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Key Points:
- New sediment provenance tool exploits E\textsuperscript{1}\textsuperscript{1}' and peroxy paramagnetic defects in quartz
- New proxy successfully differentiates quartz in loess from two different basins in Central Asia
- Potential applications for identifying climate-driven source change through time in loess and other sedimentary sequences

Supporting Information:
Supporting Information may be found in the online version of this article.

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Abstract Crystal lattice defects in quartz have long been exploited for age determination, yet also show potential for sediment provenance studies. Here, we introduce a novel method for tracking aeolian dust provenance by utilizing the natural accumulation of E\textsuperscript{1}\textsuperscript{1}' and peroxy defect centers in quartz. Our approach is based on the previously observed premise that E\textsuperscript{1}\textsuperscript{1}' and peroxy centers arise from Frenkel defect pairs, and that their concentration increases with the age of the quartz-bearing source rock. We propose that these defect centers can be utilized as a characteristic feature of the source rock and consequently, for fingerprinting sediments derived from it. We successfully apply our new protocol to distinguish fine-grained quartz extracted from loess deposits from two regions in Central Asia which are known to derive from different source material of differing age. Our method offers strong potential for identifying variability in source, both spatially and through time down sedimentary sequences.

Plain Language Summary Identifying the origins of dust deposits allows us to reconstruct sediment transport pathways which are essential for understanding past atmospheric circulation patterns. Here, we propose to exploit the characteristics of two naturally occurring defect centers in crystalline quartz, the E\textsuperscript{1}\textsuperscript{1}' and peroxy centers, as a means to distinguish sediment deriving from different origins. These centers occur as pairs and are hypothesized to increase with the age of the quartz-bearing rock. By this logic, the E\textsuperscript{1}\textsuperscript{1}' and peroxy centers can be used to determine the lithic origins of sedimentary quartz in a similar way to detrital zircon-based provenance techniques, while analyzing a more ubiquitous mineral (quartz). We apply our approach, which uses a simplified protocol for measurement in contrast to earlier studies, to successfully distinguish between loess (wind-blown dust deposits) from two different basins in Central Asia. Our new method holds great potential in its application to loess sequences as well as other sedimentary archives.

1. Introduction

Identifying the original source rocks of sediments is important for understanding sediment cycling. For aeolian sediments, pinpointing provenance has additional advantages of facilitating the reconstruction of transport pathways that relate to atmospheric circulation, and thereby changes in climate dynamics through time. Aeolian loess deposits have long been recognized as valuable archives of past climates in terrestrial environments (Kukla et al., 1988; Schaeftl et al., 2018). Loess sequences represent long-term accumulation of aeolian dust and thus identifying their provenance provides an important proxy for reconstructing changes in atmospheric circulation through time. A number of established tools are used to identify the source of dust in loess deposits. These include grain-size analysis, in particular grain sorting and end-member modeling, to elucidate transport modes and likely source area types (Li et al., 2018; Vandenberghhe, 2013); major and trace elemental composition of bulk dust (J. Sun, 2002; Újvári et al., 2008); radiogenic isotope signatures (Sr and Nd) characteristic of clay minerals (Ben-Israel et al., 2015; Chen et al., 2007; Rao et al., 2015); and detrital zircon age profiles (Pullen et al., 2011). While these methods are widely used to identify relative changes in dust sources through time, they rely either on materials found in very low concentrations (e.g., detrital zircons) or aggregated bulk measurements on multiple size fractions which are likely to have been transported from both distal and proximal sources (e.g., major
and trace elements, radiogenic isotopes). Furthermore, postdepositional weathering can alter in situ chemical signatures within loess, rendering certain analytical techniques, if uncorrected, inaccurate for provenance (Yang et al., 2001).

Given the limitations of the above-mentioned provenance techniques, there has been increasing interest in the characteristics of mineral quartz as a tool for linking dust sources and sinks. There are obvious advantages in using quartz as a provenance tool: not only is it ubiquitous, but also highly resistant to weathering and diagene sis (Goldich, 1938). A number of quartz-specific petrographic, isotopic, and geochemical provenance methods have been proposed (Ackerson et al., 2015; Bernet & Bassett, 2005; Nagashima et al., 2007, 2017; Shimada et al., 2013). Of these, electron spin resonance (ESR) signal of various defect centers in quartz has been increasingly applied to fingerprint sources in sedimentary settings (Nagashima et al., 2007, 2011; Tissoux et al., 2015; Wei et al., 2020).

ESR measures the intensity of paramagnetic species (containing an unpaired electron) in a material. Lattice defects and impurities in quartz give rise to various paramagnetic defect centers. E$_{\gamma}$' is one such center (Weil, 1984), comprising an unpaired electron in an oxygen vacancy (≡Si–O–) that are known to arise from diamagnetic oxygen vacancies (Si=Si). Jani et al., (1983). The most commonly used ESR based provenance protocol utilizes the heat treated-E$_{\gamma}$' (hereafter referred to as HT-E$_{\gamma}$') intensity of quartz by measuring the intensity of the E$_{\gamma}$' center following gamma (γ) irradiation and thermal treatment (Toyoda & Hattori, 2000; Toyoda et al., 2016). It is based on the premise that γ-irradiation and subsequent heating facilitate conversion of quartz diamagnetic oxygen vacancies into paramagnetic E$_{\gamma}$' centers resolvable by ESR and expressed as HT-E$_{\gamma}$'. The HT-E$_{\gamma}$' intensity has been observed to increase with rock age (Toyoda et al., 1992) and is assumed to reflect the total number of oxygen vacancies, which is characteristic of the rock type and consequently of the source rock from which the quartz is derived (Toyoda & Hattori, 2000; Toyoda et al., 2016). The HT-E$_{\gamma}$' intensity is utilized in combination with the crystallinity index of quartz as a common provenance tool (Nagashima et al., 2007, 2011; Toyoda et al., 2016), since the combination of these is interpreted to reflect the age, formation, and crystallization conditions of quartz in the source rock.

Here, we investigate a simplified new provenance method based solely on the analysis of two naturally occurring paramagnetic defect centers in quartz, the E$_{\gamma}$' and peroxy centers, using ESR. The E$_{\gamma}$', as previously mentioned, refers to a center with an unpaired electron in an oxygen vacancy, while the peroxy center, in general, refers to a combination of the peroxy radical (≡Si–O–O, POR) and non-bridging oxygen hole center (≡Si–O–, NBOHC) (Griscom & Friebele, 1981; Stapelbroek et al., 1979, and from recent observations by Salh, 2011; Skuja et al., 2020). We explore the use of E$_{\gamma}$' and peroxy centers as a provenance indicator of quartz based on the empirical observations by Odom and Rink (1989), which show that both these centers increase with the age of granitic host rocks. The proposed mechanism for this observation suggests that these centers arise from Frenkel defect pairs formed by alpha-recoil nuclei emitted by alpha-emitting elements (U and Th) and accumulates in the rocks through time (Rink & Odom, 1991). In this study, we investigate and apply this new method to distinguish quartz in loess sediments from inland Asia. Additionally, we compare the natural E$_{\gamma}$' with HT-E$_{\gamma}$' characteristics to elucidate differences between our new method and the previous approach (Toyoda & Hattori, 2000).

Inland Asia represents one of the world’s major atmospheric dust source, both past and present (Kok et al., 2021), and has been the regional focus for investigations using ESR signals of quartz as a provenance technique (Y. Sun et al., 2007, 2008). The basins to the north, east, and west of the Asian high mountains (the Pamirs, Alai-Altai, and Tien Shan) lie in topographic rain shadows and represent substantial sinks for glacially and fluvially derived sediment from the uplands (Schaetzl et al., 2018; Figure 1). In particular, the loess deposits of arid Central Asia (ACA) and the Chinese Loess Plateau reach hundreds of meters in thickness (Li et al., 2015; Liu, 1985) representing long-term substantial accumulation, and by extension, likely distal sources of global aeolian dust. Distinguishing between various potential dust sources in inland Asia is important at multiple scales. At regional levels, identifying changes in source down loess sequences enables reconstruction of variability in dust transport pathways and atmospheric circulation through time. At global scales, it can help identify the relative contributions of different basins to the generic “Asian” mineral dust identified in Greenland ice cores (Bory et al., 2003; Svensson et al., 2000) and Pacific Ocean marine sediments (Letelier et al., 2019; Nakai et al., 1993).

In this study, we characterize the natural E$_{\gamma}$' and peroxy center intensities of loess from two basins in ACA, the Ili basin and Tajik depression of southeast (SE) Kazakhstan and Tajikistan, respectively (Figure 1). Recent studies...
based on geochemical fingerprinting and back trajectory analysis indicate that loess in the Ili basin (Kazakhstan) is sourced from the surrounding alluvio-fluvial plains and the neighboring deserts located north of the basin (Fitzsimmons et al., 2020; Li et al., 2018), whereas the loess in the Tajik depression is sourced from the surrounding uplands as well as deserts (Kyzylkum and Karakum) located further west of the Tajikistan (Li et al., 2016, 2019). The potential significance of the region for global atmospheric dust loads past and present, coupled with the likely discrete sources for the two basins, makes them well suited for targeted spatial and temporal comparison of quartz characteristics for provenance using our new method.

2. Material and Methods

We undertook measurements on fine-grained (4–11 μm) quartz extracted from loess samples collected from five loess sections in Central Asia (Figures 1 and S1 in Supporting Information S1). Four of these sections, Panfilov (PAN), Ashubulak (ASH), Taukaraturyuk (TAU), and Malubai (MAL), are located along a c. 200 km east-west transect of the Zalisky-Alatau range in the Ili basin of SE Kazakhstan. The fifth site, Karamaidan (KAR), is a c. 60 m-thick partial section of a c. 130 m-thick loess-paleosol sequence, located in the foothills of the Gissar mountain range on the northern margins of the Tajik Depression, in southern Tajikistan (Figure 1). We analyzed 112 samples; 57 from SE Kazakhstan and 55 from Tajikistan. A detailed account of sampling, site description, and age-range, sample preparation, instrumentation, and measurement protocols are provided in the Supporting Information S1.

We performed two sets of experiments on fine-grained quartz:

(i) To test our new approach, we measured the intensity of $E'_1$ and peroxy centers for all samples. These measurements were conducted on natural quartz samples as is, without any prior treatment and we hereafter refer to these measurements as the natural $E'_1$ and peroxy intensity.

(ii) To understand the differences between our new approach and the previous ESR-based provenance method, we measured the HT-$E'_1$ intensity following published protocols (Nagashima et al., 2007; Toyoda & Hattori, 2000) and compared them to the natural $E'_1$ intensity for all our samples. This involved irradiating all the samples with a γ-dose of 2000 Gy, followed by heating them to 350°C for 15 min to obtain the HT-$E'_1$. 

Figure 1. Regional setting and location of the loess sites under study. The elevation map was created using Shuttle Radar Topography Mission data provided by AW3D of the Japan Aerospace Exploration Agency.
intensity. Further, we test our approach and evaluate the need for γ-irradiation and thermal treatment prior to E<sub>1</sub>' measurement, by investigating the variation of ESR centers (E<sub>1</sub>', peroxy, and Al-hole) with γ-irradiation (varying from 0 to 40,000 Gy) and temperature (300, 350, and 400°C) for two representative samples, one from Kazakhstan (A0016, TAU) and the other from Tajikistan (A0329, KAR). Detailed descriptions of experimental parameters are given in the Supporting Information S1.

3. Results and Discussion

3.1. Paired E<sub>1</sub>'-Peroxy Centers in Quartz: Methodological Considerations Linking Quartz Crystal Defect Dynamics to Provenance

Our measurements of the natural E<sub>1</sub>' and peroxy intensities of 112 Kazakh and Tajik samples indicate that the natural intensity of E<sub>1</sub>' and peroxy centers yield a positive correlation, with a Pearson coefficient of 0.72 (Figure 2a). This supports the hypothesis that these naturally occurring ESR centers indeed arise from Frenkel defect pairs in quartz as suggested by Odom and Rink (1989).

Although intuitive, the measurement of “natural” signals for provenance remains largely unexplored. Our provenance approach, based on natural E<sub>1</sub>' and peroxy signals, represents a simpler protocol than the hitherto established method based on HT-E<sub>1</sub>' intensity (Toyoda et al., 2016, and references therein). Figure 2b compares the natural E<sub>1</sub>' and HT-E<sub>1</sub>' results for all samples. We observe that γ-irradiation and heating simply increases the signal intensity beyond the natural E<sub>1</sub>' in all samples. This indicates that the natural E<sub>1</sub>' intensity produces the same inherited provenance characteristics as HT-E<sub>1</sub>' intensity, and implies that γ-irradiation and heating may not be necessary for our samples.

The proposed mechanism for the formation of the HT-E<sub>1</sub>' in the established provenance protocols (Toyoda & Hattori, 2000; Toyoda & Ikeya, 1991) suggests that γ-irradiation creates hole-supplying Al-hole centers (which arise from Al impurities in quartz), and post-irradiation heating causes the migration of holes from the Al-hole centers. The released holes recombine with one of the two electrons of the diamagnetic oxygen vacancies, giving rise to an oxygen vacancy with an unpaired electron, that is, an E<sub>1</sub>' center. Therefore, γ-irradiation and thermal treatment essentially converts all diamagnetic oxygen vacancies - which are characteristic of a given rock - into E<sub>1</sub>' centers, known as HT-E<sub>1</sub>' centers (Toyoda et al., 2016, and references therein). Meanwhile our results on natural E<sub>1</sub>' measurements suggest that sample pretreatment by γ-irradiation and heating is unnecessary for ESR-based quartz provenance methods. Therefore, we undertook an additional series of irradiation and heating experiments on two representative samples, A0016 (Kazakhstan) and A0329 (Tajikistan), to systematically assess the effects of γ-irradiation and thermal treatment on ESR centers (E<sub>1</sub>', peroxy, Al-hole center).
First, we tested the effect of γ-irradiation on the ESR intensity of natural \( E_1^- \), peroxy and Al-hole centers. We irradiated 11 aliquots of each sample with a γ-dose varying from 0 to 40,000 Gy and measured the intensity of each center. We observe that, overall, the natural \( E_1^- \) intensity does not change with γ-irradiation (Figure 3a). Our observations corroborate with results obtained from fine-grained quartz from other regions (Eastern Europe and North America; Figure S2 in Supporting Information S1). Likewise, the natural intensity of the peroxy center for both samples does not vary with γ-dose (Figure S3 in Supporting Information S1). We note that the uncertainty for repeat measurements on the same aliquot for peroxy signal (Figure S3 in Supporting Information S1)
is 10%–20% higher than for the natural E₁', which has an uncertainty of <1%. This is most likely due to the weak peroxy signal observed in our samples. By contrast, the natural Al-hole center intensity increases exponentially with increasing γ-dose (Figure S4 in Supporting Information S1).

Second, we tested the effect of temperature on irradiated quartz E₁' and peroxy centers for the same two representative samples (A0016 and A0329). All γ-irradiated aliquots (n = 11) of each sample were heated to 300, 350, and 400°C for 15 min, followed by the measurement of the resulting E₁' and peroxy center intensities. In both the samples, E₁' intensity increases with temperature, peaks at 350°C, and thereafter decreases (Figures S5a and S5b in Supporting Information S1). The E₁' intensity at any given temperature does not change with increasing γ-dose (Figures S5c and S5d in Supporting Information S1), which can also be seen in the constant ratio of HT-E₁' to natural E₁' intensity for both samples (Figure 3a). In contrast, the peroxy signal shows minimal change in average intensity with increased temperature (Figure S6 in Supporting Information S1). However, the generally weak peroxy signals produce high scatter in the data, rendering assessment of peroxy intensity response to increasing temperature difficult.

Our experiments on fine-grained quartz have two important implications for measuring natural E₁' as a provenance signal. First, E₁' and HT-E₁' intensity remains unchanged with increasing γ-dose, and the ratio between the two signals remains constant (Figure 3a). This suggests that heating increases net E₁' intensity, and irradiation has no effect. This is the case not only for our samples from two regions in Central Asia, but also for detrital quartz from modern river sediments (Wei et al., 2017). We conclude that natural E₁' reflects the quartz characteristics just as well as the HT-E₁' signal and argue that γ-irradiation and heating is not necessary. Second, in both our samples, the intensity of the Al-hole center increases with increasing γ-dose, in contrast to natural E₁' and HT-E₁'. If we assume the proposed formation mechanism of HT-E₁' center to be true (Toyoda & Hattori, 2000; Toyoda & Ikeya, 1991), our observations imply that the number of diamagnetic oxygen vacancies in our samples is less than the number of holes released from Al-centers upon heating, even for unirradiated samples. This further sustains our view that the γ-irradiation step is redundant for our samples.

Our work is based on the premise that naturally occurring E₁' and peroxy centers accumulate in rocks over million-year timescales, primarily due to the effect of heavy particle irradiation and thereby show an increase with rock age (Odom & Rink, 1989). Therefore, the use of these defect centers as provenance indicators relies on the fact that their intensity does not change significantly with ionizing radiation received during transport and/or burial as a result of the short time spent by quartz in sedimentary settings as compared to that in the source rock. A recent study by Toyoda and Amin moto (2021) suggests that apart from ionizing radiation received in rocks, exposure to radiation during its sedimentary history is also likely to alter the E₁' signature of quartz. We, therefore, provide further evidence that the E₁' signal is independent of ionizing radiation received during transport and/or burial by examining the natural E₁' and HT-E₁' intensity variation with depth down a c. 60 m thick loess section at KAR, Tajikistan. Based on previous (Forster & Heller, 1994) and our own magnetic susceptibility measurements, we correlated the sampled part of the KAR profile to marine oxygen isotope stages 19–9 (c. 800–300 ka, Figure S7 in Supporting Information S1; Lisiecki & Raymo, 2005). This correlation places the samples from this section beyond the limits of quartz optically stimulated luminescence dating. Hence, we estimated the minimum absorbed dose (natural burial dose) received by the uppermost sample to c. 1,000 Gy using the post-infrared infrared stimulated luminescence protocol (Table S1 in Supporting Information S1; Buylaert et al., 2012) on polynminer fine-grains (Figure S8 in Supporting Information S1). Assuming a dose rate of 3–4 Gy/ka, which is typical for loess, all samples below the uppermost sample would have received ionizing radiation corresponding to an absorbed dose of c. 1,000–3,000 Gy down the profile. The measurement of natural E₁' and HT-E₁' intensity with depth at KAR shows no discerning patterns of incremental increase or decrease (Figure 3b). While, laboratory γ-irradiation experiments on the E₁' signal show minor variations in the lower dose range (0–2,000 Gy; wherein the E₁' intensity first decreases and then increases), unlike that at higher doses (>2,000 Gy; Figure 3a). This raises an interesting observation regarding the response of E₁' signal to naturally absorbed doses down KAR, which are also likely to vary between c.1,000 and 3,000 Gy down the sequence. In Figure 3a, we observe that E₁' intensity in the lower dose range varies by 5%–20% of the natural E₁' values for the representative samples, whereas the variation in E₁' values with depth (and absorbed dose) between loess and soil horizons at KAR ranges by c. 30%–50% of the average E₁' values (Figure 3b). Here, we note that the average E₁' value is biased by the number of samples taken from the loess versus the soil horizons at KAR, hence the percent variation (if any) in E₁' signature at KAR is likely to be higher than suggested. This implies that the change in E₁' value at
KAR is not an artifact of ionizing radiation received during burial, but rather represents a change in source (see Section 3.2).

3.2. Applications in Aeolian Environments: Examples From Central Asia

The ultimate aim of sedimentary provenance analysis in aeolian environments is to identify whether parent rocks or sedimentary sources can be linked to the loess or desert deposits in question, and additionally to determine the trajectory of dust transport pathways and by extension, the most likely climate circulation patterns prevailing at the time of transport and deposition. Our study provides a critical step toward achieving the aim of identifying provenance within sediments by establishing a simplified protocol exploiting characteristic signatures based on defect centers in quartz.

Our suite of 112 samples forms two distinct spectral clusters depending on geographic region (Figure 2a). Of the two natural ESR signals measured, the natural E$_{1}'$ intensity yields the greatest difference between the two regions (Figure 2a). The more diffused peroxy signatures are a result of inherently weak peroxy signal in our samples, and may also reflect the hypothesis that “peroxy” signals, as measured, represent the overlap of the POR and NBOHC (Figure S9 in Supporting Information S1). Nevertheless, the peroxy signals from the two regions are statistically distinguishable (refer Text S3c in Supporting Information S1), and along with the natural E$_{1}'$ intensity, can be used as provenance indicators.

We observe higher natural E$_{1}'$ and peroxy signals in the Kazakh loess than the Tajik samples. Since the E$_{1}'$ and peroxy centers are known to accumulate with time (Odom & Rink, 1989), this suggests that the Kazakh quartz is sourced from older rocks than the Tajik quartz. The suggestion that the Kazakh source rock is older is consistent with the rocks of the central Tien Shan, which are of Palaeozoic age or older (Tursungaziev & Petrov, 2009), and represent the likely source material to the Kazakh loess piedmont (Fitzsimmons et al., 2020; Li et al., 2018). These are older than the predominantly Mesozoic and younger rocks of the Gissar Mountains and northwestern Pamirs (Vlasov et al., 1991), which provide the most likely source rocks for the Tajik loess (Li et al., 2019). The formation of two separate clusters for two independently sourced regions of different source rock age provides support for our proposed approach as a provenance tool.

In addition to differentiating likely source regions for different loess sites, we ultimately aim to identify potential changes in source through time down sedimentary (including loess) sequences such as those found in inland Asia. Figure 4a shows down-profile variations in natural E$_{1}'$ intensity of loessic quartz at KAR in Tajikistan, a site which preserves multiple primary loess and buried soil (paleosol) horizons (refer Supporting Information S1 for details). We observe a marked difference in the natural E$_{1}'$ and peroxy signature in loess versus paleosol horizons (Figure 4b). Recent work using trace element concentrations of loess in Tajikistan, combined with meteorological reanalysis, suggests that provenance is site-dependent and likely to be dominated by proximal montane sites (Li et al., 2016), with some contribution from distal sources such as the Karakum desert (Li et al., 2019). The relative contributions of distal and proximal sources to loess deposits in Tajikistan may have changed over glacial-interglacial timescales as a result of changes in the dynamics and intensity of atmospheric circulation. We suggest that our observed changes in paired E$_{1}'$-peroxy characteristics represent variability in the dominant source signature of quartz between the primary (glacial) loess and paleosol (interglacial) horizons. At this stage, without more targeted investigations of potential source rocks, we cannot identify whether the change in the dominant source signature through time at KAR is a result of proximal and/or distal transport of dust. Nevertheless, our observations hold promise for more focused investigations of source signature based on our provenance method.

4. Conclusion

We propose a new method for determining the provenance of sedimentary quartz based on the natural accumulation of E$_{1}'$ and peroxy centers. We confirm, based on empirical measurements of 112 fine-grained loessic quartz samples, that firstly, the natural E$_{1}'$ and peroxy signals are positively correlated and are therefore, likely to arise from Frenkel defect pairs. Secondly, the increase in intensity of these centers with the age of the source rock can be seen in the higher values obtained from the Kazakh samples, which are derived from older rocks than the Tajik loess. Therefore, quartz from the Ili basin of SE Kazakhstan yields signals distinct from quartz in the Tajik basin in Tajikistan, indicating a difference in provenance consistent with previously published studies. Furthermore, down-profile measurements at the KAR site in Tajikistan indicate a shift in source between primary loess and
paleosol horizons, most likely in response to changes in atmospheric circulation associated with climatic oscillations. Thus, our observations suggest this to be a robust new technique for sediment provenance, that can be applicable to a range of settings, exploiting the characteristics of one of the most ubiquitous minerals found in nature: quartz.

Data Availability Statement

The data supporting this study are available in the open access repository Mendeley at https://doi.org/10.17632/n4fhvswczf.1.

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Figure 4. (a) Down-profile variability in E1' intensity of fine-grained quartz at Karamaidan (KAR), Tajikistan. The magnetic susceptibility data was measured in the field (refer Supporting Information S1) while LR04 benthic δ18O data was obtained from Lisiecki and Raymo (2005); (b) Natural E1' and peroxy variation in samples from various stratigraphic sections (identified here as loess, paleosol, and weakly-developed palaeosols) at KAR based on field stratigraphic description and magnetic susceptibility data.
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