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Discovery and study of silver sulfate mineral in S_5 from the eastern suburb of Xi'an

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The paleosol samples from the fifth layer of the loess profile at Renjiapo in the eastern suburb of Xi'an are observed and analyzed using electron microscope and energy spectrum. Minerals such as $AgSO_4$ and molybdenum, which are rare to find and can indicate typical dry climate environment, are found in this layer of paleosol. Secondary mineral is usually granular form of ellipsoidal and crystallization, and has the characteristics of chemical precipitating crystallization of apertures and fracture. Molybdenum minerals have the characteristics of colloidal substances. There are two kinds of secondary minerals. One is silver sulfate mineral and the other is silver oxide mineral. The movement of secondary silver, molybdenum and cobalt minerals, new clay mineral, Fe_2O_3 and Al_2O_3 indicates that S_5 has experienced strong chemical weathering and mineral dissolution during its development. Silver, molybdenum, and cobalt can be released from primary minerals. During that period, the precipitation was abundant in Xi'an where soil reached an acidity stage of chemical weathering. At the later development stage of paleosol in the lowest part of S_5, warm and wet monsoon climate had changed to dry and non-monsoon climate. In the period of the formation of $AgSO_4$, which is easier to dissolve than $CaSO_4$, a dry and non-monsoon climate was present in the Guanzhong Plain. Strong evaporation resulted in the accumulation of SO_4^{2-} in the soil water solution and the formation of $AgSO_4$. At that time, summer monsoon of East Asia was weak and did not cross Qinling Mountains to reach Guanzhong Plain. And at that time, the precipitation in Xi'an was less than 300 mm, and it was drier then in Xi'an than at present in Lanzhou.

silver sulfate mineral, paleosol of S_5, non-monsoon dry climate, climate change, Xi'an area

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Many researches [1–9] on loess and paleosol, especially S_5 paleosol [10, 11], have been done by domestic researchers. Their results have shown that the S_5 paleosol was developed 2.5 million years ago when the climate of the Loess Plateau was the warmest and moistest, and the climate of central-southern Loess Plateau belonged to subtropical type [1, 10–12]. Moreover, previous researches also showed that loess represented cold-dry climate when winter monsoon enhanced and red paleosol stood for warm-humid climate when summer monsoon reinforced [2, 13–15], both of which climate change in Quaternary plays an important role in controlling the accumulation of loess groundwater [16, 17], which indicates that theoretical research of climate change on Quaternary has had an obvious periodicity. New research shows that important implications for discovering groundwater. In addition, many foreign scholars have conducted lots of researches on climate change of Loess Plateau and have recognized that Chinese loess is representative of climate change during glacial stage and interglacial stage, revealing the causes of the earth orbit and regularity of cli-

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mate change, and can be compared with the climate change of deep-sea sediments [18–21]. Loess also developed in other parts of the world, especially in central Asia [22, 23]; for instance, 30 layers of loess and paleosol developed in Tajikistan about 2.1 million years ago [22]. Well-developed loess also exists in Europe, such as Austria, Czech, and Hungary [24, 25]. In Austria at Krems, loess section formed about 2.4 million years ago, and in past 970000 years, the loess formation included at least 17 glacial and interglacial cycles [26].

Although a large number of researches have been done on the components of loess-paleosol, the environmental changes it indicates, the paleosol type, and the monsoonal environment it represents, reports have not appeared about silver sulfate minerals and molybdenum minerals which have important environmental significance. This article aims to explore the causes of drought climate events on the basis of the supergene silver minerals found around Xi'an.

1 Regional overview of the soil section, sampling and methods

The section is located in Renjiapo in the eastern outskirts of Xi'an and the west of Bailuyuan. It is about 8 km to the east of Xi'an City. Xi'an is in the middle of Guanzhong plain with the geomorphological type as the second terrace of Weihe River and has some loess tableland which is well-developed and has a complete range of loess from Early Pleistocene to Holocene. Xi'an area belongs to continental monsoon climate with the annual average temperature of 13.3°C. The summer is hot and winter is cold. The annual average precipitation is about 600 mm and is concentrated from June to October.

Sixty-six samples used to detect $CaCO_3$ and observed by electron microscopy were collected every 10 cm and 20 cm respectively from the clay layers (Bt layer) to leached loess layers and in the concretion sediment layers of $CaCO_3$ of the S_5 section. Methods of electron microscopy, energy spectrum analysis, X-ray diffraction and determination of $CaCO_3$ in the gas volume have been adopted in this article. Experimental analyses of the samples were conducted at the Chemical Laboratory and Environmental Laboratory of Shaanxi Normal University.

2 Experiments and observations

2.1 Soil profile layered in the field

There is well-developed S_5 paleosol with dark red-brown clay layer (Bt layer) and typical prism-like structure in Xi'an area, and the soil surface is generally wrapped with red ferruginous argillans. S_5 paleosol consists of three layers of paleosols with a thickness of about 1.5 m for each layer. Those three layers of paleosol are marked as S_5SS_1, S_5SS_2, and S_5SS_3 from top to bottom, and two loess layers among them are marked as S_5LL_1 and S_5LL_2 from top down. The concretion sediment layers of $CaCO_3$ are distributed in a particular way, not in the bottom of the clay layer, but at a 2.2 m depth below the bottom of the clay layer (Figure 1). The 2.2 m loess layer between the bottom of clay layer and concretion sediment layer of $CaCO_3$ is also eluviated strikingly. Leaching cracks have developed and soil body has broken (Figure 1). And the later measurement shows that there is almost no $CaCO_3$ in it. In the mid-upper part of the 2.2 m leached loess layer, and a lot of red ferruginous argillans appeared (Figure 1). Because $CaCO_3$ and red ferruginous argillans migrate remarkably to the lower loess layer, there is no doubt that S_5 paleosol has the characteristics of leaching type acid soil.

2.2 Observational results of silver sulfate minerals under the electron microscope

In order to observe the microstructure of S_5 paleosol, 23 samples have been observed with the electron microscope, and $AgSO_4$ mineral has been discovered about 0.5 m depth below the Bt layers. According to graphics obtained by electron microscope and chemical composition determined by the energy spectrum analysis, most of the $AgSO_4$ mineral crystals are ellipsoid and of olive shape (Figure 2), characterized by smooth crystal surface, regular shape, mosaic with each other and clear boundaries between the crystals, which indicate this mineral is formed by sediment from holes and cracks which are full of solution. $AgSO_4$ is the

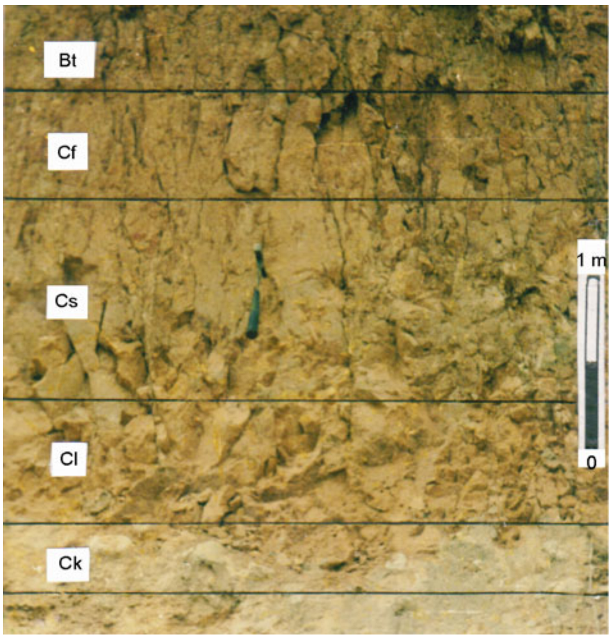

Figure 1 Layer division of the fifth paleosol at Renjiapo in Xi'an. Bt, Clay layer of paleosol S_5; Cf, weathered loess layer with red thick ferruginous argillans; Cs, weathered loess layer with thin argillans; Cl, weathered loess layer without red argillans; Ck, $CaCO_3$ nodule layer.

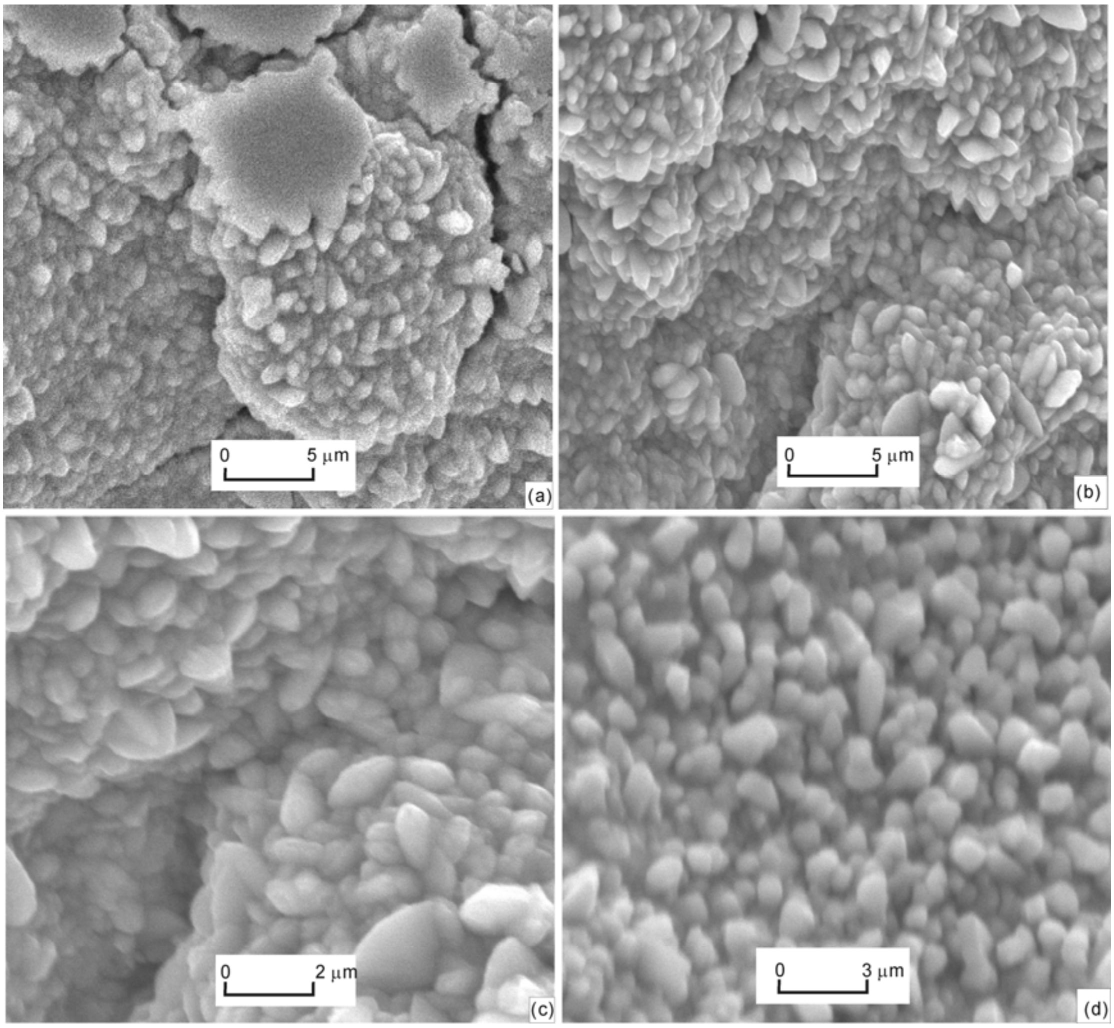

Figure 2 $AgSO_4$ mineral crystals and aggregates at Renjipo of Xi'an. (a)–(d) Silver sulfate particles with ellipsoid shaped and clear boundaries between the crystal, characterized by crystalline materials.

sulfate that is easier to dissolve and precipitate into crystals, the solubility of which is higher than that of $CaSO_4$ [27], and can be easily crystallized into good crystal. So it has clear boundaries between the crystals, smooth crystal surface, and a certain geometric pattern (Figure 2).

With the help of electron microscope and energy spectrum analysis, we can see that oxide mineral form of Mo is mostly spherical with rare ellipsoid shapes densely ordered. Crystal surface is not smooth enough, and crystal particles are in close contact (Figure 3). Crystal boundaries are not clear enough and have the characteristics of colloid aggregation. Pores between crystals can be observed with magnification of 10000 times. Crystals are filled not with debris particles but with a small amount of formless colloidal substances.

2.3 Results of energy spectrum analysis

According to the energy spectrum analysis, there are two types of silver-bearing minerals, one is $AgSO_4$ minerals, and the other is oxide minerals of silver. AgO and SO_3 account for 100% in the sample of S_5-1 (Table 1, Figure 4), and obviously the mineral in the sample is $AgSO_4$. The mineral composition in the samples of S_5-2 and S_5-3 are dominated by AgO, no S and SO_3 minerals, and SiO_2 mineral accounts for 10.59% and 12.51% respectively in those two samples with a small amount of Al_2O_3 and CaO minerals. There are 23.52% of Mo_2O_3 and a small amount of RhO in the sample of S_5-2 (Table 1, Figure 4), and sample S_5-3 contains 25.07% of Mo_2O_3 (Table 1), which indicates that there are oxide minerals of molybdenum in those two samples, and the silver minerals is also the oxide mineral. The mineral compositions in the samples of S_5-4 are also dominated by AgO and SO_3 (Table 1), and the mineral in the samples is $AgSO_4$ mineral.

2.4 Mineral and chemical composition of materials surrounding the silver minerals

In order to identify the source of new silver and molybdenum minerals and chemical weathering of soil before the

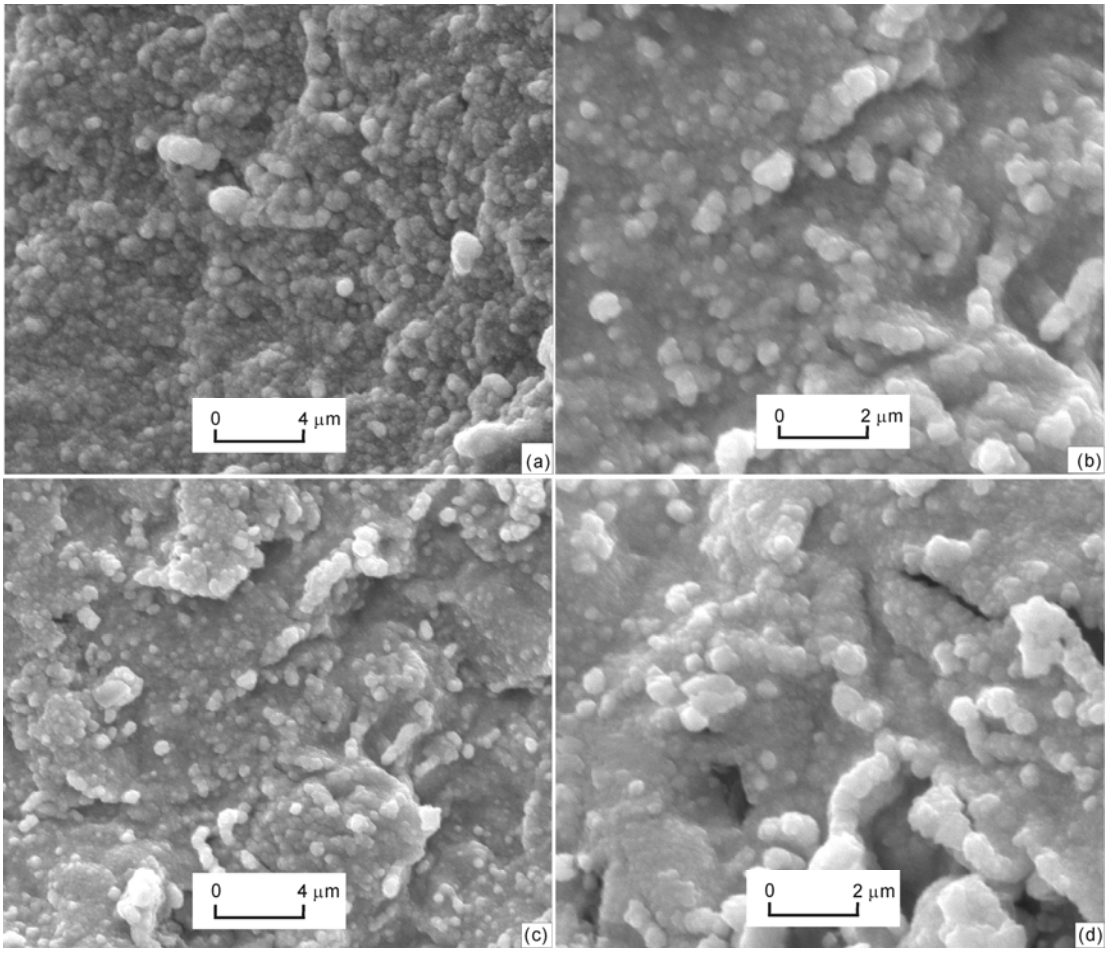

Figure 3 Mineral crystals and aggregates in S_5 at the Renjiapo of Xi'an. Molybdenum mineral crystals and not enough clear boundaries between the crystals characterized by colloidal substances.

Table 1 Chemical constituents of molybdenum and silver minerals in S_5 paleosol at Renjiapo of Xi'an (%)

Sample No.	AgO	Mo_2O_3	SO_3	SiO_2	Fe_2O_3	Al_2O_3	CoO	CaO	K_2O	RhO	MgO
S_5-1	75.36	–	24.64	–	–	–	–	–	–	–	–
S_5-2	58.93	23.52	–	10.59	–	4.17	–	2.51	–	0.028	–
S_5-3	41.95	25.07	–	12.51	6.32	5.57	0.93	7.65	–	–	–
S_5-4	59.58	–	22.58	7.83	2.95	4.34	0.25	0.82	0.37	–	1.28

formation, the clays surrounding hypergene silver minerals have been analyzed through energy spectrum analysis. According to the analysis of four samples (Table 2), the chemical compositions of the clays surrounding silver sulfate minerals are dominated by Al_2O_3 and Fe_2O_3, the former accounting for 22.4%–25.1% and the latter for 12.9%–17.2%.

Al_2O_3 and Fe_2O_3 of clays in S_5 are higher compared with the chemical composition of ferruginous concretion in the yellow-brown forest soil in Jiangsu Province, which is in South China and belongs to the subtropical region [28], which indicates that S_5 has a certain enrichment of Fe_2O_3 and Al_2O_3. $CaCO_3$ tests have shown that little $CaCO_3$, less than 1%, is in the Bt layer of S_5, even 0 in many samples. In about 2 m of earth layer between the bottom part of Bt layer and L_6, the content of $CaCO_3$ is less than 1% too, and $CaCO_3$ in the upper part of the illuvial layer reaches more than 50%.

With help of the electron microscope, micro-crystals of new clay minerals surrounding silver sulfate minerals (Figure 5) can be found, and the micro-crystals have an obvious directional arrangement. The X-ray diffraction shows that the minerals of argillans in Bt layer with relatively pure materials in S_5 are composed mainly of smectite, quartz, smalite, illite, amorphous minerals, and feldspar (Table 3, Figure 6). An 8% of smalite indicates that there was acidic environment in soil at that time, which was consistent with acidic environment shown by the migration of ferruginous

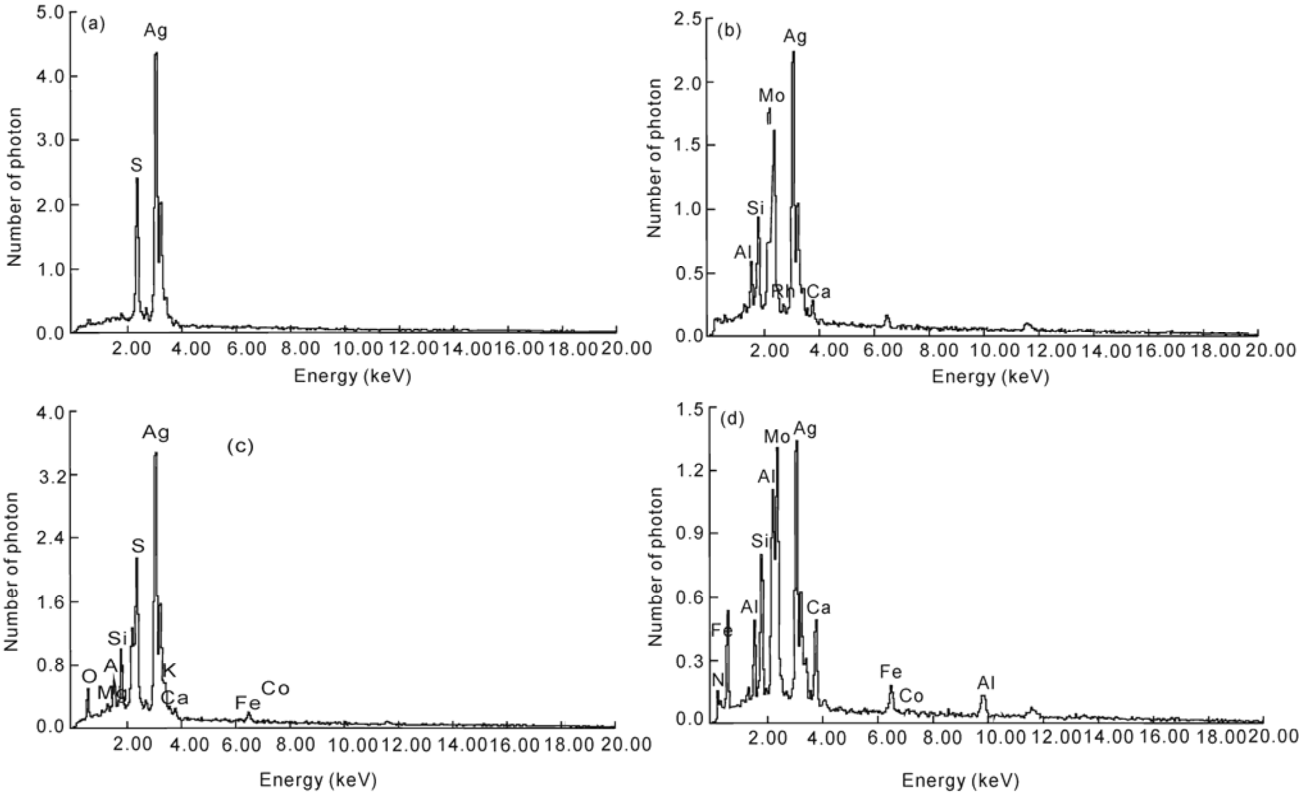

Figure 4 Energy spectrum curves of silver and molybdenum minerals in S_5 paleosol at Renjiapo of Xi'an. (a)–(d) are the energy spectrum curves of sample S_5-1, S_5-2, S_5-3, S_5-4, respectively.

Table 2 Chemical composition of clays surrounding silver sulfate minerals at Renjiapo of Xi'an (%)

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	Others
XL-1	53.4	23.8	15.4	2.3	2.1	3.0
XL-2	53.4	25.1	12.9	3.3	2.7	2.6
XL-3	51.9	22.5	17.2	3.3	2.3	2.8
XL-4	51.4	22.4	16.8	3.0	2.4	4.0

Table 3 Mineral composition of argillans in S_5 at Renjiapo of Xi'an (%)

Samples No.	Smectite	Smalite	Illite	Quartz	Feldaspar	Amorphous	Others
S_5B-1	28	9	7	25	8	19	4
S_5B-1	31	8	17	21	7	16	0
S_5B-3	26	10	11	24	8	18	3
S_5B-4	28	9	12	23	6	18	4

argillans (Figure 1).

3 Discussions

3.1 The origin and weathering of silver in the sulfate minerals

Ag is a pro-Cu element, and its content is very low in the loess [29]. The content of Mo and Co are also very low in the loess [1, 29]. The primary mineral of Ag and Mo are mainly sulfide. Co mainly exists in the basic and ultrabasic melanocratic minerals [30]. The material conditions can be provided for their precipitation and aggregation by these released elements, because forming secondary minerals needs fairly strong chemical weathering on mother rock minerals so that those elements can be released in terms of elements with low content. Consequently, the forming of secondary minerals such as Ag, Mo, Co, etc. in the layers of S_5 indicates that chemical weathering occurs more intensively. Based on minerals, chemical compositions, and migration feature of clay minerals surrounding secondary minerals of silver, we can determine the intensity of weath-

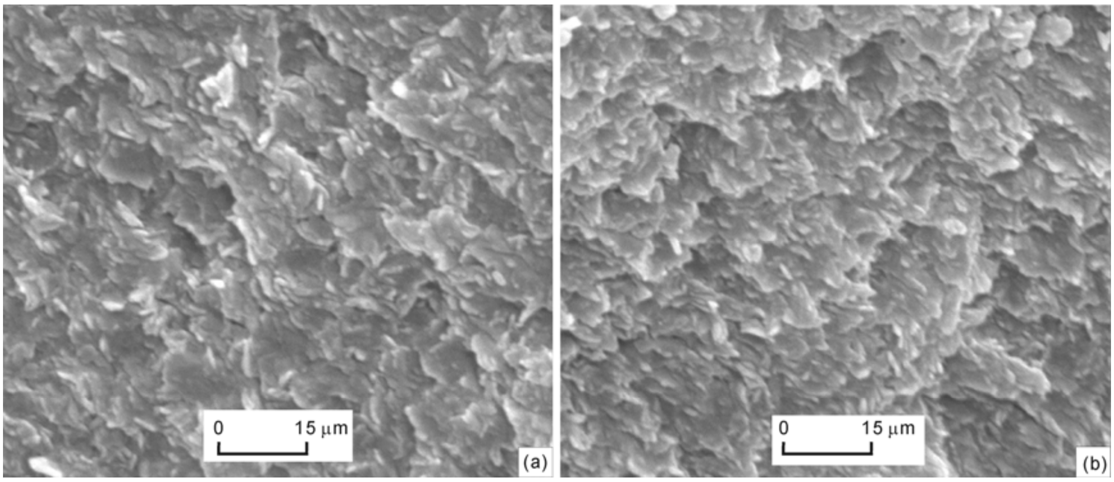

Figure 5 Newly-formed micro-crystal plates of clay mineral in Bt horizon of the fifth paleosol in Xi'an.

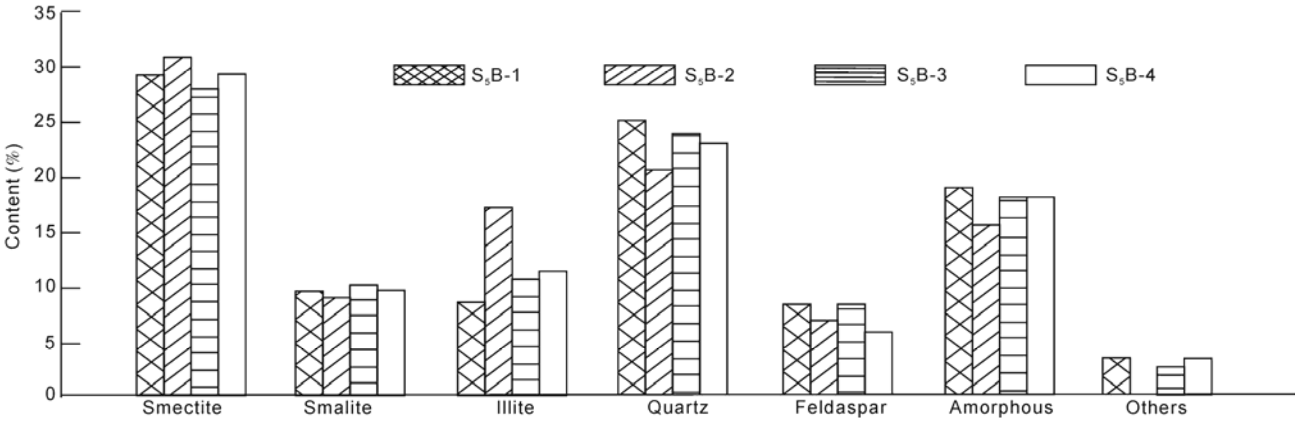

Figure 6 Mineral composition of newly-formed argillans in Bt horizon in S_5 paleosol at Renjiapo of Xi'an. S_5B-1, S_5B-2, S_5B-3, S_5B-4 are the number of the samples.

ering at that time. The above-mentioned energy spectrum analysis of clay minerals shows that the content of Al_2O_3 and Fe_2O_3 in clay minerals is very high (Table 1). The energy spectrum analysis also indicates that Al_2O_3 and Fe_2O_3 not only apparently migrate (Figure 1) and accumulate, but also form secondary clay minerals. The apparent migration of Fe_2O_3 takes place in the acidic water environment. The migration and accumulation of Al_2O_3 and Fe_2O_3 and 8% of smalite in the red argillans indicate that at least the later period of the paleosol development reaches the stage of acidic chemical weathering in Xi'an region.

3.2 The origin of SO_4^{2-} of silver sulfate minerals

The formation of silver sulfate needs a higher concentration of SO_4^{2-} contained in the paleosol. The generating condition of SO_4^{2-} is greatly different from that of silver. On the one hand, it is easy for SO_4^{2-} to leach and run off. On the other hand, the formation of SO_4^{2-} requires the arid climatic conditions. There are two probable reasons about the origin of SO_4^{2-} [31]. First, the origin is from the atmospheric precipitation, and second, it is from the sulfate brought about by the dust storm. In the arid areas and desert regions where there are less precipitation and more intensive evaporation, those ions will precipitate and accumulate in the soil [28]. The soil contains a lot of ions such as SO_4^{2-}, Cl^-, K^+, Na^+ and so on in the arid areas due to little precipitation, intensive evaporation, and the ions of easy migration cannot move and run off. The atmospheric precipitation is an important source not only of SO_4^{2-} in the soil but also of $CaCO_3$ [32]. There is less precipitation in the arid areas where brings in less ions easily dissolved, but the accumulation of long-term process could produce more soluble salt. The development of saline-alkali soil is the result of accumulated soluble salt. The transportation of dust storm may also bring about sulfate and other salts, but the content of sulfate is very low according to component analysis of dust storm, so this kind of source is inferior. Therefore, the dominant source of SO_4^{2-} in the silver sulfate mineral which exists in the lower part of the S_5 paleosol is very likely to be evaporation and sediment of atmospheric precipitation.

3.3 The great change of the climate and monsoon in the development of S_5

The migration depth of the red argillans and $CaCO_3$ in Xi'an region is deeper than that of yellow brown forest soil in Liuhe County of Jiangsu Province. Based on this as well as our previous studies [12], we can conclude that the subtropical climate prevailed when the S_5 developed in Xi'an region. We can also conclude that the mean annual precipitation at that time had reached above 900 mm. Because the summer monsoon was so strong that it could cross Qinling to provide Xi'an region with abundant rainfall. When S_5 developed, a strong summer monsoon caused the decomposition of primary minerals which provided the material conditions for the formation of the secondary materials such as Ag, Mo, Co, and so on. However, the formation of silver sulfate reveals the climate was the typically arid during the development of S_5, which indicates climate and monsoon have changed greatly. In other words, the subtropical monsoon climate changed into the arid non-monsoon climate. The solubility product of silver sulfate is 7.7×10^{-5} mol/L, and calcium sulfate is 9.1×10^{-6} mol/L [27]. The smaller the solubility of the product is, the harder the material dissolves. Therefore, the solubility of silver sulfate is greater than that of calcium sulfate under the same conditions. This indicates it is much easier for silver sulfate to dissolve and migrate than calcium sulfate. In other words, in terms of indicating dry climate, the indication of silver sulfate is stronger than that of the calcium sulfate. Moreover, the climate that silver sulfate represents is more arid than what calcium sulfate doe under the same condition of depth distribution. The modern soil with the formation and deposition of gypsum is nearly all located in the typically arid areas and desert areas. The mean annual precipitation is generally less than 360 mm in its distribution area, and there is no gypsum in the areas where the precipitation is more than 360 mm [28, 33]. The climate which is inland arid or desert is not influenced by the summer monsoon rainfall in the northwestern part of China. The mean annual rainfall is about 360 mm and the type of soil is sierozem, which is deposited by $CaSO_4$ in Lanzhou along the arid northwestern inland. As the rainfall indicated by $AgSO_4$ is less than that by $CaSO_4$, it is determined that the East Asian summer monsoon was very weak at the end of the development of S_5 in Xi'an region. At that time, this region was not influenced by the summer monsoon rainfall, and the climate was typically non-monsoon drought, and the annual precipitation was less than 300 mm. The typically warm-wet climate had changed into the typically arid climate in Xi'an region at that time. The climate in Xi'an region at that time was more arid than that of Lanzhou region today.

3.4 The formation period of silver sulfate mineral

According to migration of silver sulfate mineral and climate in which it has developed, we can estimate which layer of S_5 has developed. Sulfate illuvium of soil has formed in the arid climate [28, 33], and the migration depth of calcium sulfate is less than 2 m in modern soils of arid areas in northwestern China. The illuviation and aggregation of diffluent compositions require less precipitation than that of hard soluble constituents, and it also has a smaller migration depth. For example, soil containing halide needs drier climatic conditions than sulfate does, and the migration depth is less than 1 m and often aggregates in the upper part of soil in northwestern China [28, 33]. The migration depth of silver sulfate is less than 2 m because silver sulfate is much more diffluent than calcium sulfate.

S_5 paleosol consists of three layers, and the thickness of each layer is about 1.5 m. According to the migration depth of silver sulfate and its distribution of 0.5 m below the lowest layer of S_5, silver sulfate is the sediment of the lowest layer of S_5 (S_5SS_3), not that of upper and middle layers. As previously mentioned, there are abundant rainfall and strong eluviations at the time of S_5 formation. Silver sulfate will disappear due to effects by terminal eluviations of S_5SS_3 if it was shaped in the early and middle stages of S_5SS_3. Only silver sulfate formed in terminal stage cannot be influenced by eluviations at that time. It was initially regarded that silver sulfate was formed in the late phase of S_5SS_3 and represented noteworthy dry climate in the terminal period of S_5 formed.

4 Conclusions

(1) Silver sulfate mineral in S_5 paleosol in the eastern suburbs of Xi'an had formed in the course of the development of the lowest layer of S_5, and precipitated from the solution of soil, which is due to easy solution and crystallization of silver sulfate mineral. The oxides of molybdenum mineral have the same features as colloidal substances, due to its hard solution.

(2) Terminal period when the lowest layer of S_5 paleosol was formed in eastern suburbs of Xi'an is under the condition of typical dry climate characterized by insufficient precipitation, weak eluviations, and strong evaporation. Sulfate ion in the atmospheric precipitation can assemble in the paleosol, which leads to high concentration of it in soil water solution.

(3) Low content of silver and molybdenum in loess and the gathering of some neogenic minerals such as silver sulfate and molybdenum indicated that there used to be intense chemical weathering. The obvious migration of red argillans and calcium carbonate and plentiful smalite have manifested that there used to be warm and humid climate. When weathering was intense, it reached a stage of acidity and

chemistry weathering. The elemental differentiation was obvious and had a condition that silver, molybdenum and cobalt separated from primary mineral.

(4) The emergence of silver sulfate mineral under the bottom of the lowest of S_5 paleosol indicates the transformation of climate from warm and humid one to cold and dry one. When silver sulfate mineral was formed, typically dry non-monsoon climate appeared in Guanzhong Plain, and summer monsoon in East Asia at that time was so weak that normally could not cross Qinlin Mountain to Guanzhong Plain. The annual precipitation in Xi'an at that time was below 300 mm, and the climate was colder and drier than that of Lanzhou today.

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