Tracing subducted oceanic slabs in the mantle by using molybdenum isotopes: A case study of intraplate basalts from the Northeast China

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Abstract

Determining subducted processes is very important for understanding lithological heterogeneity because substantial quantities of slabs are recycled into the mantle. Molybdenum isotopes have been used to distinguish sources materials because of the significant Mo isotopic differences between the crust and the mantle. Here, we undertake a systematic Mo isotopic investigation on a suite of well-characterized continental basalts from Keluo and Halaha-Chaoer, located in Northeast China. $\delta^{98}$Mo of Keluo varied from -0.41 to -0.23‰ with an average of -0.34‰, and samples from Halaha-Chaoer ranged in $\delta^{98}$Mo from -0.18 to -0.12‰ with an average of -0.15‰. The former is lighter than the mean value of fresh oceanic basalts (-0.21‰), while the latter is similar to oceanic basalts. Combined with other geochemical indications (LOI, Ce/Pb, La/Yb and so on), the Mo isotopic variations cannot be interrupted by chemical weathering, continental crust contamination, or magmatic processes. The $\delta^{98}$Mo variation in this study is explained by adding various oceanic crustal materials in magma sources. The relationships of $\delta^{98}$Mo with Ba/Th, Th/U, $^{143}$Nd/$^{144}$Nd for the samples indicated that sediment and altered oceanic crust have contributed to the significant variations in these samples. This study indicates the great potential of Mo isotopes to distinguish different types of recycled oceanic crust materials in the mantle.

Keywords: Molybdenum isotopes; Subducted oceanic slabs; Intraplate basalts; Northeast China
1. Introduction

Slab subduction is widely considered to be the primary mechanism for element cycling between the crust and mantle (Hofmann, 1997; Stracke et al., 2003). The recycled materials with slabs subduction are used to either generate the magmas in the subduction zone or form different reservoirs, such as enriched mantle component I (EM I) and enriched mantle component II (EM II) (Zindler and Hart, 1986). Previous investigations suggested that the oceanic crust account for ~10% of the total mass of the mantle since the starting of subducted processes (Stracke et al., 2003; Tang et al., 2016). The converted approach took varied materials into mantle and led to lithological heterogeneity in the upper mantle (Perfit et al., 1980; Plank and Langmuir, 1998; Castillo et al., 2000). Therefore, distinguishing the contribution of subducted materials was required for understanding the lithological heterogeneity.

The previous works observed that the mobility of Mo could be negligible in the subduction zones (Noll et al., 1996; Bali et al., 2012), suggesting that Mo isotopic composition of subdued material can be recorded in the mantle (Freymuth et al., 2015). Supposing there are significant differences in δ⁹⁸Mo and Mo concentration between the upper mantle, fresh oceanic crust, altered oceanic crust, and sediment, the Mo isotope system will be a powerful tool to trace the sources of magma and calculate the contribution proportion of each source. Bezard et al. (2016) measured the Mo isotopic composition of Mid-Ocean Ridges Basalt (MORB) to establish the Mo isotopic composition of the depleted mantle (DM) as -0.21±0.02‰. Freymuth et al. (2015) analyzed the sediment and altered oceanic crust from ODP sites 801 and 802, ranging in δ⁹⁸Mo from -1.87 to 0.11‰ with an average value of -0.50‰ and from -0.88 to 0.86‰ with an average value of -0.03‰, respectively. Moreover, the Mo concentrations of the upper mantle, altered oceanic crust, and sediment are 1.65 μg g⁻¹, 0.35 μg g⁻¹, and 4.03 μg g⁻¹, respectively. The significant differences in Mo isotopic composition and Mo concentrations between mantle and crust indicate that Mo isotope has excellent potential to trace subducted processes.

Due to the natural advantage of the Mo isotope, many investigations focused on oceanic basalt using Mo isotopic system to trace the subducted processes (König et al., 2016; Willbold and Elliott, 2017; Zhang et al., 2020; Li et al., 2021). However, the current Mo isotope data of intraplate basalt is very limited, and it is debated for using Mo isotope to trace recycled
materials for intraplate basalt. Here, we measured a systematic Mo isotopic composition of a
set of well-characterized basalt samples from Keluo and Halaha-Chaoer of Northeast China.
Samples from Keluo display typical EM I characteristics, while samples from Halaha-Chaoer
have the radiogenic isotopic signatures of the focal zone (FOZO) (Hart et al., 1992; Stracke et
al., 2005). Thus, the samples allow us to use the Mo isotope to assess the contributions of
recycled crustal materials to the mantle.

2 Sample
Northeast China was set between the Siberian Craton and the North China Craton, and
considered as the eastern segment of the Central Asian Orogenic Belt (Chen et al., 2007; Zhang
et al., 1995). The plate suffered the closure of the Paleo-Asian Ocean and the amalgamation of
many microcontinent (Senger et al., 1993). Intraplate basaltic rocks in North China were formed
in the Cenozoic (Zhang et al., 1995; Zou et al., 2003). The Halaha-Chaoer and Keluo volcanic
fields were set at the Great Xing'an Mountains and the North of Songliao Basin (Zhang, 1992).
Fourteen samples were collected from two volcanic fields, including Halaha-Chaoer (2.0 Ma)
and Keluo (9.6–0.11 Ma), of Northeast China. These samples come from many works which
studied the evolution of volcanoes. Therefore, the geochemical and isotopic compositions of
more samples can be found in previous works (Sun et al., 2014, 2017), and particle samples are
measured in this study (Table S1). Here, we just provided a brief description of these samples.
The Mo concentration and MgO content of Halaha-Chaoer lavas ranged from 0.96 to 1.18 μg
with an average value of 1.05 ± 0.18 μg g⁻¹ and from 7.93 to 12.13 wt. %, suggesting
significant crystallization. And the Sr-Nd isotopic ratios of Halaha-Chaoer samples is similar
to the FOZO component (initial ⁸⁷Sr/⁸⁶Sr = 0.70354 - 0.7037 and ¹⁴³Nd/¹⁴⁴Nd = 0.51291 -
0.5193). On the contrary, the Keluo lavas are characterized by a high Mo concentration of 1.21-
2.63 μg g⁻¹ and same MgO content (7.67 to 11.28 wt.% in relation to Halaha-Chaoer lavas. The
Sr-Nd isotopic compositions of the group sample are close to the EMI component (⁸⁷Sr/⁸⁶Sr =
0.70503–0.70541 and ¹⁴³Nd/¹⁴⁴Nd = 0.51235–0.51246) (Table 1). The significant chemical
difference between Keluo and Halaha-Chaoer provided a great opportunity to check the ability
of Mo isotope to trace the contributions of recycled crustal materials to the mantle.

3 Analytical methods
3.1 Major and trace element analyses

The major and trace elements were conducted following the analytical procedures described by Qi et al. (2000). An Inductively Coupled Plasma-Atomic Emission Spectrometer measured the major elemental compositions. The loess on ignition (LOI) was analyzed after the samples were heated for 30 min at 980°C. The samples were digested using a mixture of HF-HNO$_3$-HF. The clean solutions were diluted to determine the trace element composition on an Inductively Coupled Plasma-Mass Spectrometry (ELEMENT XR, Thermo, Germany). The reproducibility for trace elements is better than 10% (2SD). All uncertainties of the major element are better than 2% (2SE).

3.3 Mo-Sr-Nd isotopic analyses

The Mo isotopic measurement of all samples was carried out at the State Key Laboratory of Isotope Geochemistry, Guangzhou Institute of Geochemistry, Chinese Academy of Sciences. Detailed processes for digestion, chemical purification and isotopic analysis can be found in Li et al. (2014) and Zhao et al. (2017). Sr-Nd isotopes were determined at the China University of Geosciences, Beijing, and a more analyzed method can be found in Xu et al. (2021).

The samples were digested using a 7 ml PFA beaker. Approximately 50-100 mg of powdered samples were mixed with 2ml of concentrated HF and 1 ml of concentrated HNO$_3$; the mixtures were heated at 185±5°C for 36 h. The dry residues were dissolved into Aqua regia to digest samples completely. The fully dissolved samples were evaporated to dryness and then redissolved in 5ml 1.5 M HNO$_3$.

The digested samples containing 100-200 ng Mo were spiked by the double spikes ($^{100}$Mo-$^{97}$Mo) in a 15ml PFA beaker to correct the potential Mo isotopic fractionation of chemical purification and measurement. The spiked samples were dried slowly at 90°C to allow the equilibration between the samples and double spike and then redissolved in 4ml mixed acid of 0.1 M HF and 1M HCl. The sample solutions were loaded to prepare BPHA resin that was clean with 6 M HCl and MQ. The matrix elements were washed out of columns by 0.1M HF-1M HCl. The fraction of Mo was collected by 6 M HF-1M HCl in a 15ml PFA beaker. The eluted solutions were evaporated to dry at 130°C. Concentrated HNO$_3$ and H$_2$O$_2$ were used to remove residual organic materials from the resin. The total bank of the procedure is 0.5ng which can be
neglected in relation to the loaded sample. Sr and Nd were purified from matrix elements using AG50W-X12 (200-400 mesh) resin and LN resin, respectively. Molybdenum isotopic measurements were performed with a Multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS, Neptune Plus, ThermoFisher). Sample solutions were introduced into the plasma by an Aridus II desolvator system. The signal sensitivity of $^{95}\text{Mo}$ was $\geq 190$ V $\mu$g$^{-1}$ mL$^{-1}$, and the bank signal of $^{95}\text{Mo}$ was less than 4 mV. The solutions and samples were dissolved at 0.5 M HNO$_3$ and diluted to 30 ng mL$^{-1}$ for measuring $^{95}\text{Mo}$ isotopes. All Mo isotope analyses were carried out on the low mass resolution mode ($\Delta M/M < 3000$). The on-peak instrument and acid matrix blank were determined by measuring 1.5M HNO$_3$ before each sample and standard to subtract from the signal intensities. Every 5 samples were bracketed by a spiked SRM 3134 with 30 ng mL$^{-1}$ to normalize the Mo isotope ratio of samples. More detailed parameters for the Mo isotopes measurements have been listed in previous work. The Mo isotopic composition is expressed relative to NIST SRM 3134 as delta ($\delta$) notation: $\delta^{95}\text{Mo}=\left(\frac{^{95}\text{Mo}^{95}\text{Mo}_{\text{sample}}}{^{95}\text{Mo}^{95}\text{Mo}_{\text{NIST SRM 3134}}} - 1\right) \times 1000$. Four geological reference materials (NOD-A-1, NOD-P-1, BHVO-2 and BCR-2) from USGS were digested, purified and measured with our samples. The Mo isotopic compositions are $-0.79\pm 0.04\%$ (2SD, n=5) for NOD-A-1, $-0.87\pm 0.05\%$ (2SD, n=5) for NOD-P-1, $-0.05\pm 0.05\%$ (2SD, n=5) for BHVO-2 and $-0.06\pm 0.04\%$ (2SD, n=5) for BCR-2, similar to previous works (Skierszkan et al., 2015; Li et al., 2016; Willbold et al., 2016). For Sr and Nd isotopic composition, the mass fractionations of chemical purification and instrument were calibrated with $^{88}\text{Sr}/^{86}\text{Sr}$=8.375209 and $^{146}\text{Nd}/^{144}\text{Nd}$=0.7219. The long-term analysis gave 0.710241 ± 24 for $^{87}\text{Sr}/^{86}\text{Sr}$ of NIST SRM 987 in the lab and 0.512434 ± 29 for $^{143}\text{Nd}/^{144}\text{Nd}$ of Alfa Aesar Nd. The Sr and Nd isotopic composition of BHVO-2 and BCR-2 determined in this study are consistent with previous values (Li et al., 2012).

4. Results

The $\delta^{95}\text{Mo}$ values of Northeast China lavas vary from -0.41‰ to -0.12‰ (Table 1). The sample from Keluo varied from -0.41 to -0.23‰ in $\delta^{95}\text{Mo}$ with an average of -0.34±0.10‰ (2SD), higher than the sediments (-0.50‰) and lighter than the altered oceanic basalts (0.03‰) (Freyimuth et al., 2015). The samples from Halaha-chaoer have homogenous Mo isotopic
composition, ranging from -0.12 to -0.18‰ with an average of -0.15±0.05‰ (2SD), similar to the fresh MORB (-0.21‰, Chen et al., 2022). The Sr-Nd isotopes, major and trace elements composition were shown in Table 1 and Table S1.

5. Discussion

Given Mo isotope can be fractionated during a range of processes, it is necessary to carefully assess the processes that may have modified Mo isotopic composition of these samples, such as chemical weathering, assimilation of shallow continental crust, magmatic differentiation and partial melting. And then, we estimate the effects of recycled crustal materials on the Mo isotopic variation of the samples.

5.1 Chemical weathering and Assimilation of crustal materials on Mo isotope

Mo isotopes were fractionated during chemical weathering, and lighter isotopes adsorbed onto secondary minerals so that the weathered residual is lighter in $\delta^{95}$Mo than fresh igneous rocks (Greaney et al., 2021). However, the samples do not observe secondary minerals (Sun et al., 2014). Moreover, the loss on ignition (LOI) ranging from 0.00 to 1.28 does not display a negative correlation (Fig. 1). Both pieces of evidences imply that the observed Mo isotopic variation cannot be attributed to chemical weathering.

These basalts erupted through the continental crust, which is about 40km. The Mo content of these basalts varies from 1.28 to 2.82 $\mu$g g$^{-1}$ with an average value of 1.83 $\mu$g g$^{-1}$, higher than the average Mo concentration of continental crust is 0.8 $\mu$g g$^{-1}$ (Rudnick and Gao, 2014). The upper continental crust is significantly different in $\delta^{95}$Mo from the basalt (Yang et al., 2017; Greaney et al., 2020). Hence, we must consider that crustal materials may modify the Mo isotopic composition of these basalts. For the scenario, a negative correlation between Mo content and $\delta^{95}$Mo were expected because of the heavy Mo isotopic composition of the continental crust. However, we do not observe the relationship (Fig. 2). On the other hand, binary mixing calculation implies that the Mo isotopic variation cannot be explain by binary mixing between continental crust and mantle melt, because of unrealistic Mo concentration ratio (Fig. 2). Hence, Assimilation of crustal materials is not the main process that leaded to the Mo isotopic variation in these basalts.
5.2 The effect of fractional crystallization on Mo isotope

The Mo isotopic composition of basalts from the Kos Island arc system ranged from 0.3 to 0.6‰, indicating that Mo isotopes are fractionated during the fractional crystallization of hornblende and biotite (Voegelin et al., 2014). However, Bezard et al. (2016) measured the Mo isotopic composition of MORB from the Pacific-Antarctic ridge and the Mohns-Knipovich ridge. The δ⁹⁵Mo did not show correlations with MgO that indicated magma differentiation, suggesting that Mo isotopic fractionation is insignificant during the fractional crystallization of MORB magma. Recently, Chen et al. (2022) analyzed some MORB glass to constrain the Mo isotopic composition of the upper mantle. The correlations between δ⁹⁵Mo and magmatic differentiation indicators were not observed, inferring that Mo isotopes were not fractionated during magmatic differentiation (Chen et al., 2022). For our samples studied here, the significant variation of MgO contents (from 6.9 to 13.0%) implies that fractional crystallization plays an essential role in changing the chemical composition of basalts. As shown in Fig. 3a, Mo concentration in basalt from Northeast China correlates well with MgO; the positive correlation indicates that Mo is a likely incompatible element enriched in solid phases during melt evolution. The unclear correlations between δ⁹⁵Mo and MgO (Fig. 3b) and Mo (Fig. 2) content suggest that the effect of fractional crystallization on the δ⁹⁵Mo of basalts is likely insignificant. The observation is similar to the previous inference (Bezard et al., 2016 and Chen et al., 2022) but different from the conclusions from Voegelin et al. (2014). Thus, the δ⁹⁵Mo variation cannot be explained simply to fractional crystallization and may be caused by partial melting.

5.3 The effect of partial melting on Mo isotopic composition of basalts

Since sodium is a moderately incompatible element at melting the with highest concentration at started melting, the Na concentration of melt decreases with the growth of melting degree. Hence, Na₈.₀ indicates the degree of partial melting without the effect of fractional crystallization (Klein and Langmuir, 1987). Bezard et al. (2016) observed the absence of correlation between δ⁹⁵Mo and Na₈.₀, explaining the trend that Mo isotopes are not fractionated during mantle melting. Since La is more incompatible than the heavy rare earth element Yb during the partial melting of mantle, (La/Yb)ₙ ratio is sensitive to the degree of partial melting.
Liang et al. (2017) observe broad correlations between $\delta^{98}$Mo and (La/Yb)$_N$ in ocean island basalts (OIB), suggesting a significant effect of melting on the Mo isotopic variations. The conclusion is contrary to Bezard et al. (2016). However, for our samples, we did not observe the clear relationships between $\delta^{98}$Mo and Na$_{8.0}$ (Fig. 4a) as well as (La/Yb)$_N$ (Fig. 4b). In addition, McCoy-West et al. (2019) accessed the Mo isotopic behaviors using a non-modal batch melting model and inferred that Mo isotopes were significantly fractionated during mantle partial melting that melt is heavier in $\delta^{98}$Mo than the residue. The fractionation was driven from as that Mo (VI) with heavier isotope is more incompatible than Mo (IV) (Leitzke et al., 2017). However, the samples (-0.33±0.10‰) produced by the high degree of partial melting have lighter Mo isotopic composition than the samples with a low degree (-0.15±0.05‰), contrary to the prediction trend by the previous model (Fig. 4, McCoy-West et al., 2019). Therefore, the evidences suggested that partial melting cannot explain the $\delta^{98}$Mo variation of these basalts.

5.4 Molybdenum isotopic variation induced by crustal recycling

The above discussions show that the Mo isotopic variation for these basalts cannot be explained by shallow-level processes, fractional crystallization and partial melting. Radiogenic isotopes, such as Nd and Sr, usually are used to reflect heterogeneous sources. The significant variations for $^{143}$Nd/$^{144}$Nd and $\delta^{98}$Mo imply that there are more than two components at least for the mantle sources of these basalts (Fig. 5). In the following, we will discuss the mechanisms that may be responsible for the processes of mantle enrichment and abnormal Mo isotopic composition.

Sediments could be recycled into the mantle with subducted slabs (Stracke et al., 2003). The $\delta^{98}$Mo of subducting sediments varied from -1.87 to 0.11‰ with an average value of -0.50 ± 0.27‰ (2SE, n=16, Freymuth et al., 2015). Since Mo is a highly incompatible element and lighter isotopes were preserved in the residual produced during metamorphic dehydration (König et al., 2016), melts derived from recycled sediments should inherit the light Mo isotopic composition. Hence, the incorporation of recycled sediments into peridotite melting could be responsible the lighter Mo isotopic composition. The $\delta^{98}$Mo of basalt studied here (-0.30‰) is lighter than fresh MORB (-0.21‰, Chen et al., 2022), and $^{143}$Nd/$^{144}$Nd is higher than sediment
suggesting that sediment is one of the sources for these basalts from Keluo. On the other hand, Th/U ratio is not fractionated significantly during magmatic processes as Th and U are incompatible elements. Both elements can be fractionated during fluid processes because of the higher mobility of U than Th (Hawkesworth et al., 1997; Bali et al., 2009; Ling et al., 2011). Thus, there is a significant difference in the Th/U ratio between fresh MORB (3.54, Gale et al., 2013) and sediment (6.64, Vervoort et al., 2011). Reasonability, the ratio is usually used as an indicator of distinguishing the mantle sources (Sun et al., 2020). The Th/U ratio of our basalts (4.94 to 6.12) falls between sediment and fresh MORB. The observation indicates that sediment may play an essential role in forming these basalts. It is noteworthy that there is not expected positive correlation between δ⁹⁸Mo and Th/U ratio (Fig. 5b), implying other sources for the basalts.

Significant Mo isotopic fractionation is observed during seawater alteration (Freymuth et al., 2015), suggesting that altered oceanic crust (-0.02‰) is heavier δ⁹⁸Mo than fresh ocean crust (-0.21‰). Thus, the incorporation of altered oceanic crust during the particle melting of mantle could produce melts with significant various Mo isotopic composition. Due to the mobility difference between Ba and Th (Weaver et al., 1986), the Ba/Th ratio of the altered oceanic crust will be increased with the growing extent of low-temperature hydrothermal alteration (Kelley et al., 2003). Furthermore, the ratio will not be changed during slab dehydration (Kogiso et al., 1997; Kessel et al., 2005). Hence, the ratio is used to trace the incorporation of altered oceanic crust in the mantle (Sun et al., 2020). The Ba/Th ratios of these basalts range from 147 to 239, higher than sediment and fresh MORB and similar to altered oceanic crust (Fig. 5c), indicating the incorporation of altered oceanic crust into the source region of these basalts.

Therefore, recycled sediment and altered both have contributed to the basalt of Keluo produced in Northeast China. The materials may have come from the westward subduction of the Pacific plate (Tang et al., 2014). The contribution of the fresh oceanic crust is insignificant because of the low Mo concentration. Mixing of melts produced from recycled sediment with the contribution of mantle peridotites derived the variation of Mo isotopic composition in the basalts.

6. Conclusion
This work suggests that the basalt from Keluo displays heterogenous Mo isotopic composition and is heavier Mo isotopic compositions than the upper mantle. At the same time, the sample from Halaha-Chaoer is homogenous in δ²⁷⁶Mo, similar to the upper mantle. The isotopic variation was explained by adding sediment and altered oceanic basalt with subducted oceanic slabs into mantle sources. Therefore, the Mo isotope is a powerful tool in investigating the effect of crustal recycling on mantle-derived magmas to understand recycling materials’ roles in mantle heterogeneity.

Acknowledgements

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Reference


Leitzke F. P., Fonseca R. O. C., Sprungb P., Mallmann G., Lagosa M., Michelya L. T. and Mu‘nker...


Table 1 Molybdenum isotopic composition of Northeast China basalt and geological reference materials

<table>
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<tr>
<th>Sample</th>
<th>Mo (μg g⁻¹)</th>
<th>MgO (%)</th>
<th>Na₂O</th>
<th>LOI</th>
<th>Ce/Pb</th>
<th>Th/U</th>
<th>Ba/Th</th>
<th>(La/Yb)ₙ</th>
<th>⁸⁷Sr/⁸⁶Sr</th>
<th>¹⁴₃Nd/¹⁴⁴Nd</th>
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Fig.1 δ^98Mo versus loss on ignition (LOI) for Northeast China basalts. Data are reported in Table 1.

Fig.2 Plots of δ^98Mo against Mo concentration for Northeast China basalts. The average Mo isotopic composition of upper continental crust is 0.14 ± 0.07‰ (Yang et al., 2017). Data are reported in Table 1.
Fig. 3 Plot of $\delta^{98}$Mo against Mo concentration (a) and MgO content (b) for Northeast China basalts.
Fig. 4 δ⁹⁸Mo versus Na₈_0 (a) and (La/Yb)₇ (b) for Northeast China basalts. Na₈_0 was calculated by Na₈_0 = Na₂O+(0.373×MgO)-2.98 (Klein and Langmuir, 1987). Data are reported in Table 1.
Fig. 5 Plot of $\delta^{98}\text{Mo}$ against $^{143}\text{Nd}/^{144}\text{Nd}$ (a), Th/U (b) and Ba/Th (C) for Northeast China basalts. The clay-rich sediments, highly altered oceanic crust, and unaltered oceanic basalts are represented by sediments from DSDP sites 294/295 (Vervoort et al., 2011), samples from the Bay of Islands ophiolite (Parendo et al., 2017), and the average E-MORB (Gale et al., 2013), respectively. The Nd isotopic ratio of clay-rich sediments is 0.512380 (Vervoort et al., 2011), whereas the Nd isotopic compositions of highly altered oceanic crust and unaltered oceanic basalts are assumed to be similar to that of the average E-MORB (0.513074) (Gale et al., 2013). The Th/U and Ba/Th ratios of these components are 6.64 and 35 (Vervoort et al., 2011), 2.5 and 166 (Parendo et al., 2017), and 3.54 and 92 (Gale et al., 2013), respectively.