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The fluctuations of fixed-NH_4^+-N content in the Luochuan loess and its paleoclimatic significance

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Abstract

The fixed NH_4^+-N content varies from 120 to 202 μg/g in Luochuan loess–paleosol sequences. This content in the interstratified paleosols is significantly higher than that of the loess. Fixed NH_4^+-N content in the loess L_2 is the lowest, varying between 144 to 147 μg/g whereas the highest in the paleosol S_5, reaching 200 μg/g. The distribution of fixed NH_4^+-N is similar to that of the <2 μm fraction and MS in the S_0–L_1–S_1 sequence. The fixed NH_4^+-N content is the highest in the paleosol S_1 developed during the last interglacial period. The content is slightly lower in the S_0 layer formed during the postglacial period. The lowest concentration occurs in the Malan loess developed during the last glacial period. Additionally, three small fluctuations between low and high value (fixed NH_4^+-N) are found in the Malan Loess. Through the detailed analysis of fixed-NH_4^+-N in the paleosol S_1, we found that fixed-NH_4^+-N is in loss in the paleosol S_1. However, fixed NH_4^+-N in the paleosol S_1 is higher than that in the loess L_1. The distribution of fixed NH_4^+-N has a close relationship with the granularity of aeolian dust in the Luochuan loess–paleosol sequence, and matches paleoclimate variation fairly. Thus, fixed-NH_4^+-N can be regarded as a better indicator which records the information of the winter monsoon variation in the Chinese Loess Plateau.

1. Introduction

Chinese loess–paleosol sequences are widely regarded as one of the best long-term records of continental paleoclimatic change (Kukla et al., 1988; An et al., 1990). Loess accumulated under a relatively cold and dry climatic regime which is associated with the East Asia winter monsoon, whereas paleosols formed during comparatively warm and humid conditions, related to the East Asian summer monsoon (Bronger and Heinkele, 1989; An et al., 1991; Rutter et al., 1991). Climate-proxy data of loess–paleosol sequences have been obtained through many ways including magnetic susceptibility (Zhou et al., 1990; An et al., 1990; Heller et al., 1993; Verosub et al., 1993), particle size (Porter and An, 1995; Vandenberghe et al., 1997), geochemistry (Han et al., 1997; Gallet et al., 1998; Chen et al., 1999; Jahn et al., 2001), amino acid analysis (Curry et al., 1994), and mineralogy (Ji et al., 1999a, b, 2001). In general, climate gradually fluctuated from warm–wet to dry–cold and multiple glacial–interglacial cycles took place in the Chinese Loess Plateau since the Early Pleistocene.

Fixed NH_4^+-N plays an important role in many science fields such as geology, environment, and agriculture. For example, study of the release and fixation mechanism of NH_4^+-N provided theoretical foundations on how to improve soil fertility in agriculture (Schneiders and Scherer, 1998; Elmaci et al., 2002). In environmental science, fixed NH_4^+-N has been drawing more and more attention from experts, associated with eutrophication in seas and lakes, and treatment of environments (Wang, 2001). Fixed NH_4^+-N provided an important theoretical guide to both genesis of rocks and ore deposits (Cooper and Bradley, 1990) and determination of rocks and source materials (Huang et al., 2001) in geology.

Because of its large ionic size and chemical similarity to potassium, fixed NH_4^+-N in minerals usually substitutes isomorphously in the structures of potassium minerals, such as feldspars and micas (Hall and Neiva, 1990). At the same time, the fixation mechanism of NH_4^+-N is similar to that of K^+ in soils (Zhu et al., 2000). Firstly, NH_4^+ enters interlayers of 2:1 clay

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minerals by diffusion, and then enters hexagonal cavities composed of oxygen ions on tetrahedrons with small quantities of hydrated energy. Subsequently, the interstices between interlayers of clay minerals decrease from 1.4 to 1.0 nm under static gravitation, so that NH_4^+ within interlayers of clay minerals is absorbed more tightly than before and cannot be easily substituted by other cations. Content and kinds of 2:1 clay minerals and NH_4^+ concentration in soils are two main factors controlling fixed NH_4^+-N content. Influences on content and kinds of 2:1 clay minerals and NH_4^+ concentration affect fixed NH_4^+-N content. Bioclimatic conditions (temperature and moisture) are an important impact factor that influences not only compositions of minerals but also fixation and release of NH_4^+-N in soils. Content of fixed NH_4^+-N varies regularly with mineral compositions with different bioclimatic conditions from soil to soil (Wei et al., 2000). Additionally, content of fixed NH_4^+-N increases with increase of the clay fraction in soils derived from the same original rocks in particular areas (Li et al., 1992). Consequently, as an important component of soil nitrogen, fixed NH_4^+-N plays an important role in the nitrogen cycle and has a close relationship with bioclimatic conditions. Although fixed NH_4^+-N in soils is used to indicate climatic variation, especially in the loess–paleosol sequence the pattern is little known. In this paper, fixed NH_4^+-N in the loess is measured, and its paleoclimatic significance is discussed in detail.

2. Materials and methods

Ninety-five samples were collected from the upper ~12 m of the Luochuan loess section (Liu, 1985) (35°45′N, 109°25′E) in Shaanxi Province, China. The section consists of Holocene Black Loam (S_0), Malan Loess (L_1) and the first paleosol of Lishi Loess (S_1) from top to bottom, spanning about 130 ka. The sampling intervals were approximately 10 cm in S_0 unit, L_1 unit, and at the L_1/S_1 boundary, and 30 cm in S_1 unit. Additionally, 3 samples in each unit from L_1 to S_7 were collected, respectively, to avoid mixture of loess and paleosol. After air-drying, the samples were ground in an agate mortar to a size-fraction of <100 mesh, and preserved.

Fixed NH_4^+-N contents were determined by using the Silva–Bremner Method (Silva and Bremner, 1966). For a sample, first, we used a 2 g sample and measured 10 times repeatedly, then reduced the amount of sample to 1 g and measured it 10 times repeatedly (Table 1). Table 1 shows that 20 ml HF–HCl solution cannot destroy a 2 g sample completely, but can destroy a 1 g sample completely. If the sample weight was insufficient (0.5 g), although these samples can be completely destroyed with 20 ml HF–HCl solution, more errors are probably caused. If the volume of HF–HCl solution increases with constant concentration, it is inconvenient for distillation. However, if the volume of HF–HCl solution decreases with constant concentration, the sample is probably incompletely destroyed. Consequently, the best scheme is to use 1 g samples and 20 ml HF–HCl solution with constant concentration.

Magnetic susceptibility was determined using the BartingtonMS2 in the City and Environment Department of Peking University. Data for particle sizes were provided by Prof. Lu Huayu, Institute of Geoenvironment, Chinese Academy of Sciences, China.

Based on magnetic susceptibility and particle size data, and data from Jiedong Yang (Yang et al., 2000), the S_0–L_1–S_1 sequence of the Luochuan loess can be divided into five units from the top downwards: S_0 (paleosol formed during the post-glacial period), L_1LL_1 (loess formed during the last stage of the last glacial period), L_1SS_1 (weak pedogenic loess formed during interstade of the last glacial period), L_1LL_2 (loess formed during early stade of the last glacial period), and S_1 (paleosol formed during the last interglacial period).

3. Results and discussion

3.1. Distribution of fixed-NH_4^+-N in the loess–paleosol cycle

Fixed NH_4^+-N content ranges from 140 to 210 μg/g in the loess and paleosols measured. The fixed NH_4^+-N content of the loess is lower than that of the interstratified paleosols (Fig. 1). Variation of fixed NH_4^+-N content basically corresponds to periodic fluctuations between cold and warm climates recorded in the Luochuan loess–paleosol sequence. The content of fixed NH_4^+-N is low in the loess formed under a dry and cold climate, but is relatively high in the paleosols formed under a warm and humid climate (Fig. 1). The fixed NH_4^+-N content varies with climate significantly. For example, the content of fixed NH_4^+-N is highest in

Table 1
Fixed-NH_4^+-N content in different weights of the same sample (μg/g)

Number	1	2	3	4	5	6	7	8	9	10	Mean value	Standard deviation
1 g	174.11	175.33	174.40	173.19	173.90	169.18	173.70	174.94	172.83	175.92	173.75	1.8
2 g	138.52	144.22	144.43	138.16	143.78	133.74	148.33	138.27	142.77	150.94	142.32	5.0

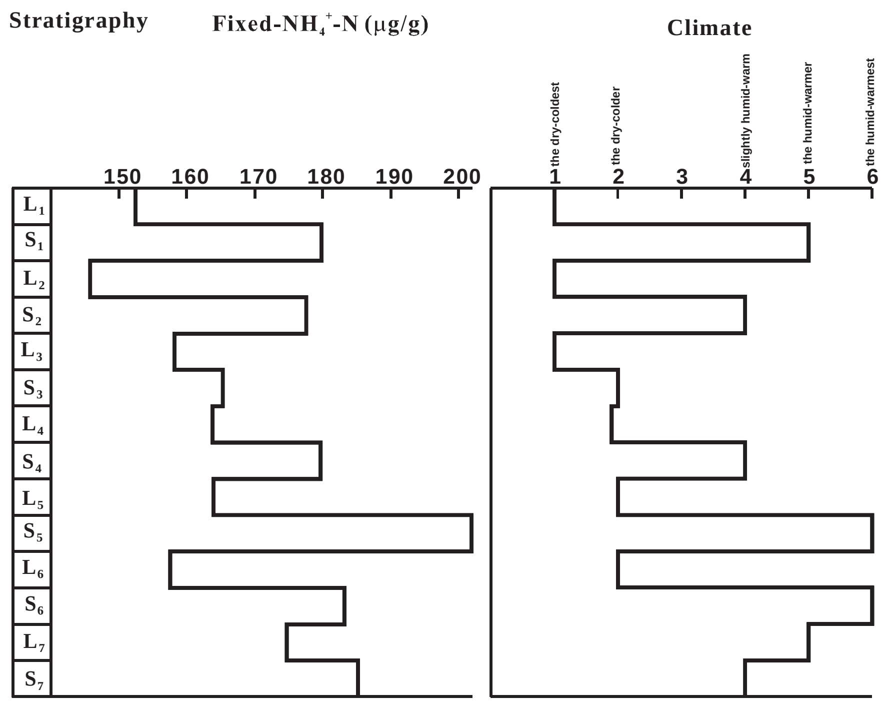

Fig. 1. Distribution of fixed-NH_4^+-N (μg/g) in the Luochuan Loess section and corresponding natural environments (Cheng and Xie, 1993).

Table 2
Fixed NH_4^+-N, MS, and the <2 μm fraction in the Luochuan loess section

Stratigraphy (the number of samples)	Fixed NH_4^+-N (μg/g)		Magnetic susceptibility (SI)		The <2 μm fraction (%)	
	Variation range	Mean value	Variation range	Mean value	Variation range	Mean value
S_0 (10)	141–187	163	67.6–155.55	102.24	8.56–9.83	9.35
L_1 (74)	134–170	158	49.1–142.5	89.63	7.47–11.58	8.5
S_1 (11)	140–187	174	53.55–233.25	180.27	8.82–13.94	12.40
S + L (95)	134–187	160	49.1–233.25	101.45	7.47–13.9	9.87
L_1LL_1 (21)	134–170	149	52.15–91.15	70.03	7.47–9.50	8.50
L_1SS_1 (29)	155–169	164	97.5–142.5	118.22	9.96–10.92	10.40
L_1LL_2 (24)	153–169	159	49.1–117.6	72.22	8.47–10.80	9.39

the paleosol S_5, developed under the most humid and warmest climate, about 202 μg/g (mean value). The fixed NH_4^+-N content is the lowest in the loess L_2 formed under the coldest and driest climate, with a mean value of about 145 μg/g.

3.2. Distribution of fixed NH_4^+-N in the Luochuan loess section during the last 130,000 years

The fixed NH_4^+-N content varies between 134 and 187 μg/g in the S_0–L_1–S_1 sequence with a mean value of around 160 μg/g. The content of fixed NH_4^+-N is high in the paleosols S_0 and S_1. In contrast, low fixed NH_4^+-N occurs in the loess L_1 (Table 2). The spatial distribution curve of fixed NH_4^+-N in the S_0–L_1–S_1 sequence exhibits three significant fluctuations between low and high values. The fixed NH_4^+-N is low in the loess L_1, medium in the paleosol S_0, and high in the paleosol S_1. Similarly, the spatial distribution curve of fixed NH_4^+-N in the Malan Loess section exhibits three small fluctuations between low and high values. The fixed NH_4^+-N in the loess L_1LL_1 is low, medium in the loess L_1LL_2, and high in the weak pedogenic loess L_1SS_1 (Fig. 3 and Table 2). Therefore, fluctuations of fixed NH_4^+-N parallel climate variation in the S_0–L_1–S_1 sequence. This can be also demonstrated by the fact that the curves of fixed NH_4^+-N and magnetic susceptibility and the <2 μm fraction exhibit a similar oscillation both in amplitude and in frequency, and there is a good correlation between fixed NH_4^+-N and magnetic susceptibility, and the <2 μm fraction (Figs. 2 and 3, and Table 2).

3.3. Impact factors on fixed-NH_4^+-N in the Luochuan loess section

According to studies on material sources of loess (Liu, 1985) and the nitrogen cycle in soils (Stevenson, 1982), fixed-NH_4^+-N in the loess–paleosol sequence has three possible sources. Firstly, native NH_4^+-N in parent materials of loess–aeolian dust; secondly, NH_4^+-N in soil solution brought by atmosphere precipitation; and thirdly, NH_4^+-N produced by mineralization of organic nitrogen. Native NH_4^+-N, which either existed with fixation by clay minerals during transport and sedimentation processes before aeolian dust accumulated in the Chinese Loess Plateau, or was native NH_4^+-N from various types of rock in the deserts and gobi areas, or both of the above, was brought into the Chinese Loess Plateau as aeolian dust, and had a close relationship with the types of loess parent materials and the Winter Monsoon. NH_4^+-N brought by precipitation and produced by mineralization of organic nitrogen results from variation of bioclimatic conditions after the formation of loess, and is related to the East Asian summer monsoon. Accordingly, fixed-NH_4^+-N in the loess–paleosol sequence is mainly controlled by three factors: primary rock sources for loess; particle sizes of aeolian dust; and intensity of weathering and pedogenesis after aeolian dust accumulation in the Chinese Loess Plateau.

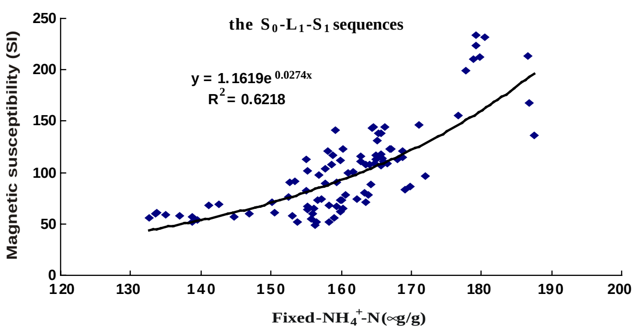

Fig. 2. Correlation between fixed-NH_4^+-N and MS, and the <2 μm fraction.

There is still no clear conclusion about the source of the loess and uniformity of its chemical composition. Most scholars have agreed that parent materials of the loess–paleosol sequence mainly come from the deserts and the gobi areas in Northern China, and have undergone sedimentation and transport before the accumulation of aeolian dust in the Chinese Loess Plateau. Through much work, most scholars (Liu, 1985; Wen et al., 1996; Gallet et al., 1998) believed that chemical composition of aeolian dust is uniform, and can be viewed to represent the mean content of the upper crust. Therefore, the fixed-NH_4^+-N content is mainly influenced by the particle size of aeolian dust and the weathering and pedogenesis after accumulation of aeolian dust in the Chinese Loess Plateau.

The particle size of aeolian dust when it accumulated mainly depended on the intensity of the East Asian winter monsoon. The particle size usually determines the amount of clay minerals that aeolian dust contains. When the winter monsoon becomes strong, the particle size in the aeolian dust is large, with inverse fining. Coarser particle assemblages contain relatively more detrital minerals such as quartz and feldspar. Fine particles are mainly composed of illite, vermiculite, chlorite, smectite and kaolinite (Liu, 1985). Fixed NH_4^+-N is mainly embedded into interlayers of silicates such as clay minerals. Accordingly, clay minerals are the main carriers of fixed NH_4^+-N in the loess. If the relative percentage of fine particles increases when aeolian dust accumulated, fixed NH_4^+-N content is relatively high, whereas it decreases with an increase of the percentage of coarse particles.

The intensity of weathering and pedogenesis after accumulation of aeolian dust is controlled by the East Asian summer monsoon, which determines the intensity of formation of secondary clay minerals and transformation

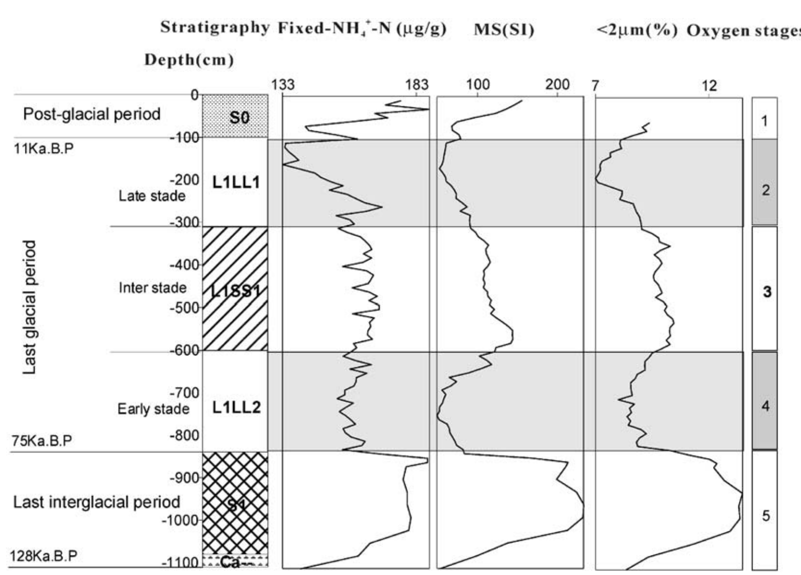

Fig. 3. Distribution of fixed-NH_4^+-N, MS, and the <2 μm fraction in the S_0–L_1–S_1 sequence.

of primary clay minerals. Consequently, the ability of NH_4^+ fixed and released by clay minerals changes with different intensity of the weathering and pedogenesis in the loess. The intensity of the East Asian summer monsoon is largely judged by precipitation and temperature in the China Loess Plateau (Yang and Ding, 2000). On the one hand, precipitation increases lead to the decrease of NH_4^+ concentration in soil solution, so that fixed-NH_4^+-N is released faster. At the same time, precipitation increases lead to alteration of minerals and leaching of carbonates, and clay minerals increase. On the other hand, if the temperature rises with sufficient precipitation, microbes become more active and the loess interchanges with the atmosphere and water more violently, and the original balance of fixed-NH_4^+-N in the loess is broken. Afterwards a new balance is established. In nature, the influence of the particle size and the weathering and pedogenesis on fixed-NH_4^+-N in the loess–paleosol sequence is focused on types, content and properties of clay minerals.

3.4. *Fluctuations of fixed NH_4^+-N in the Luochuan loess section during the last 130,000 years reflect the East Asian winter monsoon variation*

NH_4^+ fixation capacity varies with mineral composition and clay fraction contents from soil to soil. Studies of fixed NH_4^+-N have shown that NH_4^+ fixation capacity of illite is slightly less than that of vermiculite, but greater than that of smectite (Zhu et al., 2000). Kaolinite is a 1:1 clay mineral and chlorite is a 2:1:1 clay mineral, and both are not able to fix ammonium.

Clay minerals are similar in composition, but differ in content in the Luochuan loess section. Clay minerals include illite, kaolinite, smectite, chlorite, vermiculite and small amounts of mixed layer minerals (Liu, 1985), of which illite ($>50\%$) is of detrital genesis (Ji et al., 1999b). Therefore, illite is a main component of clay minerals in the Luochuan loess, closely related to fixed NH_4^+-N.

The average temperature was around 0°C and the mean annual precipitation was less than 200 mm in the Luochuan area during the Last Stade and the Early Stade of the Last glacial period. The mean annual temperature was about 3°C and the mean annual precipitation was 260 mm in the Luochuan area during the Interstade of the last glacial period (Liu, 1985, pp. 298–302). In general, it was dry and cold during the last glacial period, during which the Malan loess formed, the main form of weathering was physical weathering because of lack of water, and illite was well preserved without swelling layers (Ji et al., 1997). Furthermore, particle size determines the content of clay minerals and the fixed NH_4^+-N content is often controlled by clay mineral content, especially illite content. Therefore, the fixed-NH_4^+-N in the Malan loess is not influenced by weathering and pedogenesis but is related to particle sizes of aeolian dust.

The mean temperature was about 12°C and the annual precipitation ranged from 600 to 750 mm during the Last interglacial period, during which the paleosol S_1 developed (Liu, 1985, pp. 298–302). It was warm and humid during the Last interglacial period, during which illite possessed small amounts of swelling layers ($<3\%$) (Ji et al., 1997). So, the fixed-NH_4^+-N in the paleosol S_1 probably suffered from weathering and pedogenesis.

Regression analysis reveals that there is a good correlation between fixed NH_4^+-N and particle sizes in the Malan loess ($r=0.73$). The relationship between fixed NH_4^+-N and particle sizes is definitely expressed by a linear equation: $y=6.8957x+92.2159$ (Fig. 4).

In the paleosol S_1 where $CaCO_3$ was basically removed by precipitation, the $>2\,\mu m$ fraction is not affected by weathering and pedogenesis (Lu and An, 1998). However, the $<2\,\mu m$ fraction is likely to undergo slight pedogenesis, and its chemical composition changes to some degree, but its relative content is unchanged. It can be regarded as an original component of the paleosol S_1, subject only to the condition that $CaCO_3$ is removed. Consequently, if the weathering and pedogenesis after aeolian dust accumulation is not considered as an impact factor on fixed-NH_4^+-N in the paleosol S_1, the relationship between fixed-NH_4^+-N and particle sizes in the paleosol S_1 can be also interpreted by the linear equation mentioned above ($y=6.8957x+92.215$). In addition, the influence of $CaCO_3$ on fixed-NH_4^+-N and particle sizes can be neglected.

Therefore, the primary content of fixed NH_4^+-N can be calculated in the paleosol S_1 on the assumption of no weathering and pedogenesis (Table 3), and the difference (Δ value = calculated value − measured value) between calculated and measured values of fixed NH_4^+-N are the amounts of fixed NH_4^+-N affected completely by the weathering and pedogenesis in the paleosol S_1. As Table 3 shows, the Δ value is –9 and $-6\,\mu g/g$ in the upper of the paleosol S_1 (near the bottom of the loess L_1), indicating that NH_4^+-N is fixed there. The Δ value is above zero in the middle and lower parts of the paleosol

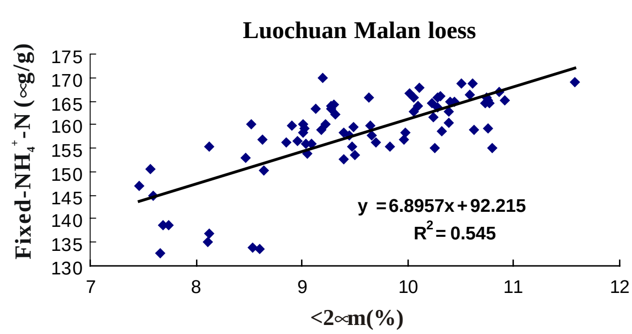

Fig. 4. Correlation between fixed-NH_4^+ and the $<2\,\mu m$ fraction in the Malan loess, Luochuan.

Table 3
Calculated and measured values of fixed NH_4^+-N (μg/g) in the palaeosol S_1

Sample no.	Calculation	Measurement	Δ (Difference)
PTXS1-3	178	187	−9
PTXS1-4	181	187	−6
PTXS1-5	180	179	1
PTS1-1	183	178	5
PTS1-2	188	179	9
PTS1-3	187	179	8
PTS1-4	188	181	7
PTS1-5	185	180	5
PTS1-6	173	165	8
PTS1-7	160	161	−1
PTS1-L2	153	140	13
Mean value	178	174	4

S_1, indicating that NH_4^+-N is relatively released or lost. In total, fixed NH_4^+-N in the paleosol S_1 is lost, implying that the release rate of fixed-NH_4^+-N is greater than its fixation rate during the release–fixation process of fixed-NH_4^+-N, because the NH_4^+-N concentration in the soil solution was diluted with much precipitation during the Last interglacial period, which caused the release of fixed-NH_4^+-N. Other causes include the rising temperature, the development of plants, and the accumulation of organic matters with absorption of NH_4^+-N in soil solution on the basis of sufficient precipitation. Additionally, the decrease of NH_4^+-N concentration in the soil solution may result from nitrification and ammonification, which caused the release of fixed-NH_4^+-N from the clay minerals.

However, Δ values change to a lesser extent in the paleosol S_1, and the most obvious change occurs in the bottom of the paleosol S_1 (Table 3), with a value of only 13 μg/g, indicating that the weathering and pedogenesis was weak during the period when paleosol S_1 developed. The conclusion supports the viewpoint (Ji et al., 1997) that illite contains small amounts of swelling layers and a very small quantity of illite was altered in the paleosol S_1.

To sum up, although there is partial loss of fixed-NH_4^+-N the fixed-NH_4^+-N in the paleosol S_1 is obviously higher than that in the Malan loess (Table 2 and Fig. 3). Therefore, fluctuations of fixed NH_4^+-N in the Luochuan loess section were closely related to particle size of aeolian dust during the last 130,000 years, which reflects periodically strong–weak variation of the East Asian winter monsoon in the China Loess Plateau.

4. Conclusions

Fixed NH_4^+-N content ranges from 130 to 200 μg/g in the loess–paleosol sequence. Fixed NH_4^+-N content is the highest in the paleosol S_5 developed in the most humid and warmest climate, with the mean value of 202 μg/g, whereas fixed NH_4^+-N content is the lowest in the loess L_2 formed during the cold–dry climate, with the mean value of 145 μg/g.

Whether on a long time scale (the loess–paleosol cycle), or on a short time scale (the S_0–L_1LL_1–L_1SS_1–L_1LL_2–S_1 sequence), the fixed NH_4^+-N in loess–paleosol sequences varies parallel to climatic variation. Fixed-NH_4^+-N of the loess is low as compared to the neighboring paleosols. Additionally, fixed NH_4^+-N of the loess during the Interstade is higher than that of the loess formed during the Late stade and Early stade in the Malan loess. Therefore, the low–high value fluctuations of fixed-NH_4^+-N in loess–paleosol sequences can reflect the variations of paleoclimate in the Chinese Loess Plateau.

Through this work, the original content of fixed NH_4^+-N in aeolian dust during the period the paleosol S_1 developed was determined by using the correlation between particle size and fixed NH_4^+-N in the Malan loess. As a whole, it was found that the release rate of fixed NH_4^+-N is greater than its fixation rate in the paleosol S_1, and fixed-NH_4^+-N is lost from the paleosol S_1. Despite this, the fixed NH_4^+-N content in the paleosol S_1 measured is higher than that of the loess. Hence, fluctuations of fixed NH_4^+-N in the Luochuan loess section are closely related to particle sizes of aeolian dust during the last 130,000 years, which reflects periodically strong–weak variation of the East Asian winter monsoon in the Chinese Loess Plateau.

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