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# Petrography and geochemistry characteristics of the lower Cretaceous Muling Formation from the Laoheishan Basin, Northeast China: implications for provenance and tectonic setting

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Abstract The petrography, mineralogy and geochemistry of sedimentary rocks from the lower Cretaceous Muling Formation  $(K_1ml)$  in the Laoheishan basin, northeast (NE) China are studied to determine the weathering intensity, provenance and tectonic setting of the source region. Petrographic data indicate the average quartz-feldspar-lithic fragments (QFL) of the sandstone is  $Q = 63$  %,  $F = 22$  %, and  $L =$ 15 %. Lithic fragments mainly contain volcanic clasts that derived from surrounding basement. X-ray diffraction (XRD) data reveal abundant clay and detrital minerals (e.g. quartz), as well as minor calcite in the fine-grained sediments. The Hf contents and element concentration ratios such as  $Al_2O_3/TiO_2$ , Co/Th, La/Sc, and La/Th are comparable to sediments derived from felsic and intermediate igneous rocks. The strong genetic relationship with the igneous rocks from the northwest and northeast areas provides evidence that the sediments of the Muling Formation  $(K_1m)$  in the Laoheishan basin have been derived from this area. The chemical index of alteration (CIA) and index of chemical variability (ICV) reveal an intensive weathering in the source region of the sediments. The multidimensional tectonic discrimination diagrams indicate that the source rocks of  $K_1$ ml are mainly derived from the collision system. However, they may also

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comprise sediments derived from the continental rift system. The results are consistent with the geology of the study area.

Keywords Provenance . Tectonic setting . Petrography . Geochemistry . Laoheishan basin . Northeast China

## Introduction

The geochemical composition of clastic sediments has been widely used to decipher the provenance (Madhavaraju and Lee [2010](#page-13-0); Migani et al. [2015](#page-13-0); Odoma et al. [2015](#page-13-0)), to evaluate the weathering history of the source area (Zaid [2013;](#page-14-0) Rahman et al. [2014\)](#page-14-0), and to infer the tectonic settings (Roser and Korsch [1986;](#page-14-0) Armstrong-Altrin [2015\)](#page-12-0). The geochemical characteristics of clastic sediments are controlled by several factors, such as the source rock composition, weathering intensity, sedimentation and post-depositional diagenesis (Nesbitt and Young [1982](#page-13-0); McLennan [1989\)](#page-13-0). Recent studies (Armstrong-Altrin et al. [2014](#page-13-0); Zhang et al. [2014](#page-14-0)) indicated that trace elements like Y, Sc, Th, Zr, Hf, Cr, Co and rare earth elements (REE) are only negligible affected by chemical weathering, denudation, and aggradation. Therefore, these elements can be used to in provenance studies and to determine the tectonic settings of sedimentary basins (Cullers et al. [1987;](#page-13-0) Condie et al. [1992;](#page-13-0) Holail and Moghazi [1998;](#page-13-0) McCann [1998\)](#page-13-0). However, because of multiple factors controlling the geochemical characteristics of sedimentary rocks, combined petrological and geochemical analyses should be applied to reveal the provenance and tectonic setting of a sedimentary basin (Hanson et al. [2001](#page-13-0); Zhang et al. [2012](#page-14-0); Xu et al. [2013a](#page-14-0), [b\)](#page-14-0).

The Laoheishan basin is located in the eastern Heilongjiang Province, northeast China (Fig. [1\)](#page-1-0), and contains abundant coal and oil shale resources. Previous studies were focused only on the calculation of coal resources, the provenance and tectonic

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Fig. 1 a Sketch map showing the geotectonic of China (Lv et al. [2015](#page-13-0)), **b** tectonic units of northeast China showing the position of the Xingkai massif (Wu et al. [2007;](#page-14-0) Xu et al. [2012\)](#page-14-0), c geological map of the

setting of clastic sediments has not been studied. In this study, the petrological and geochemical analyses are carried out on the clastic sediments of the Muling Formation to infer paleoweathering, source rock characteristics, and tectonic setting.

# Geological setting

Northeast China is formed by several micro-continental blocks separated by major faults. The micro-continental blocks are (from NW to SE) the Erguna, Xing'an, Songliao, Jiamusi and Xingkai blocks (Fig. 1a, b) (Wu et al. [2007\)](#page-14-0).

Laoheishan basin and the sampling location, d profiles of the Laoheishan basin showing the basin structure

The Laoheishan basin is situated in the Xingkai Block composed of Precambrian metamorphic basement, partly covered by carbonate, clastic and volcanic rocks of Paleozoic and Mesozoic age (Shi [2006;](#page-14-0) Xu et al. [2012](#page-14-0)). The basin covers an area of approximately 400 km<sup>2</sup>. Outcrops of the Cretaceous basin fill are very rare and occur only in northwestern part of the basin (Fig. 1c). The central and southeastern parts of the basin are covered by extensive and thick basalt layers of the Tertiary Chuandishan Formation (Fig. 1c, d).

The Lower Cretaceous basin fill is about 400 m thick and dips southeastwards with less than 5°. It comprises the Muling Formation and the overlying Dongshan Formation. The Muling Formation is subdivided into two members. The lower member is composed of conglomerate with interbedded sandstone and mudstone  $(K_1ml_1)$ . In contrast, the upper member  $(K_1ml_2)$  consists of mudstone, sandstone and limited conglomerate, with several coal and oil shale layers (Fig. 2).

#### Samples and analytical methods

Samples for the study were collected from the new-drilled well NY1, which located in the basin center (Fig. [1c\)](#page-1-0) with large thickness of  $K_1$ ml (total thickness 277.22 m). The samples were taken in  $K_1ml_2$  (total thickness 263.46 m), from 121 to 372.8 m interval and the sample location was indicated in Fig. [3](#page-3-0). Twenty-four sandstone samples have been selected for petrographic study (Fig. [3](#page-3-0); Table [1](#page-4-0)) and 8 fine-grained sediments (Fig. [3](#page-3-0); Table [2\)](#page-4-0) were analyzed for the mineral composition. Additionally, 11 (Fig. [3;](#page-3-0) Table [3\)](#page-5-0) and 35 fine-grained sediments (Fig. [3;](#page-3-0) Tables [4](#page-6-0) and [5](#page-7-0)) have been selected for major elements, trace and rare earth elements analyses, respectively. For mineral composition and geochemical analyses, samples were ground in an agate mortar and stored in the glass capillary to prevent oxidation prior to analysis.

For petrographic investigations, 24 thin sections were prepared following the procedure by Camuti and McGuire ([1999](#page-13-0)). The point counts were done using Dickinson (Dickinson, [1985\)](#page-13-0) and standard methods. In each section, we used the 300-grain-count practice originally from Dryden [\(1931\)](#page-13-0), for total quartz  $[Q = \text{monocrystalline quartz } (Qm) +$ polycrystalline quartz  $(Qp)$ ], total feldspar  $[F = K-field$ spar  $(K)$  + plagioclase feldspar (P)] and total lithic fragments  $[L = volume(Lv) + sedimentary(Ls) + metamorphic$ (Lm) rock fragments] (Dickinson [1970;](#page-13-0) Ingersoll et al. [1984](#page-13-0)), the results were presented in Table [1](#page-4-0). The mathematical background for point-counting statistics is shown in Howarth ([1998](#page-13-0)), and the statistical error is  $\pm$ 5 vol%. The practical procedures are given in Howarth and French ([1998](#page-13-0)).

X-ray diffraction analyses were conducted on a Philips PW 1830/40 device using CuK $\alpha$ -radiation (1.54 Å, 35 kV, 35 mA). Scans with a step size of 0.02。were run between 2 and 67。2θ in the air-dry state (random powder mount and oriented powder mount) and between 2 and 45。2θ in the ethylene- glycoliated state and after heating to 350 and 550。C. Qualitative processing was done using the dataset of Brindley and Brown ([1980](#page-13-0)) and Moore and Reynolds [\(1997\)](#page-13-0). Quantitative interpretation was carried out using the methods described by Schultz ([1964](#page-14-0)).

The major element compound content was determined using a Philips PW2404 X-ray fluorescence (XRF) spectrometer. Powdered samples were heated to 110。C for 6 h followed by heating in a muffle furnace at 1000。C for two hours to determine LOI (loss on ignition). Lithium tetraborate was mixed with the samples and heated to 1000。C to form a fused samples for XRF analysis. An international reference standard material (JB-2) was used to check the accuracy that was better than  $\pm$ 5 % and the precision of the measurement based on

Fig. 2 Stratigraphic division of the Laoheishan basin



<span id="page-3-0"></span>

Fig. 3 Lithologic log of well NY-1, visualizing sampling depth and principal investigations performed

duplicate was within the acceptable limit. The results are reported in Table [3](#page-5-0), CIA refers to the chemical index of alteration, which proposed by Nesbitt and Young [\(1982\)](#page-13-0), defined as CIA =  $100 \times Al_2O_3$  / (Al<sub>2</sub>O<sub>3</sub> + CaO<sup>\*</sup> + Na<sub>2</sub>O + K<sub>2</sub>O), and CaO\* refers only to the CaO in siliceous minerals. ICV refers to the index of chemical variability, proposed by Cox et al. ([1995](#page-13-0)), defined as  $ICV = (Fe<sub>2</sub>O<sub>3</sub> + K<sub>2</sub>O + Na<sub>2</sub>O + CaO +$  $MgO + MnO + TiO<sub>2</sub>)/Al<sub>2</sub>O<sub>3</sub>.$ 

Trace and rare earth elements were determined from solutions prepared by homogenized samples powder dissolved in reagent grade HF: HNO<sub>3</sub> acid mixture in screwtop vessels. A test portion (0.05 g) of samples was added to 25 ml pressure decomposition vessels. Matrix matching international certified reference materials MAG-1 (marine mud, United States) and GSR-4 (sandstone sediment, China) along with couple of procedural blanks were also prepared with the sample batch by adopting the same protocol described above to negate errors due to reagent and handling. Solutions were analyzed by high resolution inductively coupled plasma mass spectrometer (HR-ICP-MS) (Thermo Scientific X-Series). The MAG-1 was used for calibration. The analytical precision for all the elements was estimated to be <5 % based on duplicate analyses of samples and standards. The results are reported in Tables [4](#page-6-0) and [5](#page-7-0).

# **Results**

## Petrography

The sandstones from the lower Cretaceous Muling Formation comprise three components: framework gains, cements, and pores. Most of the framework grains are medium sorted, subangular to sub-round in shape and are texturally moderately mature (Armstrong-Altrin et al. [2015b\)](#page-13-0), mainly composed of quartz  $(Q)$ , feldspar  $(F)$  and rock fragments  $(L)$ , with a few heavy minerals and matrix (Table [1](#page-4-0)). The average ratio of Q-F-L is  $Q_{63}$ -F<sub>22</sub>-L<sub>15</sub>, indicate the compositionally moderately mature.

Quartz is the most abundant grain component in the sandstones and can be subdivided into two groups: monocrystalline quartz (Qm) and polycrystalline quartz (Qp) (Table [1;](#page-4-0) Fig. [4a, b](#page-8-0)). Qm contains nonundulose and undulose grains, indicating low-grade metamorphism (Basu et al. [1975\)](#page-13-0); and most Qp grains consist of more than three crystals of two types: 1) more than five elongated irregular crystals with crenulated intercrystal boundaries (Fig. [4a](#page-8-0)); 2) five or more crystals with straight to slightly curved inter-crystals boundaries (Fig. [4b\)](#page-8-0). The first type indicating a metamorphic origin (Asiedu et al. [2000](#page-13-0)) is less in the study area, whereas the second type is predominant, indicating an igneous origin (Tucker [2009\)](#page-14-0).

<span id="page-4-0"></span>Table 1 Modal point-count data of sandstone samples from the Laoheishan basin

Sample	Depth (m)	Granularity*	$Qm$ (vol $%$ )	Qp	Q	K	${\bf P}$	$\rm F$	Lv	Ls	Lm	L
$NY-B-1$	167	Medium	48	5	53	24	12	36	6	$\overline{0}$	5	11
$NY-B-2$	177	Coarse	34	14	48	19	14	33	14	$\mathbf{0}$	5	19
$NY-B-3$	188	Coarse	49	8	57	9	11	20	17	$\mathbf{0}$	6	23
$NY-B-4$	190	Silt	57	5	62	17	$\tau$	24	9	$\mathbf{0}$	5	14
$NY-B-5$	198	Fine	59	7	66	10	14	24	5	$\mathbf{0}$	5	10
$NY-B-6$	200	Fine	65	5	70	6	14	20	5	$\boldsymbol{0}$	5	10
$NY-B-7$	201	Coarse	34	18	52	15	18	33	9	$\mathbf{0}$	6	15
$NY-B-8$	202	Coarse	60	6	66	5	11	16	13	$\mathbf{0}$	5	18
$NY-B-9$	204	Coarse	29	15	44	14	13	27	17	5	7	29
$NY-B-10$	205	Coarse	26	14	40	15	23	38	14	$\mathbf{0}$	8	22
$NY-B-11$	206	Coarse	28	17	45	9	20	29	18	$\mathbf{0}$	8	26
$NY-B-12$	212	Coarse	61	12	73	6	9	15	$\overline{7}$	$\mathbf{0}$	5	12
$NY-B-13$	218.5	Medium	68	5	73	10	5	15	$\overline{7}$	$\mathbf{0}$	5	12
$NY-B-14$	241	Medium	54	6	60	$\tau$	9	16	16	$\mathbf{0}$	8	24
$NY-B-15$	242	Coarse	57	7	64	9	8	17	13	$\mathbf{0}$	6	19
$NY-B-16$	246	Fine	69	$\overline{0}$	69	15	6	21	5	$\mathbf{0}$	5	10
$NY-B-17$	248	Medium	73	$\mathbf{0}$	73	10	6	16	6	$\mathbf{0}$	5	11
$NY-B-18$	266	Medium	69	6	75	9	5	14	6	$\boldsymbol{0}$	5	11
$NY-B-19$	270	Medium	63	5	68	12	6	18	8	$\mathbf{0}$	6	14
$NY-B-20$	284	Fine	68	$\overline{0}$	68	9	10	19	$\overline{7}$	$\mathbf{0}$	6	13
$NY-B-21$	287	Coarse	70	6	76	7	7	14	5	$\mathbf{0}$	5	10
NY-B-22	293	Fine	73	$\overline{0}$	73	6	11	17	5	$\overline{0}$	5	10
$NY-B-23$	305	Medium	58	8	66	9	13	22	$\overline{7}$	$\boldsymbol{0}$	5	12
$NY-B-24$	331	Medium	68	6	74	6	7	13	$\tau$	$\boldsymbol{0}$	6	13

Abbreviations of grain parameters (after Dickinson [1970;](#page-13-0) Ingersoll et al. [1984](#page-13-0)): Qm, monocrystalline quartz; Qp, polycrystalline quartz including chert; Q, total quartzose grains  $(Q = Qm + Qp)$ ; P, plagioclase feldspar; K, K-feldspar; F, total feldspar (F = P + K); Lv, volcanic rock fragments; Ls, sedimentary rock fragments; Lm, metamorphic rock fragments; L, total lithic fragments (L = Lv + Ls + Lm)

\* Coarse,  $\langle 2 \rangle$  0.5 mm; medium,  $\langle 0.5 \rangle$  0.25 mm; fine,  $\langle 0.25 \rangle$  0.1 mm; silt,  $\langle 0.1 \rangle$  0.03 mm

Feldspar in the sandstones mainly comprise alkali feldspar (Fig. [4a](#page-8-0)) and plagioclase (Fig. [4d\)](#page-8-0). Most feldspar grains are unaltered, although some grains are altered to sericite. Orthoclase is common in alkali feldspar, whereas microcline is very rare.

Lithic fragments in the sandstones include volcanic (Lv), sedimentary (Ls) and metamorphic (Lm) rock fragments. Volcanic rock fragments comprise felsic igneous rock (such as granite, Fig. [4c](#page-8-0)) and intermediate igneous rock (such as

Table 2 Mineralogy of the samples from the Laoheishan basin as determined by XRD

Sample	Depth $(m)$	Lithology	Clay minerals $(wt\%)$					Detrital minerals $(wt\%)$	Other mineral		
			Smectite	$\rm{I/S}$	Illite	Kaolinite	Chlorite	Ouartz	K-feldspar	Plagioclase	$(wt\%)$ Calcite
$NY-X-1$	121	Silty mudstone	$\overline{0}$	29	16	13	9	30	3	$\mathbf{0}$	$\mathbf{0}$
$NY-X-2$	126.5	Silty mudstone	$\overline{0}$	19	9	10	11	32	5	14	$\theta$
$NY-X-3$	162.5	Silty mudstone	$\mathbf{0}$	11	13	11	8	34	7	16	$\theta$
$NY-X-4$	183.5	Silty mudstone	26	21	9	4	3	26	3	9	$\theta$
$NY-X-5$	195.5	Silty mudstone	53	$\Omega$	3	4	$\mathbf{0}$	40	$\mathbf{0}$	$\mathbf{0}$	$\theta$
$NY-X-6$	343	Silty mudstone	$\mathbf{0}$	14	5	28	11	29	5	9	0
$NY-X-7$	365	Mudstone	$\mathbf{0}$	9	3	38	13	37	$\theta$	$\Omega$	0
$NY-X-8$	372.8	Mudstone	$\mathbf{0}$	19	3	40	9	28	$\theta$	$\Omega$	

Abbreviation: I/S, illite/ smectite mixed layer

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andesite, Fig. [4d\)](#page-8-0); metamorphic rock fragments mainly consist of Qp; sedimentary fragments include sandstone and mudstone. The lithic fragments in the sandstones are predominantly Lv, indicating that the Muling Formation are mainly derived from igneous rock sources.

A limited content of heavy minerals is observed  $\leq$  2 % on average) in the sandstones, including zircon, rutile and tourmaline. The matrix of the sandstones consist of clay minerals, carbonate, and communited lithic fragments. However, the existence of pseudomatrices makes it difficult to distinguish the primary and the epigenetic matrix (Dickinson [1970\)](#page-13-0). Hence, the percentage of matrix may be overestimated.

The diagenesis of sandstones comprise compaction and cementation processes, the characteristics are as follows: 1) contact relation between detrital grains includes point and line contacts (Fig. [4](#page-8-0)); and 2) compaction deformation occurred in plastic grains (such as biotite) (Fig. [4b\)](#page-8-0). The cementation mainly consist of carbonate cementation, usually formed in the primary porosity (Fig [4a\)](#page-8-0).

# Mineralogy

The XRD study indicates that the fine-grained sediments of  $K_1$ ml<sub>2</sub> mainly consist of clay minerals and detrital minerals with little other minerals (Table [2\)](#page-4-0). The clay minerals on average comprise smectite (10 %), I/S mixed layers (15 %), illite  $(8\%)$ , kaolinite  $(19\%)$  and chlorite  $(8\%)$ . The detrital minerals comprise quartz (32 %), K-feldspar (3 %) and plagioclase (6 %), whereas the content of other mineral is very low (calcite  $<1$  %).

# Geochemistry

#### Major elements

Major elements concentrations in sedimentary rocks are controlled by several factors, one of the most important factors is the mineral composition (Zhang et al. [2014\)](#page-14-0). For example, the  $K_2O/Na_2O$  ratio of sedimentary rocks is controlled by the relative proportion of K-feldspar to plagioclase, and the content of  $SiO<sub>2</sub>$  is controlled by the abundance of quartz. Furthermore, weathering can also influence the major elements concentrations in sedimentary rocks. For instance, Na in weathered crust could be carried away more easily than K, causing a strong deficit of Na and an abnormally high  $K_2O$ / Na 2O ratio; and the SiO <sup>2</sup> content may increase abruptly through a hydrothermal or surface weathering process (Cullers et al. [1987](#page-13-0)). The  $K_2O/Na_2O$  ratio of fine-grained sediments from the Muling Formation ranges from 0.22 to 1.18, with an average of 0.74 (Table 3), lower than the upper continental crust  $(UCC = 0.9)$ , indicating these samples are

<span id="page-6-0"></span>Table 4 Trace element concentrations of the samples from the Laoheishan basin as determined by HR-ICP-MS

Sample	Depth $(m)$	Lithology	$Co$ (ppm)	Ni	V	Cr	<b>Sr</b>	Rb	Ba	Th	Sc	Nb	Ta	Zr	Hf	Y	U
$NY-W-1$	121	Silty mudstone	8.0	26	117	70	188	156	490	22.0	14.1	15.3	1.29	121	3.72	20.4	7.10
$NY-W-2$	126.5	Silty mudstone	13.3	32	98	81	216	123	500	16.1	13.0	16.0	1.27	172	5.17	26.6	4.34
$NY-W-3$	144.8	Silty mudstone	19.9	17	96	58	262	124	642	20.8	11.6	16.2	1.29	186	5.40	29.8	5.62
$NY-W-4$	146.2	Silty mudstone	32.7	28	110	42	202	65	332	9.5	12.7	18.0	0.61	714	5.17	52.2	11.8
$NY-W-5$	162.5	Silty mudstone	16.4	27	104	61	204	57	483	12.4	10.4	12.1	1.12	158	4.73	21.1	4.66
$NY-W-6$	168	Siltstone	18.7	21	61	64	305	134	708	14.2	13.2	17.8	1.40	173	5.66	26.1	2.85
$NY-W-7$	183.5	Argillaceous siltstone	14.7	18	75	44	399	41	452	10.4	7.1	12.5	1.19	169	5.00	16.0	4.97
$NY-W-8$	189.5	Argillaceous siltstone	4.0	5	31	15	621	20	591	9.5	3.8	20.4	1.09	95	3.53	13.0	3.68
$NY-W-9$	193.6	Silty mudstone	3.0	6	15	12	621	21	580	8.7	3.8	17.5	1.18	112	4.03	13.5	3.36
$NY-W-10$	195.5	Silty mudstone	1.7	8	59	43	300	108	426	12.2	8.1	17.1	0.97	121	3.36	12.2	3.13
$NY-W-11$	208.2	Silty mudstone	8.9	23	61	71	324	142	648	16.8	14.2	16.8	0.57	138	4.76	19.9	4.19
$NY-W-12$	218	Argillaceous siltstone	16.8	30	112	51	221	65	470	11.8	6.9	13.2	1.22	145	4.81	17.3	4.08
$NY-W-13$	230.5	Mudstone	5.7	8	141	52	215	74	364	22.8	13.5	15.7	1.48	173	5.46	23.1	6.61
$NY-W-14$	245.66	Mudstone	2.0	4	85	48	227	96	313	12.7	11.9	22.2	1.63	176	5.06	40.1	2.68
$NY-W-15$	250	Argillaceous siltstone	4.9	10	99	45	134	104	409	17.8	15.6	14.0	1.12	173	4.84	32.0	4.48
$NY-W-16$	250.4	Mudstone	5.8	20	175	58	239	73	389	13.9	20.0	16.7	1.09	518	6.07	27.3	3.15
$NY-W-17$	251.75	Mudstone	6.1	16	69	35	211	133	501	16.9	15.0	30.1	1.91	302	9.56	40.1	3.65
$NY-W-18$	255.5	Silty mudstone	8.8	17	84	47	177	121	471	22.1	18.7	19.1	1.51	219	6.47	39.3	6.16
NY-W-19	256	Mudstone	4.8	12	146	57	193	111	402	13.9	22.7	23.1	1.69	227	5.96	35.1	3.69
$NY-W-20$	256.5	Silty mudstone	14.8	16	94	41	146	99	419	18.7	14.9	21.4	1.70	296	8.31	49.2	5.41
$NY-W-21$	257	Silty mudstone	41.0	26	97	49	199	115	446	24.7	18.7	30.7	2.33	245	7.76	54.4	6.90
$NY-W-22$	257.5	Silty mudstone	5.5	8	73	42	168	94	427	18.6	14.3	31.8	2.56	396	11.1	48.1	5.34
$NY-W-23$	271	Argillaceous siltstone	15.3	16	146	68	197	150	436	19.1	19.2	20.1	1.52	212	5.70	40.8	6.06
$NY-W-24$	301.2	Mudstone	27.3	32	75	46	181	54	352	5.5	6.7	19.6	1.54	251	7.76	13.7	4.76
NY-W-25	313	Silty mudstone	15.5	25	43	20	551	60	649	6.7	5.0	8.1	0.55	113	3.87	10.8	2.35
$NY-W-26$	314.48	Siltstone	7.1	20	72	24	412	60	467	8.3	6.1	18.2	0.67	148	3.04	11.4	2.72
NY-W-27	324.5	Silty mudstone	18.4	16	107	70	314	169	609	19.5	15.6	22.7	1.55	190	5.89	33.4	9.58
<b>NY-W-28</b>	340	Silty mudstone	6.1	11	87	56	307	131	634	14.1	12.1	19.0	1.28	157	4.68	25.8	3.48
$NY-W-29$	343	Silty mudstone	6.4	14	85	48	110	38	234	6.8	5.3	15.1	1.34	169	5.83	10.4	2.55
NY-W-30	346.8	Silty mudstone	24.1	27	116	60	151	123	419	15.2	18.7	14.5	1.08	299	8.17	46.5	5.87
$NY-W-31$	360	Silty mudstone	9.6	14	119	53	100	107	426	17.2	19.1	13.6	1.05	190	5.30	25.9	2.84
NY-W-32	365	Mudstone	2.8	13	148	64	125	39	183	6.4	15.0	9.4	0.73	222	5.92	38.9	2.40
NY-W-33	368.5	Mudstone	14.6	16	133	79	113	86	295	12.1	20.3	15.9	1.14	327	7.42	41.8	4.52
$NY-W-34$	370.5	Mudstone	6.6	15	108	70	86	69	230	10.7	21.2	14.8	1.01	396	9.55	39.4	8.59
$NY-W-35$	372.8	Mudstone	10.1	17	202	64	81	94	147	10.6	8.2	12.1	1.10	436	8.67	29.5	17.05
		<b>UCC</b>	10.0	20	60	35	350	112	550	10.7	11.0	25.0	2.20	190	5.80	22.0	2.80

Abbreviation: UCC upper continental crust, data from Taylor and McLennan ([1985](#page-14-0))

affected by weathering. The medium content of  $SiO<sub>2</sub>$  $(38 \sim 63 \%$ , with an average of 55 %) indicates medium mineral maturity and medium content of quartz and/or Si-rich minerals (Zhang et al. [2014\)](#page-14-0). The relatively high ferromagnesian element concentrations (such as  $TiO<sub>2</sub>$ ,  $Fe<sub>2</sub>O<sub>3</sub>$ , and MgO) indicate some influence from basic rocks. The high  $Al_2O_3$  content (13.4 ~ 24.8 %, with an average of 18.7 %) is probably related to the high content of aluminum-rich minerals, such as clay minerals, feldspar and mica.

#### Trace elements

The selected trace elements are discussed as three groups, i.e. transition trace elements (TTE: Co, Ni, V, Cr), large ion lithophile elements (LILE: Sr, Rb, Ba) and high field strength elements (HFS: Th, Sc, Nb, Ta, Zr, Hf, Y, U). Table 4 lists the selected trace elements concentrations, the distribution pattern of these elements are presented in Fig. [5](#page-8-0).

The TTE in fine-grained sediments are enriched relative to the upper continental crust (UCC), indicating a cryptic mafic

<span id="page-7-0"></span>

Table 5 Rare earth element concentrations of the samples from the Laoheishan basin as determined by HR-ICP-MS Table 5 Rare earth element concentrations of the samples from the Laoheishan basin as determined by HR-ICP-MS

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 $(LaYb)_N$ , ratio of La<sub>N</sub> and Yb<sub>N</sub>, La<sub>N</sub> and Yb<sub>N</sub> are chondrite-normalized values  $\delta Eu = Eu_N/(Sm_NXGd_N)^{1/2}$ , Eu<sub>N</sub>, Sm<sub>N</sub> and Gd<sub>N</sub> are chondrite-normalized values

Chondrite data from Taylor and McLennan [\(1985\)](#page-14-0)

Chondrite data from Taylor and McLennan (1985)

<span id="page-8-0"></span>

Fig. 4 Photomicrographs of the Muling Formation sandstone samples (cross-polarized light): a Qp grains belonging to metamorphic rock fragments, along with K-feldspar (Kf), Qm, biotite (Bio) grains and carbonate cement (Cc); b Qp grain belonging to igneous rock fragment, Qm

or ultramafic component carried in the clay fractions of the sediments (Hossain et al. [2010](#page-13-0)). The LILE are depleted relative to the UCC. Among these, Rb shows the greatest depletion (Fig. 5). Because Rb is incorporated into clay during chemical weathering (Nesbitt and Young [1982](#page-13-0)), so it indicates strong chemical weathering in the source area. The HFS are



Fig. 5 UCC-normalized selected trace elements diagram of samples from the Laoheishan basin (UCC data from Taylor and McLennan [1985](#page-14-0))

and biotite (Bio) grain; c acid igneous rock fragment (Lv, probably granite fragment) and Qm grains; d intermediate igneous rock fragment (Lv, probably andesite fragment) along with plagioclase (Pl) and Qm grains

similar to the UCC, suggesting the source rocks are probably derived from the UCC (McLennan et al. [1995\)](#page-13-0).

#### Rare earth elements

The REE contents of the fine-grained sediments from the Muling Formation are presented in Table [5.](#page-7-0) The REE contents range from 65.99 to 336.81 μg/g, with an average of 171.65 μg/g, similar to the UCC ( $\Sigma$ REE = 146.4 μg/g), indicating a probably felsic source from the UCC (Taylor and McLennan [1985](#page-14-0)), and probably influenced by some intermediate rock sources. Of these, the total light rare earth elements (ΣLREE, include La to Eu) and total heavy rare earth elements (ΣHREE, include Gd to Lu) are 55.65–301.49 ppm and 8.03–36.08 ppm, respectively. The ΣLREE/ΣHREE ratio ranges from 3.19 to 13.44 with an average of 7.68 (Table [5\)](#page-7-0). The chondrite normalized REE patterns (Fig. [6](#page-9-0)) show LREE enrichment and relatively flat HREE. The medium  $(La/Yb)<sub>N</sub>$  value (2.55–19.02, avg. 8.44) indicates the medium extent of fractionation between LREEs and HREEs, consistent with the medium ΣLREE/ΣHREE ratio. Eu anomaly (δEu) value varies between 0.40 and 0.81, with an average of 0.63, reveals a negative Eu anomaly.

<span id="page-9-0"></span>

Fig. 6 Chondrite-normalized REE distributions of samples from the Laoheishan basin (Chondrite data from Taylor and McLennan [1985](#page-14-0))

# Discussion

#### Weathering and sediment maturity

During the weathering process, stable cations (such as  $Al^{3+}$ and  $Ti^{4+}$ ) are sorted in stable weathering products, whereas the unstable cations (such as  $Na^+$ ,  $Ca^{2+}$  and  $K^+$ ) tend to be lost (Fedo and Nesbtit [1995](#page-13-0)). Such elemental enrichment and loss commonly depend on the intensity of chemical weathering (Condie et al. [1992](#page-13-0)). In order to quantify the degree of weathering, Nesbitt and Young ([1982](#page-13-0)) proposed the Chemical Index of Alteration (CIA). The  $Al_2O_3$ -(Na<sub>2</sub>O + CaO\*)-K<sub>2</sub>O ternary diagram (Fig. 7) shows that the CIA of  $K_1$ ml<sub>2</sub> samples range from 69 to 90 with an average of 78, suggesting strong chemical weathering.

The index of chemical variability (ICV; Cox et al. [1995](#page-13-0)) is a measure to identify the compositional maturity of sediments,

which has successfully applied in many studies (Armstrong-Altrin et al. [2015a,](#page-13-0) [b\)](#page-13-0). Typical rock forming minerals like feldspars, amphiboles, and pyroxenes have ICV values of >1, whereas alteration products such as kaolinite, illite, and muscovite have ICV values <1 (Cox et al. [1995](#page-13-0); Cullers and Podkovyrov [2000](#page-13-0)). The ICV values of  $K_1ml_2$  samples range from 0.28 to 0.63 with an average of 0.47, indicating the samples are geochemically moderately mature and may derive from a strong weathered source.

## Provenance

#### The sedimentary source regions

The sandstones from the Muling Formation show medium to high quartz content, relatively low contents of feldspar and lithic fragments (Table [1\)](#page-4-0), medium sorted, and sub-angular to sub-round in shape. These features are indicative of compositionally and texturally moderately mature. The types of lithic fragments in sandstones are similar to the basement surrounding the basin. Furthermore, in conglomerates at the  $K_1ml_1$ , much gravel has been found from the basement, i.e. dacite, acid tufflava, andesite, granite, rhyolite and tuff, etc., suggesting the felsic and intermediate igneous source rocks. Dickinson ([1985](#page-13-0)) suggested that detrital modes of sandstone suites primarily reflect the different tectonic settings of provenance terranes. On the Q-F-L ternary diagram, most of the  $K_1ml_2$  sandstones plot in the recycled orogeny field (Fig. 8).



Fig. 7 Al<sub>2</sub>O<sub>3</sub>-(Na<sub>2</sub>O + CaO<sup>\*</sup>)-K<sub>2</sub>O ternary diagram of samples from the Laoheishan basin, showing the weathering status of its source rocks (after Condie et al. [1992\)](#page-13-0)



Fig. 8 Q-F-L ternary plots from the Muling Formation sandstone in the Laoheishan basin  $(Q =$ Quartz; F = feldspar; L = lithic rocks) (after Dickinson [1985\)](#page-13-0)

#### Type of the source rocks

Major and trace element concentrations of terrigenous sediments can give a reliable clue about the provenance of the source area (Armstrong-Altrin et al. [2015b](#page-13-0); Odoma et al. [2015](#page-13-0)). Among major elements,  $Al_2O_3$ and  $TiO<sub>2</sub>$  are considered as immobile during weathering, transportation, and diagenesis. Hence, the  $Al_2O_3/TiO_2$ ratio can be used to infer the source rock composition (Zhou et al. [2015](#page-14-0)). According to Hayashi et al. ([1997](#page-13-0)), the  $Al_2O_3/TiO_2$  ratios range from 3 to 8 represent mafic, 8–21 indicate intermediate and 21–70 for felsic igneous rocks. The  $Al_2O_3/TiO_2$  ratios of fine-grained samples from the  $K_1$ ml<sub>2</sub> vary between 17 and 32. The variation in  $Al_2O_3/TiO_2$  ratios indicate that  $K_1ml_2$  samples are derived from felsic and intermediate igneous source rocks.

Trace elements such as Sc, Ni, Cr and Co tend to be enriched in mafic rocks, whereas La, Th, Hf, Zr and REE are richer in acidic rocks (Cullers and Podkovyrov [2000](#page-13-0)). The fine-grained sediments from  $K_1ml_2$  show variable La/Sc ratio (0.81–7.68, avg. 3.04) and relatively constant Co/Th ratio (0.14–4.96, avg. 0.97), which plot mostly in felsic volcanic rock and andesite areas (Fig. 9a). Floyd and Leveridge [\(1987\)](#page-13-0) established a discrimination diagram using the La/Th ratio vs. Hf to determine different sources and to further distinguish the andesite and basalt sources. The samples show relatively low La/Th ratio  $\left($ <5.0) and Hf content (3.04–11.1, avg. 5.94), suggesting that they may have originated from felsic and intermediate source rocks (Fig. 9b). Thus, the geochemical analyses indicate that the source rocks of the  $K_1$ ml are mainly derived from felsic and intermediate igneous rocks, probably felsic volcanic rock and andesite.

#### The location of source area

Several igneous rocks developed surrounding the Laoheishan basin, such as the northwestern granodiorite, the northeastern quartz diorite, the northern granite porphyry and the pyroxene diorite (Cheng et al. [2006;](#page-13-0) Zhao et al. [2009](#page-14-0); Xu et al. [2013a,](#page-14-0) [b](#page-14-0)). Previous studies indicated that the granodiorite from the northwest is strong REE fractionation  $((La/Yb)<sub>N</sub> = 6.34–$ 16.68) with a negative Eu anomaly ( $\delta$ Eu = 0.53–0.74) (Zhao et al. [2009\)](#page-14-0), similar to the  $K_1ml_2$  samples (Fig. [10a](#page-11-0)). Similarly, the quartz diorite from the northeast also shows relatively strong REE fractionation  $((La/Yb)<sub>N</sub> = 5.28-8.21)$  and a negative Eu anomaly ( $\delta$ Eu = 0.63–0.82) (Zhao et al. [2009;](#page-14-0) Fig. [10b](#page-11-0)). However, the granite porphyry from the north reveals strong REE fractionation  $((LaYb)<sub>N</sub> = 8.29)$  with a weak positive Eu anomaly ( $\delta$ Eu = 1.07) (Xu [2009;](#page-14-0) Fig. [10c](#page-11-0)), and the pyroxene diorite from the north suggests weak REE fractionation and a weak positive Eu anomaly (Zhao [2013;](#page-14-0) Fig. [10d\)](#page-11-0), which are different from the studied samples.

Therefore the source rocks of the  $K_1$ ml<sub>2</sub> have strong genetic relationships with the granodiorite and quartz diorite from the northwest and the northeast, respectively. But have weak relationships with the granite porphyry and the pyroxene diorite from the north, suggesting the source rocks may contain granodiorite and quartz diorite, and probably derived from the northwest and the northeast areas.

## Tectonic setting

The distinctive elemental compositions of siliciclastic rocks in various tectonic environments have been widely used to indicate the tectonic setting of their respective sedimentary basins (Bhatia [1983](#page-13-0); Bhatia and Crook [1986](#page-13-0)). Recently, based on the geochemical composition of Neogene-Quaternary sediments,



Fig. 9 Source rock discrimination diagrams for the K<sub>1</sub>ml<sub>2</sub> samples in the Laoheishan basin; a La/Sc vs. Co/Th (after Wronkiewicz and Condie [1987\)](#page-14-0); **b** Hf vs. La/Th plot (after Floyd and Leveridge [1987\)](#page-13-0)

<span id="page-11-0"></span>



Fig. 10 Chondrite-normalised REE distribution patterns of the study area and granite surrounding the Laoheishan basin (Chondrite data from Taylor and McLennan [1985\)](#page-14-0). The grey shaded zones represent the REE distribution of the  $K_1ml_2$  samples. a Granodiorite in the northeastern

basin, with data from Zhao et al. ([2009](#page-14-0)); b Quartzdiorite in the northwestern basin, with data from Zhao et al. ([2009](#page-14-0)); c Granite porphyry in the northern basin, with data from Xu [\(2009](#page-14-0)); d Pyroxene diorite in the northern basin, with data from Zhao [\(2013\)](#page-14-0)

Verma and Armstrong-Altrin ([2013\)](#page-14-0) proposed two discriminant-function-based major-element diagrams for the tectonic discrimination of siliciclastic sediments for highsilica  $[(SiO<sub>2</sub>)<sub>adi</sub> = 63–95 %]$  and low-silica  $[(SiO<sub>2</sub>)<sub>adi</sub> = 35–95 %]$ 63 %] types, from three main tectonic settings: island or continental arc, continental rift and collision. In addition, these diagrams are already evaluated by some researchers (Nagarajan et al. [2015](#page-13-0); Verma and Armstrong-Altrin [2016\)](#page-14-0). In these high silica and low silica multi-dimensional diagrams, the  $K_1ml_2$  samples are plotted in the collision and continental rift fields (Fig. [11a, b\)](#page-12-0).

The previous studies indicated that at the end of the Late Jurassic  $(\sim] 60$  Ma), the lithosphere was affected by the early collision orogeny and intracontinental compressive deformation in the region of northeast (NE) China, caused a strong crustal shortening and thickening, and increased the lithosphere thickness (Cheng et al. [2006](#page-13-0); Xu et al. [2012;](#page-14-0) Luo et al. [2014](#page-13-0)). At the middle stage of the early Cretaceous (120–130 Ma), the rapidly oblique subduction of the Paleo-Pacific plate beneath the Eurasian continent, caused the compressed and left-lateral strike-slip shearing environment in the eastern margin of the Eurasian continent (Zhou et al. [2009](#page-14-0); Sun et al. [2010](#page-14-0)), agitated the upwelling of asthenosphere, and caused the lithospheric mantle and part of the lower crust to submergence into the asthenosphere mantle through delamination. Meanwhile the lithosphere intense thinning made asthenosphere mantle directly contact with the crust, and resulted in the large-scale magmatism and widespread development of extensional structures in the Mesozoic (Zhao et al. [2009;](#page-14-0) Liu et al. [2014](#page-13-0)). On the basis of Mesozoic volcanic rocks in NE China, Xu et al. [\(2013a,](#page-14-0) [b\)](#page-14-0) suggested the existence of late Early Cretaceous (106 ~ 133 Ma, peak age ~115 Ma) calcalkaline volcanic rocks in the eastern Heilongjiang–Jilin provinces indicated an active continental margin setting, and the coeval bimodal volcanic rocks in the Great Xing'an Range and the Songliao Basin suggested an intracontinental extensional environment related to the low-angle subduction of the Paleo-Pacific Plate beneath the Eurasian continent. Thus, the previous studies (Sun et al. [2010;](#page-14-0) Xu et al. [2013a](#page-14-0), [b](#page-14-0); Liu et al. [2014](#page-13-0); Luo et al. [2014](#page-13-0)) indicated that during the late early Cretaceous, collision and continental rift settings developed in the NE China. The Laoheishan basin located in the eastern Heilongjiang Province (Fig. [1\)](#page-1-0), combined with the results obtained from the discrimination diagrams, there is a high

<span id="page-12-0"></span>

Fig. 11 Discriminant-function multi-dimensional diagram for high-silica (a) and low-silica (b) clastic sediments (Verma and Armstrong-Altrin [2013\)](#page-14-0). The subscript m1 in DF1 and DF2 represents the high-silica diagram based on loge-ratios of major elements. The discriminant function equations are: (1)  $DF1_{(Arc-Rift-Col)ml} = (0.263 \times ln(TiO_2/SiO_2)_{adi}) +$  $(0.604 \times \ln(A1_2O_3/SiO_2)_{\text{adj}}) + (1.725 \times \ln(Fe_2O_3^{t}/SiO_2)_{\text{adj}}) + (0.660 \times$  $ln(MnO/SiO_2)_{\text{adj}}$  + (2.191 ×  $ln(MgO/SiO_2)_{\text{adj}}$ ) + (0.144 ×  $ln(CaO/$  $\text{SiO}_{2\text{)adj}}$ ) + (−1.304 × ln(Na<sub>2</sub>O/SiO<sub>2)adj</sub>) + (0.054 × ln(K<sub>2</sub>O/SiO<sub>2)adj</sub>) +  $(-0.330 \times ln(P_2O_5/SiO_2)_{adj}) + 1.588$ ; (2) DF2<sub>(Arc-Rift-Col)m1</sub> = (1.196  $\times$  $ln(TiO_2/SiO_2)_{adj}) + (1.604 \times ln(Al_2O_3/SiO_2)_{adj}) + (0.303 \times ln(Fe_2O_3^{-1})$  $\text{SiO}_2$ <sub>adj</sub>) + (0.436 × ln(MnO/SiO<sub>2</sub>)<sub>adj</sub>) + (0.838 × ln(MgO/SiO<sub>2</sub>)<sub>adj</sub>) +  $(-0.407 \times \ln(CaO/SiO<sub>2</sub>)_{\text{adj}}) + (1.021 \times \ln(Na<sub>2</sub>O/SiO<sub>2</sub>)_{\text{adj}}) + (-1.706 \times$ 

probability that clastic sediments of the  $K_1ml_2$  were derived from the collision system, and however, there were possibilities that they may comprise sediments derived from the continental rift system.

## **Conclusions**

Sedimentary rocks collected from the lower Cretaceous Muling Formation  $(K_1m)$  in the Laoheishan basin, northeast China are investigated the petrography, mineralogy and geochemistry to reveal the weathering intensity, provenance and tectonic setting.

Sandstones from the  $K_1ml_2$  have high quartz, medium feldspar and low lithic fragments contents. Lithic fragments are predominantly volcanic clasts. Much gravel has been found from the basement in  $K_1ml_1$  conglomerates. Petrological observation reveals that the sandstones are derived from the surrounding basement and recycled orogeny sources.

Petrography and geochemistry  $(AI_2O_3/TiO_2, Co/Th, La/Sc,$ La/Th, Hf) results reveal that the source rocks are mainly felsic and intermediate igneous rocks, probably felsic volcanic rock and andesite. The strong genetic relationship with the igneous rocks from the northwest and northeast indicate the sediments have been derived from this area. The



ln(K<sub>2</sub>O/SiO<sub>2)adi</sub>) + (−0.126 × ln(P<sub>2</sub>O<sub>5</sub>/SiO<sub>2)adi</sub>) - 1.068; The subscript m2 in DF1 and DF2 represents the low-silica diagram based on log<sub>e</sub>-ratios of major elements. The discriminant function equations are: (1)  $DF1_{(Arc-Riff-P)}$  $_{\text{Col} \text{m2}} = (0.608 \times \text{ln}(\text{TiO}_2/\text{SiO}_2)_{\text{adj}}) + (-1.854 \times \text{ln}(\text{Al}_2\text{O}_3/\text{SiO}_2)_{\text{adj}}) +$  $(0.299 \times \ln(Fe_2O_3^{-t}/SiO_2)_{adj}) + (-0.550 \times \ln(MnO/SiO_2)_{adj}) + (0.120 \times$ ln(MgO/SiO<sub>2</sub>)<sub>adi</sub>) + (0.194 × ln(CaO/SiO<sub>2</sub>)<sub>adi</sub>) + (−1.510 × ln(Na<sub>2</sub>O/  $\text{SiO}_2$ <sub>adj</sub>) + (1.941 × ln(K<sub>2</sub>O/SiO<sub>2</sub>)<sub>adj</sub>) + (0.003 × ln(P<sub>2</sub>O<sub>5</sub>/SiO<sub>2</sub>)<sub>adj</sub>) -0.294; (2) DF2<sub>(Arc-Rift-Col)m2</sub> =  $(-0.554 \times ln(TiO_2/SiO_2)_{\text{adj}}) + (-0.995 \times$  $ln(Al_2O_3/SiO_2)_{adj})$  + (1.765 × ln(Fe<sub>2</sub>O<sub>3</sub><sup>†</sup>/SiO<sub>2</sub>)<sub>adj</sub>) + (−1.391 × ln(MnO/  $\text{SiO}_{2}\text{)}_{\text{adj}}$ ) + (−1.034 × ln(MgO/SiO<sub>2</sub>)<sub>adj</sub>) + (0.225 × ln(CaO/SiO<sub>2</sub>)<sub>adj</sub>) +  $(0.713 \times \ln(Na_2O/SiO_2)_{\text{adj}}) + (0.330 \times \ln(K_2O/SiO_2)_{\text{adj}}) + (0.637 \times$  $ln(P_2O_5/SiO_2)_{adj}) - 3.631$ 

chemical weathering index (CIA) and index of chemical variability (ICV) suggest an intensive weathering in the source region of the sediments.

The multidimensional tectonic discrimination diagrams indicate that the source rocks of  $K_1$ ml are predominantly derived from the collision system. However, they may also comprise sediments derived from the continental rift system. These results are consistent with the geology of the study area.

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