Changes in diffuse reflectance spectroscopy properties of hematite in sediments from the North Pacific Ocean and implications for eolian dust evolution history

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Abstract: Eolian dust preserved in deep-sea sediments of the North Pacific Ocean (NPO) is an important recorder of paleoclimatic and paleoenvironmental changes in the Asian inland. To better understand changes in the dust provenances, in this study diffuse reflectance spectroscopy (DRS) was used to extract the eolian signal recorded in sediments of ODP Hole 885A recovered from the NPO. First, we systematically investigated sieving effects on the DRS data; then band positions of hematite (obtained from the second order derivative curves of the K-M remission function spectrum derived from the DRS) were used to distinguish different provenances of the eolian dust preserved in the pelagic sediments of this hole. Our results show that the sieving (38 μm) process can suppress effectively the experimental errors. Eolian signatures from Chinese Loess Plateau (CLP) sources and non-CLP-sources have been identified in the pelagic sediments of ODP Hole 885A from the late Pliocene to the early Pleistocene. The provenance differences account for the discrepancies in the eolian records recovered from the pelagic sediments in the NPO and profiles in the CLP. Temporal changes in dust provenances are caused by the latitudinal movement of the westerly jet mainstream. The hematite DRS band position is a useful tool to distinguish the provenance of eolian components preserved in pelagic sediments.

Keywords: eolian dust; diffuse reflectance spectroscopy; hematite

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1. Introduction
Eolian dust depositions in pelagic sediments of the North Pacific Ocean (NPO) (Rea, 1994; Larraoana et al., 2003; Roberts et al., 2011), terrestrial stratigraphic profiles (Liu DS, 1985; An ZS et al., 2001; Guo ZT et al., 2002; Nie JS et al., 2008, 2015, 2018; Liu XM et al., 2017; Wang XS et al., 2015), and ice cores (Bory et al., 2003) carry considerable information about Cenozoic climate changes in the Asian inland and Northern Hemisphere atmospheric circulation. The NPO has extensive deep-sea basins, lies downwind from major Asian dust sources (Tanaka and Chiba, 2006), and underlies the mainstream of westerlies; receiving abundant wind-derived particles, the NPO is thus an important eolian depositional sink.

Quantifying eolian dust recovered from different natural settings and tracing its provenance are core topics for modern eolian research. For the former, chemical separation procedures (Rea and Janecek, 1981) and rock/environmental magnetism (Thompson and Oldfield, 1986; Evans and Heller, 2003; Liu QS et al., 2012) have been widely used to extract the eolian signal preserved in marine sediments based on chemical and magnetic properties of wind-borne minerals, respectively. For the latter, isotope technology (Sr-Nd) (McCulloch and Wasserburg, 1978), and conventional geochemistry (major and trace elements) (Taylor et al., 1983), and electron spin resonance (ESR) (Ono et al., 1998) have been applied for tracing the source of eolian fractions recovered from pelagic sediments (Pettk et al., 2000; Nagashima et al., 2007; Kyte et al., 1993). Application of these techniques has contributed significantly to knowledge of changes in climate and environment of Asian inland arid and semi-arid source areas and evolutionary history of eolian dust over different time scales.

Diffuse reflectance spectroscopy (DRS) provides a rapid, non-destructive, quantitative means to determine even very low concentrations of antiferromagnetic minerals (hematite and goethite) (Balsam and Deaton, 1991). This method has been used extensively to extract hematite/goethite information from different natural settings (Deaton and Balsam, 1991; Balsam et al., 2004; Ji JF et al., 2004; Torrent et al., 2007; Zhang YG et al., 2007; Liu QS et al., 2011; Oldfield et al., 2014). The eolian fractions contain abundant hematite and goethite (Balsam et al., 1995). The DRS result is significantly affected by multiple factors, such as grain size, aluminum (Al) content, specific surface area (SSA), and crystal shape (Torrent and Barrón 2003, 2008; Liu QS et al., 2011). In order to minimize
the effect of changes in grain size, samples need to be dehydrated and ground during pretreatment for DRS test, but manual grinding may not attain homogeneous grain size, which may interfere with accurate interpretation of diffuse reflection. The process of sieving through a grid (38 μm) can improve the homogeneity of particle grain size and make the results more consistent (Balsam and Deaton, 1991); however, in most cases, especially high-resolution research with massive samples, the process of sieving is usually ignored because it is time consuming. In this study, we will systematically evaluate this effect.

A 70-meter (post-Cretaceous) record of pelagic sediment at ODP Hole 885A (44° 41′ N, 168° 16′ W; water depth = 5708.5 m, Rea et al., 1993) permits study of the evolution of eolian dust input. This hole is located downwind of Asia and underlies the scope of the westerly jet, which is sufficiently far from the continent to preclude riverine inputs and ice-rafted debris influences (Snoeckx et al., 1995). Some volcanic ash layers have been identified by lithostratigraphy (Rea et al., 1993) and environmental magnetic proxies (Bailey et al., 2011; Zhang Q et al., 2018). Several authors have recovered the evolutionary history of eolian deposition for this hole over different timescales, based on chemical extractions and rock/environmental magnetism (Snoeckx et al., 1995; Sun YB and Liu QS, 2007; Bailey et al., 2011).

Recently, we have applied DRS technique to extract eolian signals in the sediments of this hole and defined a new parameter $R_{Hm+Gt}$ to indicate the combined hematite and goethite concentration (Zhang Q et al., 2018), we found that parameter $R_{Hm+Gt}$ correlated remarkably well with the percentage content of eolian fractions obtained by chemical separation (Sun YB and Liu QS, 2007) and is thus a reliable and useful eolian dust proxy. Compared with other parameters, the $R_{Hm+Gt}$ can preclude the influence of biogenic and volcanic components. Pettke et al. (2000) made use of the Sr-Nd isotope to demonstrate that the silicates fractions of eolian dust in ODP Hole 885A have always been derived from basins north of the Himalayan-Tibetan Plateau and the Gobi Desert in central Asia over the past 12 Ma. Recently, based on the detrital zircon U-Pb ages of the red clay and loess in the Chinese Loess Plateau (CLP), it has been demonstrated that the eolian components in the ODP Hole 885A, including silicates and antiferromagnetic minerals, are derived mainly from the Asian inland (Pettke et al., 2000; Nie JS et al., 2018; Zhang Q et al., 2018); hence the hematite band position can be employed to distinguish the sources of hematite derived from Asian inland regions. It has been demonstrated that the eolian components in the ODP Hole 885A, including silicates and antiferromagnetic minerals, are derived mainly from the Asian inland (Pettke et al., 2000; Nie JS et al., 2018; Zhang Q et al., 2018); hence the hematite band position can be employed to distinguish the eolian provenance of the pelagic sediments in this hole, which can be characteristic by higher resolution.

Guided by previous results, in this study we will further investigate the changes in hematite band position obtained from DRS and the relationship of these new data to the pre-existing eolian records in the CLP and NPO, in order to decipher the provenance changes of eolian depositions with higher resolution in ODP Hole 885A from late Pliocene to early Pleistocene and explore tentatively the mechanism driving the changes.

2. Material and Methods

DRS was applied to samples obtained from deep-sea sediments in ODP Hole 885A (44° 41′ N, 168° 16′ W; water depth = 5708.5 m, 5.44 to 16.66 meters composite depth, Figure 1b) using a Cary 5000 ultraviolet-visible-infrared spectrometer equipped with BaSO₄ as the white standard. The scan rate was 300 nm/min from 300 to 2600 nm in 0.5 nm steps. DRS data were first transformed into the K-M remission function ($F(\lambda) = (1-R(\lambda))/2R$, R is reflectance), and then second derivative curves of $F(\lambda)$ were calculated following Scheinost et al. (1998) and Torrent et al. (2007). The corresponding band intensities for hematite and goethite, which are proportional to the concentrations of hematite and goethite, are defined as $I_{Him}$ and $I_{Gt}$ (Scheinost et al., 1998). A characteristic band

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Location of Lingtai/Zhaojiachuan profiles in the CLP (Sun YB et al., 2006) (a), and ODP Hole 885A in the NPO (Rea et al., 1993) (b).}
\end{figure}

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position for hematite is labeled as $P_x$ ($x$ denotes the wavelength value). All the samples for DRS measurements were sieved (38 μm) after grinding, and the $< 38$ μm portion was used for DRS test. The above-mentioned experiments have been completed in Zhang Q et al. (2018). In order to investigate the sieving effect, we selected some un-sieved samples for comparison testing.

3. Results

3.1 DRS Experiments

Figure 2 shows DRS results for representative samples before and after sieving. We have demonstrated that the antiferromagnetic minerals (hematite and goethite) with other wind-borne minerals (silicates) in the deep-sea sediments are derived from the Asian inland to the North Pacific Ocean (Zhang Q et al., 2018). Therefore, the eolian fraction separated by chemical method (Sun YB and Liu QS, 2007) can be used to evaluate the sieving effect on the DRS results.

For goethite, before sieving, $I_{Gt}$ decreases first and then gradually increases, peaking at around 2.65 Ma, then decreasing to 2.54 Ma (Figure 2a); however, the eolian fraction shows an obvious rising trend during this time interval (Figure 2a). After sieving, $I_{Gt}$ exhibits a variation tendency similar to that of eolian dust flux, and the abrupt increase in the eolian record at 2.65 Ma is also seen in $I_{Gt}$ after sieving (Figure 2b). Figure 2c and 2d indicate that sieving can effectively suppress measurement noise for hematite, the resulting $I_{hm}$ record closely mirrors the eolian dust variations (Figure 2d).

3.2 Hematite Band Position

On the basis of the changes in hematite band position (Figure 3c) and its corresponding relationship with other parameters, the

Figure 2. Comparison DRS testing results. Intensity for goethite (a) and hematite (c) before sieving. Intensity for goethite (b) and hematite (d) after sieving.
studied profile can be divided into two zones: Zone 1: 2.79 to 1.50 Ma, and Zone 2: 1.50 to 1.00 Ma.

**Zone 1** (2.79 to 1.50 Ma): For stages 1-1, 1-3, and 1-5, hematite band positions center mainly at ~560 nm (labeled as $P_{560}$) (Figure 3c), which corresponds exactly to the valleys in the records of eolian dust in the NPO (Figure 3a and 3b) and the CLP (Figure 3d). For stages 1-2 and 1-4, the band positions center at 530–545 nm (labeled as $P_{540}$), which indicates that hematite particles are non-stoichiometric. It is obvious that the value of the eolian record in the NPO (Figure 3a and 3b) is relatively high and the CLP (Figure 3d) also increases significantly at these two intervals.

**Zone 2** (1.50 to 1.00 Ma): There is an ascending tendency in the NPO eolian record (Figure 3a and 3b), while the CLP eolian record is characterised by higher amplitude fluctuations after 1.50 Ma (Figure 3d). And the peaks and valleys for MAR of eolian dust in the CLP (Figure 3d) correspond to $P_{540}$ and $P_{560}$ of hematite band positions (Figure 3c), respectively. But the relationship between the hematite band position (Figure 3c) and the NPO eolian records (Figure 3a and 3b) is vague after 1.50 Ma.

4. Discussion

4.1 Effect of Sample Sieving on DRS data

For synthetic samples, stoichiometric hematite and goethite particles have intact crystal forms and homogeneous grain sizes, and these particles strictly follow laws of diffuse reflection, which results in high-quality DRS results with small errors. However, natural minerals form in different environments and undergo different levels of weathering processes. Therefore, these minerals may possess various morphology, grain size, and substituted-element contents. Prior to DRS measurements, grinding processes for samples can disperse particles and minimize changes in reflectivity arising from grain size discrepancy, making the results more consistent and reliable (Balsam and Deaton, 1991). Although sieving ($38 \mu m$) is highly recommended for high resolution studies, it is often neglected. Our new results demonstrate that the sieving process can significantly improve the accuracy of DRS results and suppress measurement noise, especially for hematite.

4.2 Hematite Band Position in DRS and Its Implication for the Eolian Evolutionary History

Comparison of the eolian records from the major eolian depositional sinks, such as the CLP and the NPO, can improve our understanding of the changes in climate and environment for the source areas and interrelationships between the westerly jet and the winter monsoon (Sun YB and Liu QS, 2007; Nie JS et al., 2018). On the glacial-interglacial scales, the maxima and minima of eolian fraction mass accumulation rates (MAR) for core V21-146 in the Northwest Pacific Ocean since the past 600 ka corresponds to loess/paleosol layers, respectively, in the Xifeng profile of CLP (Hovan et al., 1989, 1991). On the tectonic scale, eolian inputs into the NPO (Zhang WF et al., 2016) and the Sea of Japan (Shen XY at al., 2017) since the Miocene also match well with the evolution of eolian flux in the Asian inner profiles (Guo ZT et al., 2002).

![Figure 3](image-url)  
Figure 3. Eolian proxy $R_{Hm+Gt}$ of ODP Hole 885A (a) (Zhang Q et al., 2018), mass accumulate rate (MAR) of eolian dust in ODP Hole 885A (b) (Sun YB and Liu QS, 2007), the band position of hematite obtained from DRS in the sediments of ODP Hole 885A; $P_{560}$ and $P_{540}$ indicate winter and summer patterns, respectively (c), MAR of eolian dust in Lingtai/Zhaojiachuan profiles in the CLP (d) (Sun YB and An ZS, 2005), stacked mean grain size of quartz in Lingtai/Zhaojiachuan profiles in the CLP; the lower value indicates stronger winter monsoon (e) (Sun YB et al., 2006).
However, previous studies have also revealed some discrepancies in tendency and amplitude of eolian records in the CLP and the NPO at different time-scales. For the long time scale, Nie JS et al. (2018) attributed the discrepancies to the transporting winds system: the East Asian winter monsoon for the CLP (An ZS et al., 2001; Tamburini et al., 2003) and westerly mainstream for the NPO (Wehausen and Brumsack, 2002). As for the short time scale, Sun YB and Liu QS (2007) proposed that the differences may be caused by the differences in distance from source to sink.

In addition to the above, changes in dust provenances should also be considered. As shown in satellite images, eolian dust masses can be transported from the Asian inner sources, such as the CLP-sources (Badain and Juran deserts, the Tengger Desert, the Mongolia-Gobi Desert, and the Mu Us Desert) and the non-CLP-sources (Taklimakan Desert, Qinghai, Qaidam Basin, Gansu Hexi Corridor, and the Hobq Desert) (Sun YB et al., 2013), to the North Pacific Ocean, with activity greater in springtime (Prospero et al., 2002; Husar et al., 1997), and the geochemical signatures of the Asian eolian dust have also been detected in the pelagic NPO sediments (Nakai et al., 1993; Olivarez et al., 1991; Pettke et al., 2000; Serno et al., 2014; Stancin et al., 2006). According to the meteorological data, the Asian inner arid and semi-arid regions, including CLP-sources and non-CLP-sources, all underlie the range of the westerly jet (Schiemann et al., 2009). Therefore, the eolian particles from these sources can be jointly transported into the NPO depositional sink by the westerly jet. By this point, there must be differences in the eolian provenance for the CLP and NPO.

In this study, two groups of hematite band position (P_{560} and P_{540}) have been identified in the pelagic sediments of ODP Hole 885A from late Pliocene to early Pleistocene. Previous study has found that the signal of P_{560} is the coarse-grained hematite of lithogenic origin and is mostly confined to non-CLP-sources, such as the Taklimakan Desert; the P_{540} is an intermediate phase that is present both in surface samples from CLP-sources and non-CLP-sources (Liu QS et al., 2015). The band position of hematite obtained from DRS can also be used to distinguish the eolian provenance of the pelagic sediments in this hole.

For stages 1-1, 1-3, 1-5 and 2-2, the corresponding hematite band position (P_{560}) may indicate that the eolian fractions derived mainly from non-CLP-sources. The others may be mostly from CLP-sources at stages 1-2, 1-4, 2-1, and 2-3. Therefore, high resolution DRS results indicate that the hematite band position provides more detailed information about the eolian provenance, and that the arid and semi-arid regions in southern Mongolia (CLP-sources), northern China (CLP-sources), and the northern edge of Tibet Plateau (non-CLP-sources) all provide eolian materials for the NPO. It can be implied that the discrepancies among the eolian records in the Zhaojiachuan/Lingtai profiles of CLP and the ODP Hole 885A of NPO from late Pliocene to early Pleistocene may possibly reflect differences in eolian provenance for these two major depositional sinks.

The westerly jet plays an important role in eolian transportation from source to sink. Previous study based on modern meteorological data has traced the seasonality and interannual variability of the westerly jet path in the Tibetan Plateau region (Schiemann et al., 2009), revealing that the mainstream of the westerly jet splits into two branches in January, the one located along the southern edge of the Tibetan Plateau, and the other one centered around northern Xinjiang, and a large proportion of CLP sources (such as Gobi and sandy deserts in southern Mongolia and northern China) underlie the northern branch of the westerly jet mainstream during this period; the corresponding pattern of atmospheric circulation is shown in Figure 4a (named the “winter pattern”). During the summer in the northern hemisphere, the westerly jet mainstream retreats to the northern edge of the Tibet Plateau, which is just above the Taklimakan Desert. Tall mountains, such as the Tianshan and Kunlun Mountains, block the flow of eolian materials from the Taklimakan Desert; they can be transported only by high-altitude tropospheric airflow, the westerly jet (Scheinost JM et al., 2001), hence the northern movement of the westerly jet mainstream provides the meteorological conditions to enable the eolian dust derived from Taklimakan Desert to be conveyed into the distal depositional sink. The corresponding pattern is shown in Figure 4b (named the “summer pattern”). The northward or southward movements of the westerly jet mainstream has been identified on an orbital and millennial scale in geological records preserved in glaciers and sediments (Li SH et al., 2011; Nagashima et al., 2007, 2011, 2016; Dong Z et al., 2017).

As shown in Figure 3, the values of MAR of eolian dust in the CLP (Figure 3d) and the corresponding stacked mean grain size of quartz (Figure 3e) at stages 1-1, 1-3, and 1-5 reflect the weaker East Asian winter monsoon, and the hematite band position (P_{560}; Figure 3c) in the NPO pelagic sediments indicate that the eolian fractions derive mainly from non-CLP-sources (such as the Taklimakan Desert) at these stages. It can be speculated that the westerly jet mainstream may be located at the northern edge of the Tibet Plateau and overlies the Taklimakan Desert, and that the conditions of atmospheric circulation correspond exactly to the summer pattern of Schiemann et al. (2009). Although part of the CLP-sources, such as the Badain and Juran Deserts and the Mu Us Desert, also underlie the mainstream of the westerly jet, the Taklimakan Desert may provide more eolian dust and thus dominate the DRS properties in the pelagic sediments, due to the deeper aridification compared to CLP-sources, which are closer to the range of the East Asian summer monsoon.

For stages 1-2 and 1-4, when the East Asian winter monsoon is relatively stronger, more eolian materials from CLP-sources are transported into the NPO, as suggested by the corresponding hematite band position (P_{540}; Figure 3c); the winter pattern of Schiemann et al. (2009) may help explain the path of the westerly jet mainstream during these periods. The northern branch bypasses the Taklimakan Desert and conveys eolian materials from CLP-sources, including the Badain and Juran deserts, the Tengger Desert, the Mongolia-Gobi Desert, and the Mu Us Desert, to the NPO. Therefore, on the scale of millions of years, the northward or southward movement of the westerly jet mainstream also conforms to the mechanism revealed in modern meteorological observations (Schiemann et al., 2009). Compared to the summer pattern, the range of influence of the westerly jet mainstream expands significantly in the winter pattern, the two branches of the
westerly jet mainstream conveying more eolian fractions from the Asian inner sources to the NPO; this speculation is supported by the fact that the NPO eolian records possess higher values at stages 1-2, 1-4, 2-1, and 2-3, in which the hematite band positions are centered close to ~540 nm. When the westerly jet mainstream retreats to the northern edge of Tibet Plateau at stages 1-1, 1-3, and 1-5, the shrinkage in scope of the westerly jet reduces the eolian inputs, as the NPO eolian records are characterised by lower value at these intervals.

However, the above model cannot be extended to Zone 2, especially at stage 2-2 (the loess unit L_{15}). The significant increase in grain size and eolian flux in the L_{15} may be caused by the tectonic uplift of the Tibet Plateau and nearby regions (Sun JM and Liu TS, 2000). Besides the tectonic factors, previous study has also found that there was a significant short-duration expansion of the deserts near the CLP at approximately 1.20 Ma (Ding ZL et al., 2005). The median grain-size record of the Chashmaniglar loess section in southern Tajikistan does not exhibit any distinct changes at L_{15} layers (Yang SL et al., 2006). In the meantime, the NPO eolian records show no sign of abnormality, hence the changes in properties of eolian dust at stage 2-2 is possibly a local climate event and may have been affected by multiple factors, including winter monsoon, desert expansion, and tectonic uplift. This event may not have global impact (Sun YB and Liu QS, 2007). The westerlies as the major component in the planetary wind system may be influenced insignificantly by local factors. Hence, the mechanism of longitudinal movement of the westerly jet mainstream may also be applicable in Zone 2.

Except for stage 2-2, compared with Zone 1, the mean strength of the East Asian winter monsoon is relatively stronger after 1.50 Ma; hence the location of the westerly jet mainstream still conforms to the winter pattern at stages 2-1 and 2-3: the northern branch of the westerly jet mainstream carries more eolian materials from CLP-sources into the NPO, as suggested by the P_{540} signal of hematite band position at these two stages. The value of the eolian record in the NPO at stage 2-2 is supposed to be reduced in response to the retreat and shrinkage of the westerly jet mainstream, because the hematite band position is located primarily at ~560 nm. But the ascending tendency of the NPO eolian record does not show any distinct change at this stage (Figure 3a and 3b). Besides the transportation factor, climatic and environmental conditions in source regions should be also taken into account,

Figure 4. The mode pattern of atmospheric circulation over East Asia and the North Pacific Ocean. (a) and (b) indicate the location of the westerly jet mainstream (WJM) in January and July, termed “winter pattern” and “summer pattern”, respectively. Both are modified from Schiemann et al. (2009). The red arrows, areas where the westerly jet occurs most frequently, can be treated as the location of mainstream. The light purple arrows and light green arrows indicate the East Asian winter monsoon (EAWM) and the East Asian summer monsoon (EASM), respectively. The distribution of deserts or sand deserts is adapted from Sun YB et al. (2013).
associated with the expansion of desert regions in the Asian inland at this time (Ding ZL et al., 2005); the enhanced aridification in source areas may enable the increase in eolian flux at stage 2-2 in the CLP.

5. Conclusions
(1) The sieving process after grinding can improve effectively the accuracy of DRS results and suppress measurement noise, which is an essential procedure during pretreatment for DRS testing.

(2) Two groups of hematite band position (P_{560} and P_{540}) obtained from the DRS have been identified in the pelagic sediments of ODP Hole 885A from the late Pliocene to early Pleistocene. The hematite band position can provide important information about provenance of eolian components for this hole. CLP-sources and non-CLP sources both provide abundant eolian fractions transported by the westerly jet into the NPO depositional sink. Discrepancies in the eolian records from the Zhaojiachuan/Lingtai profiles in the CLP and ODP Hole 885A in the NPO may be caused by changes in eolian provenance for this hole, based on the variation of hematite band position obtained from DRS.

(3) Changes in eolian provenance of the pelagic sediments may be a result of movement of the westerly jet mainstream. Modern meteorological observation has confirmed that northward or southward movement of the westerly jet mainstream is significantly influenced by the relative strength of East Asian winter/summer monsoon. The hematite band position obtained from DRS is a useful tool to distinguish the eolian provenance of the pelagic sediments in the NPO.

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