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Mineralogical characteristics, formation processes, and prospecting potential of Early–Middle Triassic lithium-rich claystone in the Sichuan Basin, South China

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ABSTRACT

Early to middle Triassic sedimentary units within the Sichuan Basin of southwest China contain extensive occurrences of lithium (Li)-rich clay layers (the so-called “Green bean rock”, or GBR) that have significant economic potential as a result of their substantial scale. However, a comprehensive understanding of the genesis and processes involved in the formation of these clay layers remains elusive. This study presents the results of a systematic investigation of three typical Li-rich clay profiles within the basin, using X-ray diffraction (XRD) and scanning electron microscopy (SEM) for mineralogical characterization and analysis. The presence of micron-scale primary high-temperature quartz and iron microspherules suggests that these Li-rich clay deposits have a volcanic origin. XRD analysis indicates that bulk rock samples of GBR are predominantly composed of quartz, clay minerals, and either calcite or gypsum, with the clay mineral assemblage dominated by illite and/or illite/smectite (I/S). Specifically, the GBR of the northeastern Sichuan Basin has a higher level of illitization compared to its southern counterpart, representing differences in sedimentary facies that are also found in other Li environments such as the Li deposits of Nevada. The northeastern GBR contains gypsum and clay minerals, whereas the southern counterpart consists mainly of carbonate lithologies with minor amounts of clay. This indicates that the sedimentological controls on the formation of clay mineral assemblages also influence the formation of clay-type Li deposits and Li enrichment processes. The composition of the clay components is indicative of probable derivation from smectite during diagenesis after originally being generated by the submarine modifications of volcanic materials. The later formation of I/S formation was also most likely the result of the illitization of volcanogenic smectite. The widespread presence of clay-type Li deposits indicates extensive eruptions of volcanic ash during Early to Middle Triassic volcanic events. The Li trapped within the illite and I/S is thought to have been sourced from volcanogenic materials via transportation in a composite fluid comprising meteoric, porewater, and hydrothermal fluids. These economically significant Li-rich clay minerals, found within a marine basin, represent a promising target for lithium exploration as a result of the extractable metal content. This research provides new insights into the processes involved in the formation mechanisms of clay-type lithium deposits and discusses the prospecting potential of volcanic clay-type Li deposits in the Sichuan Basin, contributing to the critical assessment of the Li resources needed for the energy transition and sustainable energy initiatives.

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1. Introduction

Lithium (Li) has a pivotal role in driving a global shift towards lower-carbon energy technologies like electric vehicles (Benson TR et al., 2023), as these technologies heavily rely on Li-ion batteries for energy storage (Wang DH, 2019). Consequently, there is currently an international “Li rush” underway to discover more lithium resources to meet the projected demand of approximately 1×10^6 t (MT) of lithium by 2040, marking an eightfold increase from global production in 2022 (USGS, 2023). Li resources are primarily hosted by pegmatites and evaporitic brines, currently the source of all current global Li production barring some small scale byproduct production that is negligible on a worldwide basis (Fig. 1; Jowitt SM, 2022; Benson TR et al., 2023). Although Volcano-sedimentary (VS) Li deposits are currently less common in the mining industry, with no successful commercial production as of 2024, they are attracting increasing attention because of their significant potential. In recent years, these deposits have garnered growing interest as an alternative source to pegmatite and brine Li deposits (Benson TR et al., 2017; Castor SB and Henry CD, 2020; Benson TR et al., 2023). VS Li deposits are typically split into carbonate-hosted clay-type, volcanic clay-type, and Jadar lithium deposits, with volcanic clay-type deposits formed in caldera settings representing the dominant sedimentary Li resources at present (Castor SB and Henry CD, 2020; Wen HJ et al., 2020). The origin of volcanic clay-type Li deposits and their related volcanic events involves the diagenesis of volcanic ash in marine and lacustrine settings (Benson TR et al., 2023). However, despite their common origin from volcanic ash, there are substantial variations in the clay mineral assemblages of volcanic clay-type Li deposits on both regional and global scales.

In South China, especially the Sichuan Basin, there is a

widespread development of Li-rich clay layers known as “Green bean rock (GBR),” which could contribute significantly to meeting the global demand for lithium resources. The GBR is well-known for its bean-shaped quartz grains and elevated lithium content, with the substantial scale of this unit indicating its potential as a lithium resource (Lin YJ et al., 2020; Feng MS et al., 2021). Specifically, Most of the GBR (75%) have lithium concentrations equal to or greater than 270×10^{-6} , with the highest reaching up to 957×10^{-6} (Fig. 2c; Sun Y et al., 2018; Ma SC et al., 2019; Feng MS et al., 2021; Peng Y et al., 2023; Wang KM et al., 2023; Zhang B et al., 2023; Chen HF et al., 2024), comparable to or exceeding the lowest potential extractive grade for clay Li deposits (400×10^{-6} , Chen ZY et al., 2023). However, the origin of the GBR remains uncertain. Clay layers within marine sedimentary sequences are often attributed to differing depositional processes, including sedimentation derived from terrigenous sources, volcanic ash deposition, and the accumulation of biogenic material in marine environments (Jeans CV et al., 1982; Drits VA et al., 2004), and extraterrestrial impacts, where the deposition of material resulting from celestial collisions contribute to the formation of these layers alongside terrestrial processes (Bohor BF et al., 1987; Ortega-Huertas M et al., 2002). Focusing on the GBR, the prevailing hypotheses suggest that the formation was generated by the expulsion and subsequent sedimentation of acidic volcanic materials with source material derived from weathering processes (Wang NZ et al., 2019). However, Zhu LJ (1994) proposed that the GBR formed by the hydrolysis of acidic volcanic ash within a basinal environment, yielding a distinct pyro-claystone composition. Although the prevailing hypothesis suggests a volcanic origin for the GBR, the precise processes involved in the formation of this lithium-enriched unit and the source of the material within these sediments remain uncertain

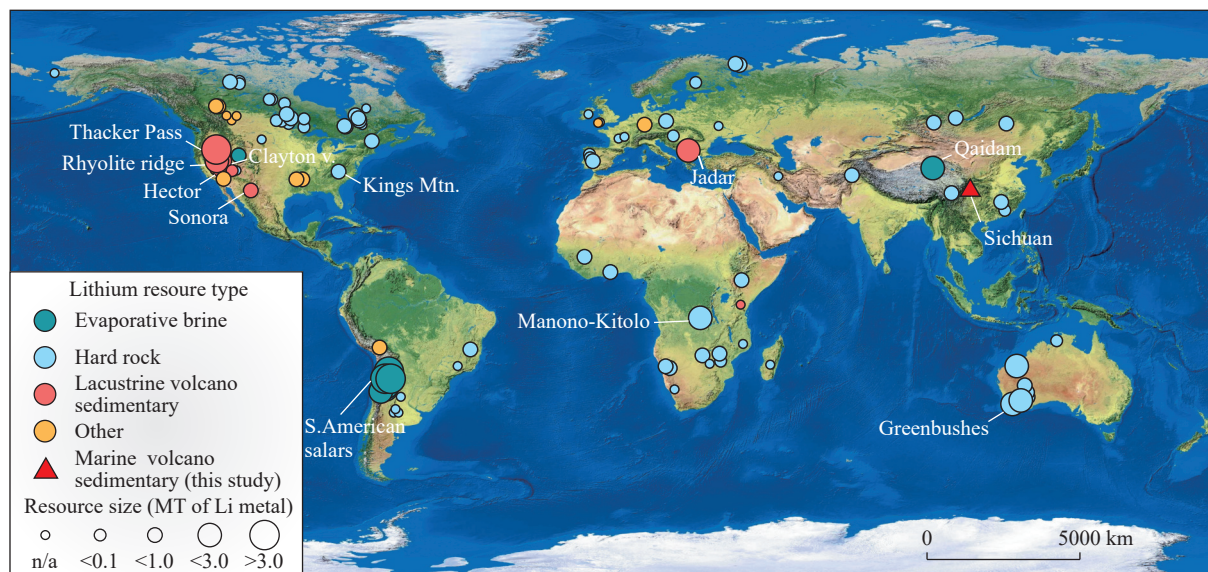


Fig. 1. Map illustrating the types and scale of Li resources (modified from Benson TR et al., 2023). This map highlights the significant resource potential of clay-type lithium deposits.

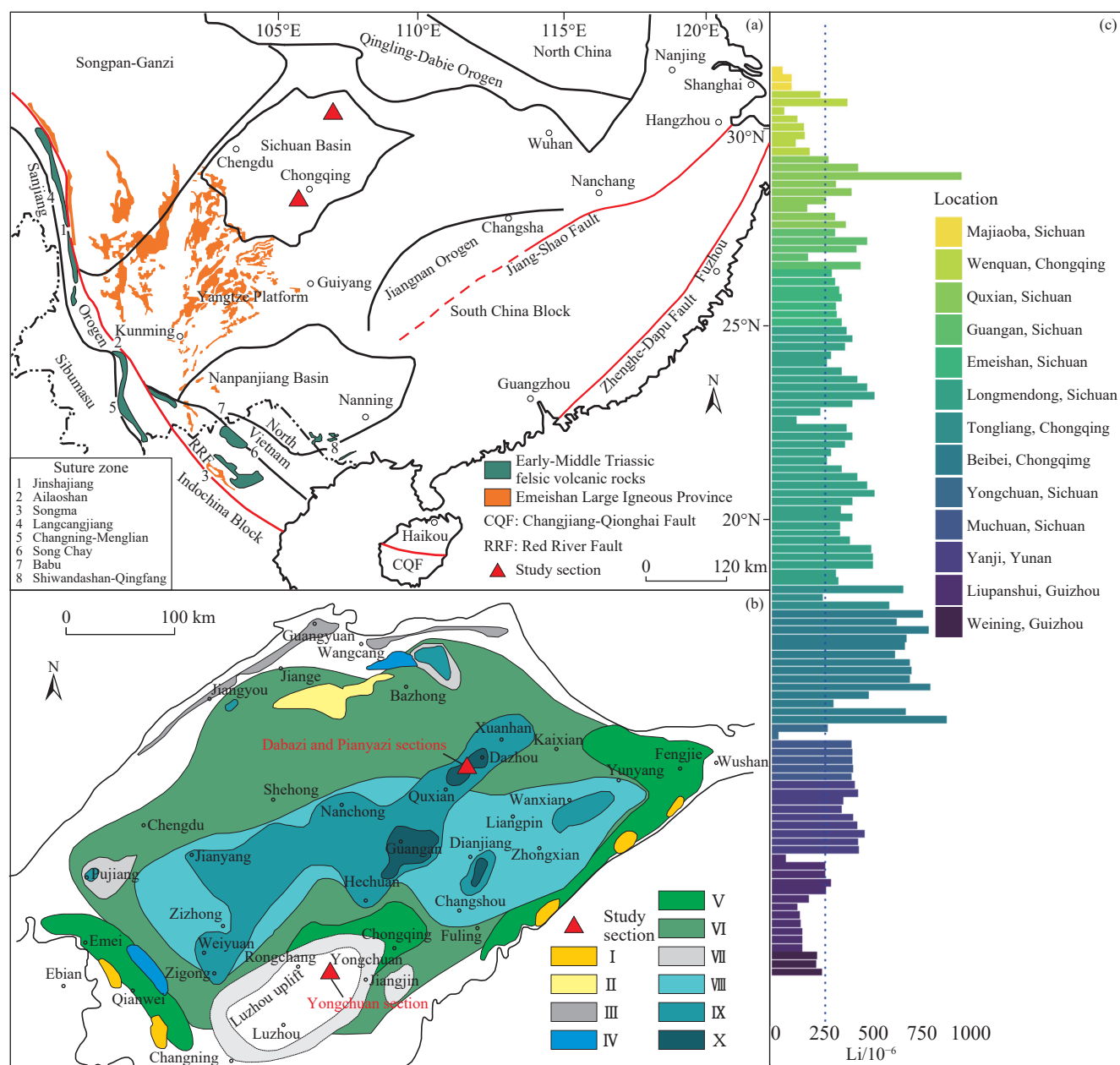


Fig. 2. a–Schematic geological map of the South China Block and the Emeishan Large Igneous Province (after Feng MS et al., 2021; Li C et al., 2021); b–Lower-Middle Triassic lithofacies paleogeography of the Sichuan Basin (after Gong DX et al., 2015). I –platform margin reef/beach; II –intraplatform shoal; III–grainy beach; IV–Lagoon behind the beach; V–paste lake; VI–intertidal flat; VII–dolomitic flat, gypsum-bearing dolomite flat; VIII–gypsum-bearing dolomite lagoon; IX–salt lake; X–salt lake center. c–Statistical analysis of lithium content in GBR across South China (Data are derived from Sun Y et al., 2018; Ma SC et al., 2019; Feng MS et al., 2021; Peng Y et al., 2023; Wang KM et al., 2023; Zhang B et al., 2023; Chen HF et al., 2024).

(Ovtcharova M et al., 2015; Feng MS et al., 2021; Zhang B et al., 2023).

The GBR also contrasts with typical volcanic clay-type lithium deposits in that it formed in a marine environment rather than in a lacustrine setting (Castor SB and Henry CD, 2020; Lin YJ et al., 2020; Feng MS et al., 2021; Benson TR et al., 2023). Sedimentary environments are a crucial control on the enrichment of Li as a result of their influence on the deposition and subsequent alteration processes affecting Li-bearing sediments (Castor SB and Henry CD, 2020; Benson TR et al., 2023). Li-rich clay minerals in volcanic clay-type lithium deposits are typically illite and illite/smectite (I/S)

mixed-layer minerals (Castor SB and Henry CD, 2020; Benson TR et al., 2023), yet their origins within a marine geological context remain uncertain. In addition, although some studies suggest that illite forms by diagenesis involving the hydrothermal alteration of volcanic ash and other precursor materials (Benson TR et al., 2017; Castor SB and Henry CD, 2020; Li C et al., 2021), alternative hypotheses outline processes such as metasomatic replacement or direct precipitation from hydrothermal fluids (Uysal IT et al., 2004; Park C et al., 2022). Understanding the relationship between the genesis of clay minerals and sedimentary environments is therefore crucial for unraveling the depositional history and

lithological evolution of volcanic clay-type lithium deposits, ultimately providing insight into their formation processes that can be used to enhance exploration targeting and further develop their economic significance.

This study focuses on clay-type Li deposits located within the well-preserved Sichuan Basin in southern China. This paper presents the results of an extensive mineralogical analysis of three representative profiles of the lithium-rich GBR within the basin, utilizing XRD and SEM approaches. The primary objectives are: (1) To discern the genesis of these deposits, differentiating between volcanic and weathering origins; (2) to conduct a comprehensive analysis of both clay mineralogy and associated non-clay minerals within the Li-enriched GBR; (3) to explore the factors governing the type and abundance of clay minerals present; and (4) to elucidate the formation and evolutionary processes that control the characteristics of the GBR throughout the Sichuan Basin. By strategically selecting representative sections, this work provides valuable insights into the geological processes and sedimentary environments during the deposition of the GBR. The comparative analysis of the differences in sedimentary facies between these sections also provides a deeper understanding of the processes involved in the development of this significant geological formation. Finally, based on these studies, discuss the prospecting potential of volcanic clay-type lithium deposits in the Sichuan Basin and identify potential target areas.

2. Geologic setting

The Sichuan Basin is located in the western part of the Upper Yangtze Plate (UYP) (Fig. 2a; Feng MS et al., 2021; Li C et al., 2021), and is bounded to the north by the Qingling-Dabie–Sula suture and the Tan-Lu Fault (Metcalf I, 2013) and to the south by the Nanpanjiang Basin. The basin is further bounded to the southwest by the Sanjiang Orogen and to the east by the Jiangnan Orogen (Lehrmann DJ et al., 2006; Metcalfe I, 2013). During the Proterozoic around 860 Ma the Yangtze Block merged with the Cathaysia Block along the Jiangnan suture to form the South China Block (Wang XL et al., 2012; Liu XF et al., 2024). Around 460 Ma, during the Early Paleozoic, the block collided with the western margin of Gondwana and was subsequently integrated into the supercontinent (Li SZ et al., 2018). During the Devonian, the South China Sea Plate broke apart from the northeast of Gondwana (Metcalf I, 2013), and drifted northward through the PaleoTethys Ocean. The northward subduction of the PaleoTethys Ocean led to collision with the Yangtze Plate with the North and South China Plates approaching each other between the Permian and the Triassic (Metcalf I, 2013), with the North China Plate eventually subducting under the South China Plate before the two plates were amalgamated along the Qinling-Dabie Orogen (Li ZX and Li XH 2007; Dong YP et al., 2012). The PaleoTethys Ocean and the Yangtze Plate underwent Early Permian-Middle Triassic collisional orogenesis (Zi JW et al., 2012), which was followed by the

Late Permian development of the Emeishan large igneous province in the southwest of the Yangtze Plate (Xu YG et al., 2004), with magmatism peaking at 259 Ma (Zhong YT et al., 2014). The tuff layer at the top of the basalt series indicates that there were still explosive volcanic eruptions occurring during the late evolution of the Emeishan large igneous province (251 ± 1.0 Ma) (Zhu J et al., 2011). During the Late Permian and Middle Triassic, weathered materials from the Yunkai Terrane and the Indochina uplift were transported into the Youjiang Basin, resulting in the formation of sedimentary units primarily composed of carbonate, clastic, and pyroclastic rocks (Chen JH et al., 2023; Liu XF et al., 2024).

The Early Triassic to Middle Triassic Jialingjiang Formation (JF, T_{1j}) records the restriction of the sedimentary environment in the study area as a result of westward sea retreat caused by the uplift of the northern margin of the UYP (Chen LQ et al., 2010; Zhang SL et al., 2018). By the end of the Middle Triassic, the whole Sichuan Basin was undergoing uplift during Episode I of the Indosinian Orogeny. The sea receded from the southern part of the basin during this period, exposing the Leikoupo Formation (LF, T_{2l}) to weathering and erosion for tens of thousands of years. Stratigraphically, the LF conformably overlies the Lower Triassic JF and is positioned beneath and parallel to the Upper Triassic Xujiahe Formation (XF, T_{3xj}) (He DF et al., 2011; Xu K and Gong HN, 2023). The JF within the UYP corresponds to the Early Triassic Olenikian stage and consists of a series of carbonates and evaporites some 300–1000 m thick (He DF et al., 2011). The stable UYP primarily served as a cratonic carbonate platform during the deposition of the Middle Triassic LF (corresponding to the Middle Triassic Anisian Stage), leading to the widespread deposition of limestone, dolomitic limestone, dolomite, gypsiferous dolomite, and gypsum (He DF et al., 2011; Xu K and Gong HN, 2023).

The GBR is a common green or yellow-green clay layer within Early and Middle Triassic sediments that is widespread across the Yangtze platform and the Nanpanjiang Basin in southern China, cropping out over an area of approximately 7×10^5 km² and occurring in beds ranging from dozens of centimeters to tens of meters in thickness (Xiao JF and Hu RZ, 2005; Lehrmann DJ et al., 2006; Ovtcharova M et al., 2015; Ma SC et al., 2019; Ju PC et al., 2020; Feng MS et al., 2021). The GBR is common within the Early-Middle Triassic sedimentary units of southern China, including the Binchuan section in the Chuan-Dian Oldland of Yunnan Province, the Bijie section within the western margin of the Yangtze Platform in Guizhou Province, the Jinya section in the Youjiang Basin of Guangxi Province, and the Tongliang section in the Sichuan Basin (Fig. 3).

3. Material and analytical methods

3.1. Sample collection and description

This study focuses on seven samples from the Sichuan Basin, South China, specifically from the Dabazi

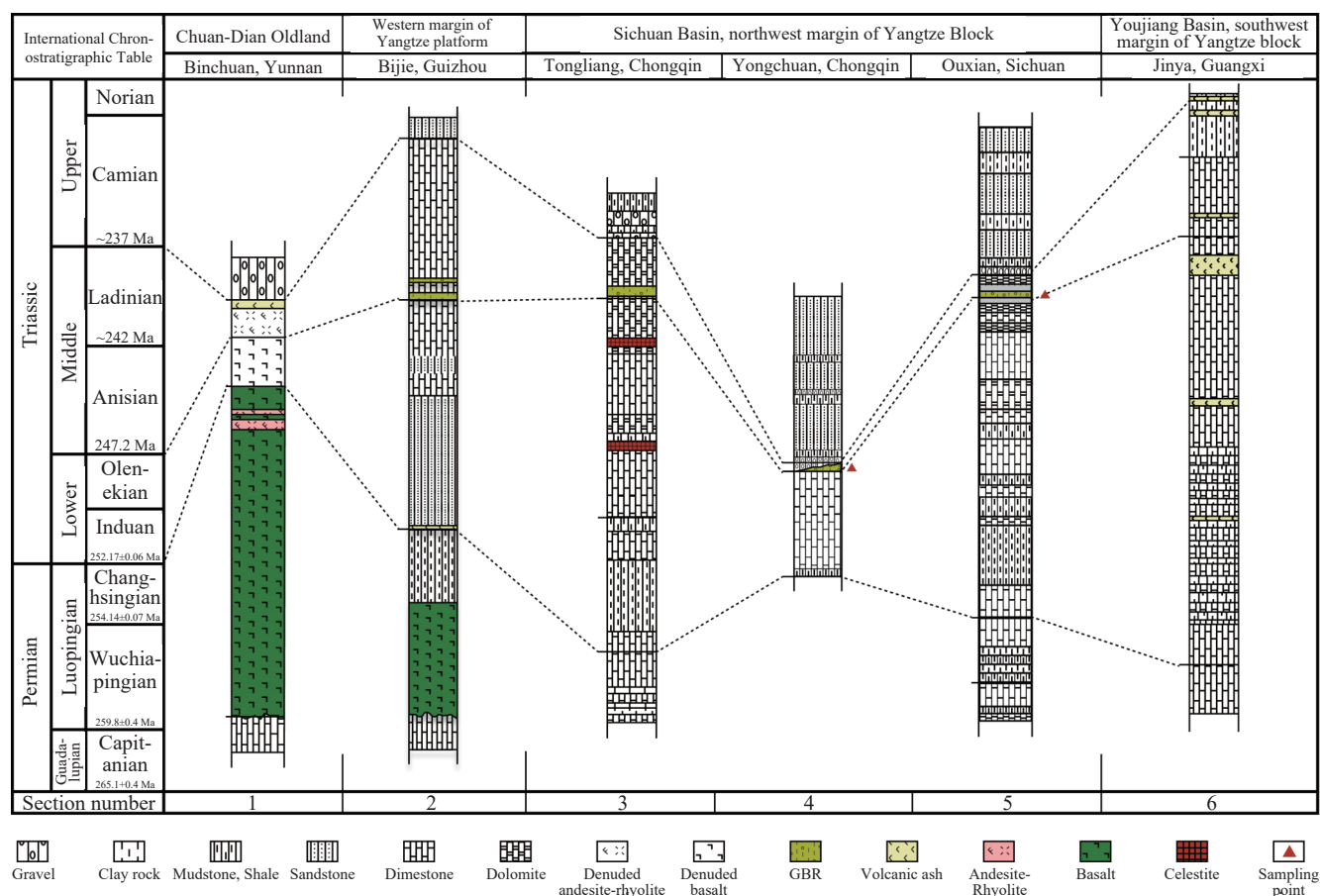


Fig. 3. Stratigraphic column map showcasing the studied section, encompassing the Yongchuan and Quxian regions (Dabazi and Pianyazi sections). The other columns provide comparisons to Lower-Middle Triassic boundary layers in southern China, including the Binchuan (after Lo CH et al., 2002; Xu YG et al., 2004; He B et al., 2007), Bijie (after Yang FQ et al., 2005; Ma SC et al., 2019), Tongliang (after Sun Y et al., 2017; Ma SC et al., 2019), and Jinya (after Ovtcharova M et al., 2006; Galfetti T et al., 2007; Huang H et al., 2012) sections. This comprehensive overview provides insights into the geological context and correlations within the specified timeframe.

(107°9'19"–107°9'40" E, 30°56'15"–30°56'53" N), Pianyazi (107°15' E, 30°50' N), and Yongchuan (105°52'54" E, 29°31' 55" N) sections. These selections were made deliberately to capture the distinct geological characteristics and depositional environments present in each section. The Dabazi and Pianyazi sections contain gypsum in both their upper and lower horizons, indicative of deposition in a closed sedimentary environment (Fig. 4). Conversely, the Yongchuan section is dominated by carbonate rocks, representing a distinctly different sedimentary environment. Both samples of Dabazi and Pianyazi area were collected from gypsum mines, where the gypsum layers are strongly folded. D2, D6, D7, and D11 are located within four small interlayers, with the strata from D2 to D11 gradually becoming older. In the Yongchuan section, YC3–1 is situated approximately 4 m above YC8, belonging to two different interlayers. The GBR layers in this study are all relatively thin, ranging from 20 cm to 30 cm. In field exposures, the GBR is yellow-green with a relatively unconsolidated texture and lacks distinct bedding. The clasts within the unit frequently have variable flattened bean-like shapes, with discernible diameters of 1–3 mm. During sampling, fresh specimens were chosen to minimize or eliminate the influence of surface weathering on analyses. Following collection,

samples were promptly stored in sealed containers to prevent contamination.

3.2. Characterisation of Li-rich clay minerals

The mineralogical investigations undertaken during this study focused on the comprehensive analysis of both the entire rock matrix and the clay mineral components within a variety of specimens prepared via the side-loading method. This analysis was facilitated by X-ray diffraction (XRD) utilizing a TTR-3 X-ray diffractometer from Rigaku Corp, Tokyo, Japan. The XRD evaluation involved the use of Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$) operating at a voltage of 45 kV and a current of 30 mA. To extract clay mineral fractions ($< 2 \mu\text{m}$), a centrifugation process was applied to powdered samples following the procedure outlined by Jackson ML and Abdel-Kader FH (1978). The XRD analyses were performed on variously treated clay samples, including air-dried oriented (AD), ethylene glycol-saturated (EG), and samples heated to 550°C. Mineral compositions and their respective proportions were determined using the Clayquan program (2016 version). This analysis relied on XRD data obtained from both non-oriented powdered samples and oriented clay samples.

The calculation of the *I/S* ratio employed the following

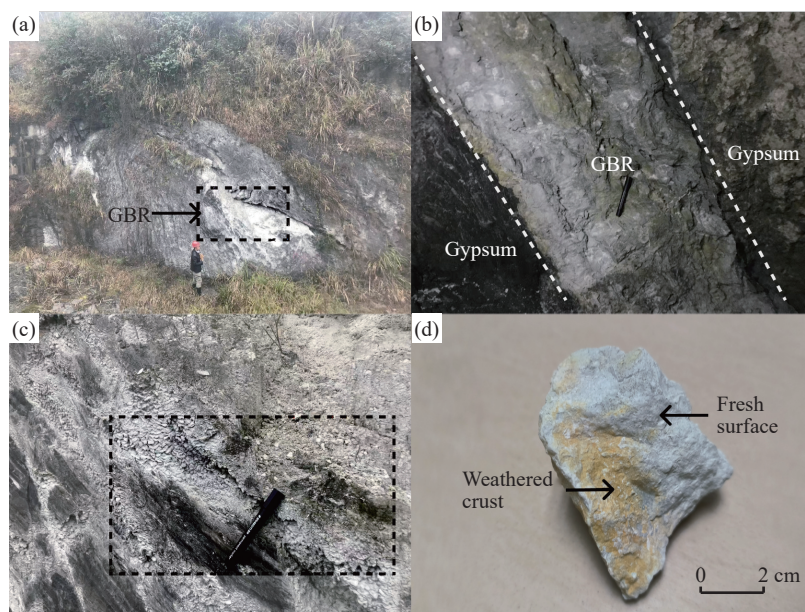


Fig. 4. Representative photographs of clay-type Li deposits within the Pianyazi (a) and Dabazi (b) sections; c–close-up photograph of the Pianyazi section; d–close-up photograph of a GBR hand specimen.

specific equation (Eq. 1):

$$\frac{I}{I+S} = \frac{I10(EG)}{I10(550) - I10(EG)} \quad (1)$$

where $\frac{I}{I+S}$ denotes the mineral content ratio of illite to the mixed layer I/S , $I10(EG)$ represents the peak area of the (001) peak in ethylene glycol-saturated clay samples, and $I10(550)$ indicates the peak area of (001) after the sample underwent heating to 550°C. A Nikon E600 POL polarizing microscope was also utilized for thin section optical microscopy.

3.3. Recovery and characterisation of microspherules and β -form quartz

All samples underwent a digestion process in 15% glacial acetic acid lasting from 2 weeks to 2 months to retrieve microspherules. Using a binocular microscope, microspherules larger than 30 μm were manually selected from the residue. The surface features of these microspherules were examined using an LEO 1530 resolution field emission scanning electron microscope (SEM). An Oxford Instruments energy dispersive X-ray analysis (EDX) system connected to the LEO 1530 was utilized to measure the main element components of different micro-areas on the surface of these microspherules. Element determinations (Na, Mg, Al, Si, S, K, Ca, Mn, Fe, Ba, Ni, Cr, Ti, P, and Cl) of the microspherules were carried out at an accelerating potential voltage of 15 kV and a probe current of 20 nA. To maintain standards, the American National Standards Institute (ANSI) provided references including hornblende (for Ti, Al, Fe, Na, and K), clinopyroxene (for Si, Mg, and Ca), fayalite (for Mn), $\text{Ba}_5\text{PO}_4\text{Cl}$ (for P and Cl), Cr_2O_3 (for Cr), and metallic nickel (for Ni). Corrections for matrix effects were applied using the ZAF software from JEOL. The accuracy of the reported

values ranges from 1% to 5% based on elemental concentrations.

The method employed for the separation of β -form quartz closely resembled the procedures outlined in (Vos K et al., 2014). Initially, 20 g of sediment from each sample was dried in an oven. Subsequently, the samples underwent a 10-minute boiling process in a solution of 15% HCl to remove carbonates and iron oxides. After this step, the samples were thoroughly washed with deionized water until the rinsing water turned clear. Following this, another 10-minute boiling treatment in a solution containing 50 g/L of tetrasodium pyrophosphate was conducted to eliminate clay, organic matter, and attached particles. The samples were again rinsed extensively with deionized water until the rinsing water became clear. Post-treatment, the samples were dried at 60°C and sequentially sieved using 600- μm , 300- μm , and 125- μm sieves for particle size fractionation. The surface features of these β -form quartz were examined using an LEO 1530 resolution field emission SEM.

3.4. Analysis of platinum group elements

The method employed for the separation of the platinum group elements (PGEs) involved tellurium coprecipitation with stannous chloride (Elson CM and Chatt A, 1983; Shazali I et al., 1987). The efficiency of this coprecipitation process in capturing PGEs is reported to be 98–100. Following coprecipitation, the resulting precipitate was filtered and dissolved in aqua regia, a solution comprising nitric acid and hydrochloric acid commonly utilized for metal dissolution (Elson CM and Chatt A, 1983). Subsequently, the dissolved precipitate was transferred to small centrifuge tubes containing 5 mL of 1.5 M HNO_3 solution for analysis (Shazali I et al., 1987). Analysis of solutions from a 5 g sample by ICP-MS (PerkinElmer) yielded detection limits of

0.1×10^{-9} for Pt and Pd; 0.01×10^{-9} – 0.05×10^{-9} for Rh, Ru, and Ir; and 0.2×10^{-9} for Au. Detection limits were calculated as three times the standard deviation for ten reagent blanks that were treated as samples. The precision of the technique, expressed as percent standard deviation for multiplicate analysis of USGS diabase standard W-1, is as follows: 13% (Pt), 7% (Pd), 6% (Ru), 0.7% (Ir), and 24% (Rh). The single Au analysis yielded 4.0×10^{-9} , close to the accepted value of 4.3×10^{-9} .

3.5. Lithium content analysis

The determination of Li concentrations was undertaken using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The sample pretreatment followed the methodologies outlined in the work of Yang XP et al. (2007). The precision of the analytical techniques was validated through the analysis of Chinese national standards GB/T 14506.14-2010 and GB/T 14506.28-2010 (Li Y et al., 2010; Wang S and Yan M, 2010), with a repeatability better than 5%.

4. Results

4.1. Whole-rock mineralogical characteristics

XRD analysis of bulk rock samples extracted from the Pianyazi and Dabazi sections within the northeast Sichuan Basin enabled the identification of distinct compositional differences when these samples were compared to the Yongchuan section (see Table 1). The bulk rock samples from Pianyazi and Dabazi are dominated by quartz, gypsum, and clay minerals (Fig. 5), contrasting with the absence of gypsum and the prevalence of calcite or dolomite in the Yongchuan section (Fig. 6a). Specifically, quartz abundance in the Pianyazi and Dabazi sections varies between 15.6% and 52.3%, whereas the Yongchuan section contains far less

quartz (1.6% to 4%). Furthermore, the maximum K-feldspar content observed in the Pianyazi and Dabazi sections is 16.4% (D11), significantly higher than the 0.3% to 0.5% present in the Yongchuan section. Gypsum was exclusively detected in the Pianyazi and Dabazi sections in abundances from 5.7% to 31.9%, whereas no traces of gypsum were found in the Yongchuan section. In addition, sample YC3-1 is dominated by dolomite, accounting for 90.2% of the sample, whereas

Table 1. Quantitative analysis results of bulk composition in the studied samples.

Sample	Quartz	K-Feldspar	Gypsum	Calcite	Dolomite	TCCM
D2	36.2	7.9	10.1	0	0	45.8
D6	52.3	8.2	11.5	0	0	28
D7	15.6	9.2	31.9	0	0	43.3
D11	20.6	16.4	16.6	0	0	46.4
PY2	42.9	5.6	5.7	0	0	45.8
YC3-1	4	0.5	0	90.2	0	5.3
YC8	1.6	0.3	0	5.9	90.9	1.3

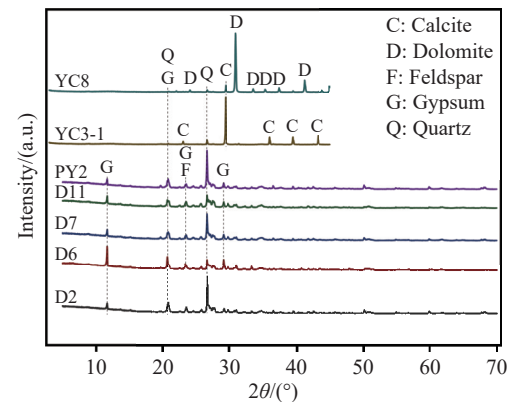


Fig. 5. Bulk rock XRD spectra of studied samples showing strong peaks of quartz in both Dabazi and Pianyazi samples, a strong calcite peak in YC3-1, and a strong dolomite peak in YC8.

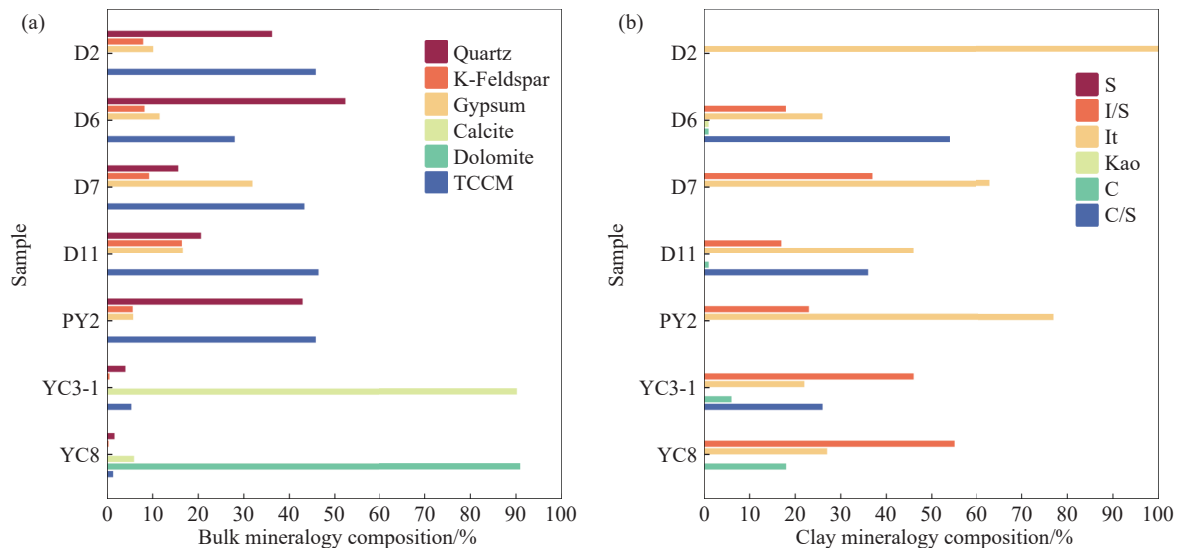


Fig. 6. Quantitative analysis results of bulk (a) and clay mineralogy (b) composition in the studied samples. Panel (a) shows the proportions of bulk minerals, including quartz, K-feldspar, gypsum, calcite, dolomite, and total content of clay minerals (TCCM). Panel (b) illustrates the clay mineral compositions, featuring smectite (S), illite/smectite (I/S), illite (It), kaolinite (Kao), chlorite (C), and chlorite/smectite (C/S), with distinct variations across different samples.

YC8 is primarily composed of calcite, constituting 90.9% of the sample. The clay minerals vary in color from yellow-green to yellow-brown and contain illite with a felt-like structure (Figs. 7a–d). The bean-like grains are predominantly composed of quartz (Figs. 7g–h) and the composition of these grains is dominated by chalcedony, with angular to sub-angular shapes prevailing (Figs. 7e–f).

4.2. Clay mineral assemblages

The XRD analysis of clay minerals within the bulk samples reveals distinctive mineral compositions across the studied sections (Fig. 8). Smectite is notably absent, with minimal amounts of kaolinite identified. Illite and I/S are the primary clay minerals (Table 2), with the exception of sample D2, which contains 100% illite (Fig. 6b). Excluding D2, the samples contain 17%–55% I/S. Clay samples from the Pianyazi and Dabazi sections contain negligible chlorite,

whereas the Yongchuan samples contain trace amounts (6% and 18%, respectively). Chlorite/smectite mixed-layer minerals (C/S) are present in only half of the samples, with abundances that vary between 26% and 54%. Swelling clays like I/S were identified through a comparison of XRD patterns of AD- and EG-oriented clay preparations, with illite-like peaks around $10^\circ 2\theta$ and additional peaks resembling smectite at 7° – $8^\circ 2\theta$ indicative of the presence of I/S.

The I/S interlayering ratio in the Dabazi and Pianyazi section samples ranges from 0% to 15%, except for samples D2 and YC3-1, and YC8 from the Yongchuan section, which have ratios of 70% and 15%, respectively. Based on the relationship between the I/S interlayering ratio and order degree (Eberl D and Hower J, 1976), the Dabazi and Pianyazi section samples report a consistent ordered R3 I/S structure within their I/S composition. In contrast, the Yongchuan section contains clays with two distinct structures: A random I/S structure in sample YC3-1 and an ordered R1 I/S structure

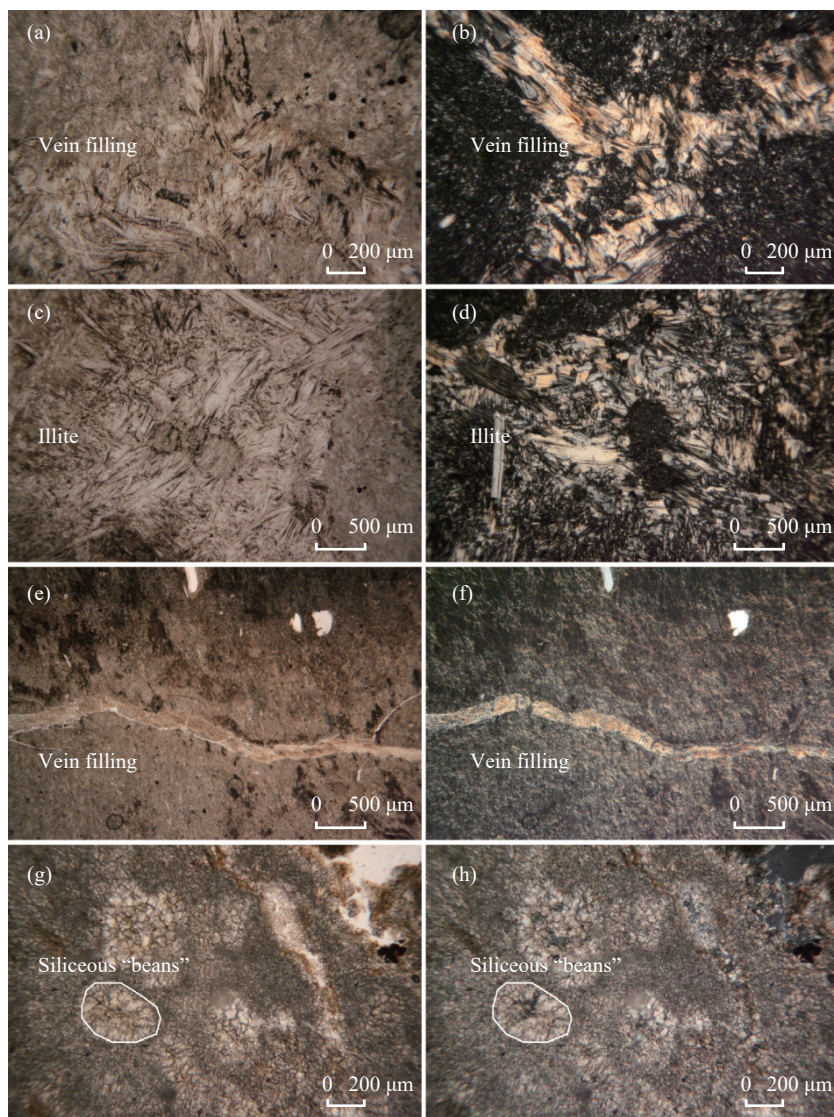


Fig. 7. Representative examples of quartz within samples from the study area. Quartz bands in sample D2 under plane-polarized light (a) and cross-polarized light (b). Illite in sample D6 under plane-polarized light (c) and cross-polarized light (d). Quartz bands in sample PY2 under plane-polarized light (e) and cross-polarized light (f). Quartz grains in sample YC3-1 under plane-polarized light (g) and cross-polarized light (h).

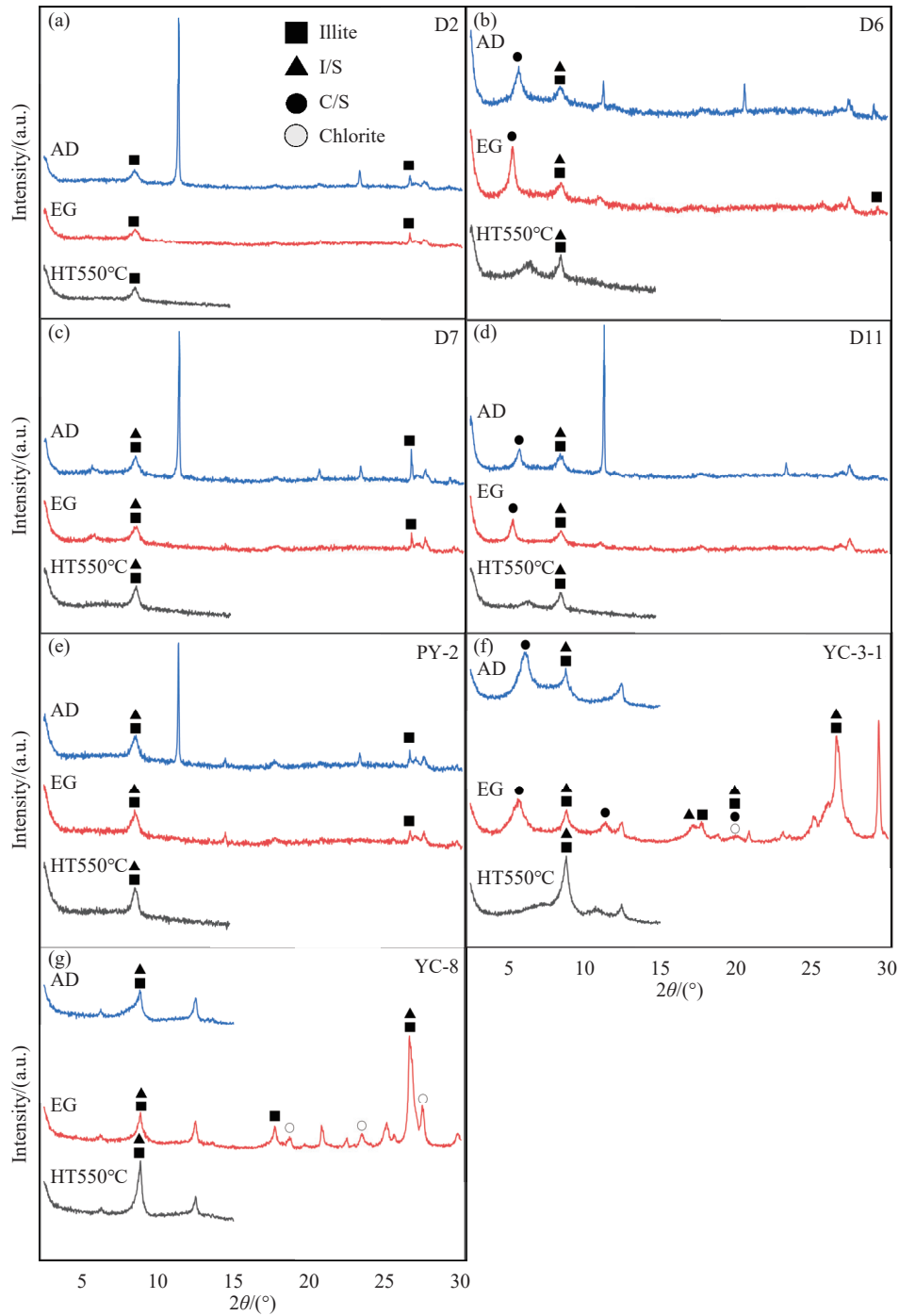


Fig. 8. XRD pattern of clay minerals in the studied samples.

Table 2. Quantitative analysis results of clay mineral composition in the studied samples.

Sample	Clay mineral/%					Mixed-layer ratio/%	
	I/S	It	Kao	C	C/S	I/S	C/S
D2	/	100	/	/	/	/	/
D6	18	26	1	1	54	5	47
D7	37	63	/	/	/	5	/
D11	17	46	/	1	36	5	46
PY2	23	77	/	/	/	8	/
YC3-1	46	22	/	6	26	70	49
YC8	55	27	/	18	/	15	/

in sample YC8.

4.3. Characterisation of β -form quartz

Natural quartz crystals have three principal faces, $m\{100\}$, $r\{101\}$, and $z\{011\}$, and quartz crystals have different crystal forms at different temperatures. Iwasaki H and Iwasaki F (1995) mapped the growth of quartz crystals according to the different growth rates of different crystal faces (Fig. 9). In this study, the authors identified high-temperature quartz paramorphs (β -form quartz) within the samples from the representative sections chosen for analysis.

The β -quartz paramorph crystals present in the samples typically have a well-defined hexagonal bipyramid shape (the purple one in Fig. 9), are colorless, transparent, and have a glassy luster, with particle sizes ranging between 200 μm and 250 μm (Figs. 10a–c). The relatively rough surface of these β -quartz paramorphic crystals is indicative of formation in a siliceous sedimentary environment. These crystals appear gray or milky white with a glassy sheen and have a fracture resembling a greasy luster, possibly indicating incomplete crystal formation as a result of volcanic eruptive activity before full crystal growth could occur. Another type of quartz with a smaller Rm/Rr value and longer crystal structure is also

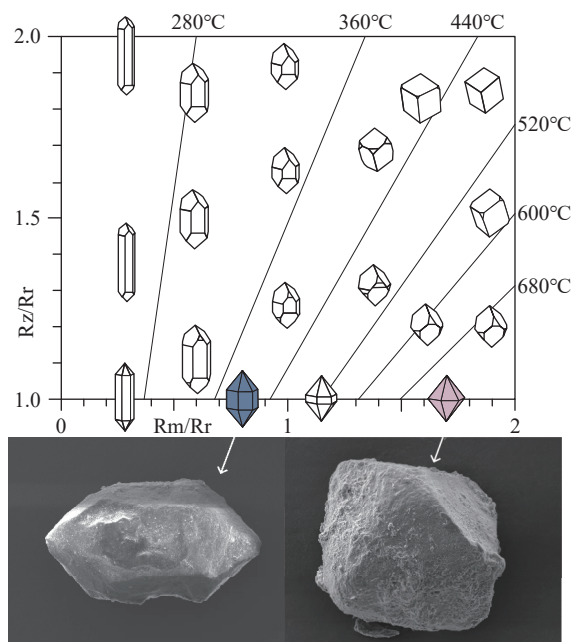


Fig. 9. Genetic diagram of quartz with different crystal types (Rm/Rr : Growth rate of m/growth rate of r; Rz/Rr : Growth rate of z/growth rate of r; modified from Iwasaki H and Iwasaki F, 1995).

present in the samples from the study area (Fig. 10c). This is not β -quartz, but the nature of this quartz also suggests a certain level of exposure to high-temperature conditions (the blue one in Fig. 9). However, no shocked quartz related to impact events was identified.

4.4. Characterisation of iron microspherules

The iron microspherules identified in the three examined sections are remarkably spherical with complex surface textures. Their crystal structure resembles that of terrapin shells with a porous arrangement. These microspherules range in size from 100 μm to 1000 μm and look like dark opaque spheres with a metallic luster when viewed under a polarizing microscope (Fig. 11). The iron content in these microspherules ranges from 18.32% to 50.37% (see Table 3). Chromium is also present in the majority of samples, ranging from 0.01% to 0.31%. The Cr/Fe ratio varies between 5.15% and 61.54%.

4.5. Lithium content and PGE concentrations

In this study, all of the GBR samples except YC8 (33.2×10^{-6}) contained relatively high lithium concentrations. The lithium concentrations of samples D2, D6, D7, D11, PY2, and YC3-1 were 401×10^{-6} , 275×10^{-6} , 179×10^{-6} , 319×10^{-6} , 375×10^{-6} , and 282×10^{-6} , respectively. The PGE concentrations determined during this study are significantly variable compared to expected concentrations for chondrites, the Primitive Mantle, and the continental crust (McDonough W and Sun SS, 1995). Samples YC3-1 and YC8 have low PGE concentrations, with samples YC3-1 and YC8 containing low concentrations of Pt (0.23×10^{-9} and 0.1×10^{-9} , respectively), significantly lower than typical chondrite values (around 1010×10^{-9} ; Fig. 12a). Samples YC3-1 and YC8 are also depleted in Ir relative to chondritic values, with concentrations of 0.01×10^{-9} in both samples compared to

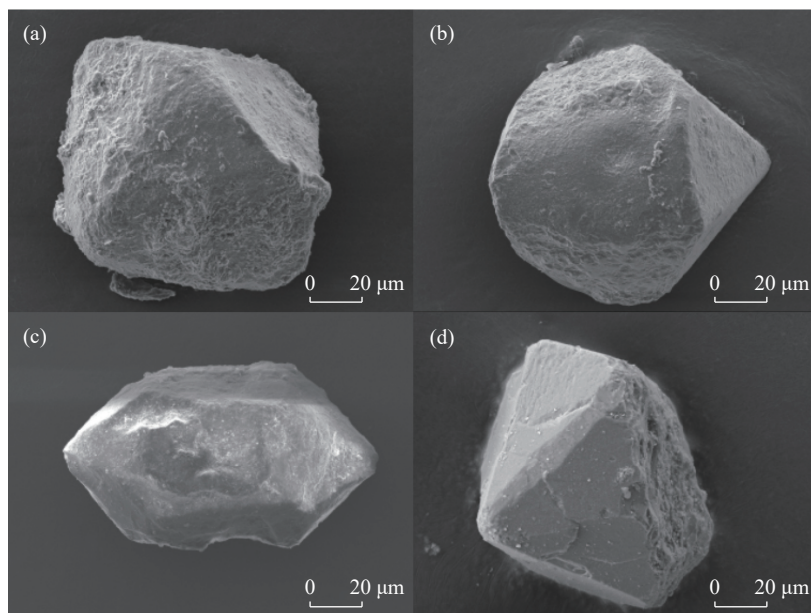


Fig. 10. SEM images of β -form quartz of the study area; β -form quartz typically retains a well-defined hexagonal bipyramidal shape.

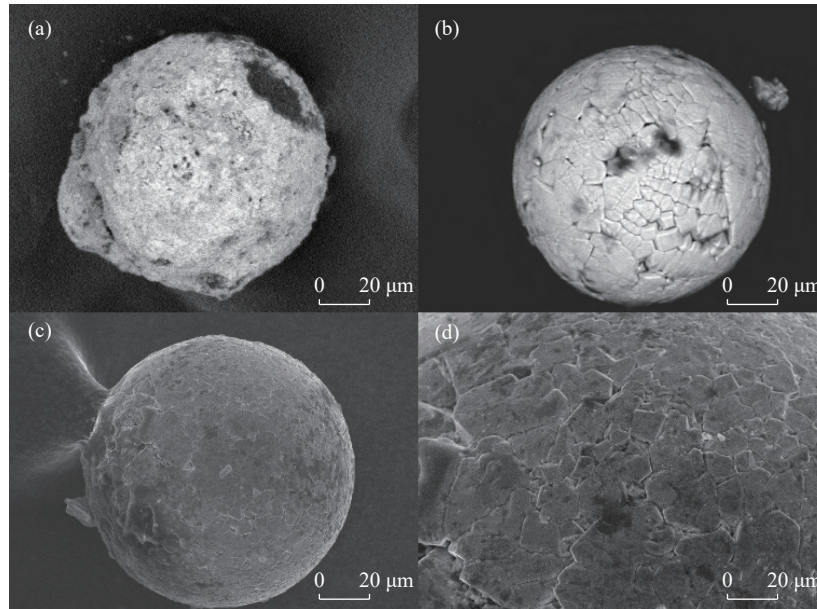


Fig. 11. SEM images of microspherulites from the samples analyzed during this study.

Table 3. Chemical composition of microspherulites (%).

Sample	Fe	O	Si	Mg	Ca	Al	Cu	Mn	Zn	Ni	Ti	Cr	Co	Cr/Fe×10 ⁴
M-1	44.28	47.52	3.61	1.98	2.44	/	/	/	/	/	0.06	0.12	/	27.10
M-2	18.32	71.74	0.88	1.07	1.23	0.29	/	0.37	/	/	/	/	/	/
M-3	44.68	45.34	0.45	2.98	0.75	0.92	0.5	/	2.96	1.4	/	0.01	/	2.24
M-4	35.14	60.16	0.1	2.23	/	/	/	/	2.16	/	0.21	/	/	/
M-5	50.37	44.78	/	/	/	/	2.29	/	/	/	0	0.31	1.64	61.54
M-6	27.46	68.4	2.08	/	0.19	1.63	/	/	/	/	0.24	/	/	/
M-7	19.41	78.73	0.79	1.38	/	0.08	/	/	/	/	0	0.01	/	5.15
M-8	34.1	59.55	1.86	1.07	3.29	/	/	/	/	/	0.12	/	/	/
M-9	45.59	45.9	4	0.55	2.57	0.19	/	/	/	/	0.24	0.23	/	50.45

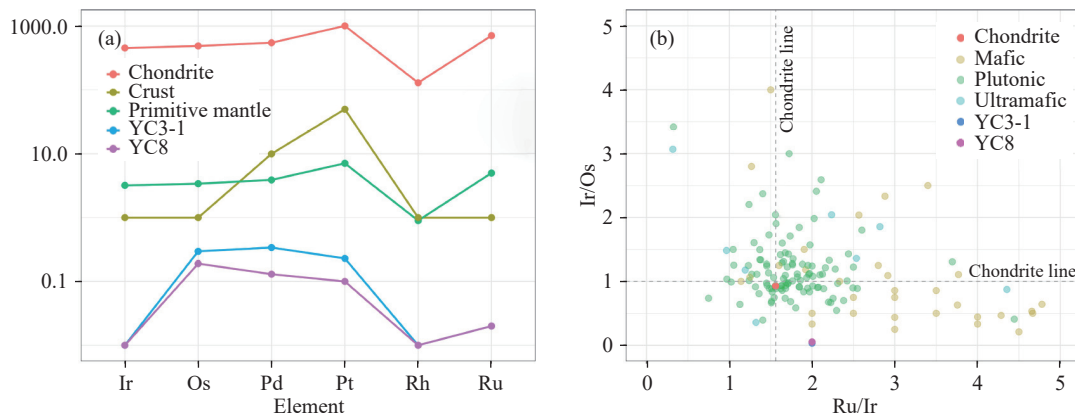


Fig. 12. a–Comparison of PGEs concentrations within the GBR samples with expected values for chondritic compositions, the continental crust, and the primitive mantle (after McDonough W and Sun SS, 1995); the vertical axis in Fig. a is on a logarithmic scale of element concentrations; b–variations in Ru/Ir and Ir/Os ratios for GBR samples, volcanic rocks from the study area (Table S1), and the expected value for chondritic compositions (after McDonough W and Sun SS, 1995).

typical chondrite values of 455×10^{-9} . These samples are also similarly depleted in the other PGE (Pd, Os, Ru, and Rh) relative to concentrations expected for chondrite, Primitive Mantle, and crustal compositions. In addition, the Ru/Ir and Ir/Os ratios of samples Y3-1 and YC8 are significantly lower than the values expected for chondrites and those typically found in volcanic rocks (Fig. 12b).

5. Discussion

5.1. Mineralogical evidence for the volcanic origin of Li-rich clays of the Sichuan Basin

Clay layers within marine sedimentary packages are thought to form as a result of various depositional processes,

which include asteroid impacts (Bohor BF et al., 1987; Ortega-Huertas M et al., 2002), volcanic events (Hong HL et al., 2019), changes in marine settings (Jeans CV et al., 1982; Drits VA et al., 2004), and weathering processes (Righi D and Meunier A, 1995; Velde B and Meunier A, 2008). In the context of the GBR, previous research has primarily linked the clays within this unit to acidic and intermediate acidic volcanic eruptions using the overall geochemical composition of the GBR (Sun Y et al., 2018; Ma SC et al., 2019; Ju PC et al., 2020; Feng MS et al., 2021). However, the geochemical composition of clay-dominated rocks can be significantly influenced by late diagenesis and diagenetic environments, often leading to element mobility and changes to the original composition of the clay-rich sediments. In this study, the discovery of β -quartz and iron microspherules has provided crucial insights into the origin of claystone originally formed within a marine setting. β -quartz, also known as high-temperature quartz, represents a polymorph of low-temperature quartz (α -quartz) that is typically indicative of derivation from quartz phenocrysts originally located within acidic and intermediate acidic volcanic exhalative rocks, consistent with the association between the source material of the GBR unit and volcanic activities (Iwasaki H and Iwasaki F, 1995; Wang SY and Yin HF, 2002). The identification of β -quartz paramorphs further corroborates this association, indicating that the clays have undergone minimal transportation after deposition and suggesting they were derived from a proximal source.

The combined analysis of morphological features and geochemical characteristics also provides insights into the source of the iron microspherules within the samples from the study area. The iron microspherules within this region have characteristics that are indicative of possible impact, cosmic, and volcanic origins. Morphologically, they share similarities with impact and cosmic microspherules but they are more geochemically similar to volcanic microspherules, as evidenced by their elemental compositions and Cr/Fe ratios (Zhang H et al., 2014). The absence of cores enriched in certain elements and their low concentrations of Ni, Cr, and

Co also support a volcanic origin (Smit J and Kyte FT, 1984; Huang C et al., 2012). Furthermore, the low PGE concentrations of the samples analyzed during this study also support a genetic model for the GBR that does not involve an extraterrestrial impact. In summary, the data presented in this study indicates that the marine GBR claystone formation is genetically associated with intermediate to felsic volcanism.

5.2. Formation of the GBR within the Sichuan Basin

5.2.1. Lithium in GBR is related to illitization

The clay minerals in sedimentary lithium deposits possess strong adsorption and ion exchange capacities, making them the primary host minerals for lithium. In Li-rich claystone, Li-bearing minerals primarily include smectite, chlorite, illite, and kaolinite (Castor SB and Henry CD, 2020; Benson TR et al., 2023; Ling KY et al., 2024). In this study, Fig. 13b displays the Li content of GBR samples and the changes in Li content concerning the TCCM, I/S, and illite. Meanwhile, the authors performed a Pearson correlation analysis on Li content, TCCM, I/S, and illite in the GBR and found strong correlations of lithium with the other three variables (Pearson K, 1895), with correlation coefficients of 0.6542051, -0.8214220, and 0.5817350, respectively (Fig. 13a). The strong positive correlation between Li and TCCM indicates that lithium in the GBR is predominantly hosted within clay minerals. The strong negative correlation between Li and I/S suggests that the degree of illitization has a significant impact on the content of Li in the GBR. Moreover, in this study, the GBR samples were found to be nearly devoid of smectite and kaolinite, with only a few samples containing trace amounts of chlorite. Coupled with the strong positive correlation between Li and illite, it can be inferred that illite is the predominant mineral hosting Li in the GBR.

5.2.2. Formation of illite and I/S and Lithium enrichment

I/S minerals are commonly found in low-temperature geological settings and represent intermediate products in the transformation from smectite (S) to illite (I). This study focuses on the GBR, which contains I/S minerals believed to

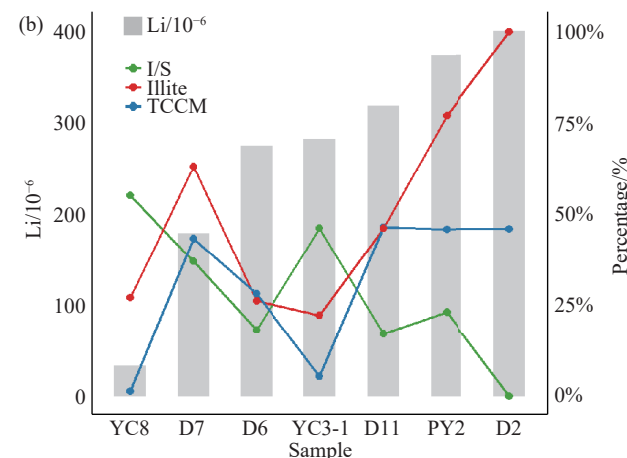
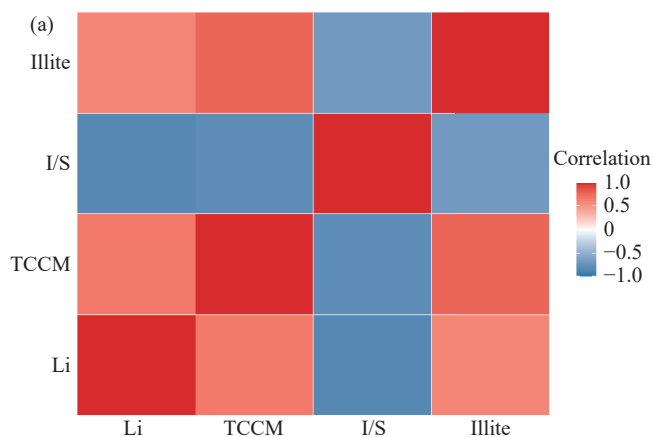


Fig. 13. a–Heatmap showing the correlation between Li content, TCCM, I/S, and Illite; b–Li content of GBR samples and the changes in Li content concerning TCCM, I/S, and illite.

have formed during a period of near-constant dacitic to rhyolitic volcanism (Lin YJ et al., 2020; Feng MS et al., 2021). During the Early and Middle Triassic periods, the Sichuan Basin incorporated significant amounts of volcanic ash and pyroclastic materials originating from various sources like the Ancient Tethys Ocean to the west, the Qinling Ocean to the north, and the volcanoes associated with Indosinian tectonism to the south side of the South China Plate (Lehrmann DJ et al., 2006; Huang H et al., 2012; Gong DX et al., 2015; Wang NZ et al., 2019). Over time, these materials underwent diagenesis, transforming into the GBR and its constituent clay minerals such as smectite and chlorite (Ma SC et al., 2019; Ju PC et al., 2020; Lin YJ et al., 2020; Feng MS et al., 2021). In this kind of closed basin, the dissolved tuff generally forms authigenic Li-rich Mg-smectite (Gagnon CA et al., 2023). Throughout this diagenesis, which occurred over time periods between several days to several years, a continuous influx of potassium (K^+) and magnesium (Mg^{2+}) ions from seawater were added to the GBR. As a result, the smectite within the GBR underwent a gradual transformation into random I/S minerals. With increasing burial depth, temperature, and time, the GBR interacted with seawater and deep brines. At the same time, the random I/S evolved into an ordered structure and eventually transformed into illite (Fig. 14; Lin YJ et al., 2020). The process of illitization typically leads to an increase in lithium content, primarily because this process reflects the degree and duration of contact between the rock and brines. The longer the contact time, the more complete the adsorption of lithium ions by the clay minerals (Castor SB and Henry CD, 2020; Benson TR et al., 2023). The transformation from random I/S to ordered I/S and the transformation from smectite to illite are the result of

prolonged geological processes that affected the GBR. Previous research extensively documented and supported the mineralogical changes observed in similar geological settings elsewhere, providing insights into the processes driving the conversion of smectite into illite within sedimentary sequences (Gong N et al., 2018).

5.2.3. Sedimentological controls on clay mineral assemblages

The clay mineral assemblages within sedimentary rocks are linked to sedimentological processes, reflecting the environmental conditions prevalent during deposition. For instance, in the McDermitt Volcanic Field, the predominant clay mineral is lithium-rich smectite. Although illite has a higher lithium content, it is only found in the southern half of the caldera at Thacker pass and adjacent to the northern part of the Montana Mountains, indicating that the sedimentary environments of these two clay minerals are distinctly different (Castor SB and Henry CD, 2020; Benson TR et al., 2023). In this study, the authors identified distinct variations in clay mineral compositions between lacustrine and marine depositional environments. Specifically, the lacustrine facies in more confined evaporative environments have a higher abundance of illite compared to the marine facies (Fig. 15). This disparity in illite content reflects contrasting sedimentological conditions and depositional settings between these two environments. The key factors influencing smectite illitization include temperature, time, and the chemical composition of pore fluids (K^+), as outlined by previous studies (Eberl D and Hower J, 1976; Turner CE and Fishman NS, 1991). In the study, since all samples share a similar age, the changes in temperature and K^+ concentrations within the pore fluids is key controls on the transformation of smectite

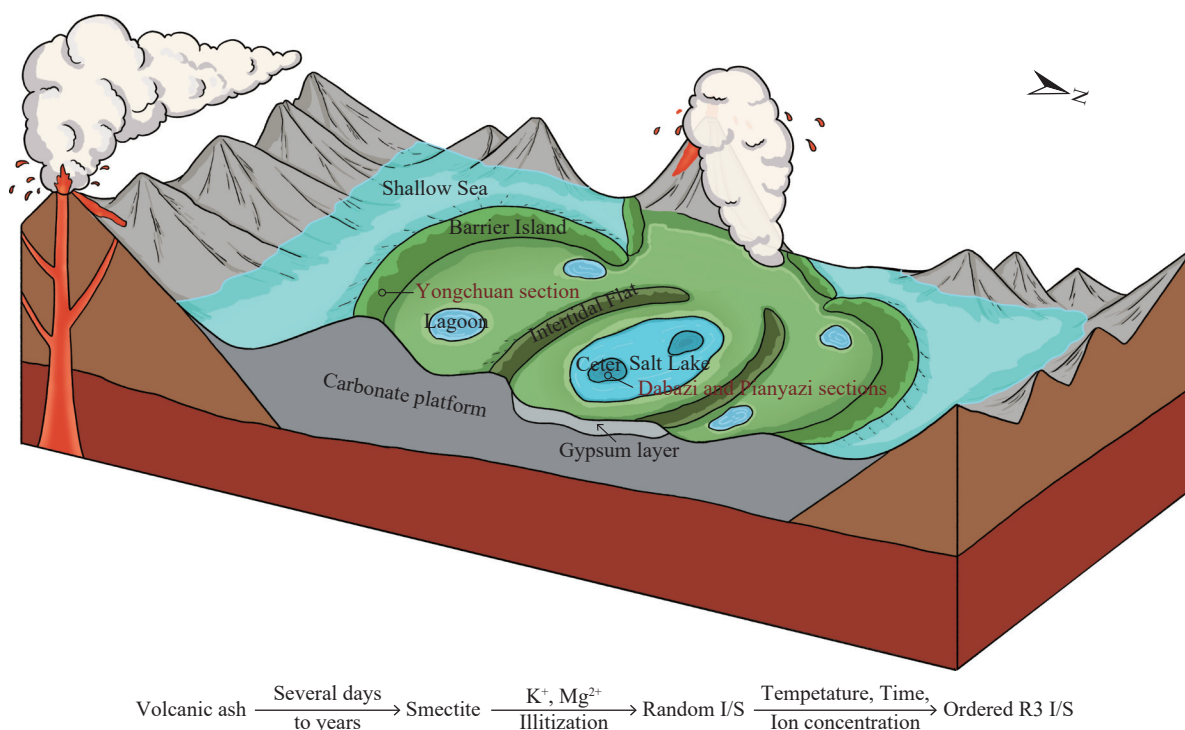


Fig. 14. Schematic diagram of T_1/T_2 GBR formation in Sichuan Basin showing the difference of sections in this study.

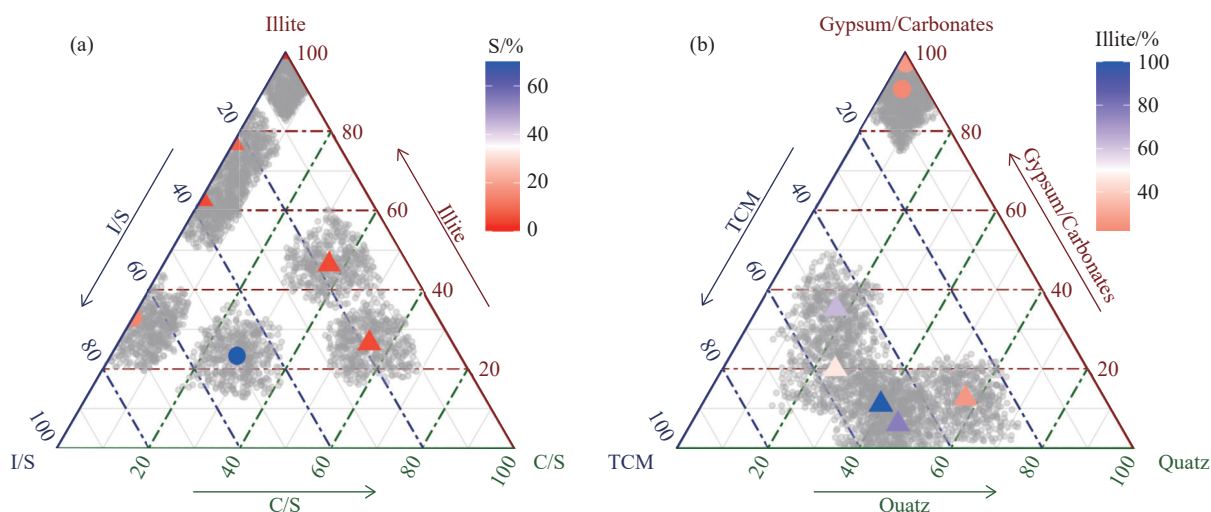


Fig. 15. a–Ternary illite-I/S-C/S plot of studied GBR; b–ternary gypsum/carbonates-TCM-quartz plot of studied GBR (circles represent marine facies, triangles represent lacustrine facies) with points simulated by Monte Carlo method.

into illite. The variation in K content is clearly a significant factor influencing the degree of smectite illitization between lacustrine and marine facies sedimentary environments (Eberl D and Hower J, 1976; Turner CE and Fishman NS, 1991; Huang WL et al., 1993). In lacustrine environments, where sedimentation occurs in relatively enclosed settings, higher K availability from terrestrial inputs or internal cycling within the water promotes smectite transformation to illite. K^+ also has a crucial role in determining the crystalline structure of clay minerals, facilitating the formation of illite (Huang WL et al., 1993). Consequently, the higher K content in lacustrine settings accelerates illitization, leading to elevated illite contents in the resulting sediments and clay mineral assemblages. In addition, smectite and illite are more abundant in low-energy settings like marine shelves or lacustrine environments, indicative of prolonged chemical weathering and diagenetic alteration under relatively quiescent conditions. Conversely, marine environments, predominantly influenced by seawater, have lower K concentrations. The hydrodynamic forces prevalent in marine settings also contribute to K dilution and transport, resulting in reduced potassium content sediments. This means that the lower potassium levels in marine facies likely impede the conversion of smectite to illite, leading to comparatively lower illite content. Thus, potassium content emerges as a key factor driving the disparity in smectite illitization observed between lacustrine and marine facies.

In this study, the main minerals within samples from the Yongchuan section are calcite and dolomite, indicative of a marine sedimentary environment. In comparison, samples from the Dabazi and Pianyazi sections contain gypsum, indicative of formation in a lacustrine sedimentary environment (Gong DX et al., 2015). Past studies have highlighted the pivotal role of these factors in the conversion from smectite to illite in various rock formations (Eberl D and Hower J, 1976; Turner CE and Fishman NS, 1991). The significant differences in sedimentary facies between the northeastern and southern Sichuan Basin influence the

mineral composition of the GBR in these different parts of the basin. For example, the rocks overlying and underlying the GBR in the Yongchuan section are carbonates, whereas the Dabazi and Pianyazi sections contain gypsum (Fig. 14). The salinity disparity arising from variations in the depositional sedimentary environments is the main control on the different degrees of illitization observed in this study. This relates to the fact that the elevated salinity in marine sedimentary environments, exemplified by differences in salt and K^+ concentrations, significantly influences the illitization process. This reflects the impact of salinity and K^+ on solution chemistry and mineral growth rates. This is demonstrated by the Dabazi section, where samples D6, D7, and D11 have similar levels of illitization despite a slight increase in depth between samples, with all three samples containing clay minerals with ordered R3 I/S structures. In addition, the shallowest sample in the Dabazi section, namely sample D2, contains clay minerals that have already undergone the complete transformation from smectite to illite, despite being within 10 m of sample D6 and with a negligible temperature difference between these samples (around 0.2°C , within the northeast Sichuan's geothermal gradient of approximately $20^\circ\text{C}/\text{km}$; Lu QZ et al., 2005; Li XL et al., 2020). This indicates that temperature differences are unlikely to account for these variations in degree of illitization, suggesting that the primary factor, as mentioned above, is likely to be changes in salinity. The sedimentary period of D2 corresponds to a period of regression within the Sichuan Basin within a hot, dry climate (Parrish JT, 1993; Qian LJ et al., 2010), causing extensive evaporation and a subsequent decrease in the salt lake's water level without seawater replenishment. This means that the salt lake's salinity likely increased at this time, accelerating the illitization process in sample D2 relative to sample D6 due to the higher K^+ concentrations within the water associated with this process.

The Pianyazi section is located within 200 m of the Dabazi section and contains sample PY2, which records a higher degree of illitization compared to the samples from the

Dabazi section expect D2. This most likely reflects differences in temperature and salinity, with sample PY2 likely representing the transgressive phase associated with waters with higher K^+ concentrations than those associated with the samples from Dabazi. There is also variable illitization recorded within the Yongchuan section, where sample YC3-1 is less illitized than sample YC8 despite the former being 5 m shallower than the latter, again suggesting that temperature differences are an unlikely control on this variation in illite development. This again suggests that these differences could be related to changes in sedimentary facies and environment. The Yongchuan section is located within the Luzhou Uplift and records a period of regression (Gong DX et al., 2015). After early diagenesis in the marine sedimentary environment, this regression caused YC3-1 to spend some time above sea level, associated with the deposition of continental sedimentary material, whereas YC8 remained near or below sea level, maintaining a marine sedimentary environment with continued K^+ recharge. This distinct sedimentary setting can explain the varying degrees of illitization between YC3-1 and YC8.

5.3. Mineral exploration potential

Volcanic clay-type lithium deposits exhibit substantial reserves and ease of extraction, positioning them as high-quality, novel lithium resources. The lithium concentrations in the study samples range from 179×10^{-6} to 401×10^{-6} , with the exception of YC8, underscoring the mineral exploration potential. The findings indicate that the strong correlation between lithium content and illitization within the GBR highlights the critical role of this process in lithium enrichment. Exploration strategies should prioritize regions where conditions favor the transformation of smectite to illite, as this process is likely to yield higher lithium concentrations. Sedimentological research and the identification of areas conducive to illitization and the formation of lithium-rich clay minerals can significantly enhance exploration efforts. Closed salt lake environments, exemplified by the Dabazi and Pianyazi sections in this study, may serve as focal points for future exploration endeavors. Peng Y et al. (2023) reported a lithium concentration of 957×10^{-6} in green bean rock from Quxian, and the Quxian sample in this study also exhibits a high lithium content, suggesting substantial potential for lithium resource exploration in the northeastern Sichuan region.

6. Conclusion

This study explores the origin and formation process of Li-rich claystone in the Sichuan Basin, and presents new mineralogical evidence supporting the volcanic origin of the Li-bearing GBR formation within the basin. Using SEM and EDX analyses, the authors identified volcanic microspherules and high-temperature quartz paramorphs within the GBR, enabling the classification of the clay-type lithium resources within the Sichuan Basin (including the GBR) as volcanic

clay-type lithium resources. The mineralogical analysis undertaken using XRD approaches during this study indicates that the GBR in northeast Sichuan contains gypsum and clay minerals, whereas the GBR in the southern Sichuan Basin contains carbonate rocks and a small amount of clay minerals. The illitization of clay minerals within the GBR in the northeastern Sichuan Basin is higher than the contemporaneous GBR sediments in the southern part of the basin, possibly reflecting differences in sedimentary depositional environments. The composition of clay minerals within these sediments also indicates that clay-type lithium deposits are likely to have undergone alteration from smectite to illite during diagenesis. This alteration is likely the result of the submarine modification of volcanic materials, leading to the subsequent formation of I/S as a result of the illitization of volcanogenic smectite. This comprehensive study not only establishes a classification for the volcanic clay-type lithium resources in the Sichuan Basin but also provides insights into the formation processes of Li-rich claystones, the primary carrier of Li within both the Sichuan Basin and other regions globally. Understanding these differences in sedimentary facies and their controls on variations in the degree of illitization provides key insights and a definitive basis for further research into the processes associated with lithium enrichments in clay and sedimentary lithium deposits globally. The study demonstrates that the Sichuan Basin holds significant potential for volcanic clay-type lithium resources, particularly in the northeastern Sichuan area, contributing to lithium exploration efforts.

CRedit authorship contribution statement

Rui Su, Yong-jie Lin and Weng-hui Huang conceived of the presented idea. Rui Su, Yong-jie Lin and Weng-hui Huang carried out the experiment. All authors discussed the results and contributed to the final manuscript.

Declaration of competing interest

The authors declare no conflicts of interest.

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Supplementary dataset

Supplementary data (Table S1) to this article can be found online at doi: [10.31035/cg2024140](https://doi.org/10.31035/cg2024140).

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