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Magnetic and diffuse reflectance spectroscopic characterization of iron oxides in the tidal flat sequence from the coastal plain of Jiangsu Province, China

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SUMMARY

In this paper, we present the results of research on a coastal lowland Holocene tidal flat sequence in order to explore the variations in magnetic mineralogy during the process of tidal flat sedimentation and subsequent land formation. Three cores (~ 6 m in length) were collected from the coastal plain in Jiangsu Province, China, and investigated with magnetic measurements, diffuse reflectance spectroscopy (DRS) and free iron oxide (Fe_d) analyses. The tidal flat sequence shows a fining-upward trend, and the top ~ 2 m of each core with redoximorphic feature was interpreted to be a salt marsh facies in origin. Unmixing of isothermal remanent magnetization acquisition curves identify magnetite and maghemite as well as high-coercivity hematite and goethite, with the latter iron oxides confirmed by DRS analysis. Enrichment of goethite, hematite and maghemite occurs in the salt marsh deposits in comparison to the lower intertidal and subtidal deposits, with goethite being the dominant iron oxide. The changes in hard isothermal remanent magnetization acquired in field above 100 mT field (HIRM₁₀₀) and the S_{-100} ratio largely reflect the presence of medium-coercivity maghemite in absolute content and relative proportions, respectively. The approach used for iron oxide characterization may offer an efficient diagnostic tool for recognizing sediments or soils subjected to redox condition oscillation elsewhere and contribute to studies of iron cycling in (palaeo-)environmental researches.

Key words: Environmental magnetism; Magnetic mineralogy and petrology; Rock and mineral magnetism.

1 INTRODUCTION

Iron oxides in the environment display different magnetic properties, and the most studied ones include ferrimagnetic minerals (e.g. magnetite) and antiferromagnetic minerals such as hematite and goethite (Thompson & Oldfield 1986; Liu et al. 2012b). Antiferromagnetic iron oxides are widespread in soils and sediments (Cornell & Schwertmann 2003). They are normally more abundant than magnetite, but due to their weak magnetism, their magnetic signals are normally suppressed by ferrimagnetic minerals (Thompson & Oldfield 1986). However, due to their ubiquitous presence and their environmental implications, proper characterization of antiferromagentic minerals has received much attention (France & Oldfield 2000; Peters & Dekkers 2003; Balsam et al. 2004; Maher et al. 2004; Rochette et al. 2005; Roberts et al. 2006; Jiang et al. 2012). Among the methods used, diffuse reflectance spectroscopy (DRS) proves to be a rapid and efficient way for detecting these weakly magnetic iron oxides (Balsam et al. 2004; Torrent et al. 2007; Liu et al. 2012b). The information gathered plays important roles in environmental change studies. For example, the signal of hematite and goethite in loess-palaeosol sequence sediments has been used to reconstruct alternating dry and wet conditions in the Asian monsoon region (Balsam *et al.* 2004; Hao *et al.* 2008, 2009), since hematite is favoured under a warm and dry environment, while goethite is favoured in a cool and wet condition (Cornell & Schwertmann 2003). It has also been found that the degree of enhancement of magnetic susceptibility in palaeosols developed on Chinese Quaternary eolian deposits is not always in accordance with the pedogenic intensity, which is especially the case in the present subtropical zone of southern China (Ding *et al.* 2001; Guo *et al.* 2001; Hu *et al.* 2011). This phenomenon can be explained by the production and preservation of hematite over magnetite/maghemite with increased weathering intensity (Zhang *et al.* 2009; Hu *et al.* 2011; Liu *et al.* 2012a; Zhang *et al.* 2012).

Tidal flats form an important wetland type developed along the coast where there is abundant supply of fine-grained sediments and tides dominate over other hydrodynamic forces (Gao 2009). In general, tidal flats comprise an upper vegetated part termed saltmarsh

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and a lower bare flat. Saltmarsh occupies the supratidal zone and upper part of intertidal zone, where sediments tend to be finest since there is a general landward trend of declining particle size due to the attenuation of hydrodynamics (Fan 2012). Tidal flats are periodically inundated by tidal water, and the drying-wetting cycle results in alternating oxidation and reduction conditions in tidal flat sediments. Hence redox cycling of iron is a common feature in tidal flat sediments, especially in saltmarsh where vegetation provides abundant organic matter input as well as subsurface oxygen delivery through root leakage (Luther & Church 1988). Since the biogeochemical cycling of iron oxides in sediments has a profound influence on the fate of carbon, nitrogen, phosphorus, sulphur, trace metals and organic pollutants (Burdige 1993), a large number of studies have paid attention to the characterization of iron oxide mineralogy in tidal flat sediments in order to address issues such as green-house gas emission, pollutant disposal and wetland remediation (Sundby et al. 2005; Church et al. 2006; Ferreira et al. 2007; Hyun et al. 2007; Roychoudhury 2007; Taillefert et al. 2007; Burton et al. 2011; Johnston et al. 2011). Research on antiferromagnetic minerals in tidal flat sediments, however, has been rare

In this study, we aim to use combined magnetic and DRS measurements to characterize antiferromagnetic iron oxides in tidal flat sequences on the coastal plain of Jiangsu Province, China. The information gathered from this research may contribute to the study of iron oxide mineralogy in sediments or soil subjected to oscillating redox conditions elsewhere.

2 STUDY AREA AND METHODS

The study area is located in the middle part of the coastal plain of Jiangsu Province, China (Fig. 1). Historical documents and sedimentological studies have indicated that the top part of the coastal plain comprises tidal flat deposits of late Holocene origin (Zhang 1984; Zhu *et al.* 1986), closely linked to sediment supply from the Yellow River in the north and the Yangtze River in the south (Li & Wang 1998). The coring sites Caoyuan (CY), Lüsi (LS) and Sanyu (SY) are located to the east of the sea dyke called Fanggongdi built



Figure 1. Study area showing the sampling sites (open circle) on the coast plain of Jiangsu Province, China. The delta formed by the southern shift of the Yellow River during 1128–1855 and its subsequent erosion has a significant impact on coastal progradation. The historic shift of shorelines is after Zhang (1984).



Figure 2. Stratigraphic logs of the studied sections of the cores.

in the Song Dynasty (about 1 ka BP; Zhang 1984). These cores are used for the purpose of studying the coastal evolution in the Holocene, especially the impact of the southern shift of the Yellow River to the study area over the period AD 1128–1855. Geographically, they have common dominant sediment sources from the old Yellow River in the last 1000 yr, but with different distance to the river mouth. Of the three sites, Sanyu is the youngest to emerge as land during the Qing dynasty (AD 1636–1912, Zhang 1984).

The study area is situated in a subtropical climate zone. The mean annual temperature is from 14.6 to 15.1°C, with mean monthly temperatures of 27.2-27.6°C in July and 1.5-2.5°C in January. Mean annual precipitation is 1000-1076 mm, with most rainfall occurring in the summer (Zhu & Gu 1990). Due to the young age of the sediments, pedogenesis is relatively weak and the soils are classified as Cambisols according to the Chinese Soil Taxonomy (Gong 1999). The soils are normally calcareous due to the high carbonate content of the Yellow River sediments (Zhu & Gu 1990). The groundwater table is 1-2 m below the surface, and the seasonal fluctuation of the water table lead to redoximorphic features in the soil (Zhu & Gu 1990). Cropland is the main land use type, especially for cotton and vegetables. The high rainfall can leach salt in the soils. When they are reclaimed for agricultural purposes, normally salt tolerant crops are planted first, such as cotton, and measures such as irrigation and ditches are used to facilitate salt leaching.

At each site, one core was obtained, namely core CY, SY and LS, which are 30, 41 and 30 m in length, respectively. The top 30 cm of the cores was human-disturbed due to agricultural activities and therefore not included for analysis. In this study, analysis is confined to the top 6-m layer, which comprises primarily tidal flat deposits (Zhu *et al.* 1986). In general, the top *ca*. 2 m of the cores is brown in colour and composed mainly of clayey silts, with abundant rootlet traces and mottles (Fig. 2). This feature is consistent with soil developed on a saltmarsh substrate in the study area, characterized by finer sediments, higher organic carbon content and redoximorphic features. The sediments below ca. 2 m depth are greyish in colour (Fig. 2), showing interlayered sands and clays, and becoming coarser with increasing depth. Such a fining-upward sequence is in accordance with tidal flat successions observed for prograding coast (Fan 2012), which reflects the spatial transition from unvegetated intertidal sediment to vegetated supratidal saltmarsh setting with increasing elevation. AMS ^{14}C dating yields an age of 625 \pm 105 cal. yr BP for the 4.28–4.30 m layer in core SY (Fig. 2). Other chronological information from the deeper layers also indicates late Holocene deposition for the upper parts of the cores, for example, an age of 2325 \pm 25 cal. yr BP for the 10.48–10.49 m layer of core CY, and 1835 \pm 115 cal. yr BP for the 16.24–16.25 m layer of core LS.

The upper 6 m of each core was sliced at 20-cm intervals, and a total of 90 samples was obtained. All samples were dried at low temperature (<40°C) before analyses. Magnetic susceptibility was measured with a Bartington Instruments MS2B magnetic susceptibility meter, at low (0.47 kHz) and high (4.7 kHz) frequencies (χ_{1f} and χ_{hf} , respectively). Frequency-dependent susceptibility (χ_{fd}) was calculated as $\chi_{fd} (= \chi_{lf} - \chi_{hf})$ in mass-specific form. Anhysteretic remanent magnetization (ARM) was acquired in a 0.04 mT dc field superimposed on a peak alternating field of 100 mT using a Dtech 2000 AF demagnetizer and was measured with a Molspin magnetometer. ARM is expressed as a susceptibility (χ_{ARM}) by normalizing ARM with the dc field. Isothermal remanent magnetization (IRM) measurements were made using a forward field of 2.5 T followed by backfields of -100 and -300 mT delivered by an MMPM10 pulse magnetizer. The IRM obtained at 2.5 T was referred to as SIRM (saturation IRM), and the backfield remenance as IRM \times mT, where \times mT indicates the backfield value. Hard IRM was defined as HIRM₁₀₀ = $0.5 \times (SIRM + IRM_{-100 \text{ mT}})$ and HIRM₃₀₀ = $0.5 \times (SIRM + IRM_{-300 \text{ mT}})$. S-ratios (S₋₁₀₀ and S₋₃₀₀) were calculated as $S_{-100} = 100 \times (\text{SIRM} - \text{IRM}_{-100 \text{ mT}})/(2 \times \text{SIRM})$ and $S_{-300} = 100 \times (\text{SIRM} - \text{IRM}_{-300 \text{ mT}})/(2 \times \text{SIRM})$, respectively. We followed Hao et al. (2008) in calculating a modified L-ratio (Liu et al. 2007) as HIRM₃₀₀/HIRM₁₀₀.

IRM acquisition curves on selected samples were measured using a combination of Princeton Measurements Corporation Model 3900 VSM and MMPM10, with the latter generating larger field from 1 to 7 T. The IRM acquisition curves were unmixed using the method of Kruiver *et al.* (2001). High temperature thermomagnetic analysis was made using a MFK1-FA Kappabridge equipped with a CS-3 high-temperature furnace. Each sample was heated from room temperature to 700°C and then cooled to room temperature in an argon atmosphere. Low temperature remanence cycling was conducted using a Quantum Design physical property measurement



Figure 3. Relationship between redness and hematite concentration in the calibration standards.

system (PPMS-9). An SIRM was imparted at 300 K in a 2.5 T field, and measurements were made while cooling the sample to 20 K and then warming back to room temperature in a near-zero field.

Particle size was analysed with a laser size analyser (Coulter LQ-100Q) with a measurement range of $0.02-2000 \,\mu$ m, following treatment with 5% H₂O₂ and 0.2 M HCl to dissolve organic matter and carbonate, respectively. Addition of 0.5 M (NaPO₃)₆ and ultrasonic dispersion were employed to ensure complete disaggregation before analysis (Ru 2000).

The diffuse reflectance spectra were obtained over the range from 400 to 700 nm at 1-nm intervals using a Perkin Elmer Lambda 950 spectrophotometer. This analysis is carried out at every 40 cm intervals. To estimate hematite and goethite concentrations, we followed the method by Long *et al.* (2011). Briefly, redness, the percent red reflectance in standard colour bands (630–700 nm, Judd & Wyszecki 1975), was used to estimate hematite content (Torrent *et al.* 1980). We used dithionite–citrate–bicarbonate (DCB) extraction to remove the iron oxides in representative samples, which were then used as the matrix into which known amounts of pigment-grade synthetic hematite (Fangyuan Nanomaterial Institute 212–19) and goethite (Hoover Color Corp. Synox HY610) were mixed to form a set of calibration standards. A linear regression models relating hematite content to redness was produced as follows (Fig. 3):

$$Hm\% = 0.05 \times redness\% - 1.15$$
 (1)

A DCB extraction was carried out to determine the content of free iron oxides (Fe_d; Cornell & Schwertmann 2003) following the method of Mehra & Jackson (1958). Ferrihydrite is commonly believed to be the main initial diagenetic oxidation product, but it is metastable and will transform into more crystalline iron oxides such as goethite. The study by van der Zee *et al.* (2003) suggests that nanophase goethite is the dominant reactive iron oxides in aquatic sediments. Lepidocrocite favours over goethite in non-calcareous soils (Cornell & Schwertmann 2003) and its occurrence in tidal flat is less commonly reported (Johnston *et al.* 2011). Therefore we suggest that iron oxides in our samples are primarily composed of goethite and hematite. Assuming that Fe_d is composed of stoichiometric hematite and goethite, then goethite can be estimated using the following equation (Torrent *et al.* 2007):

$$Gt\% = 1.59 \times (Fe_d - Hm/1.43).$$
 (2)

Extracted magnetic minerals from selected samples were examined using field emission scanning electron microscope (SEM, Hitachi S-4800) and transmission electron microscope (TEM, JEOL2100F) to check the existence of biogenic ferrimagnetic minerals.

3 RESULTS

According to the variations in lithology (Fig. 2), particle size (Fig. 4) and magnetic properties (Fig. 5), the cores can be generally divided into two units (U1 and U2), which correspond to intertidal flat and supratidal saltmarsh facies, respectively (Zhu *et al.* 1986). The lower part of core CY can be further divided into two subunits (U1a and U1b).

3.1 Particle size

The sediments in all three cores are mainly dominated by silt $(4-64 \,\mu\text{m})$ fractions (Fig. 4). In general, the particle size fractions finer than 32 µm increase upward, while the sand fraction (>64 um) shows the opposite trend. The 32–64 um fraction increases upward first and then declines toward the surface, with the transition at the boundary of U1 and U2. This transition largely reflect the aggradation of lower unvegetated intertidal flat (U1) into upper vegetated supratidal flat (saltmarsh, U2). As a result, the mean size displays an upward fining trend in all the cores. The three cores have quite similar particle size compositions, with core CY being the coarsest on average. Although the old Yellow River is the dominant sediment supply and sorting on the sediment transport pathway may exert an influence, particle size of the tidal flat sequence is largely determined by tides and waves acting at the depositional site (Fan 2012). With similar tidal flat topography and tidal range, such a similar particle size composition is expected.

3.2 Magnetic properties

 χ and SIRM generally reflect the concentration of magnetic minerals, especially ferrimagnetic minerals (e.g. magnetite; Thompson & Oldfield 1986). χ_{ARM} is particularly sensitive to single domain (SD) ferrimagnetic grains (Maher 1988). χ_{fd} reflects the presence of fine viscous grains close to the superparamagnetic (SP)/single domain boundary (Thompson & Oldfield 1986). HIRM₃₀₀ is commonly used to estimate the concentration of high-coercivity minerals (e.g. hematite and goethite), while HIRM₁₀₀ represents the abundance of medium- (e.g. maghemite) and high-coercivity minerals (Yamazaki & Ikehara 2012). In the CY core (Fig. 5a), concentration-related parameters χ , SIRM and χ_{ARM} decrease upward in unit U1, with the minimum value occurring at depth 1.8 m. In unit U2, they display increasing trends toward the surface. χ_{fd} is quite low in sediments of U1 in comparison to those in U2. HIRM₁₀₀ and HIRM₃₀₀ are relatively low in U1 and increase upward in U2 except for a decline towards the surface. Conversely, L-ratios are higher in U1 and lower in U2.

 χ_{ARM} /SIRM is commonly used as a grain size indicator of ferrimagnetic minerals, with peaks in the SD range, from which it decreases with increasing grain size (Maher 1988). χ_{ARM}/χ has also been proposed as a grain size indicator (Banerjee *et al.* 1981), but its relationship to ferrimagnetic grain size depends on whether the grain assemblages is predominantly larger or smaller than SD (Oldfield 1994). SIRM/ χ can be influenced by a number of factors, and a



Figure 4. Particle size composition of core CY (a), SY (b) and LS (c).

higher proportion of higher coercivity minerals can lead to a higher value (Thompson & Oldfield 1986). S_{-300} serves as a measure of the relative importance of low- (e.g. magnetite and maghaemite) and high-coercivity components in the total assemblage (Bloemendal & Liu 2005), while S_{-100} reflects the ratio of low-coercivity minerals to

medium- and high-coercivity minerals (Yamazaki 2009; Yamazaki & Ikehara 2012). In core CY (Fig. 5a), $\chi_{ARM}/SIRM$, χ_{ARM}/χ and SIRM/ χ are relatively low in U1 and become higher in U2. S_{-100} and S_{-300} remain relatively stable in U1a, and decrease markedly upward in U1b with minimum values present at 2 m depth. In U2,





Figure 5. Magnetic properties and iron oxides of core CY (a), SY (b) and LS (c). Hm and Gt are estimated concentrations for hematite and goethite, respectively.

their values become higher, but still lower than those in U1a. This is especially the case for S_{-100} .

The vertical changes in χ , SIRM, χ_{ARM} , χ_{fd} , χ_{ARM}/χ , $\chi_{ARM}/SIRM$, SIRM/ χ , HIRM₁₀₀ and HIRM₃₀₀ in core SY are generally similar, that is, declining slightly upwards or remaining stable

in U1, and increasing upwards in U2 (Fig. 5b). *L*-ratio and S_{-100} values are higher in U1 and become lower in U2. In comparison, S_{-300} shows no such trend.

In core LS (Fig. 5c), χ and SIRM also decline upward in U1 but they show no obvious increasing upward trend in U2. χ_{fd} is relatively



Figure 6. Representative isothermal remanent magnetization (IRM) acquisition curves and component analysis, which enable four components to be identified, that is, low-coercivity (blue) and medium-coercivity (green) components, two high-coercivity components (purple and black). Note that all components in U2 display larger mean coervcivities than their counterparts in U1.

stable and low throughout the core. χ_{ARM} , χ_{ARM}/χ , $\chi_{ARM}/SIRM$ and SIRM/ χ show slight increasing trends in U1 and are relative stable in U2. HIRM₁₀₀ is relatively low in U1 and enhanced in U2. By comparison, the contrast in HIRM₃₀₀ between U1 and U2 is not so marked. *L*-ratio and *S*₋₁₀₀ are higher in U1 and becomes lower in U2, while *S*₋₃₀₀ shows no such trend.

We tried with both a three and a four component unmixing model, and checked statistically to find the better fit (Kruiver *et al.* 2001). The three-component model has one low coercivity component while the latter one has two. The IRM acquisition curves can be better described using four dominant components (Table 1). Typical IRM acquisition curves are presented in Fig. 6. For sediments in U1, they display two low-coercivity components, with mean coercivity (B_{1/2}) as ~30 and ~60 mT, and dispersion parameter (DP) of ~0.37 and ~0.20, respectively. The two high-coercivity components have $B_{1/2}$ as ~300 and ~1600 mT, and DP as ~0.20 and ~0.20, respectively (Figs 6c, f and i). For sediment in U2, they have one lowcoercivity component and an additional medium-coercivity component. The $B_{1/2}$ values of low-, medium- and the two high-coercivity components are ~50, ~100, ~350 and ~1600 mT (except that of core LS being >2500 mT), and DP of ~0.42, ~0.20, ~0.15 and ~ 0.20 , respectively (Figs 6b, e and h). Clearly, sediments from unit U2 are magnetically harder than those in unit U1.

For sediments from unit U1, the thermomagnetic curves indicate that χ remains relative stable before 300°C. χ starts to increases from 300 to 400°C and shows a hump around 450-550°C on the heating curves (Figs 7b, d and f). This generally reflects the transformation of weakly minerals such as siderite, pyrite or clay minerals, to ferrimagnetic minerals (Roberts 1995; Pan et al. 2000). Alternatively, it could be caused by the conversion of goethite to ferrimagnetic minerals in the presence of organic carbon and calcium (Hanesch et al. 2006). The 580°C Curie temperature indicates the presence of magnetite. The cooling curves are above the heating curves, indicating the generation of ferrimagnetic minerals during the heating-cooling cycles. For sediments from unit U2, χ shows a slight increase until 300°C and an inflection between 300 and 450°C on the heating curves (Figs 7a, c and e). This increase of χ before 300°C may result from the gradual unblocking of fine-grained ferrimagnetic particles (Liu et al. 2005). The drop after 300°C is generally interpreted to be the conversion of maghemite to hematite (Liu *et al.* 1999, 2003). When the temperature reaches 580°C, χ drops significantly, suggesting the Curie temperature of magnetite.

				CY 1.05 m, U2						CY 5	i.58 m, Ul			
	Component	SIRM	$\log(B_{1/2})$	DP	Residuals			Component	SIRM	$\log(B_{1/2})$	DP	Residuals		
					LAP	GAP	SAP					LAP	GAP	SAP
Fit 1					1.03E-9	2.19E-7	2.88E-1					1.75E-9	9.75E-8	3.85E-3
	1	2.25	1.79	0.45				1	1.65	1.48	0.38			
	2	0.42	2.09	0.18				2	1.00	1.76	0.18			
	33	0.06	2.59	0.10				3	0.20	2.40	0.22			
	4	0.17	3.25	0.50				4	0.09	3.21	0.20			
Fit 2					4.31E-9	2.44E-7	4.23E-1					2.42E-9	1.06E-7	1.12E-2
	1	2.80	1.85	0.45				1	2.75	1.63	0.32			
	2	0.15	3.42	0.56				2	0.01	2.49	0.16			
	3							З	0.01	3.20	0.29			
	Test statistics	F-test	t-test	Test results					Test statistics	F-test	t-test	Test results		
	LAP	1.45	3.1	Fit 1 better					LAP	1.02	0.4	No difference		
	GAP	1.28	0.3	No difference					GAP	1.19	0.4	No difference		
	SAP	1.46	1.3	No difference					SAP	7.95	1.0	Fit 1 better		



Figure 7. Representative temperature-dependent magnetic susceptibility curves for samples from the cores. Red and blue lines represent heating and cooling curves, respectively.

The presence of maghemite in U2 is further confirmed by lowtemperature SIRM measurement (Fig. 8). When cooling SIRM from 300 K downward to Verwey transition (T_v), SIRM first increases with maximum values occurring around 175 K and then declines with further cooling. Such a feature of cooling curve above T_v , also evident on the warming curve between T_v and 300 K, is interpreted to be the signature of maghemite/oxidized magnetite presence (Özdemir & Dunlop 2010). Such a hump of maghemite presence is not so strong, which is possibly caused by a lower proportion of maghemite to magnetite in the mixed magnetic mineral assemblage (Yamazaki 2009). This feature is not observed for sediments in U1, which displays a declining trend down to T_v on the warming curve. Also the Verwey transition is sharper in U1 sediment, suggesting dominance by magnetite.

Therefore, we interpret the low-coervicity component (~30 and ~50 mT) as magnetite, medium-coercivity (~100 mT) component as maghemite, and the two high-coercivity component as hematite (~300–400 mT) and goethite (~1600 mT and larger; Kruiver & Passier 2001; Yamazaki & Ikehara 2012). The two magnetite components may indicate different grain size fractions, with the low-coercivity components of ~30 mT corresponding to coarser size. Harder magnetite with $B_{1/2}$ of ~50 mT and narrow DP of ~0.20 in U1 could be biogenic (Egli 2004). However, SEM and TEM observations reveal both micrometer and nanometer sized detrital magnetic minerals, but bacterial magnetosomes are not observed



Figure 8. Zero-field cooling and warming cycling of SIRM obtained at 300 K.

(Fig. 9). The tidal flat sediments of the Jiangsu coast plain are mainly derived from the Yellow River, which has sediments eroded predominantly from the Chinese Loess Plateau (CLP; Zhang 1984; Ren & Shi 1986). It is revealed that loess from the CLP has both finer pedogenic and coarser detrtial ferrimagnetic fractions (e.g. Zhou et al. 1990). The coarser magnetite (~30 mT) may correspond to the eolian dust component with median destructive field of IRM (MDF_{IRM}) of \sim 26 mT (Egli 2004). A harder detrital oxidized magnetite with $MDF_{ARM}\sim\!\!79\,mT$ is also present in the loess (Egli 2004). Considering the grey colour of U1, the \sim 50 mT component could result from the diagenesis of oxidized magnetite, since reductive diagenesis may lead to preferential removal of fine-grained magnetite, maghemite, and the oxidized shell on coarser magnetite (Karlin & Levi 1983; Yamazaki & Solheid 2011). The ~50 mT component in unit U2 has larger DP (~ 0.39), suggesting that it is more likely a detrital one. The softest coercivity component (\sim 30 mT) in U1 is absent in U2. The larger remanence lost after a coolingwarming cycle of SIRM in U1 in comparison to that in U2 (Fig. 8) is consist with a larger grain size of magnetite of the former (Özdemir & Dunlop 2010).

In summary, compared with HIRM₁₀₀ values in Unit U1, those in U2 are enhanced in all cores, while the S_{-100} and *L*-ratio values are lower in unit U2 (Figs 5h, j and k). Sediments in unit U2 contain a higher proportion of medium-coercivity (\sim 100 mT) minerals (Fig. 6). The magnetic mineralogy data suggests that hematite and goethite occur in both units (Fig. 6). Although core CY and SY show evidence of increased ferrimagnetic minerals in the upper part of U2, this is not the case in core LS (Fig. 5).

3.3 Fe_d and DRS estimated goethite and hematite concentrations

In each of the three cores, goethite has a larger concentration than hematite. The variation of goethite follows that of Fe_d in general (Fig. 5). Gt, Hm and Fe_d are comparatively stable in U1 and markedly increased in U2 of cores SY and LS. In core CY, the elevated Gt, Hm and Fe_d values start earlier in U1b. The Hm/(Hm+Gt) ratio is generally higher in unit U2.

3.4 Relationship between magnetic properties, iron oxides and particle size

Correlation analysis shows that diverse relationships exist between particle size, magnetic properties and DRS parameters (Table 2). A common feature is that χ_{ARM}/χ , $\chi_{ARM}/SIRM$, HIRM₁₀₀, Gt, Hm and Fe_d are positively correlated with the fractions less than 32 µm and negatively with >64 µm fraction. In contrast, S_{-100} is negatively correlated with the fractions less than 32 µm. It is found that Gt and Hm are both positively related to Fe_d, with the former showing better correlation coefficient (Fig. 10).

4 DISCUSSION

4.1 Enrichment of iron oxides in saltmarsh facies

The lower *S*-ratio and higher HIRM₁₀₀, HIRM₃₀₀ values in U2 imply that medium- and high-coercivity magnetic minerals are higher both in relative proportions and absolute concentrations. The enhanced Gt, Hm and Fe_d values in U2 indicate that both goethite and hematite, and total iron oxides increase in this layer. This increase could be caused by particle size, as Gt and Hm are positively correlated with finer particle size fractions (Table 1). As mentioned above, the tidal flat sediments in the study area are mainly derived from the Yellow River (Ren & Shi 1986). Previous magnetic studies on the Yellow River delta sediments suggests that particle size fractions finer than 4 μ m display lower HIRM₃₀₀ values but higher *S*₋₁₀₀



Figure 9. Scanning (a) and transimission (b) electron spectroscope images of extracted magnetic minerals from the 1.05 m depth of core CY.

Table 2. Correlation coefficients between particle size, magnetic properties and iron oxides.

	<4 µm	4–8 µm	8–16 µm	16–32 µm	32–64 µm	>64 µm	Mean size
x	-0.10	-0.19	-0.37	-0.44	-0.04	0.32	0.22
χfd	0.58	0.43	0.33	0.29	-0.05	-0.39	-0.42
XARM	0.45	0.35	0.26	0.25	0.03	-0.34	-0.32
SIRM	-0.06	-0.17	-0.27	-0.20	0.10	0.14	0.11
HIRM ₃₀₀	0.38	0.20	0.16	0.30	0.25	-0.39	-0.40
HIRM ₁₀₀	0.44	0.33	0.41	0.58	0.17	-0.56	-0.51
S_{-100}	-0.32	-0.39	-0.56	-0.61	-0.12	0.56	0.51
S_{-300}	-0.20	-0.25	-0.37	-0.38	-0.15	0.38	0.36
$\chi_{\rm ARM} / \chi$	0.56	0.58	0.65	0.69	-0.01	-0.65	-0.56
χ_{ARM} /SIRM	0.60	0.61	0.59	0.53	-0.03	-0.58	-0.52
L-ratio	-0.25	-0.31	-0.44	-0.55	0.01	0.43	0.35
SIRM/χ	0.16	0.14	0.30	0.53	0.18	-0.41	-0.28
Hm	0.57	0.54	0.72	0.90	0.18	-0.82	-0.71
Gt	0.42	0.49	0.64	0.61	-0.23	-0.48	-0.35
Fed	0.49	0.53	0.71	0.77	-0.09	-0.64	-0.48

Note: Bold type is significant at p < 0.01.



Figure 10. The relationships between Fe_d and (a) Hm and (b) Gt. Hm and Gt are estimated concentrations for hematite and goethite, respectively. Open circles, solid circles and triangles represent core CY, SY and LS, respectively.

values (Zhang et al. 2008), therefore a particle size effect can not fully explain the enrichment of medium- and high-coercivity minerals in U2. Dissolution of antiferromagnetic minerals in deeper layer is a possible explanation. However, reductive diagenesis in marine sediments generally suggests that antiferromagnetic minerals are more resistant to dissolution in comparison to ferrimagnetic magnetite (e.g. Karlin & Levi 1983; Yamazaki et al. 2003). If both magnetite and antiferromagnetic minerals dissolve in U1, we might expect lower S-ratios compared to the upper part U2 due to the preferential dissolution of magnetite. Yet this is not the case. Alternatively, part of the increase in goethite and hematite concentration can be linked to generation of iron oxides in saltmarsh sediments and subsequent soil forming process. Saltmarsh is periodically inundated by flooding tides and exposed when tides ebb, therefore alternation of oxidizing/reducing conditions occur with fluctuations in the water table. In comparison to the mid-, low-intertidal flat and subtidal zones, the time of tidal inundation in saltmarsh is relatively shorter, and therefore saltmarsh are more oxic. This is further influenced by vegetation and bioturbation, as root respiration and oxygen leakage around the rhizosphere and/or burrows can form oxic niches in an otherwise anoxic layer. The wetting-drying cycle of saltmarsh favors the generation of iron oxides like goethite. For example, Thompson et al. (2006) proposed that repeated redox fluctuations promoted the conversion of ferrihydrite into more crystalline goethite and hematite. The mineral formation is explained by the catalytic action of the Fe²⁺ on the structural transformation

of ferrihydrite through a dissolution-precipitation process (Hansel *et al.* 2003; Pedersen *et al.* 2005). Once formed, goethite is more resistant to dissolution in reducing conditions, as evident in a number of marine environments (Yamazaki *et al.* 2003) and soils. For example, goethite enrichment is found in the Bg and the CrBg horizons of a lowland Gleysol in northwest Germany (Mansfeldt *et al.* 2012).

Pedogenic hematite can be formed through the pathway of ferrihydrite–maghemite–hematite (Torrent *et al.* 2006). It is also possible that low temperature oxidation of magnetite leads to hematite formation through intermediate maghemite (Cornell & Schwertmann 2003). The presence of maghemite in U2 is evident in thermomagnetic curve and low temperature SIRM cycling curve (Figs 7 and 8), although a detrital component cannot be excluded. Hematite production can happen syndepositionally in the saltmarsh environment. With the progradation of the coastline, the coring sites become subaerial and groundwater tables fall, to 1–2 m below the surface at the present-day. This will promote introduction of O₂ into unit U2, and therefore favours hematite formation.

We found, in accordance with expectations, that the relationship between Gt and Fe_d is stronger than that between Hm and Fe_d (Fig. 10). Since hematite is favoured in dry environments, therefore it is expected to be less abundant compared to goethite in low-lying coastal environments. The higher Hm/(Hm+Gt) ratios in unit U2 in comparison to U1 suggests the generation and preservation of hematite in the upper drier and oxic environments.



Figure 11. The relationships between *L*-ratio and (a) HIRM₁₀₀ and (b) HIRM₃₀₀. Open circles, solid circles and triangles represent core CY, SY and LS, respectively.

4.2 HIRM, S-ratios and L-ratio as indicators of iron oxide assemblage

HIRM₃₀₀ is commonly used as a proxy for antiferromagnetic mineral concentrations, though Liu *et al.* (2007) suggested that this holds true only if HIRM is independent of the *L*-ratio. There is no significant linear relationship between HIRM₃₀₀ and *L*-ratio (Fig. 11). A recent study on Holocene soils by Quinton *et al.* (2011) suggested that HIRM reflects changes in hematite abundance even if it is correlated with *L*-ratio. Although both HIRM₃₀₀ and Hm are higher in unit U2 than those in U1, the linear relationship between HIRM₃₀₀ and Hm is not significant. This is especially the case in U1b of core CY, in which the elevated Hm is not paralleled by HIRM₃₀₀ (Fig. 5a). This could be caused by the changes in grain size, crystallinity and impurity of hematite, which can influence the magnetism of hematite (e.g. Liu *et al.* 2007).

HIRM₁₀₀ is negatively correlated with the *L*-ratio (Fig. 11). If samples in unit U1b of core CY are removed, S_{-100} is also significantly negatively correlated with Gt (Fig. 12). Considering the more or less similar HIRM₃₀₀ values throughout core SY and LS, then the higher HIRM₁₀₀ and lower S_{-100} in U2 are caused mainly by the



Figure 12. The negative relationships between S_{-100} and estimated goethite concentration (Gt), with data removed for sediments in U1b of core CY. The whole dataset is displayed in the inset. Open circles, solid circles and triangles represent core CY, SY and LS, respectively.

presence of maghemite of medium-coercivity, as evident from the component analysis of IRM acquisition curves (Fig. 6). Therefore it suggests that sediments with higher maghemite content are accompanied by higher goethite content. Considering the dynamic redox condition of saltmarsh facies and drainage condition changes after land formation, such an association of goethite with maghemite is not unexpected. However, it should be noted that such a co-existence of maghemite with goethite is only clearly evident in U2. Although goethite generation starts earlier in U1b, no concomitant generation of maghemite is found, as shown by similar lower HIRM₁₀₀ values to those of U1a. As mentioned above, this could be that U1b is near or below the groundwater table and remain relatively reducing in comparison to the overlying U2. As maghemite is easily dissolved under reducing conditions (Yamazaki & Solheid 2011), any inherited or neoformed maghemite, if existed, would disappear in comparison to goethite. Actually, sediments in U1b display an upward-decrease in χ and SIRM, as well as lowering S-ratios, suggesting reductive diagenesis of ferrimagnetic minerals (Karlin & Levi 1983). The color change also indicates such a redox condition variations across U1b, with dark layer occurring in the top of U1b (Fig. 2a).

From the definition of the *L*-ratio, that is, HIRM₃₀₀/HIRM₁₀₀, its negative relationship with HIRM₁₀₀ is expected. Normally, a higher *L*-ratio means high coercivity (Liu *et al.* 2007). However, this is not the case in our samples. As defined by Liu *et al.* (2007), *L*-ratio equals to $M_{hard}/[M_{hard} + M_{intermediate}]$, where $M_{intermediate}$ refers to intermediate IRM components acquired in a field range 100–300 mT, and M_{hard} to hard remanence obtained in fields >300 mT. From our samples, M_{hard} is quite small as the sample is almost saturated in a field of 300 mT, while the $M_{intermediate}$ component is relatively larger in U2 (Fig. 6). Therefore it is better to use *L*-ratio together with *S*-ratios to assess the coercivity of the samples.

4.3 Implications of combined magnetic, geochemical and DRS analyses

The discussion above demonstrates that combined use of magnetic, geochemical and DRS analyses can represent the iron oxide mineralogy in more detail than any single method alone. The methods reveal the iron oxide assemblages in soil developed on saltmarsh substrate of tidal flat environments. Iron oxide mineralogy can provide information regarding diagenesis/authigenesis in tidal wetlands, which is important for interpreting palaeomagnetic data (Kodama 2012). In addition, this information is helpful for biogeochemical, environmental and ecological study of tidal wetlands, since the cycling of iron and associated elements and pollutants is strongly dependent on iron oxide mineralogy (Canfield & Berner 1987; Burdige 1993; Roden & Wetzel 2002; Neubauer *et al.* 2005). For palaeoenvironmental studies, the recognition of saltmarsh facies is also crucial in the reconstruction of sea level and the transgressive/regressive history (González & Törnqvist 2009). Since alternating redox status is also common in other environments, such as floodplains or freshwater wetlands, this approach may be potentially used elsewhere for studies of iron cycling.

5 CONCLUSIONS

Combined magnetic measurements and DRS indicates that the saltmarsh facies in late Holocene tidal flat deposits are characterized by enrichment of iron oxides including goethite, hematite and maghemite, with goethite being the dominant iron oxide. The coexistence of goethite and maghemite can be linked to the oscillating redox conditions which are a feature of saltmarsh during their formation and later emergence into the inland zone of diminished tidal influence. The changes in HIRM₁₀₀ and S_{-100} largely reflect the absolute concentration and relative proportions of medium-coercivity maghemite. This combined magnetic and DRS approach may provide an efficient diagnostic tool for recognizing sediments or soils experiencing fluctuating redox conditions and may thus contribute to studies of iron cycling in different environments.

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