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Recycling of continental crust materials: Geochemical constraints from post-collisional alkaline intrusive rocks in the Dabie orogen

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Post-collisional alkaline intrusive rocks from the Dabie orogen were studied for their whole-rock major-trace elements and Sr-Nd-Hf-Pb isotopes, as well as zircon U-Pb ages and Hf-O isotopes. The results provide geochemical constraints on the nature of their mantle sources and thus insight into crust-mantle interaction in the continental collision zone. The alkaline intrusive rocks are composed of syenite and nepheline syenite. Syn-magmatic zircon U-Pb dating by LA-ICP-MS for them yielded Early Cretaceous ages of 131.3 \pm 1.4 Ma to 122.6 \pm 0.6 Ma, coeval with the post-collisional magmatism in the Dabie orogen. One relict zircon with U-Pb age of 211 Ma is consistent with the timing of metamorphism for the ultrahigh-pressure (UHP) metamorphic rocks in this orogen. They have arc-like trace element distribution patterns, such as enrichment in LILE (large ion lithophile element) and LREE (light rare earth element) but depletion in HFSE (high field strength element), and enriched whole-rock Sr-Nd-Hf isotope compositions with high $({}^{87}\text{Sr}/{}^{86}\text{Sr})_i$ ratios of 0.7077–0.7131 but negative $\epsilon_{Nd}(t)$ values of –16.0 to –9.4 and $\epsilon_{Hf}(t)$ values of -17.5 to -12.7. Their syn-magmatic zircons have three groups of Hf-O isotope compositions comparable to those of UHP metamorphic rocks in Central-South Dabie and North Dabie, which represent the upper and middle continental crust of the subducted South China Block, respectively. In this regard, slab-mantle interaction is evident during the Triassic continental collision. We suggest that the melts derived from the subducted South China Block reacted with the lithospheric mantle wedge of the North China Block, resulting in phlogopitebearing metasomatites, whose partial melting would generate the post-collisional alkaline intrusive rocks during the Early Cretaceous.

KEYWORDS

alkaline intrusive rocks, subducted continental crust, crust-mantle interaction, Dabie orogen, post-collisional

1 Introduction

Post-collisional igneous rocks are widespread within continental collision orogens, which are frequently associated with an extensional tectonic stage after collision event (Zheng et al., 2020 and references therein). Silicic alkaline igneous rocks, though accounting for a small volume among post-collisional igneous rocks, have received considerable attention about their petrogenesis due to their high alkalis, incompatible elements and volatile species (e.g., Sørensen, 1974; Marks and Markl, 2017). A number of models have been proposed, including the mantle derivation model, the fractional crystallization model, the crust derivation model, and the mixing model. The mantle derivation model suggests that they are produced by low degrees of partial melting of metasomatized mantle (Halama, 2004; Berger et al., 2014; Hou et al., 2015), which is supported by the presence of mantlederived xenoliths and silica- and alkali-rich melt inclusions in their minerals, and the lack of evidence for low-pressure differentiation (Schiano and Clocchiatti, 1994; Schiano et al., 1998; Grant et al., 2013). The fractional crystallization model suggests that their high SiO₂ parts are produced by fractional crystallization of primitive alkaline magma with or without crustal assimilation based on the coexistence of ultramafic to mafic rocks (Yang J.-H. et al., 2012; Berger et al., 2014; Hou et al., 2015; Zhu et al., 2020). For the crustal derivation model, it is suggested that they are formed by remelting of mafic alkaline rocks or other crustal rocks (Hay and Wendlandt, 1995; Kaszuba and Wendlandt, 2000; Legendre et al., 2005; Dai et al., 2017), which is also supported by the experimental phonolitic melt from the carbonated pelites at 2.5-5 GPa (Thomsen and Schmidt, 2008). The mixing model, on the other hand, suggests that they are produced by the mixing of crustal anatectic granitic and mantlederived mafic magma followed by crystal differentiation (Barker et al., 1995; Litvinovsky et al., 2002). In this regard, it is still challenging to determine the origin of evolved alkaline rocks in the presence or absence of associated ultramafic to mafic rocks.

The Dabie-Sulu orogenic belt was formed by the collision between the South China Block and North China Block during the Triassic (Li et al., 1993; 1999; Zheng, 2008). Except for widespread high-pressure (HP) and ultrahigh-pressure (UHP) metamorphic rocks, voluminous post-collisional igneous rocks of Late Jurassic to Early Cretaceous are exposed in this orogenic belt (Huang et al., 2007; He et al., 2011; Zhao et al., 2013; Zhao et al., 2017). Different from the Sulu orogen with both Late Jurassic and Early Cretaceous post-collisional igneous rocks, the Dabie orogen contains only Early Cretaceous post-collisional igneous rocks (Zhao and Zheng, 2009; Zhao et al., 2017). A large portion of these igneous rocks are felsic intrusive rocks and a small portion are mafic intrusive rocks, with volcanic rocks only sporadically found in the North Dabie and Beihuaiyang zones (Dai et al., 2017; Zhao et al., 2017). The Beihuaiyang zone contains various types of Early Cretaceous igneous rocks, such as granite, syenite, phonolite, trachyte, andesite and dacite. The Beihuaiyang alkaline intrusive rocks, though outcrop in a small area, provide good samples for understanding the cycling of subducted continental crust and tectonic evolution of this orogen. Nevertheless, it is still controversial with respect to the nature of their magma source. Zhou et al. (1995) suggested that the ancient continental crust has played a major role in the genesis of these rocks, whose whole-rock Sr-Nd isotope compositions are similar to the North China Block. In contrast, it has also been suggested that these alkaline intrusive rocks were derived from a lithospheric mantle metasomatized by the subducted continental crust materials (Yang et al., 2002; Fan et al., 2004; Xu et al., 2008; Zhou et al., 2014).

This paper presents a combined study of whole-rock major-trace elements and Sr–Nd–Pb isotopes as well as zircon U–Pb ages and Hf-O isotopes for the post-collisional alkaline intrusive rocks in the Beihuaiyang zone of the Dabie orogen. The studied rocks exhibit whole-rock Sr-Nd-Pb isotope compositions and zircon *in situ* Hf-O isotope compositions comparable to the upper and middle continental crust of the subducted South China Block, indicating their origination from the orogenic lithospheric mantle metasomatized by melts from the subducted continental crust. Therefore, our results provide insights into the nature of magma source and crust-mantle interaction for the post-collisional alkaline intrusive rocks in collisional orogens.

2 Geological setting and samples

The Dabie-Sulu orogenic belt (Figure 1A), located in east-central China, is a typical continental collision orogen formed by the collision between the South China Block and the North China Block in the Triassic (e.g., Cong, 1996; Faure et al., 2003; Zheng et al., 2003). Influenced by the Tanlu fault zone (a left-lateral strikeslip fault), the Sulu orogen has been displaced northward by about 500 km in relation to the Dabie orogen (Figure 1A; Okay, 1993; Zhu et al., 2005). The presence of ultrahigh-pressure (UHP) metamorphic minerals such as coesite (Okay et al., 1989; Wang et al., 1989; 1992) and diamond (Xu et al., 1992; 2003; 2005; Xu et al., 1998; Liu et al., 2007) in the metamorphic rocks of this orogenic belt indicate that crustal rocks have subducted to mantle depths of >100 km and subsequently exhumated to the crustal level. The UHP metamorphic rocks in the Dabie orogen are dominated by orthogneiss with subordinate eclogite, granulite, amphibolite, migmatite and marble (Zheng et al., 2003). Previous studies on zircon U-Pb geochronology and mineral O isotopes have shown that the subducted continental crust is primarily composed of the Precambrian basement and its overlying sediment of the South China Block (Zheng et al., 2005; 2006). The Dabie UHP metamorphic belt can be divided into five zones based on their different metamorphic P-T conditions. From north to south, they are the Beihuaiyang low-T/low-P greenschist-facies zone (BHY), the North Dabie high-T/UHP granulite-facies zone (NDB), the Central Dabie middle-T/UHP eclogite-facies zone (CDB), the South Dabie low-T/UHP eclogite-facies zone (SDB), and the Susong low-T/HP blueschist-facies zone (SSZ) (Figure 1B; Xu et al., 2005; Zheng et al., 2005). All of these units were intruded by the post-collisional igneous rocks composed mainly of felsic granitoids and minor mafic-ultramafic rocks (e.g., Zhao and Zheng, 2009). Geochronological studies show that post-collisional magmatism occurred in the Early Cretaceous (Zhao et al., 2017; Yan et al., 2021; and references therein).

The Beihuaiyang zone, located between the Xiaotian-Mozitan Fault and the Lu'an-Hefei Fault (Figure 1B), is composed of Late Neoproterozoic to Early Paleozoic flysch sediments of the Foziling Group and the Neoproterozoic Luzhenguan Complex, which have



(A) Geological sketch map of the Dabie-Sulu orogenic belt and the study area in the Dabie orogen; (B) Distribution of post-collisional igneous rocks in the Beihuaiyang and North Dabie zones (modified after Dai et al., 2017); (C) Geological map of the alkaline complex in the Beihuaiyang zone (modified after Zhou et al., 2014; Dai et al., 2017). Abbreviations: BHY= Beihuaiyang low-T/low-P greenschist-facies zone, NDB = North Dabie high-T/UHP granulite-facies zone, CDB = Central Dabie Mid-T/UHP eclogite-facies zone, SDB = South Dabie low-T/UHP eclogite-facies zone, SSZ = Susong low-T/HP blueschist-facies zone

experienced greenschist-facies low grade metamorphism (Zheng et al., 2005; 2007). A series of alkaline igneous rocks were intruded in the Foziling Group schist, constituting an alkaline complex at Xianghongdian in the Beihuaiyang zone (Figure 1C). The alkaline volcanic rocks consist of phonolite and trachyte, while the alkaline intrusive rocks are syenite and nepheline syenite. The alkaline volcanic rocks in the Dabie orogen were suggested to be derived from partial melting of the subducted lower continental crust of the South China Block based on whole-rock Sr-Nd-Hf and zircon in situ O isotope compositions (Dai et al., 2017). However, the petrogenesis of the alkaline intrusive rocks is still controversial.

The alkaline intrusive rocks in this study, including syenite and nepheline syenite, were collected from the Beihuaiyang zone in the Dabie orogen (Figure 1C). Syenite is mainly composed of alkaline feldspar (~55%), quartz (~15%), biotite (~20%), hornblende (~10%) and a small amount of accessory minerals such as epidote, titanite and zircon (Figures 2A, D). Alkaline feldspar is granular and subhedral (0.5 mm-2 mm in length) with dirt-brown color (Figure 2A). Hornblende, biotite and aegirine with a yellowishbrown color are the dominant mafic minerals. Hornblende (0.5 mm-2 mm in length) and biotite (0.5 mm-1.5 mm in length) generally show subhedral plate-like shape. In contrast,



Photomicrographs (under crossed polarized light) for the Beihuaiyang alkaline intrusive rocks in the Dabie orogen. Mineral abbreviations are after Whitney and Evans (2010).

aegirine has small sizes (0.2 mm–0.5 mm) and its edge is partially altered into chlorite. Nepheline syenite (Figures 2B, C) is mainly composed of alkaline feldspar and nepheline with variable degrees of alteration (Figure 2C). Alkaline feldspar (0.2 mm–0.5 mm in diameter) is subhedral plate in shape (Figure 2D), some of which are altered into clay minerals (Figure 2C). Nepheline (1 mm–3 mm in diameter) displays first-order gray interference color and has cracks, along which minerals such as calcite and albite are filled.

3 Analytical methods

3.1 Whole-rock major and trace elements

The alkaline intrusive rocks analyzed in this study are fresh with no apparent alteration. These samples were crushed to powders of 200 mesh before analysis. Whole-rock major element analysis was carried out at the Chinese Academy of Sciences (CAS) Key Laboratory of Crust-Mantle Materials and Environments at the University of Science and Technology of China (USTC), Hefei, China. A ceramic crucible with 0.5 g dried sample powder was heated in a muffler at 1050°C for 8 hours. After cooling, the loss on ignition (LOI) was calculated by the weight difference before and after heating. A mixture with 0.8 g powder and 8 g Li₂Bi₄O₇ was fused in an auto fluxer at 1050°C–1100°C, the yielded flat molten glass disk was used to take major element analysis by an X-ray fluorescence spectrometer. The analytical precision of this procedure is better than $\pm 1\%$ –2%. Whole-rock trace element analysis was carried out at ALS Chemx Co. Ltd. (Guangzhou, China). To determine the trace element contents, 0.2 g whole-rock powder was mixed with 0.9 g lithium metaborate flux and then melted in a furnace at 1000°C. After cooling, the resulting glass was dissolved in 100 mL of 4% nitric acid, and the yielding solution was then analyzed by ICP-MS. The analytical precision is better than \pm 5% for trace elements.

3.2 Whole-rock Sr–Nd–Hf-Pb isotope analyses

Whole-rock Sr, Nd, Hf and Pb isotope ratios were determined using a Thermo Scientific Neptune multi-collector (MC)-ICP-MS at the CAS Key Laboratory of Crust-Mantle Materials and Environments at USTC, Hefei, China. Chemical separation prior to analysis was undertaken using conventional ion-exchange approaches, with details of the protocol can be found elsewhere (Yang et al., 2010; 2011; Yang et al., 2012 Y.-H.; Li et al., 2016a; Chu et al., 2019; Ma et al., 2022). About 100-150 mg fine sample powder was dissolved in a mixture of 2.5 mL concentrated HF, 0.2 mL HNO3 and HClO4 in a steel-jacketed Teflon bomb and then placed in an oven at 190°C for 1 week. After complete dissolution, each sample was dried at high temperature (fuming HClO₄) on a hot plate, followed by treatment with 14 M HNO₃, evaporation overnight, and subsequent taking up in 3 M HNO₃ + 3% m/v H₃BO₃. After resealing the capsule, it was heated on a hot plate at 100°C overnight for chemical purification.



Representative zircon CL images and concordia diagrams of LA-ICP-MS U–Pb isotopic data for the Beihuaiyang alkaline intrusive rocks in the Dabie orogen. The inside smallest circles denote the analytical spots for O isotopes; the middle and outside circles denote the analytical spots for U–Pb ages and Hf isotopes. The numbers below zircon grains denote U-Pb ages, $\epsilon_{Hf}(t)$ and δ^{18} O values in order.

Using Eichrom DGA resin (50–100 μ m, 2 mL), Sr, Nd, and Pb were first separated from the matrix. After eluting and collecting the major elements fraction with 3 M HNO₃+3% m/v H₃BO₃, Sr and Pb fractions were collected for further purification. The column was then rinsed with 12 M HNO₃ to remove any residual Ca effectively before the Hf fraction was collected, followed by separation of the Hf fraction using 3.5 M HNO₃ and 0.2 M HF. A 2 M HCl was finally used to elute the Nd fraction. Further purification of the Sr and Pb fraction was performed with a Sr-specific resin (100–150 μ m, 0.2 mL) prior to mass spectrometer analysis (Yang Y.-H. et al., 2012; Li et al., 2016a; Ma et al., 2022).

An analysis of Sr, Nd, Hf and Pb isotope ratios was conducted at the CAS Key Laboratory of Crust-Mantle Materials and Environments in USTC, Hefei, China, using a Neptune Plus MC- ICP-MS. Whole procedural blanks were less than 100 pg for Sr, 50 pg for Nd and 50 pg for Pb. The ${}^{87}\text{Sr}/{}^{86}\text{Sr}$, ${}^{143}\text{Nd}/{}^{144}\text{Nd}$ and ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ ratios were normalized to ${}^{86}\text{Sr}/{}^{88}\text{Sr} = 0.1194$, ${}^{146}\text{Nd}/{}^{144}\text{Nd} = 0.7219$ and ${}^{179}\text{Hf}/{}^{177}\text{Hf} = 0.7325$, respectively, using the exponential law. During the period of data acquisition, standard analyses yielded results of ${}^{87}\text{Sr}/{}^{86}\text{Sr} = 0.710266 \pm 12$ (2σ , n=12) for NBS987, ${}^{143}\text{Nd}/{}^{144}\text{Nd} = 0.512097 \pm 6$ (2σ , n=12) for JNdi, ${}^{176}\text{Hf}/{}^{177}\text{Hf} = 0.282185 \pm 6$ (2σ , n=12) for Alfa Hf, and ${}^{206}\text{Pb}/{}^{204}\text{Pb} = 16.9397 \pm 8$ (2σ , n=8), ${}^{207}\text{Pb}/{}^{204}\text{Pb} = 15.4969 \pm 8$ (2σ , n=8), ${}^{208}\text{Pb}/{}^{204}\text{Pb} = 36.7156 \pm 2$ (2σ , n=8) for NBS981. In addition, USGS reference material BHVO-2 was also processed for Sr-Nd-Hf-Pb isotope analyses, giving ratios of 0.703474 \pm 10 (2σ , n=2) for ${}^{87}\text{Sr}/{}^{86}\text{Sr}$, 0.512982 ± 6 (2σ , n=2) for ${}^{143}\text{Nd}/{}^{144}\text{Nd}$, 0.283084 ± 6 (2σ , n=2) for ${}^{176}\text{Hf}/{}^{177}\text{Hf}$, and 18.6485 ± 8 (2σ , n=2), 15.5288 ± 20 (2σ) and 38.2379 ± 2 (2σ , n=2) for ${}^{206}\text{Pb}/{}$



²⁰⁴Pb, ²⁰⁷Pb/²⁰⁴Pb, and ²⁰⁸Pb/²⁰⁴Pb, respectively, which are consistent with the recommended values within analytical errors (Weis et al., 2005; 2006; 2007).

3.3 Zircon in situ U-Pb and Hf isotopes

Zircon grains were mounted in an epoxy resin and polished to reveal their inner centers. Cathodoluminescence (CL) imaging was conducted to select positions before in situ U-Pb dating and Hf isotope analysis. The zircon U-Pb analyses were conducted using a LA-ICP-MS at the CAS Key Laboratory of Crust-Mantle Materials and Environments, USTC, Hefei, China. According to the approach outlined by Yuan et al. (2004), a GeoLasHD ablation system equipped with a 193 nm excimer laser was employed with an Agilent 7900 ICP-MS. A carrier gas was used, helium, which was combined with argon in a homogenizer before entering the ICP. The zircon standards 91500 and GJ-1, along with the standard glasses BHVO-2G, BCR-1G, and BIR-2G, were evaluated for isotopic fractionation and trace element determinations. ICPMSDataCal (Liu et al., 2008) was used to reduce the resulting data and Isoplot version 3.0 was used to calculate ages (Ludwig, 2003). Age uncertainties are quoted at the 95% confidence level, and the GJ-1 standard zircon analysis gave an age of 602.3 \pm 4.6 Ma (2 SD, n = 30).

Zircon Lu-Hf isotope analysis was undertaken using a Thermo Scientific Neptune multi-collector (MC–ICP–MS) coupled with a 193 nm ArF excimer laser ablation system at the CAS Key Laboratory of Crust-Mantle Materials and Environments at USTC, Hefei, China. The analysis used the approach outlined by Gu et al. (2019). Analysis spots were chosen within or close to the exact zircon domains for U–Pb dating with a laser spot diameter of 44 μ m and a repetition rate of 10 Hz. Analytical quality was monitored by repeat analysis of the Qinghu and 91500 standard zircons, yielding a mean ¹⁷⁶Hf/¹⁷⁷Hf ratio of 0.282990 ± 20 (2 σ , n=5) and 0.282290 ± 22 (2 σ , n=4), respectively, consistent with the reference values for these standards (Blichert-Toft, 2008; Morel et al., 2008; Sláma et al., 2008; Li et al., 2013).

3.4 SIMS zircon O isotope analysis

Zircon in situ O analysis for samples 19BHY30, 19BHY49 and 19BHY59 was conducted by a Cameca IMS 1280-HR at the State Key Laboratory of Isotope Geochemistry, Guangzhou Institute of Geochemistry, CAS, Guangzhou. Analytical procedures are identical to those described by Li et al. (2010a) and Yang et al. (2018, 2020). The Cs+ primary ion beam with an intensity of ~2 nA was accelerated to 10 kV and rasterized over 10 µm in size diameter. For the correction of the instrumental mass fractionation (IMF), the zircon standard Penglai was used, with a recommended $\delta^{18}O$ value of $5.31\% \pm 0.10\%$ (Li et al., 2010b). The internal precision of every single analysis of δ^{18} O value is generally better than ±0.20‰. As assessed by the reproducibility of repeated analyses of the Penglai standard, the external precision during this study is 0.17‰ (2 SD, n = 25). Accordingly, the measured δ^{18} O value of Qinghu, which is used as a reference value to verify the validity of the IMF, is $5.38\% \pm$ 0.12% (2 SD, n = 26), which is consistent with the reference value within analytical error (Li et al., 2013).



Harker plots of SiO_2 versus major and trace elements for the Beihuaiyang alkaline intrusive rocks in the Dabie orogen. Literature data are same as those in Figure 4.

4 Results

4.1 Zircon U-Pb ages

In situ zircon U-Pb dating was performed on zircon grains from six alkaline intrusive rocks, and the results are listed in Supplementary Table S1. Zircon grains generally display oscillatory, band or unclear zonings in CL images, which are typical of magmatic origin (Figure 3). These samples yield U–Pb ages of 122.6 \pm 0.6 Ma (n = 25, MSWD = 1.2) to 131.3 \pm 1.4 Ma (n = 18, MSWD = 1.6) for the syn-magmatic zircons. One residual metamorphic zircon core with an age of 211 Ma is also observed with a Th/U ratio of 0.02.

4.2 Whole-rock major and trace elements

A total of nineteen alkaline intrusive rocks from the Beihuaiyang zone were analyzed for whole-rock major and trace elements, and the results are presented in <u>Supplementary Table S2</u>. All major element contents have been normalized to 100% on a loss of ignition free basis prior to plotting.

These samples display high Na_2O+K_2O contents of 11.1–11.8 wt% and plot into the syenite and nepheline syenite fields in the total alkali-silica (TAS) classification diagram (Figure 4A). They also have high K_2O/Na_2O ratios of 1.30–2.69, except for 19BHY25 with a low

 K_2O/Na_2O ratio of 0.62. On the diagram of K_2O versus Na_2O (Figure 4B), they fall into the shoshonitic and ultrapotassic fields. In the chondrite-normalized rare earth element (REE) diagram (Figure 4C), the Beihuaiyang alkaline intrusive rocks are characterized by strong LREE enrichment relative to heavy REE (HREE) with high $(La/Yb)_N$ ratios of 31.7–218.3, and either negative or positive Eu anomalies (Eu/Eu* = 0.51–1.17). In the primitive mantle-normalized trace element diagram (Figure 4D), they are characterized by the enrichment of LILE (Rb, K and Pb) but depletion of P and high field strength elements (HFSE), such as Nb, Ta and Ti.

Generally, the $Fe_2O_3^{T}$, TiO₂, MgO and P_2O_5 contents of these alkaline intrusive rocks show roughly decreasing trends with increasing SiO₂ (Figures 5A, C, E), whereas the K₂O content shows an increasing trend with increasing SiO₂ (Figure 5B). These samples have high Sr (480–3040 ppm) content and display a decreasing trend with increasing SiO₂ content (Figure 5F).

4.3 Whole-rock Sr-Nd-Hf-Pb isotopes

The Sr-Nd-Hf isotope analyses were performed on nineteen Beihuaiyang alkaline intrusive rocks, and seventeen of them were also performed for Pb isotope analysis (Supplementary Table S3). The initial (87 Sr/ 86 Sr)_i ratios, $\epsilon_{Nd}(t)$ values, $\epsilon_{Hf}(t)$ values and initial Pb isotope ratios of the Beihuaiyang alkaline intrusive rocks were



(A) Diagram of $(^{87}\text{Sr})^{86}\text{Sr})_i$ ratios versus $\epsilon_{Nd}(t)$ values for the Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen. The Sr-Nd isotope compositions for the lithospheric mantle of the North China Block (Zhang and Yang, 2007), ultra-high pressure metamorphic rocks in Central Dabie and South Dabie (Ames et al., 1996; Chavagnac and Jahn, 1996; Chen and Jahn, 1998; Li et al., 2000; Ma et al., 2000; Xia et al., 2008), ultra-high pressure metamorphic rocks in North Dabie (Ma et al., 2000; Zheng et al., 2000; Liu et al., 2005), post-collisional granitoids (Chen et al., 2002; Zhang et al., 2002; 2010; Wang et al., 2007; Huang et al., 2008), and post-collisional mafic-ultramafic instrusives (PCMI) (Chen and Jahn, 1998; Li et al., 2012) are also plotted for comparison. (B) Plot of whole-rock $\epsilon_{Hr}(t)$ versus $\epsilon_{Nd}(t)$ for the Beihuaiyang alkaline intrusive rocks in the Dabie orogen. The Terrestrial Array is after Vervoort et al. (2011). All data are calculated at t =126 Ma. Diagrams of initial ²⁰⁶Pb/²⁰⁴Pb ratios versus initial ²⁰⁷Pb/²⁰⁴Pb ratios (C) and initial ²⁰⁶Pb/²⁰⁴Pb ratios versus initial ²⁰⁸Pb/²⁰⁴Pb ratios (D) for the Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen. The Pb isotope compositions for the lithospheric mantle of the North China Block (Zhang et al., 2002), ultra-high pressure metamorphic rocks in Central Dabie and South Dabie (Zhang et al., 2002; Li et al., 2003; Shen et al., 2002; Li et al., 2002; Shen et al., 2002; Li et al., 2003; Shen et al., 2002; Li et al., 2003; Shen et al., 2002; Huang et al., 2008), and post-collisional mafic-ultramfic intrusives (PCMI) (Wang et al., 2005; Huang et al., 2007; Dai et al., 2012) are also plotted for comparison. NHRL-Northern Hemisphere reference line (²⁰⁷Pb/²⁰⁴Pb)_{NHRL} = 0.1084 × (²⁰⁶Pb/²⁰⁴Pb)_i + 13.491 (²⁰⁸Pb/²⁰⁴Pb)_{NHRL} = 1.209 × (²⁰⁶Pb)_{NHRL} = 15.207 (Hart, 1984). The literature data are from Zhou et al. (1995) and Yang (2002).

calculated back to 126 Ma based on zircon U-Pb dating results (Figure 3). These rocks have relatively high (⁸⁷Sr/⁸⁶Sr)_i ratios of 0.7092–0.7098, negative $\varepsilon_{Nd}(t)$ values of –16.0 to –14.4 (Figure 6A), corresponding to two-stage Nd model ages of 2230-2096 Ma (Supplementary Table S3). They also show negative $\varepsilon_{Hf}(t)$ values of -17.5 to -15.6 (Figure 6B), corresponding to two-stage Hf model ages of 2303-2185 Ma. As shown in Figure 6B, the samples are plotted near the Nd-Hf Terrestrial Array ($\epsilon_{Hf}(t) = 1.55 \times \epsilon_{Nd}(t) +$ 1.21; Vervoort et al., 2011) and do not exhibit significant Hf-Nd isotope decoupling. The (206Pb/204Pb)i (207Pb/204Pb)i and (208Pb/ ²⁰⁴Pb)_i ratios of the Beihuaiyang alkaline intrusive rocks are 17.232-17.452, 15.501-15.529 and 37.710-38.018, respectively. On the (207Pb/204Pb)_i versus (206Pb/204Pb)_i diagram, these alkaline intrusive rocks are plotted left of the Earth Geochron and above the Northern Hemisphere Reference Line (NHRL) (Figure 6C; Hart, 1984). In addition, they also fall above the NHRL on the (²⁰⁸Pb/ ²⁰⁴Pb)_i versus (²⁰⁶Pb/²⁰⁴Pb)_i diagram (Figure 6D).

4.4 Zircon in situ Hf-O isotopes

The zircons from the six samples that were dated by LA-ICP-MS were also analyzed for Lu–Hf isotopes by LA-MC-ICP-MS, and four samples were selected to perform zircon *in situ* O isotopes by SIMS. The analyzed domains for *in situ* U-Pb and Hf-O isotopes are the same or close to each other. The syn-magmatic zircon domains display three groups of δ^{18} O values and $\epsilon_{Hf}(t)$ values (Figure 7A). Group I has low δ^{18} O values of 3.00‰–3.82‰, high $\epsilon_{Hf}(t)$ values of –5.1 to 5.6 and young two-stage Hf model ages of 1497 to 820 Ma; Group II has medium δ^{18} O values of 4.42‰–5.17‰, variable and negative $\epsilon_{Hf}(t)$ values of –22.5 to –4.5 and variable old two-stage Hf model ages of 5.87‰–6.74‰, negative $\epsilon_{Hf}(t)$ values of –6.9 to –14.0 and medium two-stage Hf model age of 2061 to 1615 Ma (Figures 7A, B). The relict zircon with U-Pb age of 211 Ma has $\epsilon_{Hf}(t)$ value of –15.0 and two-stage Hf model age of 2187 Ma.



Zircon Hf and O isotope relationships for the Beihuaiyang alkaline intrusive rocks in the Dabie orogen. (A) δ^{18} O versus $\epsilon_{Hf}(t)$, (B) δ^{18} O versus T_{DM2} .

5 Discussion

The LA-ICP-MS zircon U-Pb dating for the Beihuaiyang alkaline intrusive rocks yielded concordant ages of 131.3 ± 1.4 Ma to 122.6 ± 0.6 Ma (Figure 3), which agree well with those dating results by previous studies (Zhou et al., 1995; Yang et al., 1999; Zhou et al., 2014). These ages are coeval with the Early Cretaceous post-collisional magmatism in the Dabie orogen, but postdate the UHP metamorphism due to the collision between the South China Block and North China Block (Zhao and Zheng, 2009). In this respect, these alkaline intrusive rocks belong to post-collisional igneous rocks.

These post-collisional alkaline intrusive rocks have arc-like trace element distribution patterns, such as enrichment in LREE and LILE but depletion in HREE and HFSE, and enriched radiogenic whole-rock Sr-Nd-Hf isotopes with high initial (87 Sr/ 86 Sr)_i and negative $\epsilon_{Nd}(t)$ and $\epsilon_{Hf}(t)$ values. These geochemical compositions show a strong affinity to the continental crust, which is generally characterized by arc-like trace element distribution patterns and enriched radiogenic isotope compositions. Furthermore, one residual zircon core with U–Pb age of 211 Ma was observed in this study, consistent with the Triassic metamorphic age for the UHP rocks in the Dabie–Sulu orogenic belt (Zheng et al., 2004; Tang

et al., 2008a; Tang et al., 2008b; Zheng et al., 2009). Therefore, the geochemical compositions of the Early Cretaceous Beihuaiyang alkaline intrusive rocks suggest that continental crust materials have played an important role in their petrogenesis.

5.1 Influence of syn/post-magmatic processes

Syn/post-magmatic processes, such as fractional crystallization and/or crustal assimilation (AFC process) and water-rock interaction, would influence the geochemical compositions of magmas after they were formed in their sources. It is necessary to evaluate these factors before discussing their petrogenesis and the nature of their magma source. The low loss on ignition (LOI) contents of 1.18–3.24 wt% and the poor relationship between the LOI and fluid-mobile element contents (not shown) indicate that later alteration has a negligible influence on the geochemical compositions of the alkaline intrusive rocks in this study.

It is generally accepted that the continental crust is characterized by high SiO₂ content, high incompatible element contents and high (⁸⁷Sr/⁸⁶Sr)_i ratios but low $\varepsilon_{Nd}(t)$ values and compatible element contents. Once influenced by crustal assimilation, the igneous rocks would exhibit covariant trends between their SiO₂ contents and trace element contents or isotopes mentioned above. Although the Beihuaiyang alkaline intrusive rocks have variable major and trace elements, they have consistent REE and trace element distribution patterns and restricted Sr-Nd-Hf-Pb isotope compositions. Additionally, there are no obvious correlations between their initial (⁸⁷Sr/⁸⁶Sr)_i ratios (Figure 8A) or $\varepsilon_{Nd}(t)$ values (Figure 8B) and their SiO₂ contents. All these pieces of evidence indicate that crustal assimilation has played a negligible role in their geochemical compositions.

The Beihuaiyang alkaline intrusive rocks have moderate SiO₂ contents of 55.5-60.4 wt%, low MgO contents of 0.25-2.07 wt% and variable Mg# number of 17.57-50.44, indicating they may experience fractional crystallization during their emplacement. The decreasing Fe₂O₃^T, TiO₂ and P₂O₅ contents with increasing SiO₂ content (Figures 5C-E) together with negative P and Ti anomalies (Figure 4D) suggest Fe-Ti oxides and apatite may be fractionated. In addition, the decreasing Sr content with increasing SiO₂ content and negative Eu, Ba and Sr anomalies for most samples (Figure 4D) indicate the fractionation of K-feldspar and plagioclase. This is because Ba is dominant in K-feldspar while Sr and Eu are mainly hosted by plagioclase. Notably, a few samples also show positive Eu, Ba and Sr anomalies and high contents of these elements, which may indicate that they did not undergo fractional crystallization but accumulation of K-feldspar and plagioclase (Figure 4D).

In summary, the alkaline intrusive rocks from the Beihuaiyang zone do not appear to be significantly affected by crustal assimilation, but they did undergo considerable crystal fractionation during magma ascent. Although the crystal fractionation can significantly affect whole-rock major and trace element compositions, it does not significantly affect their radiogenic Sr-Nd-Hf-Pb and zircon *in situ* O isotope compositions. This may explain why they show a wide range of whole-rock major and trace elements content but a restricted range



of isotope compositions. As a result, their isotopic characteristics are primarily determined by magma source and can be used to constrain the nature of magma source.

5.2 The source nature of the beihuaiyang syenites

Numerous petrological, geochemical and experimental studies have shown that alkaline intrusive rocks can be produced either by fractional crystallization of alkaline basaltic magma (e.g., Irving and Price, 1981; Eby et al., 1998) or partial melting of crustal rocks caused by an influx of volatiles and alkalis (e.g., Hay and Wendlandt, 1995; Kaszuba and Wendlandt, 2000; Legendre et al., 2005) or low degrees of partial melting of metasomatized lithosphere mantle (e.g., Laporte et al., 2014; Ashwal et al., 2016). Due to the lack of contemporaneous alkaline mafic igneous rock, we can exclude the first possibility. Despite their variable Mg# values, a few Beihuaiyang alkaline intrusive rocks have high Mg# values (up to 50.44) and MgO contents (2.02–2.07 wt%) as well as positive zircon $\epsilon_{Hf}(t)$ values, indicating their derivation from mantle rocks rather than crustal rocks. According to previous studies (Jung et al., 2007; Ding et al., 2011), K-rich alkaline intrusive rocks produced by partial melting of mafic lower continental crust under high pressure generally have high Al₂O₃ and Sr contents but low Y and HREE contents, resulting in significantly high Sr/Y ratios. However, the high K₂O contents (>4 wt%), together with negative correlations between Al₂O₃, Y contents and Sr/Y ratios (Figure 9), suggest that they are unlikely to be the product of partial melting of mafic lower continental crust.

Low-degree partial melting of pre-enriched lherzolite at pressures of 1.0–1.5 GPa can produce phonolitic melts with Mg# of 50–60 (Irving and Price, 1981; Draper and Green, 1997; Laporte et al., 2014). Because of the lack of feldspar fractionation at mantle pressures, Ba and Sr concentrations are typically very high and range from several hundred to thousands of ppm (Irving and Price, 1981). These features are consistent with those of the Beihuaiyang alkaline intrusive rocks, supporting their origination from the mantle rather



than ancient continental crust (Zhou et al., 1995). Based on the enriched radiogenic isotope compositions and arc-like trace element distribution patterns, it has been suggested that these alkaline intrusive rocks originated from the enriched lithospheric mantle metasomatized by the subducted continental crust materials (Yang et al., 2002; Xia et al., 2008; Zhou et al., 2014). Nevertheless, the origin and the nature of the subducted crustal material are still less constrained.

The Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen have more enriched Sr-Nd isotope compositions than the North China lithospheric mantle (Figure 6A), suggesting that they were not directly derived from the unmetasomatized North China lithospheric mantle. The Beihuaiyang alkaline intrusive rocks are located within the Dabie orogen, which was built by the northward subduction of the South China Block beneath the North China Block during the Triassic (e.g., Cong, 1996; Faure et al., 2003; Zheng et al., 2003). These facts suggest that the crustal materials metasomatized the overlying lithospheric mantle of North China most likely come from the subducted South China Block. Previous studies on the post-collisional mafic igneous rocks in the

Dabie-Sulu orogenic belt and the southeastern margin of North China have demonstrated that the subducted continental crust materials were involved in their mantle sources (Zhao et al., 2013; 2015). A three-layer crustal architecture was proposed for the Dabie orogen before the Early Cretaceous magmatism according to Sr-Nd-Hf-Pb isotope compositions of the metamorphic rocks and post-collisional granites (Zhang et al., 2002; Li et al., 2003; Zhao et al., 2008; 2011; Shen et al., 2014), with Central Dabie and South Dabie representing the upper layer, North Dabie representing the middle layer and the source of the post-collisional granites representing the lower layer of the subducted South China Block. The Sr-Nd-Hf isotope compositions are gradually enriched from the upper to lower layers, while the Pb isotope composition progressively becomes depleted (Figure 6).

The Sr-Nd isotope compositions of these alkaline intrusive rocks fall within the fields of the Central-South Dabie and North Dabie metamorphic rocks (Figure 6A). However, in the Pb isotope composition diagrams (Figures 6C,D), most of them fall within the fields of the Central and South Dabie metamorphic rocks, with only a few samples in the North Dabie field. In this regard, the mantle source of the alkaline intrusive rocks would be metasomatized by materials from the subducted upper and middle continental crust of the South China Block. It is generally accepted that the metasomatic agents in subduction zones are generally aqueous solutions and hydrous melts, the former is only capable of carrying water-mobile elements, such as LILE, while the latter carries not only water-mobile elements but waterimmobile elements, such as REE and HFSE (Zheng, 2019). Given that the Beihuaiyang alkaline intrusive rocks have similar Sr-Nd-Pb isotope compositions comparable to the subducted upper and middle crust of the South China Block and that their Nd-Hf isotope compositions are not decoupled (Figure 6), we suggest that the mantle source of the Early Cretaceous Beihuiayang alkaline intrusive rocks is the lithospheric mantle of the North China Block metasomatized by the melts from the subducted upper and middle continental crust of the South China Block during the Triassic.

The zircons in these alkaline intrusive rocks have three groups of Hf-O isotope compositions, suggesting the mantle source contains crustal materials with both low to high $\varepsilon_{Hf}(t)$ and δ^{18} O values. Group I zircon has low δ^{18} O values of 3.00‰-3.82‰, high $\epsilon_{Hf}(t)$ values of -5.1 to 5.6 and young two-stage Hf model ages of 1497 to 820 Ma, which are comparable to the metaigneous rocks in the Central Dabie (Figures 10A, B). Based on the result of Li et al. (2011), the $\varepsilon_{Hf}(t)$ value (t = 126 Ma) of the lithospheric mantle beneath the North China Block is about -13.1. As its O isotope composition was unconstrained, we assume that it has the normal mantle zircon δ^{18} O values of 5.3‰ ± 0.3‰ (Valley et al., 1998). Therefore, the Hf-O isotope compositions of Group I zircon can be mainly attributed to the recycling of the subducted upper crust of the South China Block, which have undergone high-T water-rock interaction during the Neoproterozoic (Zheng et al., 2003; 2004; 2009; Chen et al., 2007; 2010; Tang et al., 2008a; 2008b; He et al., 2016). Although Group II and Group III zircons have different δ^{18} O values of 4.42‰–5.17‰ and 5.87‰-6.74‰, they have similar two-stage Hf model ages of 2595 to 1463 Ma, which are comparable to the metaigneous rocks in North Dabie (Figures 10C, D) but different from those of the lithospheric mantle beneath the North China Block. The lower zircon $\varepsilon_{Hf}(t)$ values and older Hf model ages as well as higher δ^{18} O values for Group II and III zircons suggest their origination



Comparisons of zircon Hf and O isotope compositions between the Beihuaiyang alkaline intrusive rocks and the UHP metamorphic rocks in the Dabie orogen. Data sources: the UHP granitic gneisses and eclogites in Central Dabie (Zheng et al., 2004; Zheng et al., 2005; Zheng et al., 2006); the UHP granitic gneisses and granulites in North Dabie (Zheng et al., 2004; Zhao et al., 2005; 2008; Lei and Wu, 2008). Groups I (**A**, **B**) and Group II and III (**C**, **D**) zircon δ^{18} O values and Hf model ages for the Beihuaiyang alkaline intrusive rocks are comparable to those of the subducted continental crust in Central Dabie (upper layer) and North Dabie (middle layer), respectively.



from the recycled middle crust as represented by the North Dabie UHP rocks. In addition, materials from the different layers of the subducted South China Block were also suggested to be incorporated into the mantle source of post-collisional mafic to ultramafic igneous rocks in the Dabie orogen based on their zircon Hf-O isotope compositions (Dai et al., 2011).

The Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen have moderate SiO₂ contents (55.5-60.4 wt%), high K₂O contents (4.26-9.40 wt%) and high K₂O/Na₂O ratios (0.62-2.69), which require the presence of potassium-rich minerals in their mantle source (Wang H. et al., 2014). It is generally acknowledged that phlogopite and amphibole are the two major potassium-rich phases in the lithospheric mantle (Spāth et al., 1996; Turner et al., 1996; Sun et al., 2014; Conticelli et al., 2015). According to the results of previous studies (Furman and Graham, 1999), melts from phlogopite-bearing mantle source have high Rb/Sr ratios (>0.1) but low Ba/Rb ratios (<20), while melts from amphibole-bearing mantle source have low Rb/Sr ratios (<0.05) but high Ba/Rb ratios (>30). The Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen show high Rb/Sr (0.06-0.70) and low Ba/Rb (0.89-30.16) ratios (Figure 11), indicating that phlogopite is the dominant potassium-rich phase in their mantle source. Experimental studies have shown that partial melting of phlogopite-bearing mantle peridotite can directly produce potassic magmas with SiO₂ contents of 52–64 wt% (Condamine and Médard, 2014; Mallik et al., 2015; Förster et al., 2019). Therefore, the Early Cretaceous Beihuaiyang alkaline intrusive rocks may originate from partial melting of the phlogopite-bearing mantle source. Meanwhile, the presence of phlogopite in the mantle source suggests that the mantle source was enriched in potassium by metasomatism before magmatism (Laporte et al., 2014). Combined with the element and isotope compositions of the Early Cretaceous Beihuaiyang alkaline intrusive rocks, we suggest that they originated from low degrees of partial melting of the



Dabie) and middle layers (Noth Dabie) of the subducted South China Block to form the orogenic lithospheric mantle in the Triassic. **(B)** Partial melting of the metasomatized mantle to form the Beihuaiyang alkaline intrusive rocks due to the extension of the continental lithosphere and the collapse of the orogen in the Early Cretaceous.

lithospheric mantle metasomatized by the melts from the subducted upper and middle crust of the South China Block.

5.3 Crust-mantle interaction during the continental collision

The Early Cretaceous (131.3–122.6 Ma) Beihuaiyang alkaline intrusive rocks are coeval with the post-collisional magmatism in the Dabie orogen (e.g., Zhao and Zheng, 2009), indicating that they are post-collisional igneous rocks. As discussed above, they originated from partial melting of the enriched lithospheric mantle, which was metasomatized by melts from the subducted upper and middle crust of the South China Block in the Triassic. Thus, the melt-peridotite reaction would be the mechanism for producing the enriched mantle source.

Previous studies on UHP metamorphic rocks in the Dabie-Sulu orogenic belt have confirmed that the subducted continental crust were partially melted at varying degrees during the continental subduction/collision in the Triassic (Xia et al., 2008; Zheng et al., 2011; Liu et al., 2012; 2014; Chen et al., 2013; Wang L. et al., 2014). Furthermore, M-type peridotites in the Dabie-Sulu orogenic belt also record extensive evidence of melt/fluid metasomatism (Zhang et al., 2011; Zheng et al., 2011), which was demonstrated by many petrological and geochemical evidence, such as the metasomatismgenerated zircons with 206 Pb/ 238 U ages ranging from 220 ± 2 Ma to 231 ± 4 Ma (Zhang et al., 2005; Li et al., 2016b), their whole-rock arctype trace element signatures and enriched Sr-Nd isotope compositions, and the occurrence of water-bearing minerals (e.g., auriferous mica and Ti-plagioclase magnesite). All these pieces of evidence suggest that during the collision between the South China Block and North China Block in the Triassic, the continental lithospheric mantle wedge of the North China Block was metasomatized by the melts/fluids released from the subducted South China Block, forming a fertile and enriched orogenic lithospheric mantle (Zheng, 2012; Zheng and Hermann, 2014). For the Early Cretaceous Beihuaiyang alkaline intrusive rocks, the involvement of the subducted crustal materials into their mantle source can explain their arc-like trace element distribution patterns (i.e., enrichment in LREE and LILE but depletion in HREE and HFSE) and enriched radiogenic isotope compositions (i.e., high initial $({}^{87}\text{Sr}/{}^{86}\text{Sr})_i$ ratios and negative $\epsilon_{Nd}(t)$ and $\epsilon_{Hf}(t)$ values, Figures 6A,B). Meanwhile, the $({}^{87}Sr/{}^{86}Sr)_i$ ratios, $\varepsilon_{Nd}(t)$ values and initial Pb isotope compositions of the Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen fall within the fields of the Central Dabie, South Dabie and North Dabie UHP metamorphic rocks (Figure 6), which represent the upper and middle continental crust of the subducted South China Block (Zheng et al., 2005; Zheng et al., 2006; Liu and Li, 2008; Liu, 2018). This suggests that the crustal materials involved in the mantle source of the Early Cretaceous Beihuaiyang alkaline intrusive rocks mainly originated from the upper and middle continental crust of the South China Block.

Collectively, the subducted upper and middle continental crust of the South China Block was partially melted during the Triassic, resulting in felsic melts that were enriched in LREE, LILE and radiogenic isotopes, but depleted in HREE and HFSE. These melts have reacted with the lithospheric mantle wedge of the North China Block, resulting in phlogopite-bearing metasomatites that were stored for about 100 Myr (Figure 12A). During the post-collisional stage in Early Cretaceous (Figure 12B), the metasomatized lithospheric mantle was partially melted to form the Beihuaiyang alkaline intrusive rocks due to the extension of the continental lithospheric and the collapse of the orogen (Zheng and Zhao, 2017).

6 Conclusion

The Early Cretaceous Beihuaiyang alkaline intrusive rocks were formed at 123–131 Ma, which are consistent with the time of significant post-collisional magmatism in the Dabie orogen. They were produced by low degrees of partial melting of the enriched lithospheric mantle based on their element and isotope characteristics. During the continental collision between the South China Block and the North China Block in the Triassic, the subducted upper and middle continental crust of the South China Block was partially melted, and the resulting felsic melts have reacted with the lithospheric mantle of the North China Block to produce the enriched mantle metasomatites. As the lithosphere of the orogen was thinned and extended in the post-collisional stage, partial melting of these mantle metasomatites resulted in the formation of the Early Cretaceous Beihuaiyang alkaline intrusive rocks. Therefore, the Early Cretaceous Beihuaiyang alkaline intrusive rocks in the Dabie orogen provide a new perspective for understanding the crust-mantle interaction and the post-collisional magmatism in the continental collision orogen.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

Author contributions

G-CS: Conceptualization, data curation, experiments, investigation, methodology, visualization, and writing—original draft. Y-SX: Investigation and experiments. GW: Writing, zircon *in situ* Hf data processing and identification of thin sections. LQ: Zircon *in situ* Hf data processing. Xu Qingyang: writing and editing. L-QD: Writing, review and editing. Z-FZ: Conceptualization, investigation, methodology, and writing—review and editing.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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Supplementary material

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