and results range from 14.99 ± 0.12 Ma to 15.77 ± 0.03 (Supplementary material – file 1: 2.1–2.3). Note that spread in $^{40}\text{Ar}/^{39}\text{Ar}$ ages is also observed in other studies, although not necessarily in volcanic glass (De LEEUW et al., 2010, 2013; MANDIĆ et al., 2012).

The observed scatter can be explained by several potential mechanisms: 1) open system behaviour, i.e. argon (resulting in ages that are too young) and/or potassium loss (resulting in ages that are too old); 2) mixed age populations of volcanic glass implying some sort of reworking of the volcanic tuffs; 3) contamination with older detrital minerals. Based on the data obtained it is not possible to test to what extent these factors had an impact on the observed ages. Comparison of multiple vs. single grain data from Miocene tuffs is discussed by SANT et al., 2020. A reliable isochron could not be defined, but there are no indications for excess argon. The youngest dated samples might approach eruption age best if they did not suffer from argon loss. The

Table 1. Summary of the $^{40}\text{Ar}/^{39}\text{Ar}$ results.

Locality	Weighted mean age (Ma)	n	N	MSWD	Material
Nježić	14.40 ± 0.03	3	10	2.08	Volcanic glass
Čučerje	14.81 ± 0.08	4	10	1.45	Volcanic glass
Jovac	15.10 ± 0.06	4	10	0.96	Volcanic glass
Laz	15.42 ± 0.15	12	20	1.64	Volcanic glass

MSWD mean square weighted deviates, n is the number of experiments used to calculate the weight mean age, N is the total number of fusion experiments. Errors are $\pm 1\sigma$ analytical errors (not including uncertainties in standard age and decay constants).

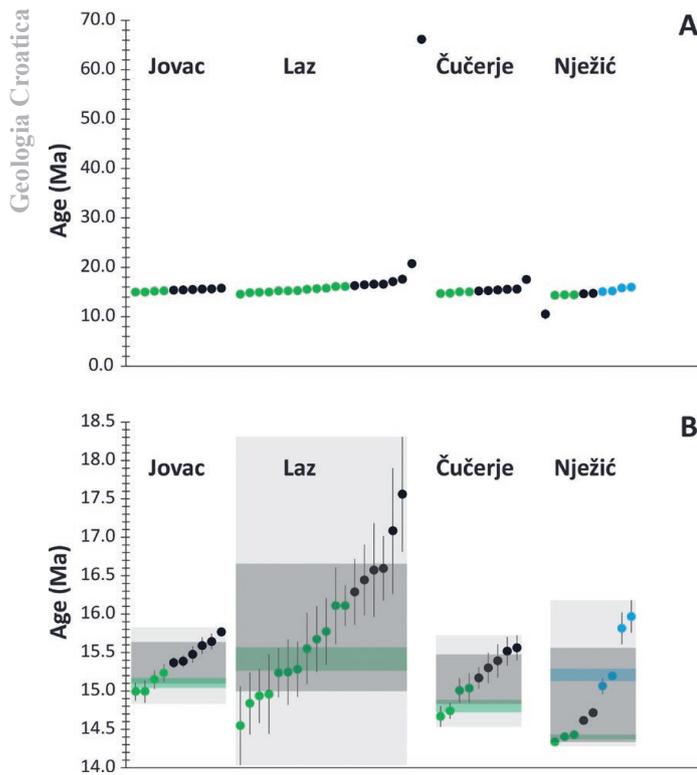


Figure 3: $^{40}\text{Ar}/^{39}\text{Ar}$ data of fusion analysis. All data are shown in (A). Data excluding outliers are shown in (B). ~5 grains are fused simultaneously for Jovac, Čučerje and Nježić; ~10 grains are fused simultaneously for Laz. 1 sigma analytical errors are shown. The analyses included in weighted mean ages are shown in green. For the Nježić locality an alternative age is represented by blue dots. The green/blue boxes represent the weighted mean age and standard error with data points excluded until MSWD < t -test statistic. The light gray box represents the total spread. The darker gray box represents the mean age and standard deviation.

weighted mean age of the 4 youngest analyses derived an age of 15.10 ± 0.06 Ma. Note that a multiple grain fraction was analysed which might obscure further complexity in the data set.

Twenty fusion experiments with ~10 grains per hole were performed on the 125–250 μm grain size fraction of volcanic glass of the Laz tuff (VU101-R6). Samples are low in K and show somewhat lower in radiogenic ^{40}Ar yields (on average ~69%). Due to the low K content beam intensities are low and analytical uncertainties are relatively large (± 0.4 Ma). Due to the spread in ages (Supplementary – file 1: 3.1.–3.3.), it is difficult to derive an eruption age accurately. When using the approach to include the highest number of analyses with a MSWD smaller than the t test statistic at the 95% confidence level, a weighted mean age of 15.42 ± 0.15 Ma was calculated, based on 12 out of 20 analyses. Note, that the inverse isochron does not show evidence of excess argon.

Ten fusion experiments with ~5 grains per hole of 250–500 μm glass from the Čuč-tuff (VU101-R7) were performed. Samples are also low in K and also somewhat lower in radiogenic ^{40}Ar (on average ~87%). Due to the spread in ages (Supplementary – file 1: 4.1.–4.3.), it is difficult to derive an accurate eruption age. The youngest dated samples might approach eruption age assuming that argon loss did not occur. Using the same approach as above, the youngest ages were included in the weighted mean age until the MSWD is larger than the t test statistic at 95% confidence level. This yields a weighted mean age of 14.81 ± 0.08 Ma based on 4 out of 10 analyses. Also for this sample the isochrons are poorly defined, but do not suggest the presence of excess argon.

Ten fusion experiments with ~5 grains per hole on 250–500 μm Nježić – A1 volcanic glass (VU101-R9) were performed. It is also difficult for this sample to arrive at an accurate eruption age (Supplementary – file 1: 5.1.–5.4.). The youngest date of ~10Ma only has 4% radiogenic ^{40}Ar and is excluded. Using the approach of including the youngest ages in a weighted mean until the MSWD > t -test at 95% confidence, the calculated weighted mean age 14.40 ± 0.03 Ma based on 3 out of 10 analyses was derived. Note that the 4 oldest analyses show excess argon and provide an inverse isochron age of 15.02 ± 0.07 Ma.

4.3. Micropalaeontology

4.3.1. Calcareous nannofossil assemblages

Calcareous nannofossil assemblages were determined for six samples from the Nježić locality and two samples from the Čučerje locality (Fig. 2A). Distributions of the calcareous nannofossil assemblages can be found in Supplementary – file 2: Table 1. and illustrations of most of the species in Fig. 4. At the Nježić locality, samples are rich in very well-preserved nannofossils. The assemblage from the sample Njež-A-b is dominated by small sized reticulofenestrids (*Reticulofenestra minuta* ROTH, 1970, *Reticulofenestra haqii* BACKMAN, 1978) and *Coccolithus pelagicus* (WALLICH 1877) SCHILLER, 1930. Presence of *Sphenolithus heteromorphus* DEFLANDRE 1953 and the absence of *Helicosphaera ampliaperata* BRAMLETTE & WILCOXON, 1967 allow determination of the NN5 Zone (MARTINI, 1971). In the sample Njež-A-t, a very similar nannofossil assemblage is present. Even though the zonal marker species, *S. heteromorphus* has not been discovered, the whole assemblage dominated by *R. minuta* indicates the NN5 Zone. The absence of *S. heteromorphus* could be due to the palaeoenvironmental characteristics of the depositional environment. The remainder of the samples from this locality have very similar nannofossil assemblages, contain zonal marker species *S. heteromorphus* and are therefore attributed to the NN5 Zone. In the sample Njež-C-b numerous diatoms were also observed, which indicate an increased concentration of nutrients.

The Čuč-b sample contains rare, relatively well-preserved forms with *Helicosphaera* cf. *waltrans* THEODORIDIS, 1984. The presence of *Sphenolithus* cf. *heteromorphus* and the appearance of *H. cf. waltrans* allow determination of the lower part of the NN5 zone. *C. pelagicus* dominates the assemblage, indicating a nearshore depositional environment, probably with strong upwelling characteristics. The Čuč-t sample is rich in well-preserved calcareous nannofossils dominated by small reticulofenestrids (*R. minuta*, *R. haqii*) and *Helicosphaera carteri* (WALLICH 1877) KAMPTNER, 1954. The assemblage has a low diversity, probably due to limited palaeoecological conditions. Numerous small reticulofenestrids, together with *H. carteri* indicate a nearshore, nutrient rich depositional environment. The lower part of the NN5 zone can be determined, although *S. heteromorphus* was not found in the sample.

4.3.2. Palynology

A total of seven samples from Laz, Čučerje and Nježić have been analyzed. At the Laz locality, four samples were analyzed. All samples are dominated by gymnosperm pollen (mostly *Pinus*, Fig. 5b), phytoclasts and resin. Beside conifers in the sample Laz-b1, the fern spores *Polypodium* and *Lygodium* were determined. Laz-b2 contains, beside conifers, fern spores *Polypodium* and the chlorophyte algae *Botryococcus* sp. (Fig. 5a), that is tolerant to brackish conditions.

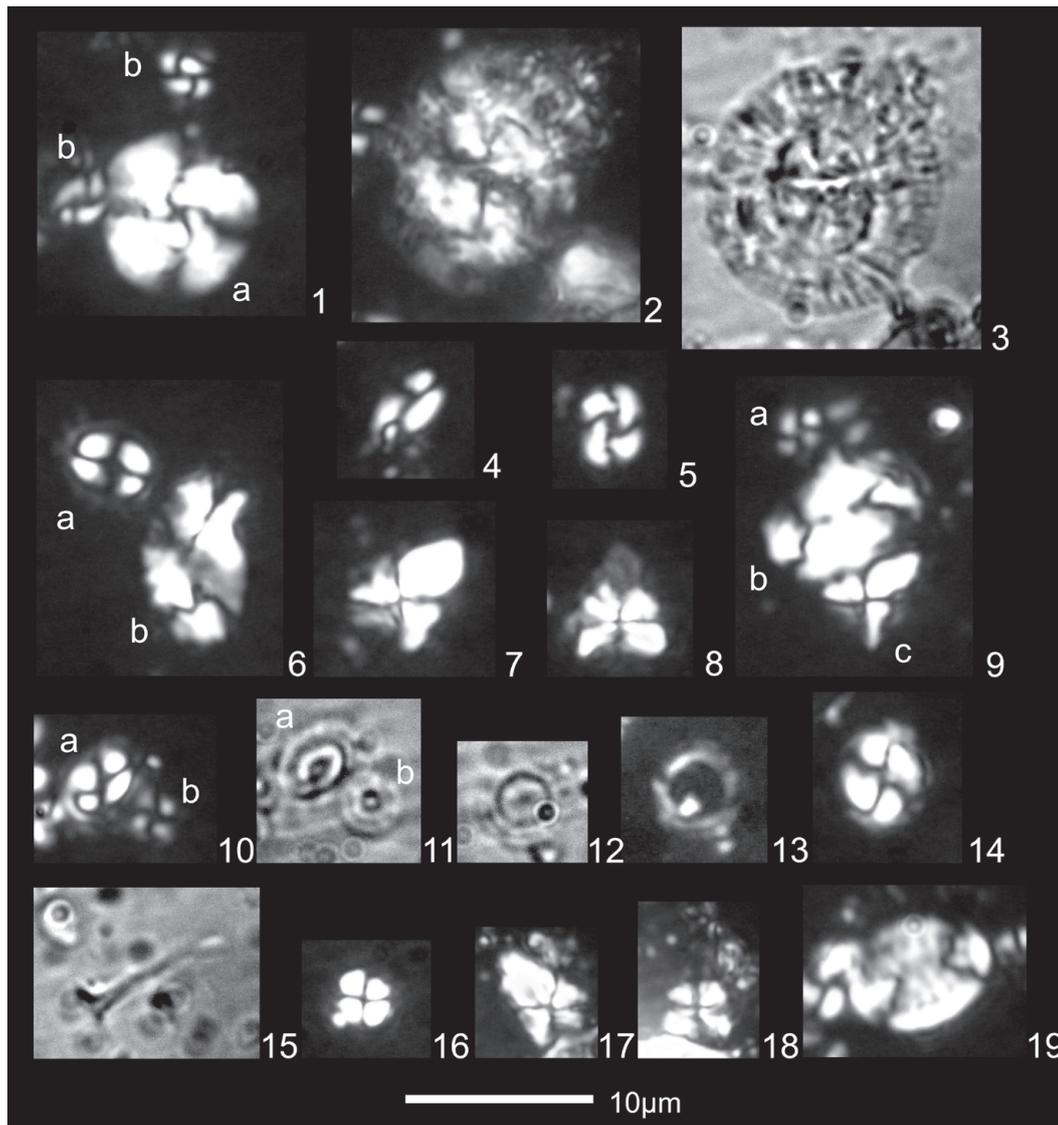


Figure 4. Photomicrographs of calcareous nannoplankton species: (1a) *Cyclicargolithus floridanus* (ROTH & HAY, in HAY *et al.*, 1967) BUKRY, 1971 (1b) *Reticulofenestra minuta* ROTH, 1970; (2, 3) *Coccolithus miopelagicus* BUKRY, 1971; (4) *Helicosphaera walbersdorfensis* MULLER, 1974; (5) *Reticulofenestra gelida* (GEITZENAUER 1972) BACKMAN 1978; (6a) *Coccolithus pelagicus* (WALLICH 1877) SCHILLER, 1930 (6b) *Helicosphaera carteri* (WALLICH 1877) KAMPTNER, 1954; (7, 8) *Sphenolithus heteromorphus* DEFLANDRE 1953; (9a) *Reticulofenestra minuta* ROTH 1970; (9b) *Helicosphaera carteri* (WALLICH 1877) KAMPTNER, 1954 (9c) *Sphenolithus heteromorphus* DEFLANDRE 1953; (10, 11a) *Coccolithus pelagicus* (WALLICH 1877) SCHILLER, 1930 (11b) *Umbilicosphaera jafari* MULLER, 1974; (12, 13) *Umbilicosphaera rotula* (KAMPTNER, 1956) VAROL, 1982; (14) *Coccolithus pelagicus* (WALLICH 1877) SCHILLER, 1930; (15) *Rhabdosphaera sicca* (STRADNER, 1963) FUCHS & STRADNER 1977; (16) *Sphenolithus moriformis* (BRÖNNIMANN & STRADNER, 1960) BRAMLETTE & WILCOXON, 1967; (17, 18) *Sphenolithus heteromorphus* DEFLANDRE 1953; (19) *Pontosphaera multipora* (KAMPTNER, 1948 ex DEFLANDRE in DEFLANDRE & FERT, 1954) ROTH, 1970. Nr. 1 – 15 & 19 are from sample: Njež-C-t; 16, 17 & 18 are from sample Čuč-b.

The Laz-t1 sample has the same assemblage, while the Laz-t2 sample also contains fern spores *Verrucingulatisporites* cf. *rugosus* (Fig. 5c). At the Čučerje locality, only one sample (from the marl overlying the tuff) was analyzed. Among the abundant pollen of conifers, common pollen of the riparian tree *Carya* and relatively rare fern spores *Polypodium* were determined. Infrequent findings of the marine dinocyst *Spiniferites pseudofurcatus* (Fig. 5e) and *Homotryblum tenuispinosum* (Fig. 5d) point to unstable coastal environments.

At the Nježić locality, only two samples from the middle part of the sequence were suitable for analysis, marls from the underlying and overlying beds of the Nježić B1/B2 tuff. From the underlying marl, sample Njež-B-b contains, besides a rare tropical-subtropical marine, the lagoonal dinocysts *Polysphaeridium zoharyi*, also a chlorophyte algae *Botryococcus* sp. that is tolerant to brackish conditions. High concentrations (> 90%)

of the cysts *P. zoharyi* (Fig. 5f) and rare prasinophyte phycocyst *Hidasia* sp. in the overlying bed, from sample Njež-B-t have also been found.

4.3.3. Ostracods

Five samples were used for ostracod analyses, four samples from the Laz locality (Laz-b1, Laz-b2, Laz-t1, Laz-t2) and one sample from the Jovac locality (Jov-b). Ostracods were found in all samples, and the ostracod specimens were differently preserved. Carapaces and valves are well preserved in the sample from the Jovac locality, while all samples from the Laz locality contain deformed and strongly calcified ostracods. Eleven ostracod taxa were identified of which most of the species remained in open nomenclature. Distributions of ostracod species recorded in the Miocene sediments of the investigated locality are summarized in the Supplementary – file 2: Table 4, listed alphabetically, and most of the species are illustrated in Fig. 6.

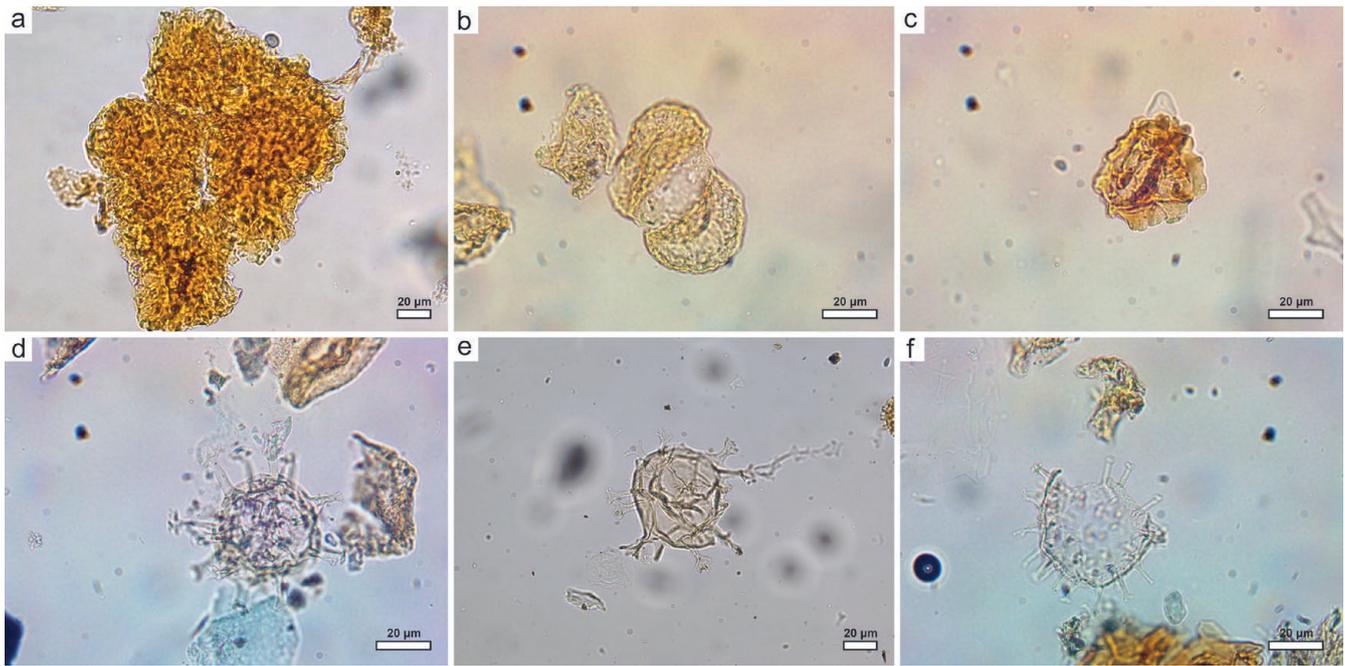


Figure 5. Photomicrographs of palynomorphs from samples a: *Botryococcus*, Laz-b2; b: *Pinus*, Laz-b2; c: *Verrucingulatisporites* cf. *rugosus*, Laz-t2; d: *Homotryblium tenuispinosum*, Čuč-t; e: *Spiniferites pseudofurcatus* Njež-B-b; f: *Polysphaeridium zoharyi*, Njež-B-t)

Ostracod fauna recorded in the five samples included eleven ostracod species that represent four families: *Cypridopsis* cf. *bipplanata*, *Herpetocypris* sp., *Herpetocypris* cf. *reptans* and *Hete-*

rocypris sp. within Cyprididae; *Candona* sp., *Pontoniella* sp. and *Cypria* sp. (Candonidae); *Paralimnocythere* cf. *rostrata* within Limnocytheridae; and *Amnocythere* sp. and *Leptocythere* ? sp.

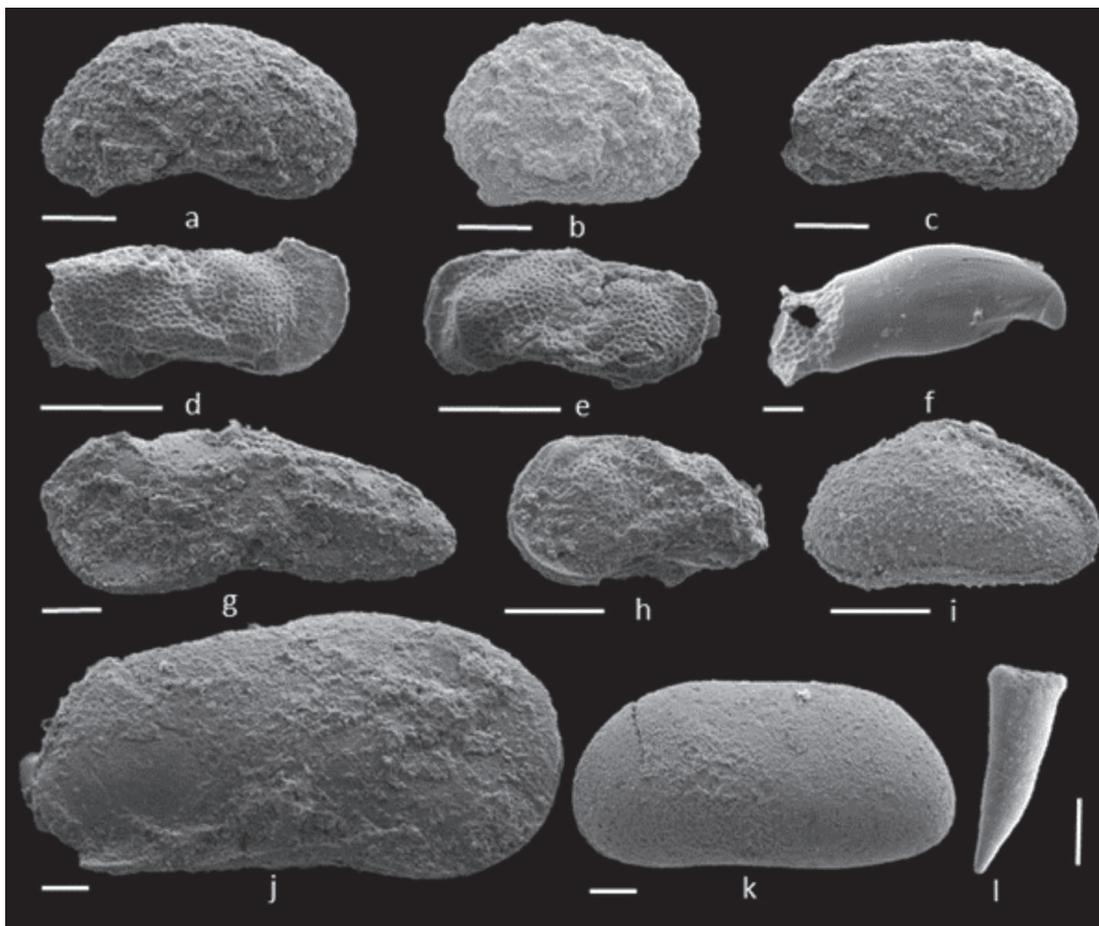


Figure 6. SEM photomicrographs of selected microfossils from Laz and the Jovac locality: a, c *Candona* sp., sample Laz t1; b *Cypria* sp., sample Laz t1; d *Paralimnocythere* cf. *rostrata*, sample Laz t1; e *Leptocythere* ? sp., sample Laz t2; f claw of freshwater crab, sample Laz b2; g, *Pontoniella* sp., sample Laz t1; h *Amnocythere* sp., sample Laz t1; i *Potamocypris* sp., sample Jov b; j *Herpetocypris* sp., sample Laz b1; k *Herpetocypris* cf. *reptans*, sample Jov b; l fish tooth, sample Laz b1. Scale bars: 100 μ m.

within Leptocytheridae. The most common records are of the genus *Herpetocypris* (present in 5 samples), and *Candona* (present in 3 samples), followed by *Amnicythere*, *Heterocypris* and *Cyprina* (in 2 samples). Only one genus *Herpetocypris* is present with two species, while another genus is represented by only one species. Species richness is generally low.

In addition to the ostracods, the investigated samples contained additional micropalaeontological remains, such as planktonic and benthic foraminifera, algal thalli (sample Jov-b) and rhizoliths (sample Jov-b), fish teeth (samples Laz-b1 and Laz-t2) and the claws of freshwater crabs (Laz-b2 and Laz-t2).

4.3.4. Foraminifera

4.3.4.1. Planktonic foraminifera

At the Nježić locality six samples have been analyzed. The distribution of the foraminifera from the marl samples is given in the Supplementary – file 2: Table 2. The foraminiferal assemblage in the Njež-A-b sample is diverse and well preserved. Representatives of the *Trilobatus* genera (*T. trilobatus*, *T. quadrilobatus*, *T. sicanus*) are very frequent in the planktonic foraminiferal association. Specimens of *Paragloborotalia mayeri* and *Praeorbulina curva* have also been observed. Planktonic foraminifera species determined in the Njež-A-b sample have a somewhat wider stratigraphic range and suggest a late early Badenian to middle Badenian age (M5-M6 Zone – according to WADE et al., 2011 and MMi4c to MMi5 – according to LIRER et al., 2019). Sample Njež-A-t has a poorly preserved and less diverse planktonic foraminifera assemblage suggesting the same age (late early Badenian to middle Badenian). In the sample Njež-B-b the foraminiferal assemblage is relatively well preserved, with a slightly lower degree of biodiversity. Specimens of the genus *Paragloborotalia* predominate but *T. trilobatus*, *Praeorbulina curva*, *Globigerina tarchanensis* etc. have also been identified. The assemblage indicates the late early Badenian to middle Badenian (M5 to M6 Zone – according to WADE et al., 2011 and MMi4c to MMi5 – according to LIRER et al., 2019). In the sample Njež-B-t planktonic foraminifera are relatively well preserved. In the planktonic foraminiferal association, beside numerous representatives of the genera *Paragloborotalia*, the species *Praeorbulina curva*, *P. glomerosa* and *P. cf. circularis* have been observed. The assemblage suggests a middle Badenian age (M6 Zone – according to WADE et al., 2011; MMi4d to MMi5 – according to LIRER et al., 2019). In the Njež-C-b sample a well-preserved assemblage of planktonic foraminifera (*Praeorbulina glomerosa*, *P. circularis*, *P. curva*, *Trilobatus trilobus*) indicates a middle Badenian age (M6 Zone – according to WADE et al., 2011 and MMi4d to MMi5 – according to LIRER et al., 2019). In the sample Njež-C-t, the planktonic foraminiferal association is diverse and well preserved with numerous specimens of the genera *Praeorbulina* and *Orbulina*. The presence of the index fossil *Orbulina suturalis* suggests a middle Badenian age – MMi 5a Zone (LIRER et al., 2019) and the M6 Zone (WADE et al., 2011).

At the Čučerje locality, the marl from the overlying bed of the tuff (Čuč-t) has been analyzed. The sample contains *Globigerina bulloides*, *G. tarchanensis*, *G. cf. subcretacea*, *Globigerinita cf. uvula*, *Globoturborotalita drury* and *Tenuitella* sp. Determined species of planktonic foraminifera indicate a Badenian age, most likely the middle Badenian, however a typical middle Badenian assemblage has not been observed.

At the Laz locality, few findings of planktonic foraminifera have been observed (samples Laz-b1 - ? *Trilobatus* sp., Laz-b2 and Laz-t2 - *Globoturborotalita cf. pseudopraebulloides*).

4.3.4.2. Benthic foraminifera

Benthic foraminiferal assemblages at the Nježić locality are well preserved and rich in individuals. The species richness (number of species level taxa) ranges from 27 to 36 per sample (total 54 species, Supplementary – file 2: Table 3.). Along the entire section, the dominating species are *Cibicidoides ungerianus* (D'ORBIGNY), *Cassidulina laevigata* D'ORBIGNY and *Bolivina dilatata* REUSS. The abundance of planktonic foraminifera in the samples varies from 80.9% to 94.2%. According to the benthic foraminifera, the studied section belongs to the Lagenidae Zone (Moravian substage of the Badenian), middle Badenian (sensu HOHENEGGER et al., 2014), traditionally early Badenian, but no more precise stratigraphic placement is possible within the Lagenidae zone. The index species for the Lagenidae Zone is *Uvigerina macrocarinata* PAPP & TURNOVSKY, while *Uvigerina grilli* SCHMIDT is the index species for the Lagenidae Zone and the *Spirorutilus* Zone. Also, the determined species *Vaginulinopsis pedum* (D'ORBIGNY) and *Uvigerina bulbacea* (GALLOWAY & HEMINWAY) stratigraphically do not cross the upper part of the Lagenidae zone.

Samples from the Čučerje locality did not contain enough specimens to support a palaeoecological interpretation. Only rare specimens of *Cibicidoides pseudoungeriana* (D'ORBIGNY) and *Bolivina dilatata* (REUSS) have been observed. At the Laz locality, the benthic foraminifera *Cibicides* sp. has been observed.

5. DISCUSSION

5.1. $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology

$^{40}\text{Ar}/^{39}\text{Ar}$ dating of tuffs at four localities in the NCB yielded ages of between 14.4–15.4 Ma. This is consistent with the main volcanic activity in the PBS (LUKÁCS et al., 2015, 2018; SZAKÁCS et al., 2018) during the syn-rift phase of basin development in the early Miocene and the lower part of the middle Miocene (PAVELIĆ, 2001; PAVELIĆ & KOVAČIĆ, 2018). According to the microfossil assemblages from the underlying and overlying layers of the dated tuffs, a shift from terrestrial to marine environments occurred in parallel with the volcanic activity. The oldest tuffs analysed in this paper, were found in the north-western part of the NCB at the Laz locality on Mt. Medvednica (Fig. 1B), where the tuff yielded a $^{40}\text{Ar}/^{39}\text{Ar}$ age of 15.42 ± 0.15 Ma (Tab. 1, Fig. 3). A similar age was obtained for volcanoclastic turbidites from Požeška gora in the central part of the NCB, where BRLEK et al. (2020) dated sanidines and reported a $^{40}\text{Ar}/^{39}\text{Ar}$ age of $15.34 \pm 0.32 / 15.43 \pm 0.32$ Ma and CA-ID-TIMS U-Pb zircon age of 15.345 ± 0.02 Ma, respectively, from the same material. Similar ages of pyroclastic material were found south of the NCB in the Dinaride Lake System area (DLS). For example, dating of feldspar from tuff deposited in the Gacko Basin yielded a $^{40}\text{Ar}/^{39}\text{Ar}$ age of 15.31 ± 0.16 Ma (MANDIĆ et al., 2011; De LEEUW et al., 2012) and tuff deposited at the Lučane locality in the Sinj Basin dated on sanidine yielded an $^{40}\text{Ar}/^{39}\text{Ar}$ age of 15.43 ± 0.05 Ma (De LEEUW et al., 2010, 2012). A slightly younger age is determined along the southern part of the NCB in the Banovina area, where the tuff layer at the Jovac locality (Fig. 1B) is dated at 15.10 ± 0.06 Ma. A similar age has not been previously determined in the NCB. In contrast, a tuff with an age corresponding to 14.81 ± 0.08 Ma at the Čučerje locality on Medvednica Mt. (Fig. 1B) has been described at several localities in the PBS and its surroundings (LUKÁCS et al., 2018 and references therein). According to chronostratigraphic similarity, the Čučerje tuff can be correlated with the Demjén ignimbrite Bükkalja Volcanic Field (BVF) erup-

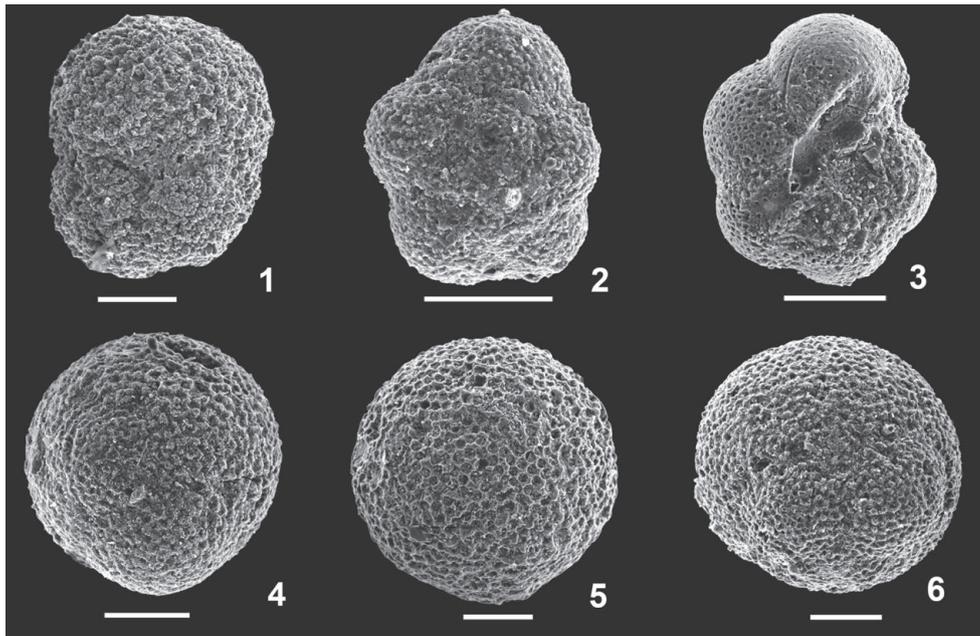


Figure 7. Planktonic foraminifera (Nježić) – (1) *Trilobatus trilobus*, Njež-A-b; (2) *Globigerina tarchanensis*, Njež-B-b; (3) *Paragloborotalia mayeri*, Njež-B-t; (4) *Praeorbulina glomerosa*, Njež-C-b; (5) *Praeorbulina circularis*, Njež-C-b; (6) *Orbulina suturalis*, Njež-C-t. Scale bar – 100 μm .

tion unit described by LUKÁCS et al. (2018). The youngest age of volcanic activity in this paper was determined in Slavonia in the central part of the NCB. There, at the Nježić locality, the oldest tuff layer from the lower part of the sequence is dated at 14.40 ± 0.03 Ma. The thickness of the tuff layers at the Nježić locality, reaching up to 18 m, as well as their number in a sequence one hundred metres thick, indicates strong and long-lasting volcanic activity in the middle of the Badenian in the NCB area. The fact that the mid-Badenian is characterized by strong volcanic activity throughout the PBS area is confirmed by layers of volcanic ash of similar age described from several localities. For example, HANDLER et al. (2006) report a $^{40}\text{Ar}/^{39}\text{Ar}$ age of 14.39 ± 0.12 Ma on sanidine crystals from the Retznei locality in the Styrian Basin, while DE LEEUW et al. (2013) report a $^{40}\text{Ar}/^{39}\text{Ar}$ age of 14.37 ± 0.06 Ma on sanidine crystals from the Dej tuff complex in the Transylvanian Basin. According to chronostratigraphic similarity, the Nježić tuff can be correlated with the Harsány ignimbrite BVF eruption unit described by LUKÁCS et al. (2018).

5.2. Biostratigraphy and depositional environments

The microfossil assemblages from the underlying and overlying beds of the studied tuff layers indicate that tuff deposition occurred in different sedimentary environments. For example, the assemblage of ostracods (*Herepetocypris* sp. and the genera *Cyprina* and *Potamocypris*) determined in the underlying bed of the tuff layer at the Jovac locality in Banovina indicates a shallow freshwater lacustrine environment. Based on the mollusc and ostracod fauna for the tuffs intercalated with marls at the Paripovac locality in the Glina sub-depression, and the Sjeniĉak locality in the Karlovac sub-depression, a freshwater environment was determined (MANDIĆ et al., 2012; HAJEK-TADESSE et al., 2016). The age range of the afore-mentioned tuffs from 16.03 ± 0.06 Ma at the Paripovac locality and 15.91 ± 0.06 Ma at the Sjeniĉak locality (MANDIĆ et al., 2012) to 15.10 ± 0.06 Ma at the Jovac locality suggests that the freshwater lacustrine environment existed in the early Badenian along the southern boundary of the NCB for at least one million years. South of the NCB, the

same sedimentary environments existed in the early Badenian, in the DLS area, where tuff layers intercalate with freshwater lacustrine sediments in the Gacko and Sinj basins (MANDIĆ et al., 2011; De LEEUW et al., 2010, 2012).

At the same time, i.e. 15.42 Ma ago, a slightly different sedimentary environment existed at the Laz locality on Medvednica Mt., in the north-western part of the NCB, according to tuff dating results. Findings of the freshwater but tolerant to brackish water chlorophyte algae *Botryococcus* as well as the discovery of euhaline ostracod species from family *Leptocytheridae*, indicate the existence of a freshwater to brackish water environment. Brackish water and the possible ingress of seawater in the Laz locality is further supported by findings of a few planktonic (*Globobulimina* cf. *pseudopraebulloides*, ? *Trilobatus* sp.) and benthic foraminifera (*Cibicides* sp.) in samples Laz-b1, Laz-b2 and Laz-t2. Findings of the fern spores *Verrucingulatisporites* cf. *rugosus* (Fig. 5c) indicate a late Karpatian – early Badenian age (NAGY, 1985). According to the palynomorph assemblage, deposition occurred in a subtropical coastal freshwater – brackish environment. A similar ostracod assemblage was described in the central part of the NCB on Požeška gora (HAJEK-TADESSE et al. 2009; MANDIĆ et al., 2019a). Such a brackish environment could be the result of occasional seawater ingress into the lake or a consequence of climate change from subtropical humid to more arid conditions (MANDIĆ et al., 2019a).

The fossil assemblage of calcareous nannoplankton and planktonic foraminifera from the underlying and overlying beds of the tuffs at the Čučerje locality on Medvednica Mt. and the Nježić locality on Papuk Mt. shows that their deposition took place in a marine environment. The presence of *Sphenolithus* cf. *heteromorphus* and the occurrence of *Helicosphaera* cf. *waltrani* in the calcareous nannofossil assemblages of the Čučerje locality enable the determination of the lower part of the NN5 zone, i.e. the upper part of the Early Badenian according to KOVAČ et al. (2018a) and Middle Badenian according to HOHENEGGER et al. (2014), which is consistent with the $^{40}\text{Ar}/^{39}\text{Ar}$ age of the tuff layer dated at 14.81 ± 0.08 Ma (Fig. 8). The dominance of *Coccolithus*

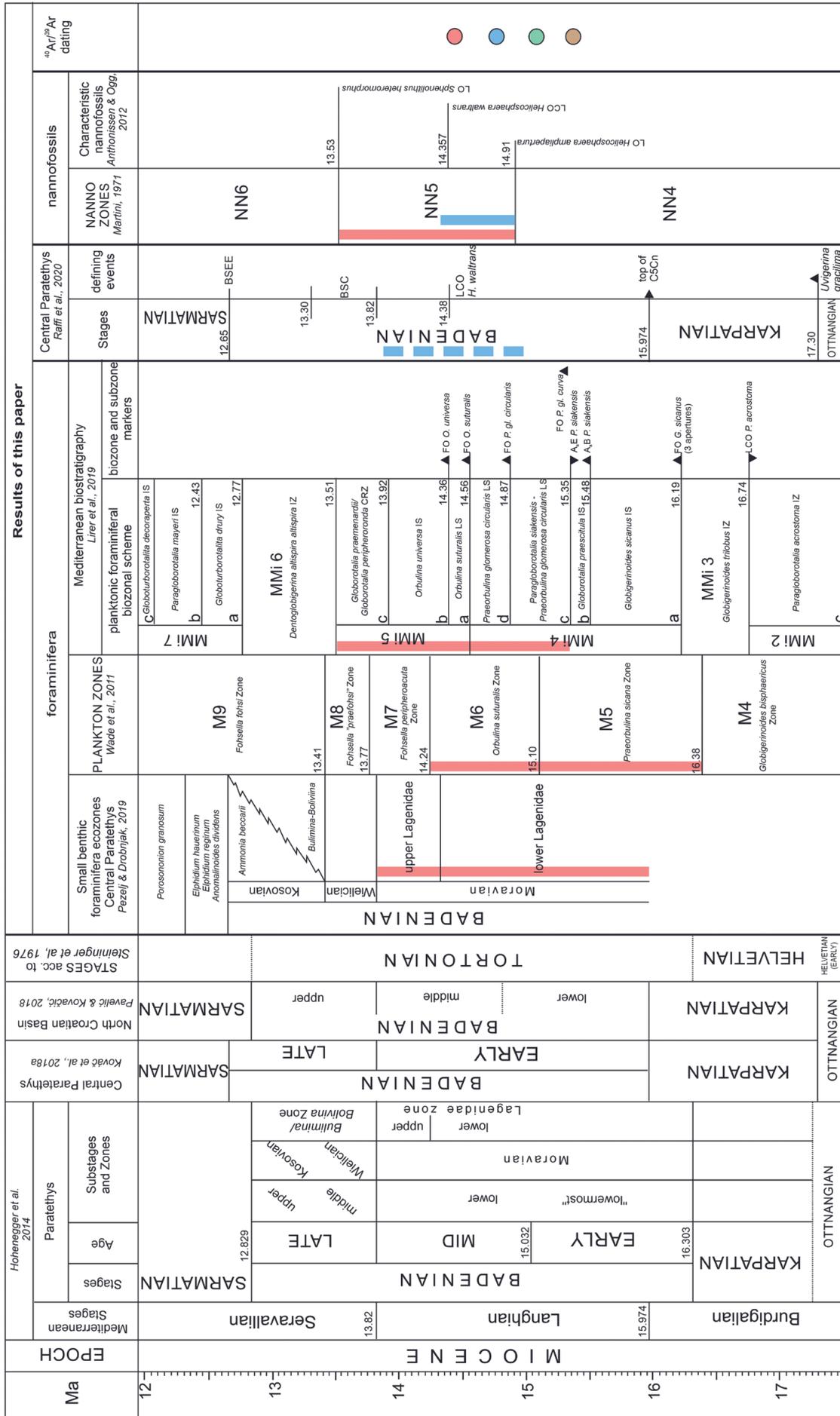


Figure 8. Compilation of the results obtained by microfossil analysis and ⁴⁰Ar/³⁹Ar dating for the investigated localities. Localities are represented in different colours: Nježić – red; Čučerje – blue; Jovac – green and Laz – brown. Boxes represent the stratigraphic position obtained by microfossil analysis and the circles represent the results obtained by ⁴⁰Ar/³⁹Ar dating. Abbreviations: LO – Last Occurrence, BSC – Last Common Occurrence, FO – First Occurrence, BSEE – Badenian-Sarmatian Extinction Event, AB – Acme Base, AE – Acme End

pelagicus in the underlying bed and numerous small reticulofenestrids as well as the presence of *Helicosphaera carteri* in the overlying bed, also indicate a nearshore sedimentary environment. The Badenian age, most likely the middle part of the Badenian, is also suggested by the planktonic foraminiferal assemblage. However, the assemblage typical of this period was not found. The palynofacies also suggests that deposition occurred in a marine environment in a subtropical climate during the Badenian. At the Nježić locality, where the oldest tuff layer is dated at 14.40 ± 0.03 Ma, the calcareous nannofossil assemblages allow the determination of the NN5 zone, i.e. the middle part of the Badenian (Fig. 8). This assemblage is also rich in reticulofenestrids of small dimensions ($3 \mu\text{m}$), indicating an epicontinental marine environment and a stratified water column. A similar assemblage was observed from the scientific borehole Baden-Soos from the southern part of the Vienna Basin (ČORIĆ & HOHENEGGER, 2008). The NN5 zone was also attributed to the Dej Tuff in the Transylvanian Basin (MÉSZÁROS & ŠURARU, 1991). The assemblage of planktonic foraminifera indicates the M6 zone - according to WADE et al. (2011) and the MMi4c to MMi5 zones - according to LIRER et al. (2019), i.e. also the middle part of the Badenian. According to the benthic foraminifera, the studied samples from the Nježić locality belong to the Lagenidae Zone (Moravian substage of the Badenian). Correlation with the identified calcareous nannoplankton zone NN 5 (Fig. 8), facilitates the conclusion that the studied time interval certainly indicates the Upper Lagenidae Zone, i.e. Middle Badenian sensu HOHENEGGER et al. (2014) or the Early Badenian sensu KOVÁČ et al. (2018a). A similar *Cibicidoides-Bolivina-Cassidulina* assemblage of benthic foraminifera with clear dominance of the species *Cibicidoides ungerianus* (D'ORBIGNY), *Cassidulina laevigata* D'ORBIGNY and *Bolivina dilatata* REUSS was identified throughout the section. The high proportion of planktonic foraminifera (>80%) in the Nježić samples indicates an upper bathyal environment (MURRAY 2006), while the overlap of depth ranges of benthic foraminifera indicates a deep-water environment of the outer shelf (ca. 200 m). High concentrations (~90%) of *Polysphaeridium zoharyi* cysts (Fig. 5f) indicate blooms of a toxic dinoflagellate *Pyrodinium bahamense* (motile stage of *P. zoharyi* cyst). Increased productivity in both modern and palaeo-oceans has been associated with volcanism (ACHTERBERG et al., 2013). Abundances (> 50%) of *P. zoharyi* cysts occur where summer upper temperatures are >29.1°C and winter upper temperatures are >19.1°C (ZONNEVELD & POSPELOVA, 2015). Rare prasinophyte phycoma *Hidasia* sp. were also found, indicating shallow-sea and nearshore conditions. According to these findings, a Badenian age could be suspected. Palaeoenvironmental preferences of "Homotryblium Complex" taxa (*Pyrodinium/Polysphaeridium*, *Eocladopyxis* and *Homotryblium*) are discussed by de VERTEUIL & NORRIS (1996). They concluded that "Homotryblium Complex" species are often dominant in dinocyst assemblages with moderate to low diversity, typically in unstable coastal environments. This trend suggests an opportunistic, bloom-forming ecology. This group also appears to reflect warm temperate to tropical palaeoceanographic provinces. *Pyrodinium bahamense* is euryhaline and can be abundant in both low salinity and high salinity waters (DYBKJÆR, 2004).

HAJEK-TADESSE (2006); HAJEK-TADESSE & PRTOLOJAN (2011) and VESEL LUKIĆ & HAJEK-TADESSE (2017) published a detailed description of the Badenian marine ostracods from the Banovina area, Čučerje and Nježić locality. In the Slavonia region, marine deposits were also described on Požeška gora Mt., interbedded with a tuff layer and dated to 15.34 Ma

(BRLEK et al., 2020). This age suggests that the marine environment in the central part of the NCB also existed during the Early Badenian sensu HOHENEGGER et al. (2014).

5.3. Timing of the initial marine transgression in the NCB

The results of radioisotope dating of tuffs integrated with the results of calcareous nannoplankton, foraminifera, ostracods and palynomorph assemblages from the immediately underlying and overlying beds of the tuffs at four localities in the NCB area showed that tuffs older than about 15 Ma were deposited in a lacustrine freshwater environment (Jovac) and in a lacustrine freshwater/brackish water environment (Laz), while tuffs younger than 15 Ma were deposited in a marine environment (Čučerje and Nježić). These results suggest that the NCB area became part of the marine realm of Central Paratethys about 15 Ma ago, i.e. in the Early Badenian sensu KOVÁČ et al. (2018a), or at the transition from the Early to the Middle Badenian sensu HOHENEGGER et al. (2014). Determination of the calcareous nannoplankton zone NN5 at the Nježić and Čučerje localities and the M6 *Orbulina suturalis* (WADE et al., 2011) zone and MMi4c to MMi5 (LIRER ET AL., 2019) at the Nježić locality, respectively, confirm the results of radioisotope dating (Fig. 8). These results are consistent with the results of the studies on the onset of marine transgression in the NCB based on biostratigraphy ČORIĆ et al., 2009. In particular, they concluded that the onset of the marine transgression was at least one million years later than 16 Ma, which was the prevailing opinion at that time. This early Badenian sensu KOVÁČ et al. (2018a) or Middle Badenian sensu HOHENEGGER et al. (2014) marine transgression could be related to the TB 2.4 cycle and their deposits are described in Slovenj Gradec Basin, Styrian Basin and Novohrad Basin (HOHENEGGER et al., 2014; IVANČIĆ et al., 2018; HUDÁČKOVÁ et al., 2020; RUMAN et al., 2021). Alternatively, PREMEC FUČEK et al. (2017) determined the *Globigerinoides trilobus* Zone in wells from the Sava Depression in the southern part of the NCB, suggesting that the initial marine flooding corresponds to the TB 2.3 3rd order sequence. The same initial marine flooding indicates marine sediments underlying and overlying tuff beds dated to 15.35 Ma in the northern margin of the Sava Depression (BRLEK et al., 2020). This flooding could be the result of a marine transgression from the Mediterranean towards the Central Paratethys domain through Dinaride gateway in present-day Slovenia (KOVÁČ et al., 2018a; MANDIĆ et al., 2019a). During this period, (early Badenian; upper part of NN4), the sediments of the Modrý Kameň Fm. were deposited in the Western Carpathians Novohrad Basin (KOVÁČ et al., 2018; HUDÁČKOVÁ et al., 2020; RUMAN et al., 2021), and possibly also the "schlier" of the Cerová-Liesková site in the Vienna Basin (e.g. KOVÁČ et al., 2017b). In a deep well from the central part of the Sava Depression HERNITZ KUČENJAK et al. (2018) determined the *Globigerinoides bisphericus* Zone, suggesting an even earlier onset of the marine transgression, corresponding to the TB 2.2 3rd order sequence. This transgression was most likely the consequence of sea-level rise and deepening gateways with the Mediterranean Sea through the Alpine foreland (HOFMAYER et al., 2019) and over Slovenia (IVANČIĆ et al., 2018) during the TB 2.2 cycle (sensu HARDENBOL et al., 1998). Marine deposits associated with the TB 2.2 cycle are well described in the Styrian and Vienna Basins in Austria (HOHENEGGER et al., 2014; HARZHAUSER et al., 2019, 2020) and in Western Carpathians, Novohrad Basin in Slovakia (RUMAN et al., 2021).

Considering the results of this paper and those published in ČORIĆ et al. (2009), PREMEC FUČEK et al. (2017), HERNITZ KUČENJAK et al. (2018) and BRLEK et al. (2020), it can be assumed that different parts of the NCB were flooded at different times as a result of various marine transgression events. The central parts of the NCB in the Sava Depression became part of the Central Paratethys Sea as early as the Karpatian during the TB 2.2 cycle, while marginal parts of Sava Depression were inundated in the Early Badenian, during the TB 2.3 cycle. Marginal parts of the NCB were terrestrial, freshwater or brackish lacustrine environments until 15 Ma, when they were finally inundated during the TB 2.4 cycle.

6. CONCLUSIONS

Tuffs from four localities in the NCB yielded the following ⁴⁰Ar/³⁹Ar ages: Jovac (15.10 ± 0.06 Ma), Laz (15.42 ± 0.15 Ma), Čučerje (14.81 ± 0.08 Ma) and Nježić (14.40 ± 0.03 Ma).

At the Jovac locality, a freshwater lacustrine environment was determined. At the Laz locality, a freshwater/brackish lacustrine environment was determined. At the Čučerje and Nježić localities, deposition took place in a fully marine environment, in a subtropical to moderately warm climate.

Calcareous nannofossil data as well as planktonic foraminiferal assemblages match the data obtained by the ⁴⁰Ar/³⁹Ar dating method at Čučerje and Nježić.

According to chronostratigraphic similarity, the Čučerje tuff can be correlated with the Demjén ignimbrite BVF eruption unit and the tuff from the Nježić locality can be correlated with the Harsány ignimbrite BVF eruption unit.

Based on the presented results, the initial marine transgression in the marginal areas of the western part of the NCB can be constrained to a period from 15.10 ± 0.06 Ma to 14.81 ± 0.08 Ma i.e. at the early/middle Badenian boundary.

Considering these results with the existence of lower Badenian marine sediments in the Sava depression (in the southern part of NCB), it is possible to conclude that during the early Badenian in the NCB freshwater lacustrine and marine environments co-existed.

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