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Long-term effects of soft rock amendment on changes of soil aggregate cementing agents of sandy soil by SEM-EDS

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Soil aggregates are a crucial constituent of soil and have a significant function in regulating water, nutrients, air, and heat within the soil. The development of soil aggregates is influenced by various factors, including the soil's parent material and human activities. Understanding the formation and the mechanism of stabilization of soil aggregates is of great significance in the study of soil development, in regulating and managing organic carbon pools in soils, and in promoting soil fertility. In this study, aeolian sandy soil with a low degree of soil development and compound soil formed by combining soft rock and aeolian sandy soil were selected as the research objects. We selected three time points from 0 to 9 years after amendment by soft rock in order to investigate the changes of soil aggregate cementing agents. The shape of soil aggregates in both types of soils was analyzed by environmental scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), which were also used to assess the appearance of soil aggregates and quantify the composition of mineral elements on a cross section of the aggregate. The results show that when the soft rock and the aeolian sandy soil are compounded and mixed, the clay minerals in the soft rock change the microstructure of the original aeolian sandy soil from a single granular barrier to one characterized by a cumulative porous structure, indicating that clay minerals promote soil development and form aggregates with good structural properties. The cementing agents in the compounded soil aggregates are mainly clay minerals, aluminum, iron, and calcium. In comparison to aeolian sandy soils, the presence of iron and calcium in compounded soils is notably elevated. The iron oxides present in compounded soils serve a similar function to "bolts" in the formation of soil aggregates. These findings establish a theoretical foundation for investigating the process of soil aggregate formation and the mechanisms by which cementing agents contribute to their stabilization.

KEYWORDS

aeolian sandy soil, compound soil, soil aggregate, cementing agent, environmental scanning electron microscopy

1 Introduction

Soil aggregates are an important component of soil and are widely regarded as the basic unit as well as an important parameter of soil structure (Barral et al, 1998; Hou et al, 2018). Aggregates play important roles in coordinating water, nutrients, air, and heat in soil, affecting the types and activities of soil enzyme, maintaining and stabilizing soil, and loosening and curing layers (Haynes et al, 1993; Jastrow et al, 1996). The formation of soil aggregates is a very complex process involving a range of physical, chemical, and biological effects that depend primarily on the quantity and nature of the various materials of composition in the soil (Chaney et.al., 1986a; Janalizadeh et al, 2019b; Lado et al, 2004). During the process of soil particle aggregation, not only organic compounds play an important role in particle cementation, but inorganic compounds such as iron and aluminum oxides and hydroxides, as well as silica and calcium carbonate also play important roles (Arya et al, 1972; Falsone et al, 2007; Wiesmeier et al, 2012). The parent material is the primary material that makes up soil, and has a great influence on the composition and action of inorganic soil cementing agents (Bosch-Serra et al, 2017; Oades et al, 1993). The formation of organic cementing agents is related to the quantity of microorganisms, their activity and metabolites, plant root exudates, and organic matter input (Chaney et.al., 1986b; Fakhrabadi et al, 2021; Gale et al, 2000).

Soil cementing agents may be inorganic, organic, or a combination of organic and inorganic (Giovannini et al, 1976; Shi et al, 2002) Although these three types of cementing agents may be found at the same time in different soil types, the composition of organic and inorganic cementing agents in the soil differs due to differences in parent materials, bioclimatic conditions, and agricultural management. In soils with high organic matter content and low clay and oxidized iron and aluminum, the role of organic matter is dominant (Guénet et al, 2016; Ren et al, 2011); in soils with low organic matter content, but high clay and oxidized iron and aluminum content, the formation of soil aggregates is mainly due to the cohesive force between clay particles and due to cementation by iron and aluminum oxides (Barral et al, 1998; Roshan et al, 2022; Skjemstad et al, 1993). Numerous studies have investigated the quantity, distribution, stability, and other factors that influence the formation of soil aggregates (Chaney et.al., 1986a; Terpstra et al, 1990; Diaz et al, 1994; Schomburg et al, 2018). However, such studies cannot describe the internal structure of soil aggregates, explain the process of aggregate formation, or the cementation mechanism. The basic physical indicators of aggregates contain very limited information and do not reflect the complex internal structural differences of the aggregates, but it is difficult to distinguish and discriminate the shape and internal composition of soil aggregates.

Soft rock, formed in continental clastic rock system of late Paleozoic Permian, Mesozoic Triassic, Jurassic, and Cretaceous (Sun and Han, 2018), has the characteristics of low diagenetic degree, poor intergrain bond and low structural intensity and is the fundamental reason for serious water loss and soil erosion in soft rock area. To solve the problems of the sand soil sharing common root but with different qualities and that it is hard for sandy to become soils, Han et al (2012) found that soft rock had great potentials as sandy remediating material. They prepared "new type soil" by mixing soft rock sandstone with sand at certain proportion, which not only could solve the problems of the sandy being unable to form colloidal materials due to poor resistance and fertility, but also could alleviate the trouble of nonlocal soil dressing. Applied in Mu Us Desert land consolidation projects, this technology helped increase the arable areas of more than 200,000 ha. Corns and potatoes have been planted in the "compound soil" with high yield. In this way, utilization of sand and soft rock is realized, which has increased effectively the arable areas and guaranteed food security with good ecologic, economic and social benefits (Sun et al, 2021). To study furthermore the improvement of soft rock of aeolian sandy soil as well as the formation of this new type of "artificial soil" mixed with soft rock and sand and to realize multi-purpose application of the compound soil, it is hard to diagnose and study relying only on some routine features of cross section form and the fundamental physical and chemical analysis; therefore, it requires also to study profoundly the micro morphology of the compound soil as well as the formation and development of the aggregate cementing materials in the compound soil.

With the advancement of soil morphology research, research on the structure of soil aggregates has evolved from qualitative observation by scanning electron microscopy (SEM) to quantitative analysis combined with image processing technology (Garbout et al, 2013; Hapca et al, 2015; Wang et al, 2012; Wilson et al, 2013). SEM has the characteristics of high magnification, high resolution, and large depth of field. It can be used to directly observe the original microstructure of soil aggregates and can be used together with X-ray diffraction spectroscopy to achieve simultaneous analysis of morphology at a microscopic scale and elemental composition (Sayen et al, 2009; Jiménez-Pinilla et al, 2016; Yu et al, 2017). It has become an important tool for the analysis of the morphology of clay mineral particles. The combined analysis of the qualitative and quantitative microstructure data provides information on the various nutrients, microorganisms, and cementing agents in soil aggregates and is an ideal method by which the state and mechanism of soil aggregate formation may be explored. For this research, the selected objects of study were aeolian sandy soil with low soil development and a compound soil created by combining aeolian sandy soil and soft rock. Understanding the nature of organic cementing materials, the concepts behind the formation of soil aggregates, the mechanisms of soil aggregate stability, and the mechanism for renewal and turnover can help to further explore changes in soil development and succession, organic carbon pool, water stability of soil aggregates, and other soil fertility indexes once virgin land is first opened for cultivation, as well as the mechanism of the influence of soil structure on soil fertility (Yu et al, 2017; Watteau et al, 2012; Zhang et al, 2015). The main purpose of our study is to determine, through the use of SEM-energy dispersive spectroscopy (EDS):

- the main composition of cementing agents on the surface of soil aggregates during soil development
- to quantify the distribution of major mineral elements on the surface of soil aggregates

TABLE 1 Basic characteristic of sandy soil and soft rock^a.

Material	Texture	Sand	Silt	Clay	BD	рН	CEC	Organic matter	Fe	Al	Ca
		(%)	(%)	(%)	(g cm⁻³)		(cmol kg ⁻¹)	(g kg ⁻¹)	(%)	(%)	(%)
Aeolian sandy soil	Sand	95.4	4.1	0.5	1.6	7.4	3.86	2.93	1.81	5.04	2.25
Soft rock	—	34.9	58.1	7	1.4	8.1	45.79	1.74	4.82	8.40	1.16

^aPercentage of clay (<0.002 mm), silt (0.002–0.05 mm), and sand (0.05–2 mm) particles, measured by Pipette Method; BD, bulk density, measured by undisturbed soil core method; soil pH was measured with a soil: water ratio of 1:2.5 using an ion pH meter; CEC, cation exchange capacity, measured by shaking 1 mm of air-dried soil with 1 M NH4OAc at pH 7.0.

TABLE 2 Mineral composition of sandy soil and soft rock.

Material	Quartz	Kaolinite	Montmorillonite	Feldspar	Calcite	Dolomite	Amphibole					
		(%)										
Aeolian sandy soil	82	4	—	10	2	—	2					
Soft rock	57	—	30	10	—	3	_					

• to elucidate the mechanism of action of cementing agents during the formation of soil aggregates.

2 Materials and methods

2.1 Sites and soil sampling

The aeolian sandy soil and compound soil selected for the experiment were collected from the Mu Us desert, Jingkeliang, Daji Khan Village, Yuyang District, Yulin City, China (38°27′53″N, 109°28′58 E). The area is located in northwestern Shaanxi at an altitude of 1,210 m above sea level and is a typical warm temperate monsoon climate. The annual average temperature is 6.0°C-8.5°C and the annual precipitation is 400-440 mm. The compound soil is composed of materials from soft rock (also called Pisha sandstone or Feldspathic Sandstone) and aeolian sandy soil in a volume ratio of 1: 2 [Detailed field experiments design were shown in Sun et al (2018)]. The physical and chemical properties of the aeolian sandy soil and the soft rock materials are shown in Tables 1, 2. The soft rock is composed of Paleozoic Permian, Mesozoic Triassic, Jurassic and Cretaceous thick layer sandstones, sand shale, and argillaceous sandstones (Bazhenov et al, 1993; Martin et al, 1999). Its rock layer is thin, with low pressure, low diagenesis, and poor cementation between sand grains and poor structural strength (Wang et al, 2009). In recent years, some scholars have used the complementary properties of soft rock and aeolian sandy soil to form a new compound soil (the soil at this time is only at the beginning of its development) which is an improvement on aeolian sandy soil (Sun et al, 2018; Han et al, 2015; Han et al, 2012; Wang et al, 2013; Sun et al, 2019). As the years of cultivation increase, the compound soil tends to ripen. Therefore, this study selected aeolian sandy soil, compound soil that has undergone 3 years of planting, and compound soil that has undergone 9 years of planting as research materials. Undisturbed soil was collected from topsoil and the soil samples were taken back to the laboratory and naturally airdried. The dry sieve method was used to screen the 1-2 mm soil aggregates that were the objects of observation in this study.

2.2 SEM and X-ray diffraction methods

The German LEO1430VP scanning electron microscopy (SEM) and the OXFORD7353 energy dispersive spectrometer (EDS) were used in combination. The SEM was operated with a test voltage of 30 kV, a secondary electron resolution of 3.5 nm, and a maximum magnification of 900,000 times. The EDS was used for quantitative analysis of the mineral elements in the sample, with an error of less than 5%. SEM-EDS was used to observe and analyze the soil aggregates (1–2 mm). The relatively flat interparticle cementation was used as the location of analysis for surface cementation, which standardized the locations where analyses were performed. In this study, 10 typical cementation points were selected on the surface of each observed soil aggregate for energy spectral point element analysis.

Soil aggregates 1.0–2.0 mm were selected for cross-sectional elemental analysis. The selected aggregates were embedded and fixed with polyester resin and sample columns with a diameter of 2 cm and a height of 1 cm were cut out, and the sample was mechanically polished. The cross section of the compound soil was observed by SEM, and the elemental distribution of the soil aggregate section was analyzed using the element mapping mode of EDS. Firstly, the typical cross section of the target aggregate was found at low magnification and then the overall element distribution analysis was performed by using the mapping mode. Finally, the overall regional elements are collected and analyzed quantitatively using pattern mode analysis. The SEM was set on fast mode, working distance 10 mm, acceleration voltage 15 kv, dead time is at 30%–50%, and then the result was subjected to zaf wt% normalization and correction.





2.3 Statistical analysis

Statistical analysis was performed using the PROC GLM process in SAS for equation analysis. The mean comparison was performed using the least significant difference (LSD) method with a significance level of p < 0.05 between treatments.

3 Analysis result

3.1 Aggregate trait analysis

The microaggregates of aeolian sandy soil are mainly composed of single particles of irregular shape, with high degree of rounding, smooth surface, almost no sharp edges and corners, and no bonding surface (Figures 1A, C). Compared with the aeolian sandy soil aggregates, the compound soil aggregates have a certain number of bridging barrier structures formed by the bonding of small mineral particles as cementing agents to the core structural particles; the coarse core structural particles are filled by fine particles, which either form filler-like junction structures filling the voids of the core structure or an envelope-like structure encapsulating core structural particles (Figures 1B, D). The differences in morphological characteristics between compound soil and aeolian sandy soil can be clearly observed on the surface of compound soil microaggregates, where $2-40 \,\mu m$ morphologically different pore structures can be observed (Figure 2A). The presence of hyphae was also observed on the surface of compound soil aggregate structure.



FIGURE 3

SEM-EDS analysis of aeolian sandy soil aggregate. SEM image of the sample (A), and corresponding X-ray maps of O (B), C (C), Al (D), Si (E), Fe (F), Ca (G), Mg (H), Na (I), and K (J).

3.2 Soil aggregate cross-section elemental distribution

The aeolian sandy soil aggregate section has regions of significant Si and Al enrichment, and only a small amount of Fe

and Ca (Figure 3). The distribution of Al, Ca, and Fe in aeolian sandy soil aggregates are intermittently in the soil aggregate cross section. Areas of highly concentrated Si, Al, Fe, and Ca appeared in the cross section of compound soil aggregates that has undergone 3 years of planting (Figure 4). The Al-Mg distribution zones in the



FIGURE 4

SEM-EDS analysis of compound soil aggregates that has undergone 3 years of planting. SEM image of the sample (A), and corresponding X-ray maps of O (B), C (C), Al (D), Si (E), Fe (F), Ca (G), Mg (H), Na (I), and K (J).

compounded soil have strong correlation and connectivity, and run through the entire soil aggregate cross section and form the basic shape of the aggregate section. Al has obvious regions of enrichment that exist mainly as semi-joined points. The areas of Al enrichment are mostly concentrated on the pores in the cross section and at the edge of the core structural particles. Ca is distributed independently in some parts of the section, and is relatively far from the areas containing Al-Mg.

Treatment	Analyzed sites	Element (%)								
		Ο	Na	Mg	Al	Si	К	Ca	Fe	
3 year aeolian sandy soil		49.98ª	0.78ª	1.91 ^b	11.87ª	26.93ª	2.72ª	1.73°	4.08 ^c	
3 year compound soil		43.56 ^b	1.34ª	2.72ª	11.05 ^a	28.50 ^a	2.09 ^a	3.47 ^b	7.27 ^b	
9 year compound soil		41.07 ^c	0.83 ^a	2.52ª	10.58ª	27.26ª	2.60 ^a	4.40 ^a	10.74 ^a	

TABLE 3 Analytical results of X-ray energy spectrum characteristics of the aggregates in aeolian sandy soil and compound soils.

Mean values in the table, and columns within the same soil layer and with the same letters are not significantly different at p < 0.05 according to a protected LSD test. a, b, c: Columns within the same soil layer and with the different letters are significantly different at p < 0.05 according to a protected LSD test.

3.3 Analysis of elemental content on the surface of soil aggregates

The relative contents of Fe, Ca and Mg in the compound soil aggregates were significantly higher than those in the aeolian sandy soil (Table 3). It suggests that Fe, Ca, and Mg elements play an important role in the cementation process during the formation of soil aggregates. From this, it can be inferred that compared with aeolian sandy soil, the cementing materials on the surface of compound soil aggregates mainly include ferric oxide, calcium carbonate, and magnesium carbonate. After 9 years of cultivation, there are more iron cementation points on the surface of large aggregates in compound soil, and the content of Fe super-enriched zones is higher. The relative content of Fe increased from 7.27% to 10.74% and the distribution area is more extensive and tight. The Ca enrichment area also increased and the relative content of Ca increased from 3.47% to 4.40% (Table 3).

4 Discussion

Soil minerals are important constituents of soil and can be divided into three types: primary minerals, secondary minerals, and soluble minerals (Gislason et al, 1996; Ghadakpour et al, 2020). Primary minerals in soft rock and compound soils are silicate minerals such as quartz and feldspar. The main elements are Si, Al, and O. Secondary minerals include clay minerals, secondary oxides, and salts. The clay minerals are mostly in the form of cementing agents (Afrakoti et al, 2020; Roshan et al, 2020). The elemental composition is mostly Si, Al, O, Mg, K, Ca, and Fe. Secondary oxides are iron oxide or oxidized alumina, and its elemental composition is mostly O, Fe, and Al. Salt minerals include carbonates and other substances, the main elements of which are O, C, and Ca. In this study, the resin embedding technique was used for the cross-section analysis of the soil aggregates. The constituent elements of the resin were C and O. Therefore, C and O were not analyzed in this study.

This study analyzed the mapped image of the elements found on the cross-section of 1–2 mm soil aggregates in compound soil. According to the distribution characteristics of the Fe-rich region using iron oxide as the indicator, iron oxide is mainly in the form of an organic-inorganic composite formed by the iron oxide cemented by adsorption or co-precipitation with organic matter, and is mostly present around the core structural particles of the soil aggregate and at the pores as a clear semi-continuous envelope (Janalizadeh et al, 2019a; Koutenaei et al, 2021). Iron oxides act like "bolts" in the development of compound soil aggregates. In addition, the high degree of association between Fe-Ca distribution zones and Fe



FIGURE 5

SEM-EDS analysis of compound soil aggregates that has undergone 9 years of planting. SEM image of the sample (A), and corresponding X-ray maps of O (B), C (C), Al (D), Si (E), Fe (F), Ca (G), Mg (H), Na (I), and K (J).

enrichment zones in the cross section of compound soil aggregates cultivated for 3 and 9 years indicates that good clay mineral-metal oxide-organic matter composite cementing agents is formed in the compound soil (Figures 4, 5). The composite cementing system is more stable as the years of cultivation increases.

The interaction between iron-aluminum oxide and clay minerals is closely related to the cementation of soil aggregates (Barral et al, 1998; Amézketa et al, 1999; Duiker et al, 2003; Molina et al, 2001). For the new "artificial soil" with low organic matter content and high content of clay minerals and iron-aluminum oxides (Al_c:

Treatment	Al _f	Fe _f	Al _c	Fe _c	Clay mineral content (%)	Relative content of clay minerals (9		nerals (%)	
	g/kg		mg/kg			I/S	Illite	Kaolinite	Chlorite
3 year aeolian sandy soil	0.59	5.06	0.61	1.03	13.7	45	27	12	16
3 year compound soil	0.48	7.66	2.17	4.92	21.2	47	28	12	13
9 year compound soil	0.08	7.42	2.07	3.97	12.9	52	24	11	13

TABLE 4 Analytical results of	f cementing materials	content in aeolian	sandy soil and	compound soils ^a
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^aAl_f is free aluminum oxide, Fe_f is free iron oxide, Al_c is complex aluminum, Fe_c is complex iron, I/S is illite smectite mixed layers.

2.17-2.07 mg/kg; Fec: 4.92-3.97 mg/kg) in the compound soil (Table 4), the formation of soil aggregates mainly depends on the cohesive force within the clay and the cementation of ironaluminum oxide (Barral et al, 1998; Choobbasti et al, 2017; Skjemstad et al, 1993). This study found that iron oxides have obvious distribution characteristics in the compound soil aggregates: Fe is mainly concentrated at the outside of the aggregates, and the surface Fe aggregates become more and more abundant with increased years of cultivation (Figure 5). The free iron oxide in the compound soil aggregates showed a small downward trend with the increase in years of cultivation (Table 4). This is mainly because the soft rock contains minerals rich in Fe, adding the soft rock to the aeolian sandy soil increases the content of free iron oxide in the compound soil. Since iron oxide is very active in the soil environment and can move, under the continuous action of soil roots, leaching and redox, free iron oxide continuously enters the soil solution and adsorbs to the surface of the aggregate, effectively promoting the cementation of soil aggregates (Choobbasti et al, 2015; Muggler et al, 1999; Barberis et al, 1991). On the other hand, as a redox sensitive element, Fe is continuously enriched at the surface of the aggregate due to the difference of redox conditions on the inside and the outside of the compound soil aggregates and due to the lack of pores in the aggregate (Yaghi et al, 2013; Huang et al, 2016). This also leads to positively charged iron oxides on the surface of the compound soil aggregates becoming more easily cemented together with the negatively charged aggregates by electrostatic forces, causing the soil aggregate particle size to increase (Choobbasti et al, 2018). With increased years of cultivation, the free iron oxide in the compound soil ages, the free iron oxide is continuously reduced, and the iron oxide in the cemented state is gradually increased, which also reveals the soil formation process of the compound soil. In general, iron plays an important role as a "bridge builder" in the development of compound soil aggregates (Giovannini et al, 1976; Jiang et al, 2015).

Compared with compound soil, the distribution characteristics of clay minerals and aluminum oxides in aeolian sandy soils are not significantly different, but iron oxides exhibit a certain distribution on the surface and inside of aeolian sandy soil aggregates (Table 3), showing the difference in the distribution of cementing agents between the aeolian sandy soil and compound soil. The content of free iron oxide and complexed iron oxide is small, indicating that the aggregate cementation strength in aeolian sandy soil is low, which also illustrates in a different way the difference between the redox system and the pore channels inside soil aggregates from aeolian sandy soil and compound soil. When soft rock is compounded and mixed with the aeolian sandy soil, the microstructural characteristics of the original aeolian sandy soil with a single-grain barrier are changed, which promotes the development of "new man-made soil" to form bridges, fill microaggregates, and promote good structural characteristics, mainly due to the positive effect of the cementing materials in the soft rock on soil formation and development of the compound soil.

With the same number of cultivation years, the clay mineral content in the compound soil is higher than that in the aeolian sandy soil, and the clay minerals in the compound soil and the aeolian sandy soil are mainly water-sensitive illite smectite mixed layers. This is mainly because soft rock contains abundant water-sensitive clay minerals (Li et al, 2014; Ma et al, 2016). These clay minerals have strong hydrophilicity, specific surface area and cation exchange capacity (Van et al, 1995; Joussein et al, 2004). The surface of the clay particles has strong hydrophilicity, which can absorb water and nutrients in the soil, forming a cohesive substance and promoting the formation of soil aggregates (Ghadakpour et al, 2020). In addition, clay particles can also provide support force to promote the stability of soil aggregates. After soft rock is used as a remediation material and compounded and mixed with aeolian sandy soil, under the action of continuous leaching and weathering, the soft rock gradually disintegrates, and the clay minerals are continuously released and redistributed (Li et al, 2017; Ribeiro et al, 2018). The clay minerals adsorb to the minerals in the aeolian sandy soil and improve its surface characteristics, which promotes the development of microaggregates in compound soil.

5 Conclusion

In this study, SEM-EDS was used to characterize the microstructure and elemental distribution of soil aggregates in aeolian sandy soils and compound soil containing a mixture of soft rock and aeolian sandy soil, and the formation and development and aggregation of cemented materials in compound soil aggregates were studied. The results show that when soft rock and aeolian sandy soil are compounded and mixed, the micro-structure of the original aeolian sandy soil, which is primarily single granular barrier, is changed, promoting the formation and development of soil and forms soil aggregates with good structural characteristics. Compared with aeolian sandy soil, the cementing agents in the compound solid aggregates are mainly clay minerals, aluminum cements, iron cements, and calcium cements. During soil development, many aggregates are fixed by combining with organic matter, which further

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strengthens the stability and development of soil aggregates. The iron oxides in the compounded soil play a role similar to "bolts" during the development of soil aggregates. This study provides a theoretical basis for the study of the formation process and stabilization mechanism of soil aggregates driven by cementing agents. This study also has some shortcomings. It only analyzed the surface morphology characteristics of the complex soil, and did not detect the structures below the surface. Quantitative research needs to be strengthened in future studies.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author.

Author contributions

ZS and ZL wrote the main manuscript. JH and ZS designed the experiment. ZL performed the experiments and collected the data. HW and HZ prepared the figures. ZS and JY contributed the statistical analyses. All authors contributed to the article and approved the submitted version.

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Conflict of interest

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