



Article The Late Triassic Molasse Deposits in Central Jilin Province, NE China: Constraints on the Paleo-Asian Ocean Closure

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Abstract: This paper presents a new detailed study including zircon U-Pb-Hf isotopic, whole-rock geochemical and Sr-Nd isotopic analyses of conglomerate and granitic pebbles from the molasse deposits in central Jilin Province, NE China. These data are used to better constrain the Late Permian-Triassic tectonic evolution regarding particularly the final closure of the Paleo-Asian Ocean (PAO) along the Changhun-Yanji suture (CYS). Zircon U-Pb data indicate that the granitic pebbles formed in the end-Permian (254-253 Ma). The youngest detrital zircon age of 231 Ma from the conglomerate, and presence of the overlying Upper Triassic Sihetun Formation suggests that the molasse deposits on the Jin'gui Island formed during the Late Triassic. The end-Permian granitic rocks display high SiO₂ (66.07-74.77 wt %), with low MgO (0.55-2.05 wt %) and Mg# (31.61-43.64) values, together with depleted Hf and Nd isotopic values ($\varepsilon_{Hf}(t) = +1.61$ to +11.62; $\varepsilon_{Nd}(t) = +3.3$ to +4.2; $({}^{87}Sr/{}^{86}Sr)i = 0.706458-0.706842)$ and juvenile second-stage Hf model ages (1148-512 Ma), suggesting that they were probably generated by the partial melting of a Meso-Neoproterozoic juvenile metabasaltic lower crust. They are characterized by enrichments in large ion lithophile elements (LILEs) and depletions in high field strength elements (HFSEs), with affinities to igneous rocks forming in a subduction-related setting. This, combined with regional coeval subduction-related magmatic rocks, indicates that the PAO still existed along the CYS. In addition, the identification of Late Triassic molasse deposits on the Jin'gui Island in this study, coupled with occurrences of many Early Triassic syn-collisional granitoids along the CYS, indicates that the final closure of the PAO took place prior to the Late Triassic.

Keywords: molasse deposit; zircon U-Pb-Hf isotopes; geochemistry; NE China; Paleo-Asian Ocean

1. Introduction

Northeast (NE) China, also called the Xing'an-Mongolian Orogenic Belt (XMOB), is located between the North China Craton (NCC) and Siberia Craton (SC) and is an important component of the Central Asian Orogenic Belt (CAOB; Figure 1a) [1–8]. It formed as a result of multiple subduction- and collision-related processes during the closure of the Paleo-Asian Ocean (PAO) and the amalgamation of micro-continental blocks with different origins and geological histories (Figure 1b) [4–15]. It is widely accepted that the Solonker-Xra Moron-Changchun-Yanji suture belt tectonically separates the NCC in the south from the micro-continental blocks in the north, and it marks the final closure of the Paleo-Asian Ocean [4–6,16–20]. Therefore, the timing of collision and formation of the Solonker-Xar Moron-Changchun-Yanji suture plays a significant role for us to understand the evolution of the PAO.



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). So far, although a large number of studies have investigated the sedimentology, paleobiogeography, magmatism, metamorphism, paleomagnetism, structural deformation, and tectonic evolution in the regions around the Changchun-Yanji suture (CYS) [21–53], some important aspects of the geology of this region, especially the precise time of the final closure of the PAO, still remain controversial. In one school of thought, the final closure occurred prior to the Permian, mainly including before the Late Devonian and between the Late Devonian [54–56] and Early Carboniferous [57–59]. In an alternative view, the closing of the PAO occurred during the Permian–Triassic, which can be reclassified as during the late Early Permian [60,61], during the Middle–Late Permian [62–65], during the Late Permian [4,34,48,66], during the Late Permian–Early Triassic [3,10,11,21,33,35,40,41,50,67–70], during the Late Permian–Middle Triassic [5,22,25,26,28,32,42,71,72], and during the Middle– Late Triassic [20,24,37,49,73]. Therefore, substantial controversy remains regarding the closure time of the PAO along the CYS.



Figure 1. (a) Tectonic outline of the Central Asian Orogenic Belt (CAOB) and surrounding regions (modified from [74]); (b) Tectonic sketch map of NE China (modified from [5]).

Molasse generally provides ample evidence, present a record of regional tectonic evolutions, and have been widely applied to constrain the collision timing in the study of orogenic belt [46,75–77]. In this contribution, we focus on the Yangjiagou Formation in central Jilin Province, since this stratum tectonically experienced the evolution of the CYS and has the nature of molasse and carbonate deposits [78,79]. Here, we present novel zircon U-Pb dating, zircon Lu-Hf isotopic data, and whole-rock geochemical and Sr-Nd isotopic data for conglomerate and granitic pebbles within, from the molasse deposits of the Yangjiagou Formation in order to provide constraints on the Triassic tectonic evolution of the CYS and the timing of the final closure of the PAO.

2. Geological Setting and Sample Descriptions

2.1. Geological Setting

The central Jilin Province in NE China is located in the western part of the CYS at the junction of the Songnen Block and the NCC (Figure 1b). This region is adjacent to the Dunhua–Mishan Fault and the Songliao Basin to the east and west, respectively (Figure 1b). The CYS is composed mainly of Phanerozoic granitoids with a series of subduction-related tectonic mélanges, including the Kaiyuan, Xia'ertai, Hulan, Seluohe, Qinglongcun, and Kaishantun tectonic mélanges [12,15,21–23,25–28,31,35,47]. In addition, minor Paleozoic and Mesozoic sediments are also distributed in the "granite ocean" [39,43–46,48–50].

The study area is to the southeast of Jilin City, in central Jilin Province (Figure 2). According to the 1:200,000 geological map, this area mainly contains the Lower Permian Daheshen, the Upper Permian Fanjiatun and Yangjiagou, and the Triassic Xiaomifengdingzi formations [80] (Figure 2). The intrusive rocks include Triassic–Jurassic granitoids, in which the Jurassic monzogranite and granodiorite are the major components [80]. The Yangjiagou Foramtion is dominated by gray to greenish slate, siltstone, sandstone, conglomerate and tuffaceous breccia. This formation has a thickness of 500–1200 m and shows the nature of molasse formation.



Figure 2. Geological map of the Songhua Lake area.

2.2. Sample Descriptions

The samples in this study were all collected from the Jin'gui Island in the Songhua Lake (Figure 2). Field observations suggest that pebble-bearing tectonic schist and conglomerate with complicated and high pebble content mainly crops out on the island. The rocks on this island were previously assigned to the Late Permian Yangjiagou Formation. The pebbles in the conglomerate are composed of granitic rocks, volcanic rocks (e.g., andesite), quartzite,

marble, and sandstone with poor sorting and roundness (Figure 3b–g). The lengths of pebbles generally range from 5 to 135 cm. Most pebbles are stretched and aligned in parallel to the fabric of the rock, which is indicative of plastic deformation and a strong strain effect on them.



Figure 3. Field photographs of the Jin'gui Island showing (**a**) the sample locations and (**b**–**g**) different types of pebbles in the conglomerate.

To constrain the maximum depositional age of the sedimentary rocks on the Jin'gui Island, we collected one conglomerate sample (JG-6) and three granitic pebble samples (> 60 cm; JG-1, JG-2, and JG-3) whose locations are shown in Figure 3a.

The pebbles in the conglomerate sample (JG-6) are dominated by sandstone (90%) with minor granitic rocks (8%) and marble (2%). The matrix is grayish-green in color, crumbly, and shows well-developed foliations. It is composed mainly by quartz, plagioclase, biotite, sericite, chlorite, and opaque minerals (Figure 4a–c). Some of the quartz and feldspar grains exhibit brittle fracturing. Rotated quartz, feldspar and biotite porphyroclasts also can be observed.

Among the three granitic pebble samples, Sample JG-1 is a biotite monzogranite that consists mainly of plagioclase (40 vol %), alkali feldspar (30 vol %), quartz (25 vol %), and biotite (5 vol %) (Figure 4d). Sample JG-2 and Sample JG-3 are granodiorites that consists mainly of plagioclase (45–55 vol %), quartz (20–30 vol %), alkali feldspar (5–10 vol %), and hornblende (5 vol %) (Figure 4e,f). The granitic rocks also contain accessory zircon and Ti-Fe oxides.



Figure 4. Photomicrographs of (**a**–**c**) matrix of the conglomerate and (**d**–**f**) granitic pebbles in the conglomerate. Mineral abbreviations according to [81]. plagioclase (Pl), quartz (Qz), biotite (Bt), hornblende (Hbl), orthoclase (Or).

3. Analytical Methods

The analytical techniques employed for zircon morphology, U–Pb geochronology, trace element compositions, in situ Lu–Hf isotopes, as well as whole-rock major and trace elements and Sr-Nd isotopes are presented in the Supplementary Materials.

4. Results

4.1. LA-ICP-MS Zircon Dating

Four samples including a conglomerate (JG-6) and three granitic pebbles (JG-1, JG-2, and JG-3) were selected for zircon U-Pb isotopic analyses. Representative cathodoluminescence (CL) images of analyzed zircons are shown in Figure 5. The analytical results are listed in Table S1. For statistical purposes, 207 Pb/ 206 Pb ages are used for grains >1000 Ma, whereas 206 Pb/ 238 U ages are used for grains <1000 Ma.

4.1.1. Conglomerate Sample JG-6

The majority of the zircons from this sample are euhedral to subhedral and exhibit a clear internal structure and fine-scale oscillatory growth zoning (Figure 5a), suggesting a low degree of reworking. However, a small number of zircons are rounded to subrounded and display blurry oscillatory or cloudy zoning (e.g., #81 and #183; Figure 5a), suggesting that they experienced multiple transportation events.

A total of 228 detrital zircon U-Pb ages were obtained, and all the ages are concordant (Figure 6a). The analyzed zircons range from 40 to 260 μ m in size with length/width ratios of 1:1 to 3:1, and they contain Th and U contents of 8 to 793 ppm and 19 to 1519 ppm, respectively (Table S1). Generally, the Th/U ratio of metamorphic zircon is <0.1 and magmatic zircon is >0.4 [82–86], which can be used to distinguish the origin of zircons. A total of 228 detrital zircons were analyzed, most of which have Th/U ratios >0.4, and there were 26 grains with ratios of 0.1–0.4 (predominately between 0.3 and 0.4; Table S1 and Figure 7a), indicating main magmatic origin. The concordant ages range between 2291 Ma and 231 Ma. Two grains generated 207 Pb/ 206 Pb ages of 2291 Ma and 1044 Ma, and one grain generated 206 Pb/ 238 U age of 802 Ma. Others have 206 Pb/ 238 U ages ranging from 529 to 231 Ma, and two primary peak ages are noted from the relative age probability diagram: (1) a remarkable peak value at 473 Ma in the range 529–426 Ma and (2) a remarkable peak value at 254 Ma in the range 334–231 Ma (Figure 6b).



Figure 5. Representative CL images of zircon from the (**a**) conglomerate and (**b**–**d**) granitic pebbles in the conglomerate. The solid-line and dash-line circles represent the locations for U-Pb dating and in situ Lu-Hf isotope analysis, respectively. The numbers show the ages of the zircons (Ma) and the $\varepsilon_{Hf}(t)$ values.



Figure 6. (a) U–Pb concordia and (b) probability diagrams of zircon ages for the conglomerate sample JG-6.



Figure 7. Th/U ratio vs. U-Pb ages diagram for zircons from the (**a**) conglomerate and (**b**) granitic pebbles in the conglomerate. The shaded area denotes values for typical metamorphic zircons with Th/U < 0.1.

4.1.2. Granitic Pebble Samples

Zircons from the three granitic pebble samples are generally ally euhedral–subhedral in shape and have the length ranging from 50 to 260 μ m, while the length-to-width ratios are from 1:1 to 2:1. CL imaging revealed that these zircons show clear fine-scale oscillatory growth zoning, showing the features of magmatic zircons (Figure 5b–d), which are further supported by their high Th/U ratios (0.34–2.06, average 0.92; Table S1 and Figure 7b).

Sample JG-1 (Biotite Monzogranite)

Forty-two analyses were made on 42 zircons from sample JG-1, and 39 concordant ages were obtained (Table S1). With the exception of three spots that yielded ages of 506 ± 10 Ma, 276 ± 7 Ma, and 270 ± 4 Ma, a total of 36 analyses yielded a weighted mean 206 Pb/ 238 U age of 254 ± 2 Ma (MSWD = 0.065, n = 36; Figure 8a), representing the emplacement age of the biotite monzogranite; the three older ages represent the ages of inherited or captured zircons entrained by the granitic magma.



Figure 8. U–Pb concordia diagrams of zircon ages for the granitic pebbles in the conglomerate. (**a**) sample JG-1; (**b**) sample JG-2; (**c**) sample JG-3.

Sample JG-2 (Granodiorite)

Thirty-six zircon grains from sample JG-2 were analyzed, and 33 of them gave ${}^{206}\text{Pb}/{}^{238}\text{U}$ ages ranging from 256 to 251 Ma, yielding a weighted mean ${}^{206}\text{Pb}/{}^{238}\text{U}$ age of 253 \pm 3 Ma (MSWD = 0.024, *n* = 33; Figure 8b), which is interpreted as the emplacement age of the granodiorite. The other three spots gave ${}^{206}\text{Pb}/{}^{238}\text{U}$ age of 351 \pm 5 Ma, 317 \pm 7 Ma and 282 \pm 10 Ma, representing the ages of inherited or captured zircons entrained by the granitic magma.

Sample JG-3 (Granodiorite)

Out of thirty analytical spots, 27 analyses from sample JG-3 are concordant and yielded 206 Pb/ 238 U ages from 255 to 250 Ma, which produces a weighted mean age of 253 \pm 2 Ma (mean squared weighted deviation (MSWD) = 0.057, *n* = 27; Figure 8c), representing the emplacement age of the granodiorite.

4.2. Zircon Hf Isotopic Compositions

In situ Lu-Hf isotopic compositions were measured in a total of 41 grains from the conglomerate sample JG-6 and 45 grains from the granitic pebble samples (JG-1, JG-2, and JG-3). The results are listed in Table S2 and are shown in Figure 9.

Forty-one detrital zircon Hf spot analyses from the conglomerate sample JG-6 were obtained, which include 38 Paleozoic–Early Mesozoic zircons that have initial ¹⁷⁶Hf/¹⁷⁷Hf ratios of 0.282608–0.282999, $\varepsilon_{\rm Hf}$ (t) values of +4.47 to +14.17, single-stage Hf model ages (T_{DM1}) of 891 to 350 Ma, and two-stage Hf model ages (T_{DM2}) of 1146 to 383 Ma. The remaining three Precambrian (2291, 1044, and 802 Ma) zircons yield relatively lower initial

 176 Hf/ 177 Hf ratios of 0.281270–0.282221, with negative $\epsilon_{\rm Hf}(t)$ values of -3.78 to -1.65. The $T_{\rm DM1}$ and $T_{\rm DM2}$ ages of these three zircons are 2701, 1702, and 1436 Ma and 2947, 2104, and 1807 Ma, respectively.



Figure 9. $\varepsilon_{Hf}(t)$ versus t diagram of the rocks from the Jin'gui Island. XMOB: Xing'an–Mongolia Orogenic Belt; YFTB: Yanshan Fold and Thrust Belt (Yang et al., 2006 [87]).

Forty-five Paleozoic magmatic zircons from the granitic pebble samples were dated for Hf isotopic compositions, with initial 176 Hf/ 177 Hf ratios of 0.282655–0.282954, $\varepsilon_{\rm Hf}(t)$ values of +1.61 to +11.62, $T_{\rm DM1}$ values of 829–421 Ma, and $T_{\rm DM2}$ values of 1148–512 Ma, respectively.

4.3. Major and Trace Elements

Five samples, listed in Table 1, were analyzed to know the geochemical features of the Late Permian granitic pebbles from the Jin'gui Island. All the data are plotted in Figures 10 and 11.



Figure 10. Major element diagrams for the granitic pebbles in the conglomerate. (**a**) K₂O versus SiO₂ diagram (after Peccerillo and Taylor, 1976 [88]); (**b**) TAS classification diagram (after Middlemost, 1994 [89]); (**c**) ANK versus ACNK diagram (after Maniar and Piccoli, 1989 [90]).

The granitic pebble samples from the Jin'gui Island have high SiO₂ (66.07–74.77 wt %) and Al₂O₃ (13.64–16.28 wt %) but display low FeO^T (1.67–2.09 wt %) and MgO (0.55–2.05 wt %) contents (Table 1). They show relatively lower K₂O (0.25–0.77 wt %) and higher Na₂O (6.03–7.04 wt %) contents (Table 1), therefore having low K₂O/Na₂O ratios (0.04–0.12 wt %) and showing low-K calc-alkaline characteristics (Figure 10a). In the total alkali-silica (TAS) diagram (Figure 10b), these samples exhibit subalkaline characteristics and plot in the granodiorite and granite fields. They have high A/CNK ratios (1.08–1.24) and are peraluminous (Figure 10c).



Figure 11. (a) Chondrite-normalized rare earth element (REE) patterns and (b) primitive mantle-normalized trace element patterns of the granitic pebble samples. Normalizing values are from [91].

Table 1. Chemical compositions of major elements (wt %) and trace elements ($\times 10^{-6}$) of the Late Permian granitic pebble samples from the Jin'gui Island.

Sample	JG-1(1)	JG-1(2)	JG-2(1)	JG-2(2)	JG-3	Sample	JG-1(1)	JG-1(2)	JG-2(1)	JG-2(2)	JG-3
SiO ₂	74.77	74.55	66.07	66.67	66.75	Zr	84.09	221.60	55.20	245.70	73.41
Al_2O_3	13.82	13.64	15.99	16.28	15.44	Nb	5.94	9.22	5.49	9.55	5.43
TiO ₂	0.37	0.32	0.78	0.59	0.74	Cs	0.24	0.96	0.63	1.19	0.70
FeO	0.85	0.89	2.37	1.43	1.18	Ba	85.27	688.05	238.40	593.47	247.30
Fe ₂ O ₃	0.98	2.14	2.26	4.36	3.68	La	14.19	25.20	7.38	24.80	13.42
CaO	0.54	0.85	1.04	1.35	1.12	Ce	26.46	50.19	15.82	46.32	28.00
MgO	0.55	0.73	1.79	2.05	1.95	Pr	3.58	5.98	2.08	6.36	4.02
K ₂ O	0.25	0.35	0.77	0.64	0.67	Nd	12.92	22.20	8.43	24.70	16.36
Na ₂ O	7.04	6.03	6.18	6.06	6.24	Sm	2.24	4.26	1.78	4.89	3.58
MnO	0.03	0.12	0.06	0.09	0.06	Eu	0.35	0.75	0.41	1.10	0.76
P_2O_5	0.06	0.08	0.15	0.19	0.16	Gd	2.11	3.81	1.75	4.40	3.63
LOI	0.55	1.26	1.80	2.02	1.59	Tb	0.37	0.62	0.35	0.71	0.72
Total	99.79	100.96	99.26	101.73	99.58	Dy	2.47	3.74	2.34	4.39	4.77
A/CNK	1.08	1.15	1.24	1.24	1.19	Ho	0.47	0.70	0.46	0.80	0.98
A/NK	1.17	1.32	1.45	1.53	1.40	Er	1.53	2.07	1.45	2.47	3.08
σ	1.67	1.29	2.09	1.90	2.01	Tm	0.26	0.33	0.23	0.39	0.45
FeO ^T	1.73	2.82	4.40	5.35	4.50	Yb	1.54	2.13	1.50	2.50	3.03
K ₂ O/Na ₂ O	0.04	0.06	0.12	0.11	0.11	Lu	0.24	0.32	0.22	0.40	0.45
Mg#	36.02	31.61	41.95	40.57	43.64	Hf	2.91	4.06	2.19	5.31	3.00
Be	0.77	1.26	1.00	1.82	1.23	Ta	0.58	0.89	0.45	0.74	0.49
Sc	3.89	5.93	6.86	12.72	8.98	Pb	3.46	28.00	5.21	24.10	6.17
V	24.68	24.20	86.40	67.70	82.60	Th	5.91	13.20	2.98	8.42	5.03
Cr	213.50	26.30	268.40	16.10	234.10	U	0.33	1.32	0.41	1.34	0.60
Co	2.81	5.55	5.20	10.07	6.24	δΕυ	0.48	0.56	0.70	0.71	0.64
Ni	6.79	13.70	8.10	11.50	4.53	(La/Yb) _N	9.08	12.53	8.82	14.71	17.84
Cu	14.88	36.20	8.91	20.10	9.69	(La/Sm) _N	14.63	27.84	11.60	31.96	23.41
Zn	11.69	30.40	26.04	54.20	36.60	(Gd/Yb) _N	10.28	18.54	8.50	21.41	17.68
Ga	6.83	13.50	8.24	18.20	10.25	∑REE	4.09	3.82	2.68	3.27	2.42
Rb	1.02	13.80	4.55	19.40	6.98	∑LREE	1.13	1.48	0.96	1.46	0.99
Sr	160.40	186.00	240.70	341.00	276.40	∑HREE	6.60	8.49	3.53	7.12	3.17
Y	12.31	19.00	11.11	21.90	27.05	$T_{Zr}(^{\circ}C)$	737	820	705	830	727

Note: A/CNK = mole [Al₂O₃/(Cao + Na₂O + K₂O)]; A/NK=mole [Al₂O₃/(Na₂O + K₂O)]; δ Eu = 2 × Eu_N/(Gd_N + Sm_N); LREE = La + Ce + Pr + Nd + Sm + Eu; HREE = Gd + Tb + Dy + Ho + Er + Tm + Yb + Lu; Σ REE = Σ LREE + Σ HREE.

With regard to trace elements, the granitic pebble samples have uniform rare earth element (REE) patterns in the chondrite-normalized diagram (Figure 11a) and are characterized by light rare earth element (LREE) enrichment ($(La/Yb)_N = 3.17-8.49$) and flat

heavy rare earth element (HREE) patterns ((Gd/Yb)_N = 0.96-1.48) with negative Eu anomalies (δ Eu = 0.48-0.71). In the primitive mantle-normalized trace element spider diagrams (Figure 11b), these samples show Nb, Ta, and Ti negative anomalies, and display positive Ba, Th, and Pb spikes, thereby indicating typical characteristics of arc-related magmatic rocks [92–97].

4.4. Sr-Nd Isotopic Analyses

The results of the Sr-Nd isotopic analyses of the granitic pebbles from the Jin'gui Island are listed in Table 2. The initial ⁸⁷Sr/⁸⁶Sr and $\varepsilon_{Nd}(t)$ values were calculated based on their zircon U-Pb ages. For the Late Permian granitic rocks, (⁸⁷Sr/⁸⁶Sr)i = 0.706458–0.706842 and $\varepsilon_{Nd}(t) = +3.3$ to +4.2, and the two-stage Nd model ages (T_{DM2}) range from 758 to 685 Ma. These values are consistent with those of the Phanerozoic felsic rocks in the CAOB [25,39].

Table 2. Sr–Nd isotopic data for the Late Permian granitic pebble samples from the Jin/gui Island.

Sample	t(Ma)	⁸⁷ Rb/ ⁸⁶ Sr	⁸⁷ Sr/ ⁸⁶ Sr	2σ	(⁸⁷ Sr/ ⁸⁵ Sr) _i	¹⁴⁷ Sm/ ¹⁴⁴ Nd	¹⁴³ Nd/ ¹⁴⁴ Nd	$\pm 2\sigma$	$\varepsilon_{\rm Nd}(t)$	T _{DM2} (Ma)
JG-1(1)	254	0.214835	0.707270	0.000009	0.706494	0.116010	0.512671	0.000006	3.3	758
JG-2(1)	253	0.164735	0.707051	0.000009	0.706458	0.119688	0.512724	0.000006	4.2	685
JG-3	253	0.073123	0.707105	0.000010	0.706842	0.132294	0.512736	0.000005	4.0	699

5. Discussion

5.1. Age Interpretations

The sedimentary rocks on the Jin'gui Island were previously assigned to the Yangjiagou Formation, the depositional period of which has long been a controversial topic, since neither the top nor the bottom surface of the strata has been found [80]. Recent LA-ICP-MS dating documented maximum depositional ages of 262, 264, 258, 254, and 245 Ma for the sandstones from the Yangjiagou Formation in the same/adjacent areas [48–50]. However, the formation age of the conglomerate in the Yangjiagou Formation has not been well constrained.

In the present study, zircons in the granitic pebble samples from the conglomerate display typical oscillatory growth zoning, with high Th/U ratios (0.34–2.06, average 0.92), indicating a magmatic origin. Therefore, we conclude that the LA-ICP-MS U-Pb ages for these zircons represent the emplacement age of the granites. The weighted mean 206 Pb/ 238 U ages for three granitic pebbles are 254 ± 2 Ma, 253 ± 3 Ma, and 253 ± 2 Ma, indicating that the granitic rocks formed in the end-Permian. Consequently, the formation timing of the conglomerate on the Jin'gui Island should be not earlier than the end-Permian. Furthermore, a new youngest, concordant 206 Pb/ 238 U age obtained from sample JG-6 is 231 Ma, which suggests that the age of the conglomerate on the Jin'gui Island is Late Triassic or younger, because a sedimentary unit can be no older than the youngest detrital zircon(s) within that unit [98]. This, combined with presence of the overlying Upper Triassic Sihetun Formation, suggests that the conglomerate on the Jin'gui Island formed during the Late Triassic.

5.2. Petrogenetic Implications for the Granitic Pebble Samples

The granitic pebble samples in this study are peraluminous, with A/CNK between 1.08 and 1.24. They have relatively high content of Na₂O (6.03–7.04 wt %) but low initial ⁸⁷Sr/⁸⁶Sr ratios (0.706458–0.706842) and positive ε_{Nd} (t) ratios (3.3–4.2) (Tables 1 and 2), which suggest that these rocks may be I-type and/or A-type, but not S-type granites according to the criteria of Chappell (1999) [99] and Chappell and White (2001) [100–102]. Additionally, these rocks are characterized by low contents of Nb (5.43–9.55 ppm), Ta (0.45–0.89 ppm), Y (11.11–27.05 ppm), and Zn (11.69–54.20 ppm) alongside low 10,000*Ga/Al (0.93–2.11) and FeO^T/MgO (2.30–3.86) values (Table 1), similar to the widely distributed I-type granites in NE China [21,22,25,28–30] but distinct from those A-type granites [103].

This result is also supported by the absence of mafic alkaline minerals such as arfvedsonite and riebeckite.

Zircon saturation temperatures (T_{Zr}) calculated from whole-rock compositions can be used to estimate peak melting temperature experienced by magmatic rocks under the prerequisite that zirconium is saturated in the melt [104–107]. In this study, the presence of inherited zircons in the granitic pebble samples indicates that zirconium was saturated in the parent magma and T_{Zr} is thus applicable. The calculated T_{Zr} for the granitic pebble samples are in the range of 705–830. These values are distinctly lower than those of Atype granites (calculated average T_{Zr} values was 839 °C) [104,105] but consistent with those of I-type granites (calculated average T_{Zr} values for fractionated I-type granites and unfractionated I-type granites were 764 and 781 °C, respectively) [106,107], which further suggests that these granitic pebble samples are I-type granites.

The high SiO₂ concentrations and significant enrichment in large ion lithophile elements (LILEs) and depletion in high field strength elements (HFSEs) in the primitive mantle-normalized multi-element diagram (Figure 11) imply that the primary magma of the above I-type granitic rocks was derived from the partial melting of crustal material [21,22,25,28–30]. The higher SiO₂ but lower MgO and Mg# of these rocks are similar to those of the experimentally derived partial melts from metabasaltic rocks [108]. This, combined with the negative Nb-Ta-Ti anomalies suggests residues of garnet and hornblende in the source [109]. In addition, the granitic samples display low initial ⁸⁷Sr/⁸⁶Sr ratios (0.706458–0.706842), positive $\varepsilon_{Nd}(t)$ (3.3–4.2) and zircon $\varepsilon_{Hf}(t)$ (1.61–11.62) values, and young second-stage Hf model ages from 1148 to 512 Ma, which are consistent with the isotopic features of widely distributed Permian felsic igneous rocks along the CYS and indicate crustal growth during the Meso–Neoproterozoic [21,22,25,28–30]. Therefore, the primary magma of the I-type granitic pebble samples from the Jin'gui Island likely originated from the partial melting of a Meso-Neoproterozoic juvenile metabasaltic lower crust, with garnet and hornblende as the major residue mineral phases in the source region.

5.3. Provenance Analysis of the Conglomerate on the Jin'gui Island

It is widely accepted that age dating and Hf compositions of detrital zircon is a proven and effective method in determining the provenance of clastic sedimentary rocks [42–50,110–112]. Apart from three zircon grains that yield Paleoproterozoic (2291 Ma), Mesoproterozoic (1044 Ma), and Neoproterozoic (802 Ma) ages, the remaining 225 detrital zircons from the conglomerate analyzed in this study yield two populations: 529–426 Ma (Early Paleozoic) with a peak at 473 Ma and 334–231 Ma (Late Paleozoic–Early Mesozoic) with a peak at 254 Ma (Figure 6b). Therefore, the material deposited within the Late Triassic conglomerate on the Jin'gui Island was predominantly derived from a Paleozoic–Early Mesozoic source, with minor contributions from Paleoproterozoic to Neoproterozoic sources.

The Paleoproterozoic (2291 Ma) zircon has a negative $\varepsilon_{Hf}(t)$ value of -1.65, which is similar to Precambrian detrital and magmatic zircons from the NCC [16–19,42–50]. This suggests that the Paleoproterozoic zircon was sourced from Precambrian basement rocks of the NCC. In addition, this zircon is rounded in shape, indicating that it underwent long distance transportation prior to deposition.

The NCC experienced the Columbia supercontinental final break at approximately 1.35–1.32Ga, leading to absence of the Grenvillian (\approx 1200–800 Ma) magmatic events in the NCC, especially in the northern margin [113,114]. Therefore, it is impossible that the source region of the Mesoproterozoic and Neoproterozoic zircons was the NCC. In contrast, the two zircons have negative $\varepsilon_{Hf}(t)$ values and are similar to those Mesoproterozoic–Neoproterozoic isotopic ages identified in the Xing'an-Mongolian Orogenic Belt (XMOB) [115,116], suggesting that they were derived from microcontinents to the north [44].

The Early Paleozoic and Late Paleozoic–Early Mesozoic zircons display the characteristics of magmatic zircons, indicating intense magmatic activity during these periods. As a result of initial and ongoing subduction of the Paleo-Asian oceanic plate, an Early Paleozoic intra-oceanic arc abutting the northern margin of the NCC was proposed [117]. This view is supported by some Early Paleozoic mid-ocean ridge basalt (MORB)-like and arc-like magmatic rocks identified in the study area and adjacent regions, including the Zhangjiatun meta-diabase dyke (493 Ma), hornblende gabbro (486 Ma), quartz diorite (476 Ma), and tonalite (443 and 440 Ma) [117,118]; the Xiaosuihe serpentinite (494 Ma) and pyroxene andesite (467 Ma) [118]; the Toudaogou metamorphic intermediate-mafic and ultramafic rocks (474, 468, 466, and 465 Ma) [13]; and the Fangniugou rhyodacite (425 Ma) [12]. In addition, the Early Paleozoic zircons in the conglomerate have positive $\varepsilon_{Hf}(t)$ values (Figure 9a), similar to those in the above magmatic rocks but distinct from the Phanerozoic zircons from the NCC that generally have negative $\varepsilon_{Hf}(t)$ values [87]. Moreover, these zircons are subhedral–euhedral in shape, indicating that they underwent minimal transportation prior to deposition. Thus, zircons with ages of 529–426 Ma were most likely derived from the Early Paleozoic arc at the northern margin of the NCC.

The Late Paleozoic–Early Mesozoic zircons also display the characteristics of magmatic zircons and are subhedral–euhedral in shape, indicating that they were locally derived from magmatic rocks. The 16 zircons with ages of 334-300 Ma closely match the ages recorded in Permian–Triassic strata (e.g., the Shoushangou, Fanjiatun, Yangjiagou, Huangyingtun, Dongnancha, Xiaohekou and Dajianggang formations) in central Jilin Province [43,44,46–50]. The 87 Permian–Triassic (298–231 Ma) zircons are commonly recorded from the widely distributed magmatic rocks in the study area and adjacent regions, including the Daheshen volcanic rocks (299 Ma) [39]; the Fangniugou monzogranite (270 Ma) [21]; the Xiaolihe monzogranite (260 Ma) [22]; the Youyi syenogranite (259 Ma) [21]; the Shuangfengshan olivine gabbro (258 Ma) [21]; the Fangshendingzi monzogabbro (257 Ma) [21]; the Doushantouzi syenogranite (257 Ma) [25]; the Sanmenmojia granodiorite (255 Ma) [21]; the Daheishan volcanic rocks (255–253 Ma) [25]; the Shanhe monzonite (252 Ma) [22]; the Tuding hornblende gabbro (252 Ma) [22]; the Jianpingzhen monzogranite (249 Ma) [21]; the Nantiancun granodiorite (235 Ma) [20]. In addition, all the Late Paleozoic–Early Mesozoic zircons have positive $\varepsilon_{Hf}(t)$ values (Figure 9a), which are similar to the compositions of zircons in the above sedimentary and magmatic rocks. Therefore, zircons with ages of 334–231 Ma were locally derived from the Late Paleozoic-Early Mesozoic sedimentary and magmatic rocks in the study area and adjacent regions.

In summary, the provenance of the conglomerate on Jin'gui Island was mainly the Early Paleozoic magmatic arc and Late Paleozoic–Early Mesozoic sedimentary and magmatic rocks in the study area and adjacent regions, and to a lesser degree the interior of the NCC and the XMOB.

5.4. Implications for the Tectonic Evolution of the PAO

As stated above, the timing of the final closure of the eastern segment of the PAO has been controversial, and various dominant views have indicated that the final closure of the PAO occurred during the late Early Permian [60,61], the Middle–Late Permian [62–65], the Late Permian [4,34,48,66], the Late Permian–Early Triassic [3,10,11,21,33,35,40,41,50,67–70], the Late Permian–Middle Triassic [5,22,25,26,28,32,42,71,72], and the Middle–Late Triassic [20,24,37,49,73]. The determination of the age of the molasse deposits on the Jin'gui Island provides new evidence for in-depth understanding of the Late Permian–Late Triassic tectonic evolution of the PAO.

The petrological and geochemical data show that the Late Permian granitic pebbles were formed by partial melting of the lower crustal-derived juvenile component. They display depletion in HFSEs and LILEs enrichment, and they also have low initial ⁸⁷Sr/⁸⁶Sr ratios and positive $\varepsilon_{Nd}(t)$ values, which are akin to representative island arc-like magmas [119]. In addition, all the granitic samples plot within the field of volcanic arc granite within the Ta vs. Yb and Rb vs. Y + Nb discrimination diagrams (Figure 12a,b), suggesting that they were generated in an arc-related tectonic setting. Moreover, all the samples plot within or close to the typical arc-rock field in the Sr/Y vs. Y and (La/Yb)_N vs. Yb diagrams (Figure 12c,d), which are further indicative of arc-type magmatism in association with subduction process. Therefore, a subduction-related tectonic setting linking with the sub-

duction of the Paleo-Asian oceanic plate likely created the above-mentioned end-Permian granitic rocks, as well as some previously reported contemporaneous magmtic rocks, which exhibit arc-related geochemical signatures along the CYS, such as the Doushan-touzi syenogranites (256 Ma), the Daheishan volcanic rocks (255–253 Ma), the Kaiyuan metamorphosed basaltic volcanic rocks (258 Ma and 254 Ma), and the Kaishantun basalt (252 Ma) [17,25,26,31]. In addition, coeval high-Mg andesites (252 Ma and 250 Ma) that formed in a subduction-related setting also distributed along the CYS [23,34]. Thus, we conclude that the eastern segment of the PAO was undergoing subduction in the end-Permian, implying that the PAO still existed along the southeastern margin of the XMOB in the end-Permian.



Figure 12. (a) Ta versus Yb diagram (after Pearce et al., 1984 [120]) (b) Rb versus Y+Nb diagram (after Whalen et al., 1987 [103]), (c) Sr/Y versus Y diagram (after Defant and Drummond, 1990 [121]), and (d) (La/Yb)_N versus Yb_N diagram (after Martin et al., 2005 [122]) for the granitic pebble samples. Abbreviations: syn-COLG, syn-collision granite; WPG, within plate granite; ORG, ocean ridge granite; VAG, volcanic arc granite.

The occurrences of many Early Triassic syn-collisional granitoids (such as the Jianpingzhen, Liushugou, Dayushan, and Fudongzhen plutons) along the CYS record the collisional events between the NCC and combined NE China blocks [20–22,24,27,28]. These granitoids, which display high SiO₂, low Mg#, low Cr and Ni, and notably high La/Yb and Sr/Y, were proposed to have originated from thickened lower crust that resulted from collision [20–22,24,27,28]. Some recently reported Early Triassic ages from igneous rocks or metamorphic complexes (e.g., the Daheishan volcanic rocks and the Kaiyuan and Hulan complexes) in the adjacent areas probably correspond to the above collisional events [10,25,26,31]. Importantly, molasse sequences provide reliable evidence for the end of an orogeny [5,46]. The identification of Late Triassic molasse deposits on the Jin'gui Island in this study, together with previously identified contemporaneous molasse sequences in the adjacent regions along the CYS (e.g., the Xiaohekou, Xiaoyngzi, and Dajianggang formations), indicates that final closure of the PAO took place prior to the Late Triassic [46]. Considering the above, we suggest that final suturing of the eastern segment of the PAO took place during the Early–Middle Triassic in central Jilin Province.

6. Conclusions

Our new geochemical and geochronological data for the granitic pebbles and conglomerate from molasse deposits of the Jin'gui Island in NE China led us to the following conclusions.

- (1) The zircon U-Pb data indicate that the granitic pebbles included in the conglomerate formed in the end-Permian (254–253 Ma). The youngest detrital zircon age of 231 Ma from the conglomerate on the Jin'gui Island, together with the overlying Upper Triassic Sihetun Formation, indicates that the molasse deposits formed during the Late Triassic.
- (2) The primary magma of the end-Permian granitic pebble samples was derived from the partial melting of a Meso-Neoproterozoic juvenile metabasaltic lower crust.
- (3) The provenance of the conglomerate on the Jin'gui Island was mainly the Early Paleozoic magmatic arc and Late Paleozoic–Early Mesozoic sedimentary and magmatic rocks in the study area and adjacent regions, and to a lesser degree the interior of the NCC and the XMOB.
- (4) The end-Permian granitic pebbles on the Jin'gui Island represent the final stages of arc magmatism related to subduction of the Paleo-Asian oceanic plate. These rocks, together with the identification of Late Triassic molasse deposits along the CYS, suggest that the final closure of the PAO in central Jilin Province occurred during the Early–Middle Triassic.

Supplementary Materials: The following are available online at https://www.mdpi.com/2075-163 X/11/2/223/s1, Table S1: LA-ICP-MS zircon U-Pb data for the samples from the Jin'gui Island, Table S2: Lu-Hf isotopic data of zircons from the samples from the Jin'gui Island. References [25,123–128] are cited in the supplementary materials.

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