Cretaceous Research 186 (2026) 106424

Contents lists available at ScienceDirect

Cretaceous Research

journal homepage: www.elsevier.com/locate/CretRes

High-precision U–Pb zircon age of the early angiosperm herbaceous assemblage from the Frentsevka Formation, Far East of Russia and its implication for the timing of angiosperm diversification in Eastern Asia

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A R T I C L E I N F O

Article history:
Received 26 August 2025
Received in revised form
20 April 2026
Accepted in revised form 1 May 2026
Available online 7 May 2026

Keywords:
Early angiosperms
Early Cretaceous
Isotopic dating
Primorye
Russia

A B S T R A C T

Isotopic data (U–Pb ID-TIMS dating) provides new evidence for the age interpretation of the upper part of the Frentsevka Formation (Partizansk coal basin, Primorye, Russia). These deposits contain numerous remains of early herbaceous angiosperm and ferns. The early Albian age (109 ± 0.5 Ma) is assigned to plant-bearing layers from the section near Bolshoy Kuvshin Cape, town of Bolshoy Kamen. Isotopic data clarified previous age estimates (early-middle Albian) based on palaeobotanical, palynological and mollusks data.

The early angiosperm assemblage of the Frentsevka Formation was correlated with other early angiosperm floras from northeastern China using absolute dating. This correlation showed that the angiosperm assemblage from the Bolshoy Kuvshin locality existed at the same time as the early angiosperms from the Chengzihe Formation, Heilongjiang, China.

1. Introduction

Investigation of fossil angiosperms provides important data for reconstruction of the early radiation of this group. The lack of precise dating of early angiosperm-bearing floras precludes a more exhaustive study about evolutionary and dispersion patterns of this group of plants (Hochuli et al., 2006). The most diverse assemblages of fossil angiosperms occur in extended terrestrial sequences without key marine fossils, which makes determination of their precise age problematic. Usually the palynological data are used for this purpose, but quality of preservation of palynomorphs in marine sediments are poor. Besides that, diversification and appearance of the different pollen types may have been not synchronous in different sections.

Isotopic techniques, which yield highly accurate absolute ages, were infrequently applied until recently, largely because volcanic ash layers were scarce in stratigraphic sections containing significant early angiosperm fossils. However, the widespread presence of volcanic deposits in the terrestrial Lower Cretaceous strata of Eastern Asia has recently enabled increased absolute dating of these sequences for this critical time interval (Wan et al., 2007; Xi et al., 2019).

In this paper, we present the U–Pb age (ID-TIMS and LA-ICP-MS methods) of zircon from plant-bearing deposits of the upper part of the Frentsevka Formation (Primorye, Russia) based on new discovery of rhyolite tuff layer. These deposits contain diverse herbaceous angiosperms and ferns assemblage. The locality, named Bolshoy Kuvshin (Golovneva et al., 2018), is situated on the east coast of the Ussuri Bay on the Bolshoy Kuvshin Cape near the town of Bolshoy Kamen (Fig. 1A). It was discovered by geologist A.

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https://doi.org/10.1016/j.cretres.2026.106424

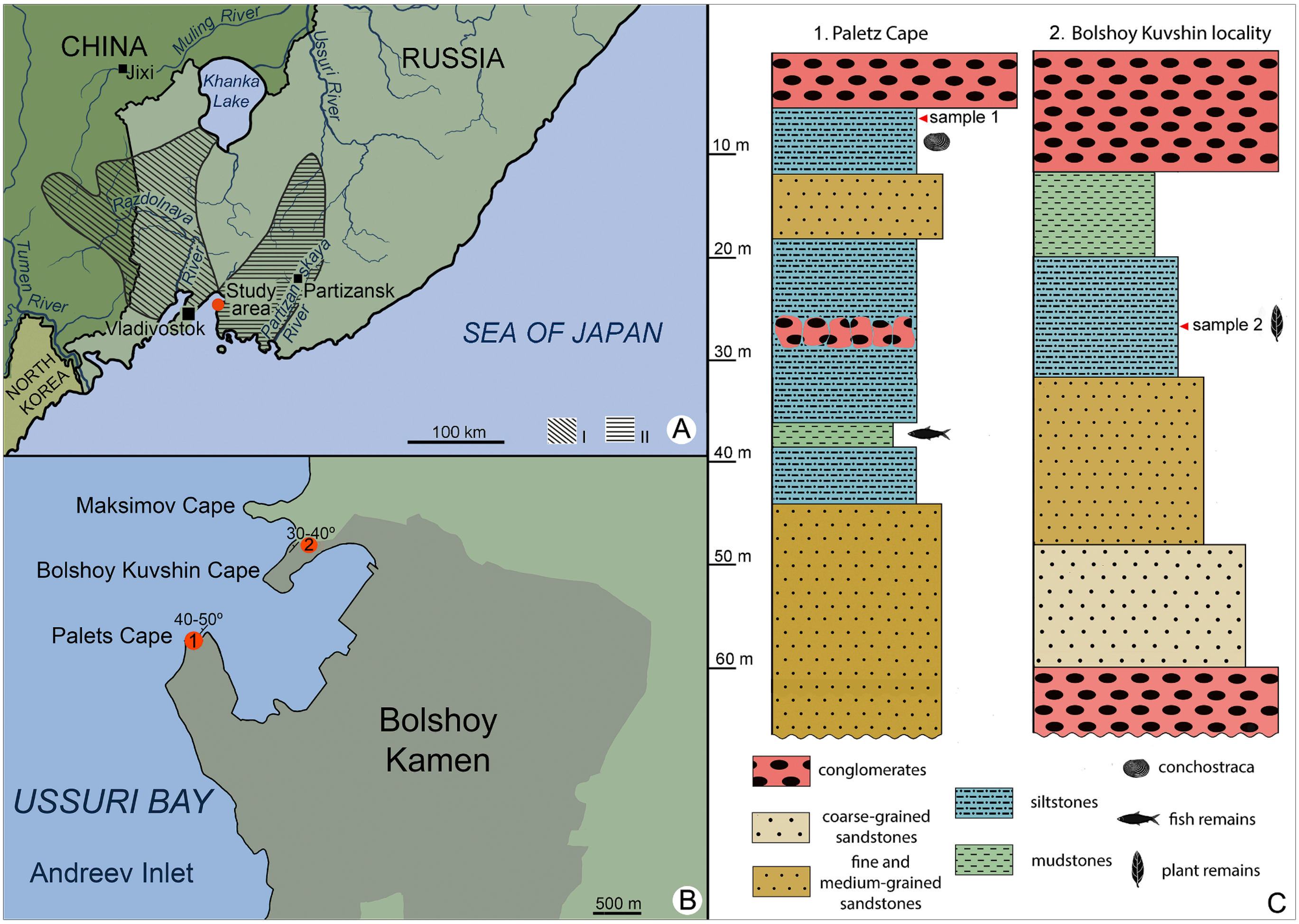

Fig. 1. A – map of Primorye, Russia: I – Razdolnaya coal basin; II – Partizansk coal basin; B – sections of the upper part of the Frentsevka Formation near town of Bolshoy Kamen: 1 – section along northern coast of the Palets Cape (43.12312 N. 132.31935 E); 2 – section in the locality Bolshoy Kuvshin, exposed along the railroad in the base part of the Bolshoy Kuvshin Cape (43.13159 N 132.33472 E); C – stratigraphic columnar sections of the upper part of the Frentsevka Formation near town Bolshoy Kamen, showing the sampling locations for zircon dating in this study: sample 1 - the tuff layer at the Palets Cape, sample 2 - plant-bearing tuffaceous fine-grained sandstone at the locality Bolshoy Kuvshin.

Oleynikov, then studied by Krassilov and Volynets (2008), who described two species of tiny herbaceous angiosperms from there: *Achaenocarpites capitellatus* Krassilov et Volynets, 2008 and *Ternaricarpites floribundus* Krassilov et Volynets, 2008. New excavations at this locality were made from 2017 to 2024.

Angiosperm fossils are represented by almost complete plants or big fragments with attached roots, leaves and fruits (Golovneva et al., 2018, 2020). These plants were small (10–30 cm high) and very delicate. Their excellent preservation in rather coarse sediment (fine-grained sandstone) indicates the absence of long water transport. Stronger fern leaves were often left standing in organic connection to their rhizomes. Some fern specimens are represented by almost entire young plants.

The plant fossils in the Bolshoy Kuvshin locality were buried during several flooding events. They formed a pioneer open herbaceous community, consisting of ferns and angiosperms with a predominance of the latter (Fig. 2), and adapted to colonize fresh sediments in periodically flooded areas (Golovneva et al., 2018).

2. Geological setting

In southern Primorye the Barremian–Albian interval is represented by the coal-bearing non-marine deposits of the Partizansk and Razdolnaya coal basins (Fig. 1A). The history of biostratigraphic investigation of these basins also associated with Perepechina et al. (1958); Likht (1961); Konovalov (1964), Vereshchagin (1977), Oleynikov et al. (1990), Likht (1994), Markevich et al. (2000) and many others. In the monograph on the Early Cretaceous flora, Krassilov (1967) elaborated the detailed stratigraphy of the Lower Cretaceous of Primorye, which is still relevant.

The Lower Cretaceous deposits of the Razdolnaya coal basin deposits are represented by the Nikan Group, consisting of the Ussuriysk, Lipovtsy and Galenki formations (Kosenko et al., 2021).

The locality Bolshoy Kuvshin is confined to the Partizansk coal basin. The Lower Cretaceous deposits of this basin were combined in the Suchan Group, which rests on the Lower Paleozoic complex of gabbroid and granitoid rocks, as well as on the marine and nonmarine Valanginian–Hauterivian deposits (Golozubov, 2006; Bugdaeva and Markevich 2009; Bugdaeva et al., 2014; Kosenko et al., 2021). The Suchan Group is conformably overlain by variegated or red-colored volcaniclastic deposits of the Korkino Group.

According to Likht (1961), Krassilov (1967) and Likht (1994), the Suchan Group consists of the Starosuchan, the Severosuchan and the Frentsevka formations. The Starosuchan Formation is composed of coarse-grained sandstones, conglomerates, rare siltstones, claystones, and coal beds. Its thickness varies from 280 m at the northeast to 800 m in the central area of the basin.

Fig. 2. Reconstruction of environment with herbaceous angiosperms from deposits of the Frentsevka Formation, Bolshoy Kuvshin locality.

The Severosuchan Formation is subdivided into the lower coal-free and upper coal-bearing subformations. The first one is 80–220 m thick and is composed of fine-grained sandstones with rare interlayers of medium-to coarse-grained sandstones. The second subformation is 120–280 m thick and consists of sandstones, siltstones, claystones, coal beds, and rare conglomerates. The main industrial deposits are located in the eastern part of the basin, near Partizansk city, where they have been mined almost from the beginning of the last century. The upper boundary of the Severosuchan Formation is marked by the last thick coal layer, known as the Velikan (Giant).

Only one angiosperm species is known from the Severosuchan Formation. This is *Aralia lucifera* Kryshtofovich, 1929, which was found by geologist M. A. Pavlov in the Velikan coal layer in the top of the Severosuchan Formation (Kryshtofovich and Pavlov, 1928). Later this species was transferred to the fossil genus *Araliaephyllum* Fontaine (Golovneva, 2018).

The best sections of the Frentsevka Formation are located on the Kamenka River in the city of Partizansk, along the Kangauz River and along the seacoast in the Andreev Inlet.

On the Kamenka River, the Frentsevka Formation includes three measures: marine *Trigonia* beds, nonmarine black shales and a measure of greenish-gray plant-bearing sandstones and siltstones (Golozubov, 2006). Total thickness of the Frentsevka Formation near city of Partizansk is about 600 m.

Trigonia beds usually begin from a bedset (about 30 m) of fine-grained grey or greenish-grey marine heterogranular sandstones with the remains of trigoniids following by beach deposits with fragments of *Isognomon* sp., *Lima* sp., *Ostrea* sp., and *Callista pseudoplana* Yabe et Nagao, 1925, representing deposits of a short-term marine ingression. It is overlain by fine-grained sandstones and siltstones with an admixture of conglomerates and rare thin coal layers. The thickness of this bedset is 70–80 m. These sediments include different plant fossils (Krassilov, 1967). The overall thickness varies between 85 and 250 m. The Albian age of this measure was confirmed by the occurrence of *Inoceramus concentricus* Parkinson, 1819 in the south part of the Partizansk coal basin from the Vladimirskaya Unit (Oleynikov et al., 1998) which are considered as deposits from the same transgression (Markevich et al., 2000).

The black shales measure is 100–150 m thick and consists of interbedded siltstones and mudstones with beds of fine-grained sandstones, representing mostly lacustrine deposits. These sediments yields freshwater mollusks, ostracods and conchostracans (Markevich et al., 2000).

The overlaying measure is 60–150 m thick and consists of intercalations of greenish-gray tuffaceous sandstones and siltstones with an admixture of coarse-grained sandstones and conglomerates. It yields remains of ferns and conifers, and also small leaves of angiosperms *Sapindopsis orientalis* Volynets, Golovneva et Zolina, 2023; Golovneva et al., 2023).

In the western part of the Partizansk coal basin, along the seacoast in the Andreev Inlet, the Frentsevka Formation is represented mostly by upper measure of greenish-gray tuffaceous sandstones. These deposits contain remains of plants and freshwater mollusks. Marine bivalves were not found in this area.

Detailed lithological and facies description of the Lower Cretaceous sediments of the Partizansk coal basin is presented in the paper by Sharudo (1960). Deposits of the Frentsevka Formation reflect mostly nonmarine sedimentation and are represented by alluvial-proluvial, lacustrine, paludal and coastal facies. Near the Palets Cape and the Bolshoy Kuvshin Cape, deposits of the Frentsevka Formation are represented by alluvial-lacustrine floodplain facies intercalated with coarse-grained sandstones and conglomerates, representing deposits of braided rivers flowing down from the upland where is now located the Ussuri Bay (Sharudo, 1960).

Based on plant megafossils and palynological data, the age of the Starosuchan Formation was estimated as Hauterivian–early Aptian or the Barremian–early Aptian and the age of the Severosuchan Formation as the late Aptian or late Aptian–early Albian (Krassilov 1967; Turbin 1994; Markevich et al., 2000; Volynets 2005), The marine *Trigonia* beds in the bottom of the Frentsevka Formation were dated as middle Albian (Markevich et al., 2000). The sediments of the Korkino Group contain few fossil plants (Volynets, 2005), and its age is inferred as the late Albian–early Cenomanian.

3. Material and methods

Volcanic rocks for isotopic dating were sampled in the upper part of the Frentsevka Formation in two sites (Fig. 1B): 1) below the conglomerates at the Palets Cape (sample 1, rhyolite tuff); 2) at the locality of angiosperms Bolshoy Kuvshin (sample 2, tuffaceous fine-grained sandstone). Sampling horizons are marked in Fig. 1C.

The locality Bolshoy Kuvshin is situated in separate outcrop exposed along the railroad in the base part of the Bolshoy Kuvshin Cape. The sequence commences and ends with thick layers of conglomerates (Fig. 1C). Sample 2 comes from a layer of plant-bearing tuffaceous fine-grained sandstone and siltstone about 15 cm thick, which is located 14 m above the lower conglomerate layer (Fig. 3).

The outcrop along the northern coast of the Palets Cape demonstrates similar stratigraphic sequence (Fig. 1C). The upper conglomerates of both sites are on the same stratigraphic level. Sample 1 was taken 0.5 m below the upper conglomerate from tuff layer that is 10–30 cm thick (Fig. 3).

Some authors interpreted the lower conglomerate layer on the Bolshoy Kuvshin Cape as marking a regional erosion event (Markevich et al., 2000) and considered the overlying layers as lower part of the Kangauz Formation of the Korkino Group (Perepechina et al., 1958; Sharudo, 1960; Markevich et al., 2000).

However, the deposits of the Frentsevka Formation are characterized by significant facies variability and all layers of conglomerates in its upper part are lenticular, as shown by the sections study near Palets Cape, where the lower conglomerates are exposed at different stratigraphic levels and have different thicknesses. Krassilov (1967) interpreted all conglomerates of the Frentsevka Formation as marking only local disconformities. In this paper, we follow Krassilov's point of view on the volume of the Frentsevka Formation.

Both samples for zircon study were processed at the Institute of Precambrian Geology and Geochronology in Saint Petersburg. The standard procedure of zircon extraction includes crushing, separation in heavy liquids, electromagnetic separation and then selection under a binocular microscope.

The samples were analyzed using two methods. The U–Pb (ID-TIMS) technique, which is the most precise and accurate, was applied to the tuff layer under the assumption of a uniform zircon population in sample 1. In situ U–Pb LA-ICP-MS method, which can be used to characterise age populations for given minerals, was applied to zircons from sample 2 (tuffaceous fine-grained sandstone) assuming heterogeneous sources of detrital zircons.

The U–Pb (ID-TIMS) geochronological study has been performed for zircons from the rhyolite tuff of sample 1 (Palets Cape). Hand-picked zircon crystals were subjected to a preliminary chemical abrasion (Mattinson, 2005). After the preliminary treatment, zircons were analyzed using standard technique (Krogh, 1973). Isotope measurements were carried out using ^{202}Pb–^{235}U spike on a multicollector TRITON TI mass spectrometer in static and dynamic modes (using ion counters). The accuracy of U/Pb ratios and U and Pb contents was 0.5%. The blanks was <3 pg Pb and 1 pg U. Experimental data were processed using PbDAT and ISOPLOT softwares (Ludwig, 1991, 2003). Ages were calculated using uranium decay constants (Steiger and Jager, 1976). Corrections for common lead were introduced using Stacey-Kramers model (Stacey and Kramers, 1975).

U–Th–Pb isotopic analyses of zircons from the volcanoclastic rocks of sample 2 (Bolshoy Kuvshin) were performed using the LA-ICP-MS. Measurements were carried out on the ESI/New Wave Research NWR213 ablation system equipped with a 213 nm Nd:YAG laser and TV2 two-volume ablation cell attached to a magnetic sector ICP-MS ThermoFinnigan Element XR. Laser spot size was 25 μm at energy output densities of 3.5–4 J/cm^2 and 10 Hz repetition rate. Before each measurement, the background (gas blank) was measured for 30 s. The ablation time was 60 s followed by 30 s wash out time. The isotopes ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{238}U and ^{232}Th were measured in E-scan mode. Calibration was performed using the GJ-1 zircon standard (Jackson et al., 2004), with a precisely established CA-ID-TIMS ^{207}Pb/^{206}Pb age of 607.7 ± 0.67 Ma (2σ) and ^{206}Pb/^{238}U age of 601.86 ± 0.37 Ma (2σ) (Horstwood et al., 2016). The primary reference zircon GJ-1 was measured twice, at the beginning and at the end of session, which included twelve unknowns and the secondary zircon standards 91500 (Wiedenbeck et al., 1995) and Plešovice (Sláma et al., 2008) for data quality control. All U–Th–Pb isotopic ratios were calculated using the GLITTER 4.0 (GEMOC) software (Van Achterbergh et al., 2001), with common lead correction applied following Andersen (2002). Calculations are based on the decay constants of Steiger and Jager (1976). Our ^{207}Pb/^{206}Pb and ^{206}Pb/^{238}U ages of zircon 91500 obtained during the period of analysing samples are 1072 ± 14 Ma (2σ, MSWD = 0.72, probability = 0.63, n = 7) and 1065 ± 15 Ma (2σ, MSWD = 0.0040, probability = 1.000, n = 7), respectively, which are in good agreement with the values reported by Horstwood et al. (2016) using CA-ID-TIMS methods (^{207}Pb/^{206}Pb age of 1066.01 ± 0.61 Ma (2σ) and ^{206}Pb/^{238}U age of 1063.51 ± 0.39 Ma (2σ). The obtained weighted average ^{206}Pb/^{238}U age for the Plešovice zircon is 338 ± 5 Ma (2σ, MSWD = 0.24, probability = 0.96, n = 7) and matches the corresponding age of 337.16 ± 0.11 Ma (Horstwood et al., 2016). Concordia ages (Ludwig, 1998) were calculated using IsoplotR software (Vermeesch, 2018). Only concordant ages (p-value >0.05) have been used for the probability density plots and calculation of peak ages (Gehrels, 2012). The obtained ages and isotopic ratios are quoted in the text and Table 1 at 1σ absolute.

Graphic reconstruction of fern-angiosperm herbaceous community was made using Terragen 4 and Xfrog programs by P. Alekseev.

4. Results of U–Pb geochronological studies

Zircon crystals from the rhyolite tuff of sample 1 are predominantly prismatic, faceted by a prism {100} (rarely in combination with {110}) and bipyramids {101}, {111}, {211}. The crystals are corroded up to smoothing of edges and faces (Fig. 4A-D). Zircon is

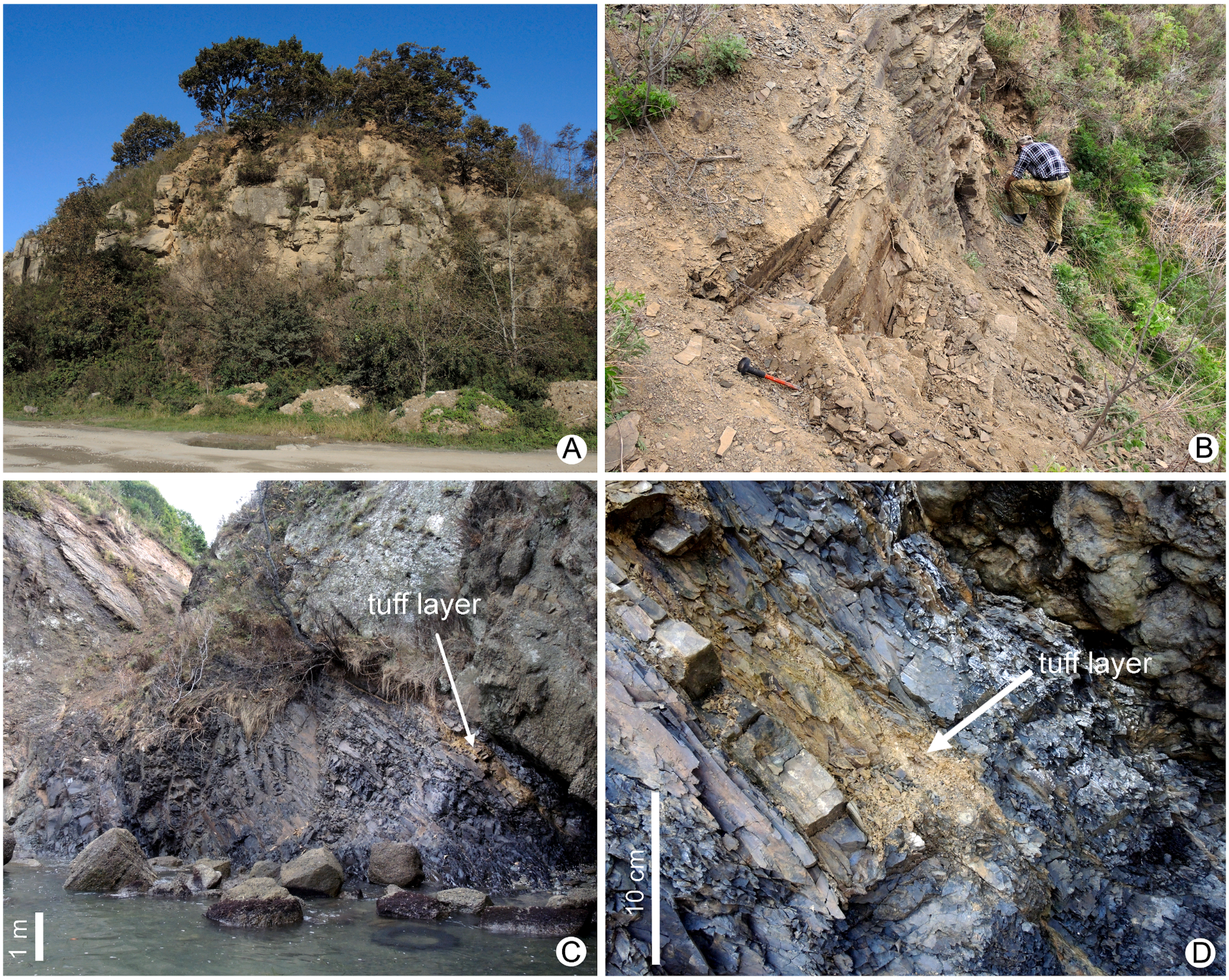

Fig. 3. A, B – section of the Frentsevka Formation in the locality Bolshoy Kuvshin: A – lower conglomerate and coarse-grained sandstones; B – plant-bearing tuffaceous fine- and middle-grained sandstones in the middle part of the section, sample 2 site. C, D - section along northern coast of the Palets Cape: C - tuff layer in upper part of Frentsevka Formation below upper conglomerate, sample 1 site; D - same section, enlarged view.

Table 1
Results of U–Pb ID-TIMS geochronological zircon studies.

N#	Characteristics: Size (μm), number of grains, colour and habit	U/Pb[a]	$^{206}Pb/^{204}Pb$[b]	Isotopic ratios corrected for blank and common Pb				Rho	Age, Ma		
				$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{207}Pb/^{235}U$	$^{206}Pb/^{238}U$		$^{207}Pb/^{235}U$	$^{206}Pb/^{238}U$	$^{207}Pb/^{206}Pb$
1	>100, 20 gr., CA 2/220 °C	50.43	556	0.0482 ± 1	0.1839 ± 1	0.1133 ± 2	0.0170 ± 1	0.65	109 ± 1	109 ± 1	107 ± 3
2	<75, 32 gr., CA 4/180 °C	48.80	263	0.0485 ± 2	0.1914 ± 1	0.1136 ± 6	0.0170 ± 1	0.37	109 ± 1	109 ± 1	126 ± 13
3	<75, 20 gr., CA 4.5/220 °C	53.23	718	0.0487 ± 2	0.1855 ± 1	0.1176 ± 6	0.0175 ± 1	0.38	113 ± 1	112 ± 1	136 ± 10
4	<75, 22 gr., CA 2/220 °C	50.27	738	0.0489 ± 1	0.2054 ± 1	0.1197 ± 4	0.0177 ± 1	0.59	115 ± 1	113 ± 1	145 ± 6

[a] U/Pb ratios for unweighted grains were determined using $^{202}Pb/^{235}U$.
[b] measured isotopic ratios; Rho – correlation coefficient of $^{207}Pb/^{235}U$ versus $^{206}Pb/^{238}U$ ratios; uncertainties (2σ) refer to last digits of corresponding ratios; CA2/220 °C – chemical abrasion during 2 h at 220 °C.

transparent and translucent, light yellow in color. The internal structure of the crystals is characterized by fine magmatic zoning and the presence of relics inherited cores in individual crystals (Fig. 4E-H).

U–Pb ID TIMS geochronological studies were carried out for the most transparent and idiomorphic zircon from four micro-samples, selected from size fractions <75 μm and >100 μm (Table 1). The selected crystals were subjected to preliminary chemical abrasion. The studied zircon is characterized by a concordant age of 109 ± 0.5 Ma (MSWD = 0.92, probability = 0.34) or is slightly discordant (Fig. 5). All analyzed data points form a discordia, the lower intersection of which with the concordia corresponds to the age of 108 ± 2 Ma (the upper intersection is 684 ± 250, MSWD = 2.2).

For geochronological study of sample 2 (Bolshoy Kuvshin), 89 zircon grains from volcanoclastic rocks were randomly selected. Detrital zircon is represented mainly by subidiomorphic short prismatic crystals and their fragments (Fig. 6). The grains are

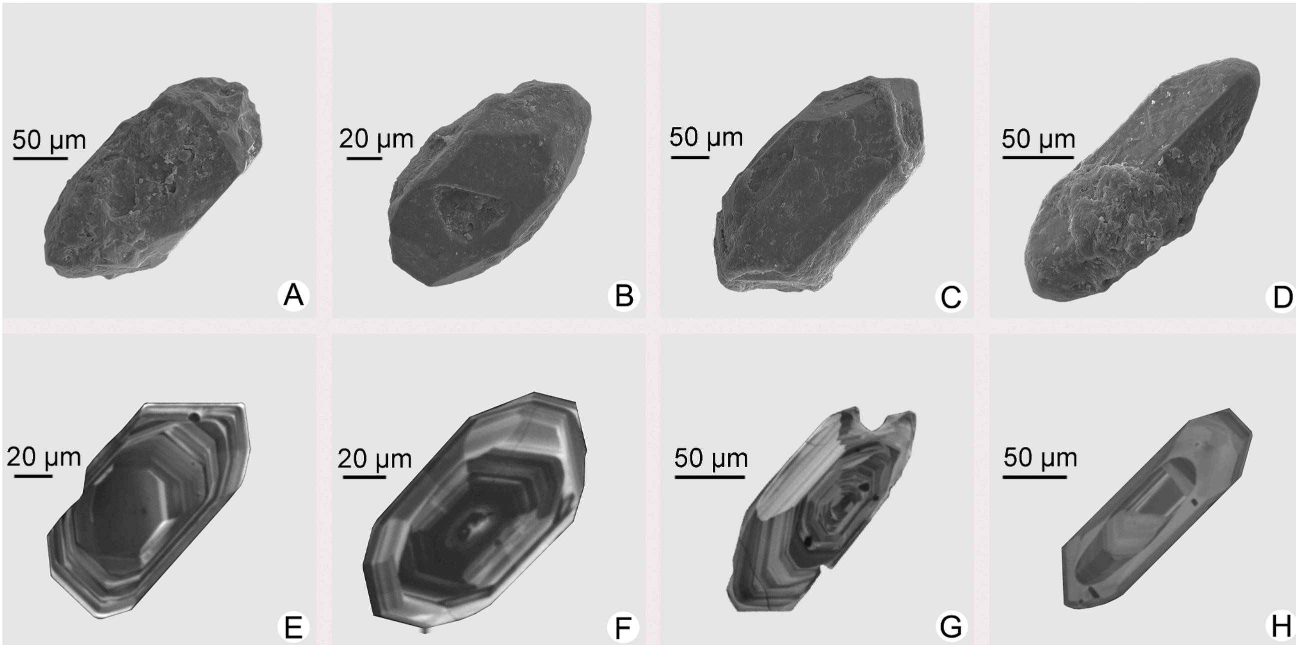

Fig. 4. Secondary electron (A–D) and cathodoluminescence (E–H) images of zircon crystals from the sample 1 (Palets Cape).

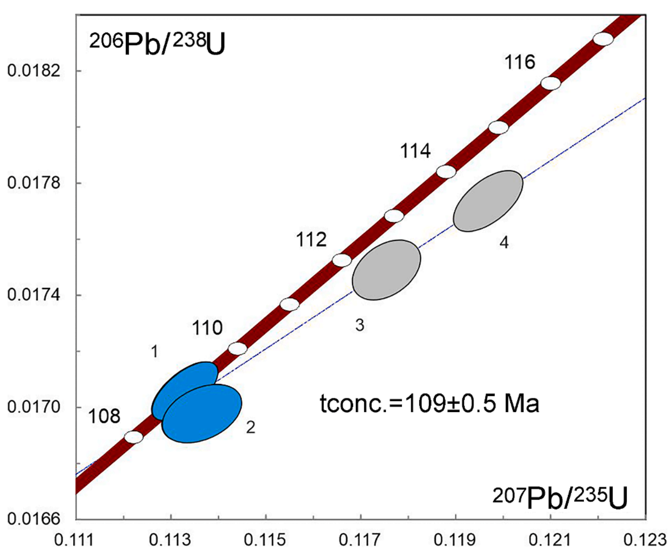

Fig. 5. Concordia diagram for zircons from the sample 1 (Palets Cape).

poorly rounded with well-preserved prismatic and dipyramidal facets. The morphology of these grains indicates that they have not been transported far from the parental rock. The majority of zircon grains show fine oscillatory zoning, while grains with sectorial zoning, which contain primary melt and mineral inclusions, are less common. The dimensionality of individual zircons of different ages varies (Fig. 6A, B).

We analysed 84 grains, yielding 30 concordant age estimates. Concordia ages for all studied grains span the intervals 171–200 and 268–275 Ma with the age peaks of ca. 185 Ma ($n = 22$) and 275 Ma ($n = 3$) on the probability density plot. Additionally, two grains showed a concordant age of 317 Ma and 108 Ma (Table 1). Thus, the maximum age of deposition of the tuffaceous fine-grained sandstone of Frentsevka Formation defined by detrital zircon age data is ca. 185 Ma.

In the plant-bearing layer tuffaceous zircons coeval with the host sandstone are poorly represented. The main sources of the tuffaceous sandstones could be the Late Paleozoic to Early Mesozoic granitoids that are widespread in the Bureya-Jiamusi terrane of the Central Asian mobile belt.

Thus, based on these results, the Frentsevka Formation can be assigned to the early Albian (109 ± 0.5 Ma), according to the current Geologic Time Scale (International Chonostratigraphic Chart, 2024).

5. Discussion

Previously, the age of the Frentsevka Formation was based mainly on marine mollusks from *Trigonia* beds. Konovalov estimated the age to be the middle Albian (Markevich et al., 2000). However, in reality this bivalve assemblage includes many endemic species. Other species have a rather wide stratigraphical range during the Albian (Tashiro and Matsuda, 1983; Matsukawa et al., 1997).

Freshwater mollusks, ostracods and conchostracans from black shales of the Frentsevka Formation are in general insufficiently studied. At present, they suggest an age range from the Aptian to the Albian (Markevich et al., 2000).

The Early Cretaceous palynoflora of Primorye is dominated by diverse fern spores and gymnosperm pollen grains. The angiosperm pollen is usually scarce in the late Aptian and Albian palynospectra. Markevich (1994, 1995) established seven palynological zones for the Lower Cretaceous deposits of the Razdolnaya and Partizansk coal basins.

According to palynological data, the first appearance of early angiosperm in Primorye was recorded in the upper part of the Lipovtsy Formation of the Razdolnaya coal basin. In this time monosulcate, tricolpate, and pentachotomocolpate angiosperm pollen appeared in the geological record of Primorye simultaneously.

Angiosperm megafossils from the upper part of the Lipovtsy Formation are represented by linear serrate leaves of *Pandanites*

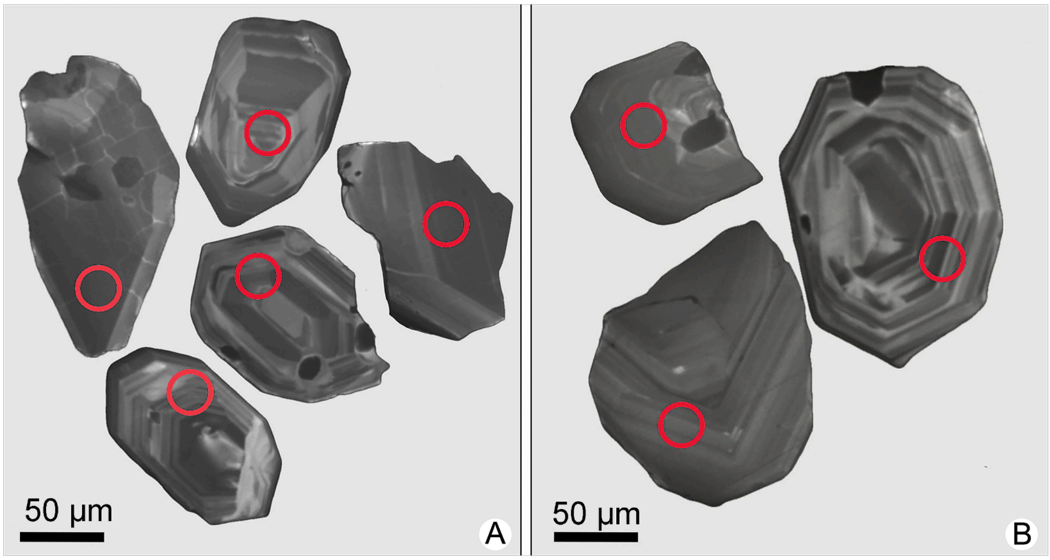

Fig. 6. Cathodoluminescence images of representative zircons from the sample 2 (locality Bolshoy Kuvshin): A - typical grains of zircon for the age group with the age peaks of ca. 185 Ma; B - typical grains of zircon for the age group with the age peaks of ca. 275 Ma. LA-ICP-MS analytical spots (25 μm) are indicated, spot numbers and Concordia ages (±1σ) refer to Table 1.

ahnertii (Kryshtofovich, 1929) Golovneva, 2021 and rather big leaf fragments of trilobate leaves with craspedodromous venation and serrate margin and ovate leaves with pinnate secondary venation and dentate margin, described as *Dicotylophyllum* spp. (Golovneva et al., 2021).

Age of the upper Lipovtsy Formation was clarified using isotopic U–Th–Pb LA-ICP-MS method (Kovaleva et al., 2016; Golovneva et al., 2021) and was estimated as 118 ± 1.4 Ma that corresponds to the middle–late Aptian (Fig. 7, 8).

The late Aptian angiosperm assemblages are unknown in China (Fig. 9). But similar age (118.07 ± 0.98 Ma, U–Pb zircon LA-ICP-MS dating, late Aptian) was obtained for a rhyolite tuff of the Jiufotang Formation in western Liaoning, Fuxin Basin (Ying et al., 2025). The Jiufotang Formation represents the third evolutionary stage of the Jehol Biota, when it reaches its peak in development and spreads over a large area (Zhou et al., 2021). Unfortunately, the section in the Fuxin Basin does not contain pollen or macrofossils of

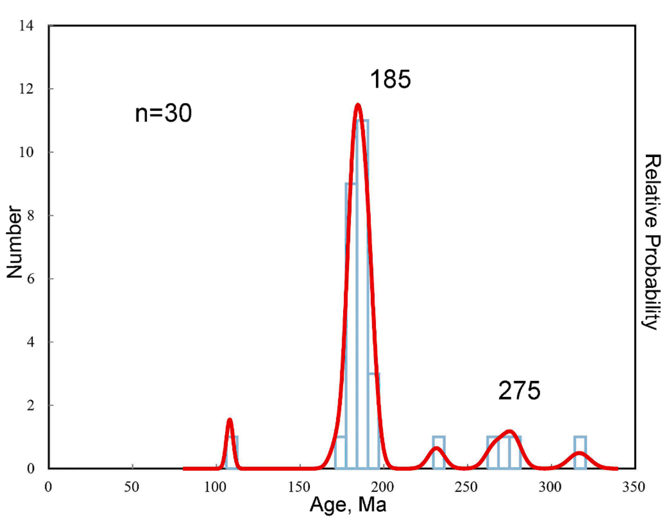

Fig. 7. Probability plots and age peaks values in Ma (Gehrels, 2012) for detrital zircons ages from sample 2 (locality Bolshoy Kuvshin).

flowering plants, since angiosperm pollen from inland areas is relatively scarce (Zhang et al., 2015).

The same age estimate (118.23 ± 0.09 Ma, U–Pb CA-TIMS method) was reported for the early angiosperm assemblage from the Anfiteatro de Ticó Formation, Patagonia, South America (Loinaze et al., 2013). Recently, Archangelsky et al. (2009) recognized three stages in the evolution of the Cretaceous angiosperms in southern South America. The angiosperm pollen record from the Anfiteatro de Ticó Formation was referred to the earliest Stage I. Pollen of this stage is represented by the genera *Clavatipollenites, Retimonocolpites, Monocolpopollenites* and *Asteropollis*, and leaf remains correspond to three morphotypes.

We supposed that earlier phase of angiosperm evolution will be revealed in Primorye in the future with a more thorough study of the Barremian and lower–middle Aptian parts of the sections (Starosuchan, Severosuchan and Ussuriysk formations). In adjacent regions (Transbaikalia, North Korea, China, Japan) angiosperm pollen was recorded in sediments of this age (Vakhrameev, Kotova, 1977; Pu, Wu, 1985; Jiang, Yang, 1996; Shi et al., 2021). In Japan, the oldest occurrence of angiosperm pollen was reported from the Barremian Nishihiro Formation (Legrand et al., 2014). In China, the late Barremian–earliest Aptian angiosperm pollen was recorded from the Huolinhe Formation, Huolinhe Basin, eastern Inner Mongolia (Guo, 1995; Shi et al., 2021). Age of the angiosperm pollen assemblages from last locality (*Clavatipollenites, Asteropollis* and *Tricolpites*) was confirmed by U–Pb zircon geochronology (SIMS and LA-ICP-MS methods), yielding 125.6 Ma (Shi et al., 2021).

The most ancient angiosperm macrofossils in Asia are known from the Yixian Formation in western Liaoning, China (Fig. 7). This is famous aquatic plant *Archaefructus* and several other genera (Duan, 1998; Sun et al., 1998, 2001, 2002; Leng and Friis, 2003, Ji et al., 2004; Leng and Friis, 2006; Wang and Zheng, 2009, 2012; Han et al., 2013, 2017; Liu and Wang, 2018).

Initially, the age of the Yixian Formation was determined as the Jurassic (137–145.3 Ma) using K–Ar, Rb–Sr and $^{40}Ar/^{39}Ar$ dating (Wang and Diao, 1984; Lo et al., 1999). Later the age of the Yixian Formation was defined as the Hauterivian–Barremian using $^{40}Ar/^{39}Ar$ (Swisher et al., 1999; Wang et al., 2001; Swisher et al., 2002) as well as U–Pb (TIMS) methods (Peng et al., 2003; Meng

Fig. 8. Localities of early angiosperms in Primorye and northeastern China: 1 – Yixian Formation, Daxinfangzi Bed; 2 – Yixian Formation, Jianshangou Bed; 3 – Dalazi Formation; 4 – Chengzihe Formation; 5 – Lipovtsy Formation; 6 – Frentsevka Formation.

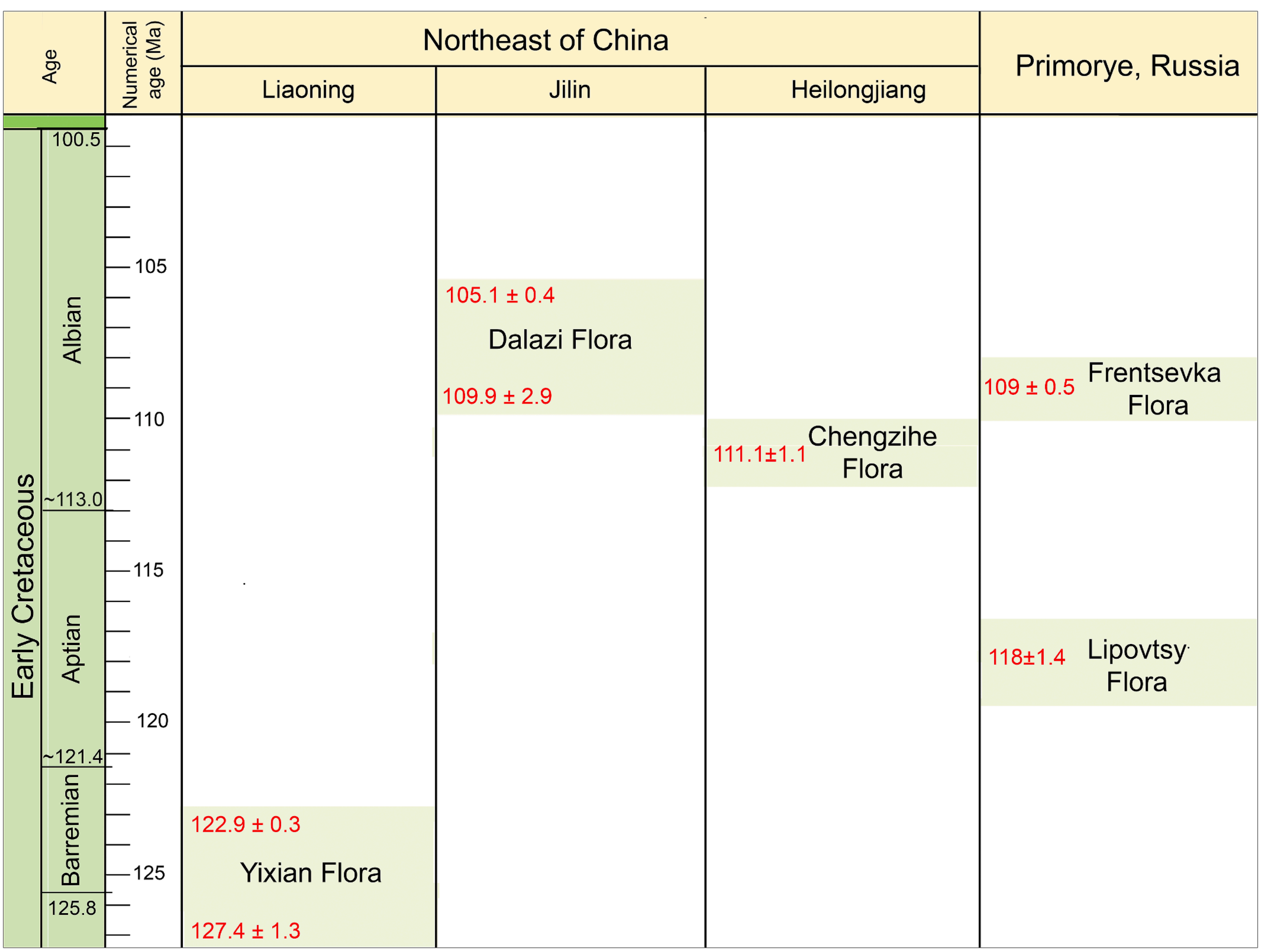

Fig. 9. Correlation of Early Cretaceous floras with early angiosperms based on isotopic data.

et al., 2008; Chang et al., 2009; Zhong et al., 2021). The remains of the early angiosperms of the Yixian Formation comes from the Daxinfangzi Bed in Lingyuan and Jianshangou Bed in Beipiao (Sun et al., 2011). The age the Daxinfangzi Bed with *Archaefructus sinensis* Sun, Dilcher, Ji et Nixon, 2002, *Hyrcantha decussata* (Leng et Friis, 2003) Dilcher, Sun, Ji et Li, 2007 and *Leefructus mirus* Sun, Dilcher, Wang et Chen, 2011 was estimated from 122.9 to 124.4 Ma (Swisher et al., 1999; Zhang, 2006; Meng et al., 2008). And the age of the Jianshangou Bed with *Archaefructus liaoningensis* Sun, Dilcher, Zheng et Zhou, 2002 and *A. eoflora* Ji, Li, Bowe, Liu et Taylor, 2004 was estimated from 125.2 to 127.4Ma (Wang et al., 2001; Swisher et al., 2002).

Stratigraphically higher occurrences are known in the uppermost part of the Chengzihe Formation, Heilongjiang, China (Fig. 8, 9). This assemblage includes *Asiatifolium elegans* Sun, Guo et Zheng, 2002, *Jixia pinnatipartita* Guo et Sun, 2002, *Jixia chengzihensis* Sun et Dilcher, 2002, *Jixia* sp., *Shenkuoa caloneura* Sun et Guo, 2002, *Zhengia chinensis* Sun et Dilcher, 2002, *Xingxueina heilongjiangensis* Sun et Dilcher, *Xingxuephyllum jixiense* Sun et Dilcher, 2002 and poor preserved reproductive organs (Sun and Dilcher, 2002).

The geologic age of the Chengzihe Formation has been debated for a long time, because this terrestrial sequence has no key marine fossils. Freshwater mollusks, dinoflagellates and conchostracans from these deposits are in general insufficiently studied and suggested age ranged from the Hauterivian up to Aptian (Gu et al., 1997; Sun and Dilcher, 2002; Sha et al., 2003). New isotopic data (U–Pb LA-ICP-MS) yield age 111.1 $\pm$ 1.1 Ma that correspondent to the early Albian (Chen et al., 2018).

This age corresponds to our isotopic age of the upper part of the Frentsevka Formation (109 $\pm$ 0.5 Ma). This means that the Bolshoy Kuvshin assemblage of herbaceous angiosperms and the Chengzihe flora existed at the same time (Fig. 9). It should be noted, that the Bolshoy Kuvshin assemblage includes two common species with the flora of the Chengzihe Formation: *Jixia pinnatipartita* and *Asiatifolium elegans*.

The diversity of leaf morphology of angiosperms of the Frentsevka Formation increases significantly in comparison with late Aptian Lipovtsy angiosperm assemblage (Golovneva et al., 2021). More than 10 different morphotypes were recognized (in Lipovtsy assemblage only 3 morphotypes). Various ternate, pinnatifid and pinnately compound leaves appeared, also nymphaeaphylls and trochodendrophylls (Golovneva et al., 2021).

The early Albian age (109 $\pm$ 0.5 Ma) assigned to the upper part of the Frentsevka Formation, clarified previous age estimates (early–middle Albian) based on palaeobotanical, palynological and mollusks data.

Even younger angiosperm remains in China were found in the Dalazi Formation in the Yanji basin, which is located in the Jilin Province, northeastern China, near the China-North Korea border (Tao and Zhang, 1990; Golovneva et al., 2023).

The geologic age of the Dalazi Formation was regarded from the Aptian (Tao and Zhang, 1990) up to Albian (Cao, 1994) and Cenomanian (Nichols et al., 2006) based on plant micro- and macrofossils. The isotopic age of the Dalazi Formation was estimated from 105.1 $\pm$ 0.4 Ma (Zhong et al., 2021; Shen et al., 2021) up to 109.9 $\pm$ 2.9 Ma (Li et al., 2015) that corresponds to the middle-late Albian.

As the above review has shown, the age of most of the localities with early angiosperms in the territory of Primorye and northeastern China has long been debated and now has been precisely dated by a series of recent radiometric analyses. Currently this is the best dated succession of early angiosperm assemblages in the interval between the Barremian and late Albian. It reflects the most important phases in the evolution of angiosperms in this region. Compared to other dating methods, isotopic dates are usually younger. These data change not only our understanding of the age of individual assemblages of early angiosperms, but also the correlation of various events in the development of angiosperms in different sections.

6. Conclusion

1. Isotopic data (U–Pb ID-TIMS dating) are indicated early Albian age (109 $\pm$ 1 Ma) for the early angiosperm assemblage from the upper part of the Frentsevka Formation, the Partizansk coal basin, Primorye Region, Russia.
2. The early angiosperm assemblage from the locality Bolshoy Kuvshin is coeval with angiosperm assemblage from upper part of the Chengzihe Formation, Heilongjiang.

CRediT authorship contribution statement

Lina B. Golovneva: Writing – review & editing, Supervision, Conceptualization. **Anastasia A. Zolina:** Writing – original draft, Validation, Investigation. **Ekaterina B. Salnikova:** Writing – original draft, Methodology, Investigation. **Eugenia V. Bugdaeva:** Writing – original draft, Investigation. **Elena B. Volynets:** Writing – original draft, Investigation. **Elena V. Adamskaya:** Methodology, Formal analysis. **Viktor P. Kovach:** Formal analysis, Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors are grateful to anonymous reviewers for their helpful comments. This study was supported by the Russian Science Foundation (grant number № 24-24-00492).

Data availability

Data will be made available on request.

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