



Geochronological study on the Sanjiaochengzi fossil locality: Age constraints on a Late Jurassic flora of western Liaoning, Northeast China

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Abstract

Some fossil plants, previously identified as angiosperm species, have been reported from the “Jiulongshan”/Haifanggou Formation at the Sanjiaochengzi fossil locality in Huludao City, western Liaoning, Northeast China. However, the precise ages of these fossil plants remain uncertain. To address this issue, we conducted LA-ICP-MS zircon U-Pb dating on two tuffaceous interlayer samples from the fossil-bearing horizon of this section. The resulting ages of 159.9 ± 0.5 Ma and 160.1 ± 0.5 Ma constrain the depositional age of the fossil-bearing horizon to the Late Jurassic (Oxfordian stage). By integrating these chronological results with regional stratigraphic correlations, we determine that the fossil-bearing horizon of the Sanjiaochengzi section belongs to the lower part of the Tiaojishan Formation.

Keywords: Jurassic, Yanliao biota, Tiaojishan Formation, fossil plants

Introduction

Over the past two decades, three fossil species which were attributed as potential Jurassic angiosperm by authors have been reported from the same bed of Jurassic strata in western Liaoning, Northeast China: *Schmeissneria sinensis* (Wang *et al.*, 2007; Wang, 2010), *Xingxueanthus sinensis* (Wang & Wang, 2010) and *Euanthus panii*

(Liu & Wang, 2016). These fossils were discovered in the “Jiulongshan”/Haifanggou Formation at north of Sanjiaochengzi village, Baimashi Township, Lianshan District, Huludao City, Liaoning Province (Wang & Wang, 2010; Liu & Wang, 2016; Huang, 2016). While their taxonomic placement remains a subject of debate (Herendeen *et al.*, 2017), they have been considered to offer possible new insights into understanding the origins and early evolution of angiosperms (Wang & Wang, 2010; Liu & Wang, 2016). Despite the scientific significance of these fossil-bearing strata, geochronological analyses and systematic stratigraphic correlations have yet to be published (Huang, 2016). In this study, we present two LA-ICP-MS zircon U-Pb ages from tuffaceous interlayer samples within the fossiliferous horizon, providing new age constraints for these fossil plants.

Geological setting

Well-preserved fossil plants have been discovered from the Middle–Late Jurassic terrestrial strata throughout northeastern China, including Liaoning, Hebei, and Inner Mongolia (Fig. 1A). They are well known as the Yanliao biota, which is further subdivided into the early Assemblage Daohugou biota and the late Assemblage Linglongta biota (Huang, 2015; Huang *et al.*, 2018). Representative stratigraphic sections of the Yanliao biota are illustrated in Fig. 1B.

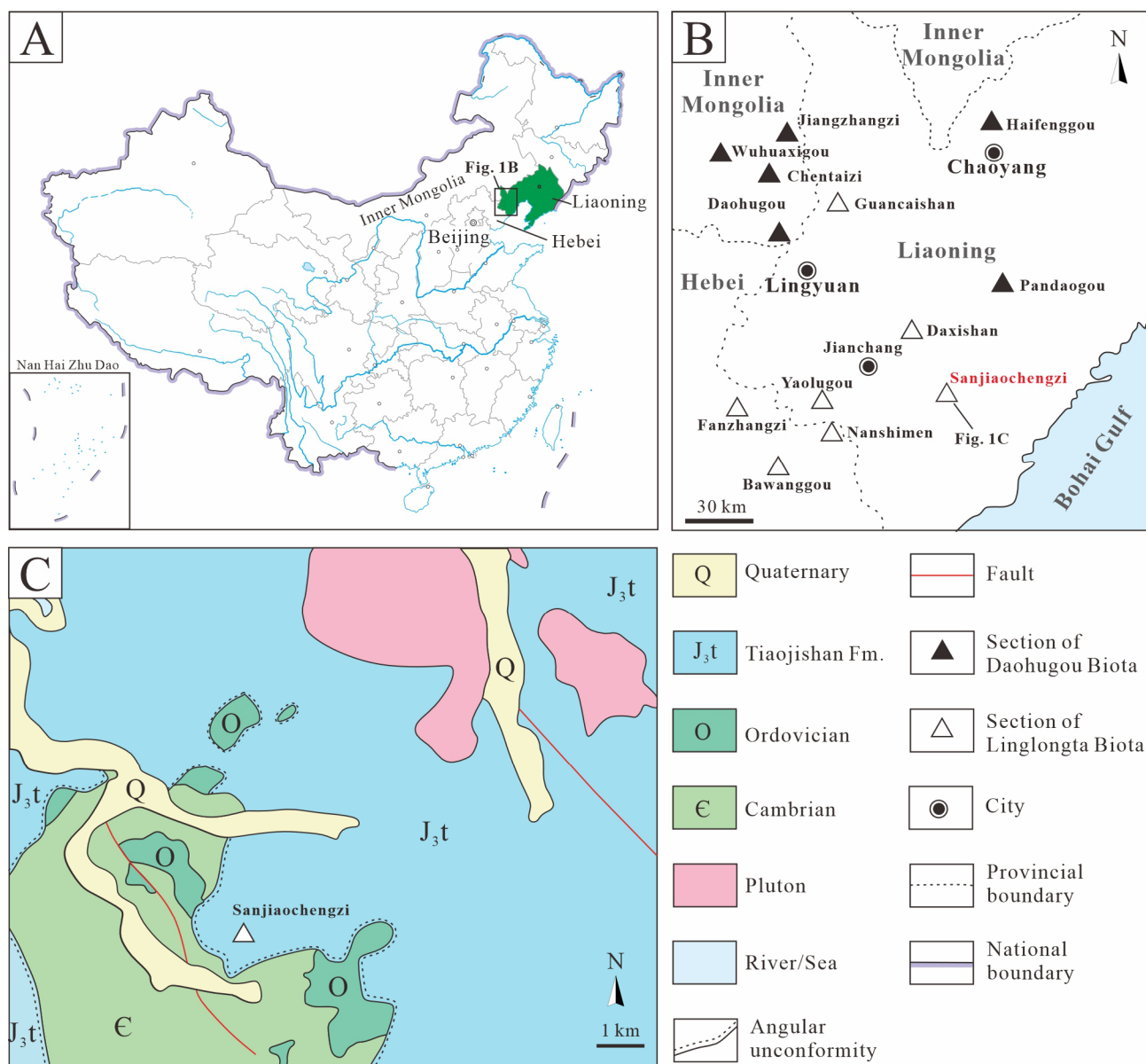


FIGURE 1. The location and geological maps of the study area. **A**, The location of the study area. **B**, Type sections of the Daohugou and Linglongta Biotas. **C**, Geological maps of the Sanjiaochengzi section, Huludao City, western Liaoning (GPS: 120°22'5.75"E, 40°58'7.25"N; modified after Liu & Wang, 2016).

The Jurassic succession in western Liaoning comprises, in ascending order, the Xinglonggou, Beipiao, Haifanggou, Tiaojishan and Tuchengzi formations. Fossils of the Daohugou biota are preserved in the Middle Jurassic Haifanggou Formation, whereas the Linglongta biota occurs in the overlying Upper Jurassic Tiaojishan Formation (Huang, 2015). The Haifanggou Formation, composed of conglomerates and volcanoclastic deposits, is distributed across western Liaoning and the Ningcheng area of Inner Mongolia. In contrast, the Tiaojishan Formation is characterized by voluminous andesitic volcanic rocks during active eruptive phases, and interbedded tuffaceous

siltstone and shale units that accumulated in a lacustrine phase during volcanic quiescence (Huang, 2019). The sub-molasse formation of the bottom Haifanggou/Longmen formations and an angular unconformity separates the Tiaojishan Formation from the underlying Haifanggou Formation, marking Episode A of the Yanshan Movement—a major compressional tectonic event first documented by Wong (1927). This episode of tectonic event induced significant paleogeographic restructuring and geographical shifts, driving distinct differentiation between the Daohugou and Linglongta biotas (Huang, 2015).

Material and methods

The Sanjiaochengzi section predominantly consists of light-white tuffaceous layers and interbedded lacustrine greyish-black shales, with localized occurrences of subvolcanic rocks (~ 30 cm thick, Fig. 2 B). The fossil plants are preserved in the greyish-black shales. Two tuffaceous samples (SJCZ-Z-254 and SJCZ-Z-255), used for the LA-ICP-MS zircon U-Pb analysis (Fig. 2 B), were collected from layers a few meters higher than the dark shales, thereby constraining the age of the fossil plants.

Before isotopic analysis, bulk rock samples underwent sequential cleaning and mechanical fragmentation to liberate zircon crystals. The zircon grains were separated by hand-picking under a binocular microscope. These

grains were embedded in 25 mm epoxy mounts, polished to expose grain interiors, and sputter-coated with gold films to ensure conductivity. Cathodoluminescence (CL) imaging combined with transmitted and reflected light microscopy (Fig. 3) was employed to evaluate zircon internal structures, enabling discrimination of inherited cores, metamorphic overgrowths and radiation-damaged domains unsuitable for analysis.

The zircon U-Pb isotopic analyses were conducted using an Agilent 7500 LA-ICP-MS instrument at the State Key Laboratory of Continental Dynamics, Northwest University, Xi'an, China. The off-line selection and integration of background and signals, time-drift correction and quantitative calibration were conducted using GLITTER 4.0 software. Zircon U-Pb concordia and

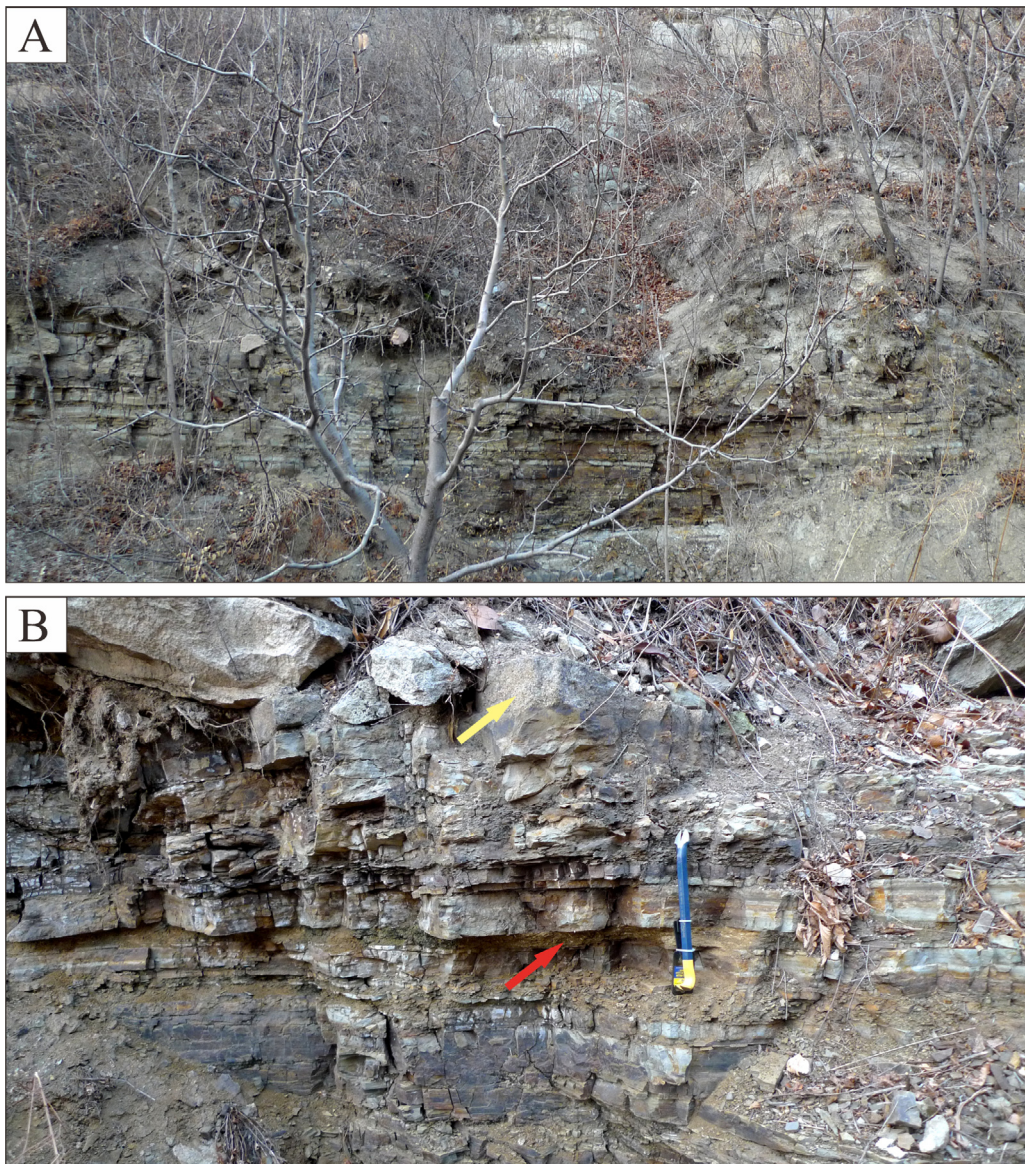


FIGURE 2. Field photographs of the Sanjiaochengzi section, Huludao City. **A**, Overview of the outcrop. **B**, Close-up view of the lower part of the so-called boundary of the Jiulongshan-Tiaojishan formations by Liu & Wang, 2016. The chisel is approximately 30 cm in length. Arrows indicate subvolcanic rocks (yellow) and a tuff layer (red). This tuff layer product very tiny zircons (*ca.* 10 microns in length) that remains difficulty for geochronological dating.

TABLE 1. Results of LA-ICP-MS zircon U-Pb analyses for the tuff from the Tiaojishan Formation in the Sanjiaochengzi village, Huludao City, western Liaoning.

Analyses	Pb	Th	U	Th/U	²⁰⁷ Pb/ ²⁰⁶ Pb		²⁰⁷ Pb/ ²³⁵ U		²⁰⁶ Pb/ ²³⁸ U		²⁰⁷ Pb/ ²³⁵ U		²⁰⁶ Pb/ ²³⁸ U		Concordance (%)		
					Ratio	1σ	Ratio	1σ	Ratio	1σ	Age	1σ	Age	1σ			
SJ CZ-Z-254																	
01	2	51	63	0.8	0.04949	0.00375	0.18047	0.01334	0.02645	0.00044	171.3	167.9	168.5	11.5	168.3	2.8	99.9
02	3	71	71	1.0	0.04967	0.00336	0.19898	0.01404	0.02906	0.00047	179.7	160.5	184.3	11.9	184.7	2.9	99.8
03	10	141	295	0.5	0.05111	0.00183	0.17694	0.006	0.02511	0.00023	245.7	80.2	165.4	5.2	159.9	1.5	96.7
04	14	219	414	0.5	0.04919	0.00151	0.17052	0.00487	0.02515	0.00021	157.0	69.7	159.9	4.2	160.1	1.3	99.9
05	21	843	486	1.7	0.04987	0.00191	0.17141	0.00627	0.02493	0.00025	189.1	86.8	160.6	5.4	158.8	1.5	98.9
07	4	57	113	0.5	0.04928	0.00282	0.17056	0.00949	0.02511	0.00032	161.1	128.8	159.9	8.2	159.9	2	99.9
08	16	190	224	0.8	0.054	0.00157	0.36121	0.00986	0.04852	0.00041	370.9	64.2	313.1	7.4	305.4	2.5	97.5
09	7	89	177	0.5	0.0581	0.00194	0.21418	0.00677	0.02674	0.00024	532.9	72.0	197.1	5.7	170.1	1.5	86.3
10	8	127	235	0.5	0.04907	0.00198	0.17091	0.00661	0.02527	0.00025	151.1	91.9	160.2	5.7	160.9	1.6	99.6
11	3	84	86	1.0	0.04954	0.00372	0.17064	0.01248	0.02499	0.00043	173.4	166.5	160	10.8	159.1	2.7	99.4
12	3	60	71	0.8	0.05193	0.0039	0.18005	0.01316	0.02515	0.00043	282.2	162.9	168.1	11.3	160.1	2.7	95.2
14	5	140	120	1.2	0.07859	0.00156	2.17596	0.03866	0.20083	0.00161	1161.8	38.81	1173.4	12.36	1179.8	8.64	99.5
15	3	73	66	1.1	0.04981	0.00363	0.20276	0.0144	0.02953	0.00049	186.0	161.4	187.5	12.2	187.6	3.1	99.9
16	5	126	117	1.1	0.04937	0.00262	0.16999	0.00873	0.02498	0.00031	165.2	119.5	159.4	7.6	159	1.9	99.7
18	11	208	299	0.7	0.0517	0.00181	0.17707	0.00588	0.02484	0.00023	272.2	78.3	165.5	5.1	158.2	1.4	95.6
19	5	123	116	1.1	0.04985	0.00339	0.17351	0.01147	0.02524	0.0004	188.2	151.0	162.5	9.9	160.7	2.5	98.9
22	10	141	230	0.6	0.05044	0.00205	0.21823	0.00851	0.03138	0.00033	215.5	91.6	200.4	7.1	199.2	2	99.4
23	4	110	93	1.2	0.05081	0.00328	0.17569	0.01101	0.02508	0.00037	232.1	142.3	164.3	9.5	159.7	2.3	97.2
25	11	154	341	0.5	0.04752	0.00147	0.15707	0.00457	0.02397	0.0002	74.6	72.5	148.1	4	152.7	1.2	96.9
27	6	83	188	0.4	0.04909	0.00193	0.17124	0.00643	0.0253	0.00025	151.9	89.4	160.5	5.6	161.1	1.6	99.6
SJ CZ-Z-255																	
02	21	627	554	1.1	0.0504	0.00124	0.17283	0.00391	0.02486	0.00018	213.7	56.0	161.9	3.4	158.3	1.1	97.8
03	15	180	437	0.4	0.04944	0.00143	0.18122	0.00489	0.02658	0.00021	168.9	66.1	169.1	4.2	169.1	1.3	99.9
04	23	809	588	1.4	0.04924	0.0011	0.16616	0.00338	0.02447	0.00017	159.2	51.6	156.1	2.9	155.8	1.1	99.8
07	33	1335	744	1.8	0.04922	0.00102	0.17038	0.00316	0.0251	0.00017	158.3	47.9	159.8	2.8	159.8	1	99.9
08	8	120	240	0.5	0.0497	0.0023	0.17141	0.00763	0.02501	0.00028	181.1	104.3	160.6	6.6	159.2	1.8	99.1
09	5	82	163	0.5	0.05045	0.00267	0.17391	0.00889	0.025	0.00032	215.8	118.0	162.8	7.7	159.2	2	97.8
10	10	136	309	0.4	0.04967	0.00163	0.17232	0.00534	0.02515	0.00022	179.7	74.6	161.4	4.6	160.2	1.4	99.3
11	9	121	276	0.4	0.04918	0.0016	0.17146	0.00529	0.02528	0.00022	156.6	74.6	160.7	4.6	160.9	1.4	99.9
13	20	400	583	0.7	0.05099	0.0015	0.17444	0.00482	0.02481	0.0002	240.4	66.5	163.3	4.2	158	1.3	96.8
14	5	140	121	1.2	0.04944	0.00242	0.17293	0.00819	0.02537	0.0003	168.7	110.7	162	7.1	161.5	1.9	99.7
15	9	142	265	0.5	0.05422	0.00158	0.20109	0.0055	0.0269	0.00023	379.9	64.3	186	4.7	171.1	1.4	92.0
16	9	318	219	1.5	0.0492	0.00163	0.17581	0.00552	0.02592	0.00023	157.3	75.7	164.4	4.8	164.9	1.4	99.7
17	12	232	347	0.7	0.04958	0.00142	0.17848	0.00479	0.02611	0.00021	175.2	65.5	166.8	4.1	166.2	1.3	99.6
18	8	124	240	0.5	0.04931	0.0019	0.18026	0.00665	0.02652	0.00026	162.4	87.9	168.3	5.7	168.7	1.7	99.8
19	6	84	82	1.0	0.05082	0.00198	0.35135	0.0131	0.05015	0.00052	232.7	87.4	305.7	9.9	315.4	3.2	96.8
20	5	127	145	0.9	0.04917	0.00258	0.16967	0.00861	0.02503	0.00032	156.2	118.4	159.1	7.5	159.4	2	99.8
21	14	260	409	0.6	0.04925	0.00181	0.17116	0.006	0.02521	0.00024	159.9	83.8	160.4	5.2	160.5	1.5	99.9
23	3	83	68	1.2	0.04924	0.00362	0.17159	0.01232	0.02528	0.00041	159.2	163.7	160.8	10.7	161	2.6	99.9

Analyzers are Agilent 7500, the laser is 32µm and the error is 1σ. Calculated by Glitter

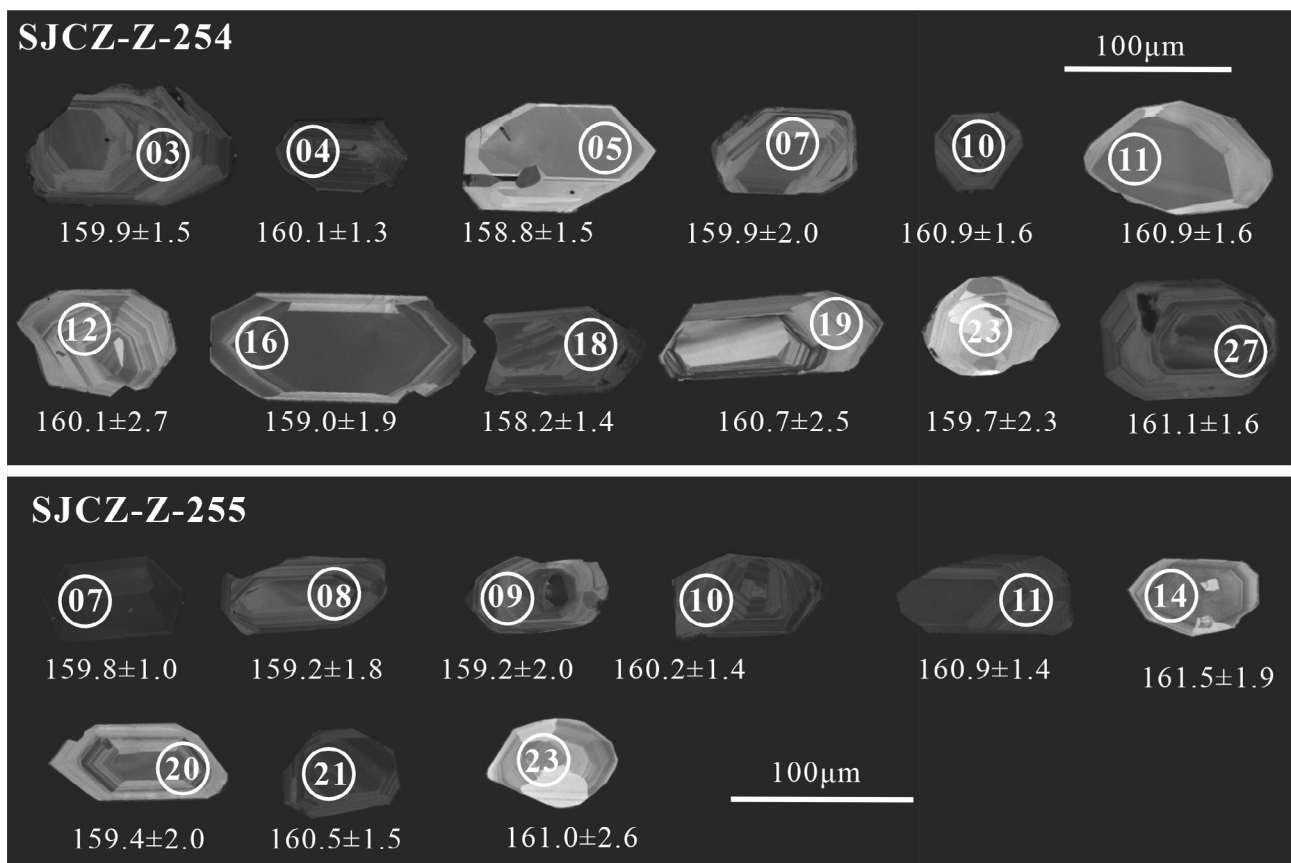


FIGURE 3. CL images of representative zircons from the tuffaceous samples from the Sanjiaochengzi section, Huludao, western Liaoning. White circles show the locations of analysis spots, and U-Pb ages (Ma) are provided nearby.

weighted mean age plots were produced using Isoplot/Ex_ver3 (Ludwig, 2003), with uncertainties reported at 1σ confidence (Table 1). Discussion of the age data is based on $^{207}\text{Pb}/^{206}\text{Pb}$ ages for analyses older than 1000 Ma, and $^{206}\text{Pb}/^{238}\text{U}$ ages for younger grains.

Results

Zircon grains separated from the tuff samples exhibit euhedral morphologies with typical grain lengths ranging from 50 to 100 μm . CL imaging reveals that most crystals display well-defined oscillatory zoning patterns, characteristic of magmatic growth (Fig. 3). The concentrations of Th and U range from 51–1335 ppm and 63–744 ppm, respectively. The calculated Th/U ratios of 0.4–1.8 support a magmatic origin for these zircons, consistent with established magmatic zircon discrimination criteria (Rubatto & Gebauer, 2000). In constructing concordia diagrams and calculating weighted mean ages, we applied rigorous data filtering by excluding analyses showing either excessive scatter in U-Pb isotopic systematics or concordance values below 95%.

Twenty-seven zircon grains from the sample

SJ CZ-Z-244 were analysed, of which ten analyses were excluded due to discordance exceeding 5%. Among the remaining concordant analyses, eleven grains yielded a concordia age of 159.9 ± 0.5 Ma (Fig. 4A) and a weighted mean age of 160.0 ± 1.1 Ma (Fig. 4B). The remaining six grains display ages spanning 168–305 Ma. Twenty-three grains from the sample SJ CZ-Z-245 were analysed and eight analyses were rejected for discordance ($>5\%$). Nine concordant spots yielded a concordia age of 160.1 ± 0.5 Ma (Fig. 4C) and a weighted mean age of 160.2 ± 1.0 Ma (Fig. 4D). Another six analyses show old ages ranging from 165 Ma to 315 Ma.

Discussion

Age of possible pre-Cretaceous angiosperms

Zircon U-Pb geochronological analysis of two tuff samples (SJ CZ-Z-254 and SJ CZ-Z-255) from the fossiliferous horizon in the Sanjiaochengzi section yields concordia ages of 159.9 ± 0.5 Ma and 160.1 ± 0.5 Ma, respectively. These ages are consistent within the error and constrain the depositional age of the fossiliferous horizon to the Late Jurassic (Oxfordian stage).

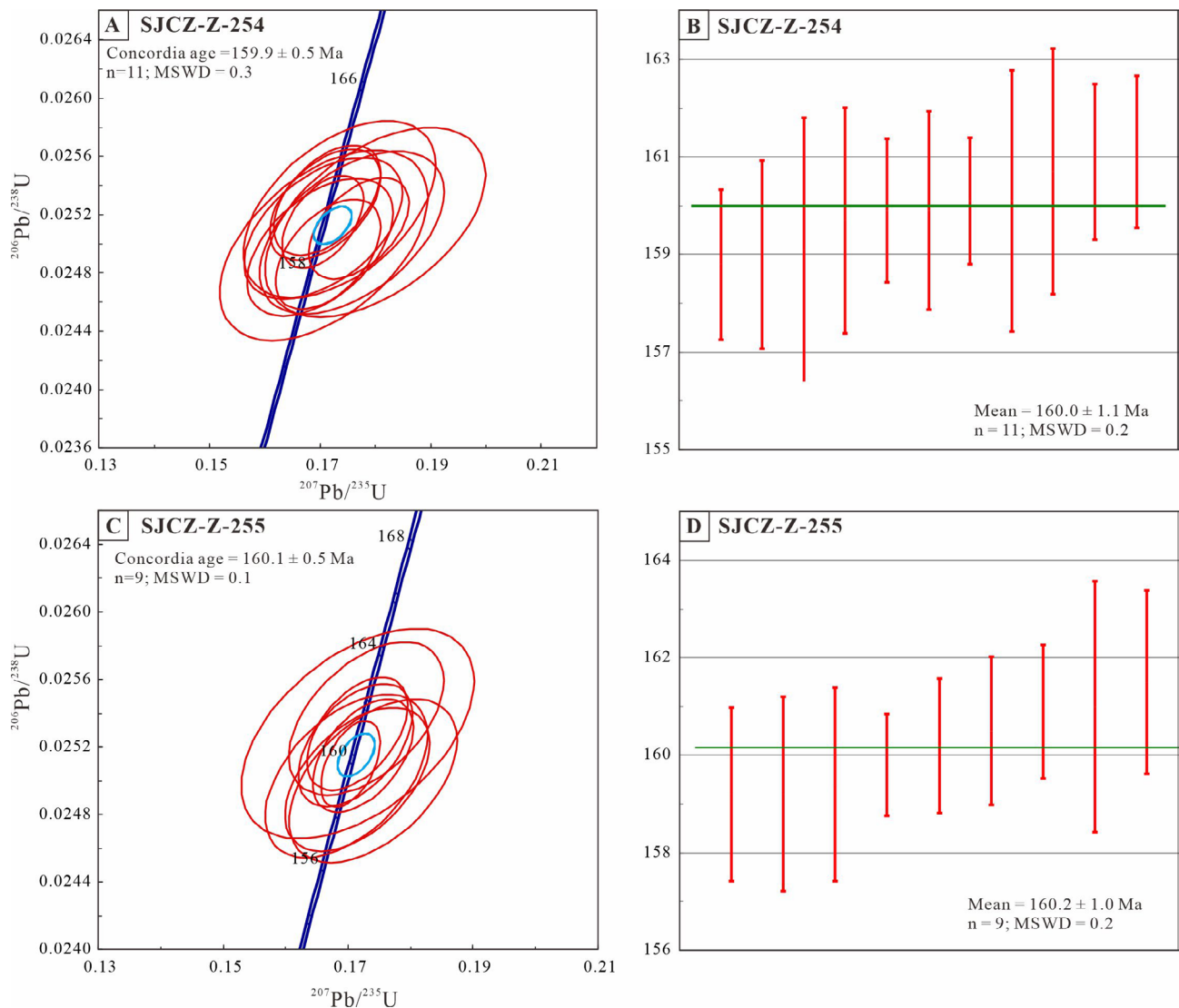


FIGURE 4. Age plots for tuffaceous samples from the Tiaojishan Formation of the Sanjiaochengzi section, Huludao City, western Liaoning Province. **A, B**, Zircon U-Pb concordia and weighted mean age plots for the sample SJCZ-Z-254. **C, D**, Zircon U-Pb concordia and weighted mean age plots for the sample SJCZ-Z-255.

Stratigraphic correlation

Some researchers have proposed defining the boundary between the Haifanggou and Tiaojishan formations at the first occurrence of volcanic rocks in this section (Fig. 2B, yellow arrow; Wang & Wang, 2010). Based on this criterion, they suggest that the fossil-bearing stratum lies a few meters below this boundary, placing it either within the uppermost part of the Jiulongshan Formation (Liu & Wang, 2016) or assigning it to the Haifanggou Formation (Huang, 2016). Huang (2019) further argued that the so-called “Jiulongshan Formation” in northern Hebei Province actually corresponds to the Longmen Formation, which underlies the Tiaojishan/Jiulongshan formations. Additionally, it has been suggested that the Tiaojishan and Jiulongshan formations represent contemporaneous heterotopic facies of volcanic deposits (Huang, 1960; Huang, 2019), with the base of the Tiaojishan Formation

typically consisting of a thick sequence of extrusive rocks or lava breccia. Our field observations indicate that the “volcanic rocks” at this site are actually subvolcanic intrusions emplaced along weak bedding planes. This intrusive unit is relatively thin, measuring only about 30 cm, and the lithology of the overlying and underlying tuffaceous sequences remains consistent. The observed lithostratigraphic continuity suggests that defining the boundary at this intrusive contact is geologically inappropriate.

The lithology of the Sanjiaochengzi section closely resembles that of the Daxishan section in Jianchang County, with both sections comprising andesite, various types of tuffaceous sandstone, siltstone, mudstone, and fossil-bearing shale. Furthermore, our dating results support the stratigraphic correlation between the Sanjiaochengzi and Daxishan sections. Age constraints

for key fossil horizons in the Daxishan section, determined by CA-ID-TIMS and SIMS, range from 160.89 to 160.25 Ma (Wang *et al.*, 2013; Chu *et al.*, 2016), which closely aligns with our zircon U-Pb ages of 159.9 and 160.1 Ma. Additionally, dark, fossil-rich shales are a distinctive feature of the Tiaojishan Formation, occurring in multiple localities such as Guancaishan and the Liujiang Basin. Given these similarities, the fossiliferous horizon of the Sanjiaochengzi section can be confidently assigned to the lower part of the Tiaojishan Formation.

Abundant well-preserved fossil plants have been reported discovered at this outcrop, including *Selaginellites* Zeiller, *Equisetites* Sternberg, *Coniopteris* Brongniart, *Cladophlebis* Brongniart, *Pterophyllum* Brongniart, *Nilssonia* Brongniart, *Anomozamites* Schimper, *Zamites* Brongniart, *Ctenis* Lindley et Hutton, *Cycadolepis* Saporta, *Taeniopteris* Brongniart, *Ginkgoites*, Seward, *Baiera* Braum emend. Florin, *Sphenobaiera* Florin, *Cephalotaxopsis* Fontaine, *Podocarpites* Andrae, *Brachyphyllum* Brongniart, *Pagiophyllum* Heer, *Pityophyllum* Nathorst, *Cupressiocladius* Seward, *Schizolepis* Braun, *Carpolithus* Wallerius and *Yanliaoa* Pan (Wang & Wang, 2010).

Current research on impression fossil plants within the Yanliao biota predominantly focuses on the Haifanggou Formation, particularly those documented in the Beipiao area of western Liaoning and the Daohugou area of Inner Mongolia (*e.g.*, Zheng *et al.*, 2012; Huang, 2016). In contrast, the silicified fossil woods from the Tiaojishan Formation have been extensively studied (*e.g.*, Wang *et al.*, 2005, 2017), while the research on impression fossil plants in this stratigraphic unit remains comparatively limited. The Sanjiaochengzi section yielded multiple types of well-preserved fossil plants dating to the early Late Jurassic, showing a unique plant community assemblage, which is of great significance for understanding the plant evolution of the Yanliao biota.

Conclusion

Our zircon U-Pb dating of tuffaceous samples from the Sanjiaochengzi section in Huludao, western Liaoning Province, provides robust age constraints for these plant fossils. The fossil-bearing layer is part of the lower Tiaojishan Formation and dates to approximately 160 Ma, placing it within the Oxfordian Stage of the Late Jurassic. This new evidence identifies another lacustrine fossil-bearing horizon within the Upper Jurassic Tiaojishan Formation, offering additional potential for exceptional preservation and providing valuable insights into the evolutionary history of life during the middle Mesozoic.

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