Particle size separation and evidence for pedogenesis in samples from the Chinese Loess Plateau spanning the past 22 m.y.

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ABSTRACT

A comparison between particle size distributions determined by laser diffraction and the pipette method confirms previous studies that point to a variable underestimate of the clay fraction using laser diffraction, relative to the values obtained by pipette analysis. In order to yield four particle-sized fractions (<2 μm, 2–4 μm, 4–8 μm, >8 μm) for further analysis, the pipette method was used on loess and paleosol samples from sites on the Chinese Loess Plateau spanning the past 22 m.y. In all cases, a subsidiary mode in the <2 μm fraction is identified. The clear separation of this from the coarse mode in the >8 μm fraction provides a basis for evaluating the relative contributions of pedogenic and detrital components. Pretreatment using acetic rather than hydrochloric acid for carbonate removal results in no significant loss of ferrimagnetic minerals and allows magnetic characterization of the separate fractions. This confirms the isolation of almost all the finest, pedogenic ferrimagnetic grains in the clay fraction and their negligible contribution to the magnetic properties of the coarse fractions in all the samples from early Miocene time onward. Because similar conclusions may be drawn from previous studies of late Pleistocene samples from the east-central and extreme western parts of the Chinese Loess Plateau, we conclude that this approach provides a basis for separately characterizing eolian deposition and pedogenesis throughout the region and for the whole time span of loess accumulation.

Keywords: loess, pedogenesis, granulometry, pipette analysis, magnetic properties.

INTRODUCTION

After comparing contrasting methods for establishing particle size distributions in loess and paleosol samples from the Chinese Loess Plateau, we identify a method for evaluating pedogenic and detrital components within the sample, and illustrate this using magnetic measurements. Samples of loess and intervening paleosols have geophysical and geochemical properties that reflect both eolian deposition and in situ weathering and pedogenesis. One of the challenges in research designed to generate paleoenvironmental inferences from loess profiles is to distinguish and characterize both types of processes. One of the many tools used in this type of research is particle size analysis. The published literature includes two types of approaches. In most recent studies, samples are disaggregated using a variety of methods, subsequent to which particle size distributions are calculated using commercially available particle size analyzers based on laser diffraction. This type of equipment has largely replaced sedimentation-based instrumentation, such as the SediGraph. In the main alternative to instrumentally determined granulometry, samples are separated into size classes after disaggregation using a combination of sieving for the coarsest material and pipetting from suspension for the finer fractions. The former instrument-based approach has the advantage of speed. The latter, though time consuming, yields particle-sized separates that can be further analyzed.

Research pointing to the underestimation of the clay grade in sediments by laser diffraction was first published in the 1980s (McCave et al., 1986). Since then, several investigations have been conducted to compare the differences between laser diffraction analysis and the classic sedimentation method (Blott and Pye, 2006; Fedotov et al., 2007, and references therein). It is important to address this issue in Chinese loess because there is an increasing tendency to interpret grain size changes in more and more detail (e.g., Sun et al., 2004; Qin et al., 2005; Prins and Vriend, 2007). Here we compare the results of the Chinese eolian deposits spanning the past 22 m.y. using laser granulometry and pipette analysis: the comparison highlights the advantages arising from the latter method.

PREVIOUS RESEARCH

Studies using the instrument-based approach provide granulometric data from sections ranging in age from Miocene to Holocene. These have been used to provide a basis for confirming the eolian origin of the deposits (e.g., Guo et al., 2002), differentiating loess and paleosol layers (Ding et al., 1994; Evans et al., 1996), and identifying variations in particle size within loess layers (e.g., An and Porter, 1997; Lu et al., 1999). Many studies treat the resulting grain size distributions as unimodal, although several authors use mathematical decomposition to identify up to three modes, including a fine, rather poorly defined mode that includes clay-size particles (e.g., Sun et al., 2004; Qin et al., 2005; Prins and Vriend, 2007). Those authors favored the view that this inferred fine mode, mainly reflecting eolian deposition during periods of minimum atmospheric turbulence, rather than pedogenesis. These inferences are inconsistent with paleoenvironmental reconstructions attributing the clay fraction to postdepositional weathering and pedogenesis (e.g., Kemp, 2001; Feng and Wang, 2006). In contrast with laser granulometry, studies using the sieve and pipette method show a bimodal particle distribution with a peak in the medium-coarse silt range and a clearly defined subsidiary peak in the clay fraction (Zheng et al., 1991; Chen et al., 1995; Sartori et al., 2005). The contrast between the results obtained by the two methods is apparent in

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late Quaternary samples from both the central and western parts of the Chinese Loess Plateau (Zheng et al., 1991; Chen et al., 1995; Sun et al., 2004; Sartori et al., 2005) (see Fig. DR1 in the GSA Data Repository[1]). In each case where pipette analysis has revealed a bimodal distribution with a peak in the clay fraction, the authors have inferred a pedogenic origin from its magnetic properties. These different approaches to particle size determination generate conspicuously different distributions, and therefore interpretations require further evaluation, especially since there is no evidence that the contrast in the results obtained by the two methods can be attributed to differences in pretreatment and disaggregation methods in this or previously published research (e.g., Beuselinck et al., 1998).

SAMPLING STRATEGY

The 26 samples (Table 1) used as the main basis for this study were selected from a suite of more than 100 samples representing successive magnetic mineral assemblages produced by loess accumulation and paleosol formation over the past 22 m.y. on the Chinese Loess Plateau. They come from three sites, Xifeng (0.5–2.7 Ma), Dongwan (4.2–7 Ma), and QA-I (6.4–21.9 Ma) (Fig. 1), and represent the main variations in the magnetic properties of both loess and paleosol layers at these sites (Hao et al., 2008). Wherever possible, they represent loess-paleosol couplets.

RESULTS

Figure 2A shows the results of pipette analysis after disaggregation using acetic acid to remove carbonates, followed by dispersal in Calgon. Buffered acetic acid was used for carbonate removal rather than HCl because it does not significantly affect the magnetic properties (e.g., Freeman, 1986). The low contribution of material >63 μm in all but the youngest Pleistocene loess rendered it unnecessary to sieve the samples prior to pipette analysis. Only four size classes were extracted: <2 μm, 2–4 μm, 4–8 μm, and >8 μm. The mass percentage of the <2 μm fraction varies from 12.0% to 24.9% for the Quaternary loess samples and from 20.5% to 42.9% for the Neogene samples. The percentages of the 2–4 μm and 4–8 μm fractions are <12.0% and 16.1%, respectively, for all samples. The fraction >8 μm accounts for 58.5%–78.6% of the Quaternary loess samples and 37.4%–64.8% of the Neogene samples.

TABLE 1. AGE, SAMPLE NUMBER, AND LITHOLOGY OF THE SAMPLES USED IN THIS STUDY

Section	Age (Ma)	Sample number	Lithology	Section	Age (Ma)	Sample number	Lithology
Xifeng	0.51	XF-1314	Soil	QA-I	7.61	GJ-278	Loess
	0.64	XF-1385	Loess		9.46	ZW-138	Soil
	1.84	XF-324	Soil		13.01	ZW-578	Soil
	1.87	XF-331	Loess		13.07	ZW-590	Loess
	2.62	XFRC-61	Red earth		15.03	QW-1510	Loess
	2.69	XFRC-86	Red earth		15.11	QW-1533	Soil
Dongwan	4.26	DW-738	Loess		15.44	QW-1603	Soil
	4.88	DW-856	Soil		15.48	QW-1612	Loess
	5.55	DW-980	Loess		17.49	QW-1999	Soil
	6.96	DW-1302	Soil		18.61	QW-2170	Soil
QA-I	6.41	GJ-32	Soil		18.69	QW-2178	Loess
	6.44	GJ-36	Loess		21.73	QW-3481	Soil
	7.60	GJ-275	Soil		21.83	QW-3497	Loess

The graphs shown in Figures 2B and 2C show the particle size distributions determined by laser granulometry using a Coulter LS 200. In the case of Figure 2B, in order to replicate more closely the pretreatment used in pipette analysis, carbonate was removed by acetic acid. For the subset of 10 samples from the same set shown in Figure 2B, HCl was used for carbonate removal (Fig. 2C).

Figure 3 shows selected magnetic properties for the clay (<2 μm) and coarse (>8 μm) fractions, calculated as the total contribution of each particle size fraction to the magnetic properties of 100 g bulk samples. The frequency-dependent magnetic susceptibility (χ_{fd}) is defined as the difference between susceptibility measurements at 0.47 and 4.7 kHz. Saturation isothermal remanent magnetization (SIRM) was imparted in a field of 1 T.

DISCUSSION

Both the previous results and those of our study (Fig. DR2) demonstrate a variable underestimate of the clay contributions determined by instrumental analysis relative to those separated by pipette analysis (cf. McCave et al., 1986; Loizeau et al., 1994; Konert and Vandenberghe, 1997; Beuselinck et al., 1998; Buurman et al., 2001; Blott and Pye, 2006; Fedotov et al., 2007). The significant clay content in Chinese eolian deposits determined by the pipette method, especially for the Neogene samples with an average clay content of ~34% ± 5% (n = 22), raises fundamental sedimentological questions for future studies on formation processes of clay-sized particles in the mid-latitude Asia region.

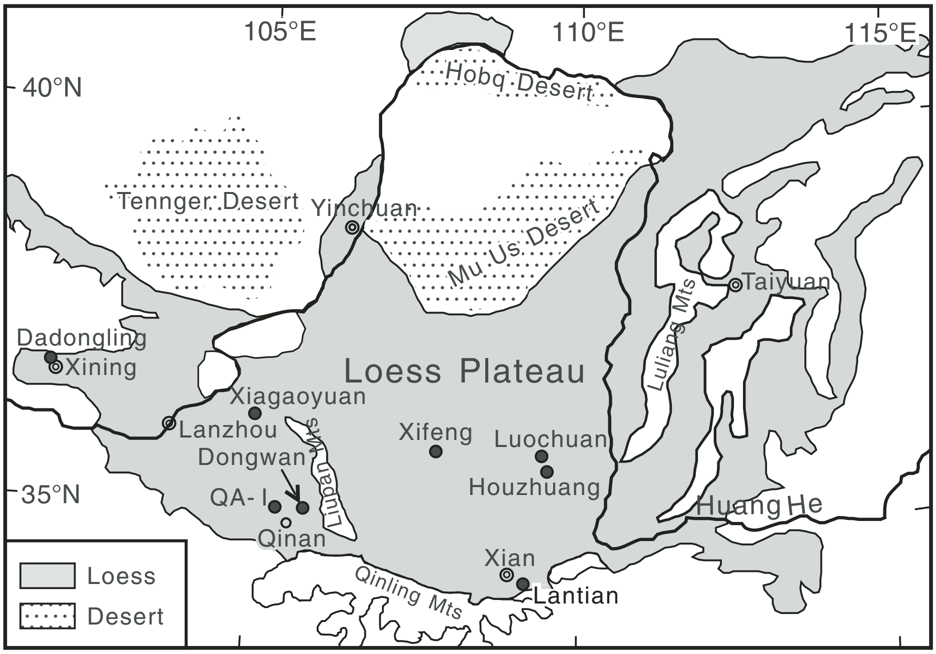

Figure 1. Location map showing Chinese Loess Plateau and all sites mentioned in text.

The magnetic properties shown in Figure 3 illustrate the distinctions between the finest and coarsest fractions. Since the initial studies by Zhou et al. (1990) and Maher and Thompson (1991), many papers have confirmed that

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[1] GSA Data Repository item 2008182, Figure DR1 (particle-size distributions of loess and paleosol samples from Luochuan section determined by sieve and pipette method and laser diffraction), and Figure DR2 (crossplot of the particle-size fraction measured by the pipette method and laser diffraction), is available online at www.geosociety.org/pubs/ft2008.htm, or on request from editing@geosociety.org or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

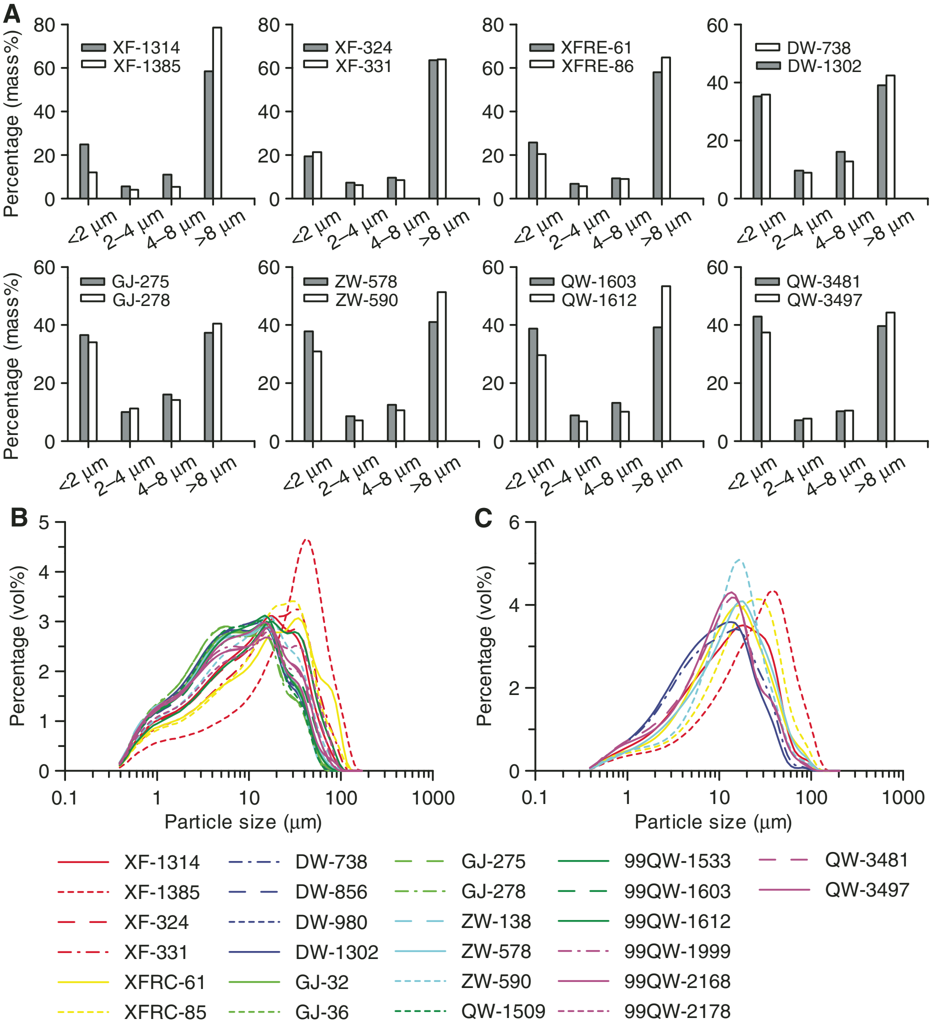

Figure 2. A: Particle size distribution determined by pipette method. B, C: Particle size distribution determined by laser granulometry using a Coulter LS 200. B: Results for all 26 samples after carbonate removal using acetic acid. C: Results for subset of 10 samples after carbonate removal using HCl. Gray bars denote paleosol samples, and light bars denote loess.

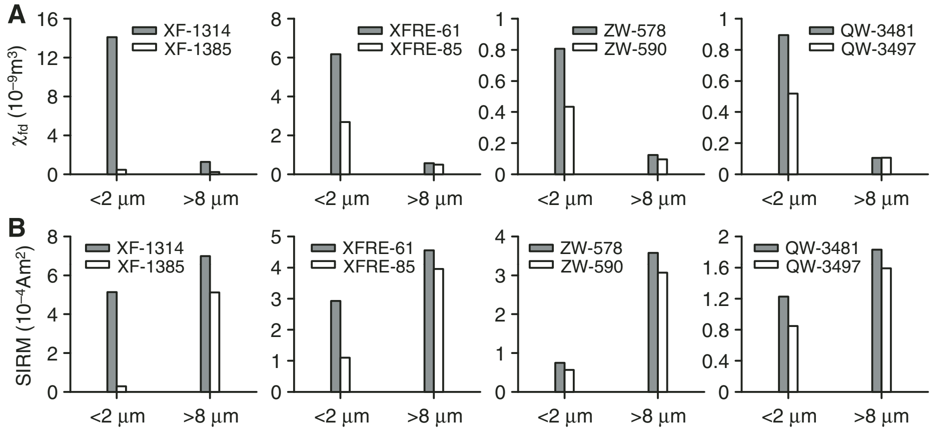

Figure 3. A: Total contribution of <2 µm and >8 µm fractions of 100 g bulk loess and soil samples to χ_{fd}. B: Total contribution of <2 µm and >8 µm fractions of 100 g bulk loess and soil samples to saturation isothermal remanent magnetization (SIRM).

frequency-dependent susceptibility, χ_{fd}, reflecting the presence of grains close to the superparamagnetic and stable single domain boundary, may be used as an indicator of the formation of fine-grained secondary ferrimagnetic oxides as a result of pedogenesis. The figure shows that the values in the clay fraction are consistently higher than in the >8 µm fraction, even when, as in the case of late Quaternary loess samples, the latter forms >70% of the sediment. SIRM values will include contributions not only from some of the fine pedogenic grains, but also from coarser ferrimagnetic grains, as well as from imperfect antiferromagnetic minerals, mainly hematite. SIRM therefore represents both pedogenic and detrital magnetic minerals. However, the extremely low values for χ_{fd} in the >8 µm fraction suggest that this coarse component is largely devoid of pedogenic material. The χ_{fd} signal in the coarse fraction is probably caused by the presence of small amounts of clay-sized particles in the coarse grade, which inevitably occurs in the pipette methods (Zheng et al., 1991; Chen et al., 1995). The SIRM peak in the coarse grade therefore mainly reflects the detrital minerals that compose the bulk of the sample in each case, suggesting a major contribution of depositional remanent magnetization to the natural remanent magnetization in Chinese eolian deposits.

These results reinforce the view that these extreme fractions separated by pipette analysis can be used to characterize, respectively, the magnetic signatures of the pedogenic and the detrital components in each sample. Subsequent research may indicate that a more complete representation of the pedogenic fraction could be achieved by using 3 µm or 4 µm as the upper size limit. Because in this sample set, the 2–4 µm fraction is never >12% of the bulk sample, this would not greatly affect the results presented here. In some earlier papers, where a fine-grained mode is identified by mathematically decomposing the particle size distributions produced by laser granulometry (Sun et al., 2004; Qin et al., 2005; Prins and Vriend, 2007), the mode so defined is on average coarser, much less well defined, and bears no resemblance to the well-defined <2 µm peak found in every sample subjected to pipette analysis, whether from the three sites represented in Figure 2A, from the Dadongling section in the far west of the Chinese Loess Plateau (Chen et al., 1995), or the Luochuan (Zheng et al., 1991) and Houzhuang (Sartori et al., 2005) sections to the east of our studied sites.

CONCLUSIONS

Pipette analysis and, where necessary, separation by sieving show that particle size assemblages in loess and paleosol samples throughout the area of the Chinese Loess Plateau and over the full temporal span of loess accumulation invariably include a well-defined mode in the clay fraction. This is never well defined and often not identified at all by laser diffraction analysis. The present results provide a direct basis for discriminating between pedogenic and detrital fractions in loess and paleosol samples and for exploring their physicochemical characteristics. From the results presented here, the approach appears to be applicable over most if not all of the Chinese Loess Plateau and throughout the period of loess deposition. It avoids destructive chemical treatments (e.g., Verosub et al., 1993; Deng et al., 2005) for which the claimed discrimination is based on less direct evidence. Moreover, the method leaves particle sized samples available for a range of subsequent analyses. The present results have important implications for research on all continental records of past environmental change based on loess-paleosol sections or other eolian depositional sequences. They are also relevant to the interpretation of records from other depositional archives containing a mixture of weathered and unweathered source materials.

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