

High-field magnetic susceptibility (χ_{HF}) as a proxy of biogenic sedimentation along the Antarctic Peninsula

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Abstract

High-field mass-normalized magnetic susceptibility (χ_{HF}) is presented as a proxy for biogenic sedimentation in glacial marine sediment from the western Antarctic Peninsula. χ_{HF} is measured at field strengths that exceed the saturation field of ferrimagnetic minerals. These measurement conditions exclude the contributions of ferrimagnetic iron oxides that dominate low-field volume-normalized susceptibility (k) measurements. Therefore, χ_{HF} allows for closer examination of paramagnetic and diamagnetic minerals, the latter of which includes biogenic silica and biogenic calcite. In sedimentary sequences from the western Antarctic Peninsula, the main processes affecting the abundance of diamagnetic material in sediment is biological productivity and the subsequent flux of biogenic silica to the seafloor. χ_{HF} profiles were measured on two biosiliceous sediment cores from the western Antarctic Peninsula. Comparisons with quantitative biogenic silica measurements indicate that χ_{HF} tracks % opaline silica very well, and reveals the presence of century-scale cycles in sediment composition in intervals where k appears featureless. χ_{HF} is limited as a quantitative measure of biogenic sediment flux, since terrigenous paramagnetic and diamagnetic minerals also contribute to the measurement. The interpretation of χ_{HF} can also be complicated by the presence of unsaturated high-coercivity minerals such as hematite and goethite, or by the presence of ultra-fine superparamagnetic (SP) particles. However, in sediment sequences where the condition of saturation is met, χ_{HF} is very well suited for the rapid identification of temporal trends in biogenic sedimentation.

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1. Introduction

Low-field volume-normalized magnetic susceptibility (k) is one of several flexible magnetic tools that is useful for rapid reconnaissance of sediment core composition, for correlation amongst several sediment cores, and for the detection of temporal cycles in sedimentation. Profiles of k from Holocene sediment sequences

collected along the Antarctic Peninsula (Fig. 1) reflect the interplay of several processes including variable dilution of terrigenous material with biogenic silica, shifts in the particle size of the magnetic minerals, changes in magnetic mineralogy, and possibly post-depositional diagenesis (Fig. 2) (e.g., Leventer et al., 1996; Brachfeld and Banerjee, 2000; Brachfeld et al., 2002). No single process is responsible for controlling the shape of the entire k profile, and different processes dominate the k signal at different intervals in time. Nevertheless, k is often utilized as a rapid reconnaissance measurement of fluctuations in the proportions of terrigenous and

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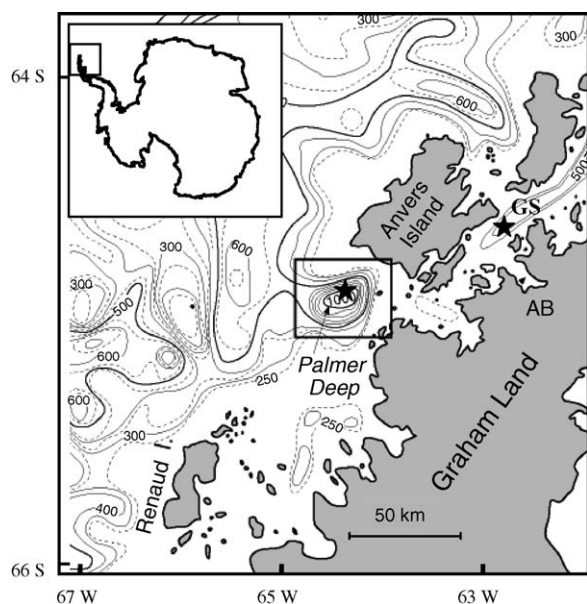


Fig. 1. Location map showing the Palmer Deep (PD, Ocean Drilling Program Site 1098), the Schollaert Drift within the Gerlache Strait (GS, JPC28), and Andvord Bay (AB). Stars denote sediment core locations. Bathymetry from Rebessco et al. (1998).

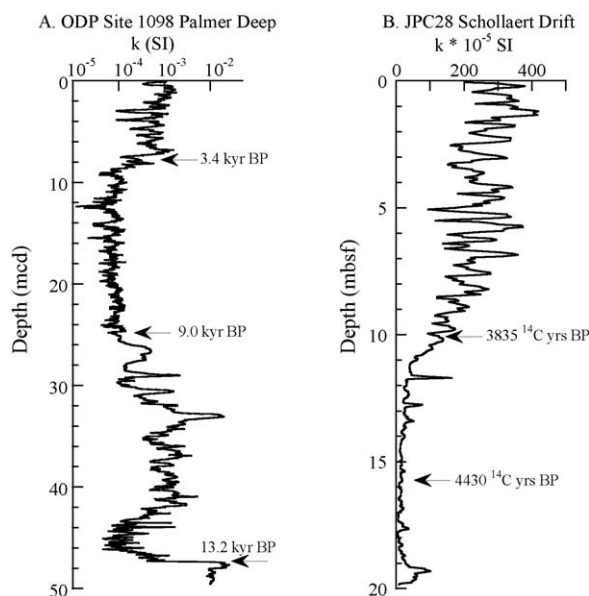


Fig. 2. Low-field volume-normalized magnetic susceptibility profiles (k) of sediment cores from the Palmer Deep (ODP Site 1098) and the Schollaert Drift (JPC28), measured with Bartington MS2C 80-mm and 125-mm diameter sensors, respectively. The late Holocene interval (0 to ~ 3.5 ka) is characterized by strong but variable k . The middle Holocene interval (9–3.5 ka) is characterized by uniformly weak k . Calibrated radiocarbon dates for ODP Site 1098 are from Domack et al. (2001). Uncorrected radiocarbon dates for JPC28 are from Domack et al. (2003).

biogenic sediment, and interpreted as a tracer of paleo-productivity (Domack et al., 2001).

The use of k as paleoproductivity proxy stems from the desire for a rapid, inexpensive tool to track this important paleoenvironmental processes. Primary production in the surface ocean is a key component of the global carbon cycle, affecting CO_2 drawdown from the atmosphere and the export of organic carbon and silica to burial at the seafloor. In biosiliceous sediments, records of export production are generated using the opaline silica mass accumulation rate. The percentage of opaline silica in sediments is determined using a selective dissolution experiment described by DeMaster (1979, 1981) and since modified by others (e.g., Mortlock and Froelich, 1989). While this technique is very precise and has excellent reproducibility for duplicate samples, it is time-consuming and requires constant attention during the analysis. Hence the continued use of physical properties such as k and gamma-ray attenuation porosity evaluated bulk density (GRAPE) to track paleoproductivity.

There are several challenges associated with the use of k as a tracer of paleoproductivity, particularly along the Antarctic margin. Downcore variations in magnetic mineralogy and magnetic particle size cause very large variations in k that mask the productivity-driven dilution effects of biogenic silica (Brachfeld and Banerjee, 2000; Brachfeld et al., 2002). This study presents high-field magnetic susceptibility (χ_{HF}) as a tool for monitoring biogenic sedimentation in glaci-marine sediment. Brachfeld et al. (2002) observed that century-scale cycles were present in glacial–marine sediment χ_{HF} profiles, even where k profiles were featureless, and suggested that χ_{HF} was the more appropriate parameter for tracking biogenic sedimentation. This parameter has several advantages over k . The χ_{HF} measurement is made on discrete samples of dry sediment. This eliminates the dilution effect of water, and each discrete sample is truly an independent measurement rather than a convolution over a several-cm interval, as is the case with pass-through k sensors. Most importantly, χ_{HF} targets the diamagnetic and paramagnetic minerals in sediment and eliminates the contributions of the ferrimagnetic minerals that dominate low-field susceptibility measurements. This study presents the χ_{HF} measurement technique, error estimates, caveats, and comparisons with quantitative biogenic silica measurements in order to provide a framework for assessing the utility of this parameter as a tracer of paleoproductivity along the Antarctic continental margin.

2. Samples and methods

This study compares χ_{HF} and quantitative opaline silica measurements on cores from two sites along the western margin of the Antarctic Peninsula (Fig. 1). The first site, Ocean Drilling Program Leg 178 Site 1098, was triple APC cored at $64^{\circ}51.7235'$ S, $64^{\circ}12.4712'$ W, in 1012 m of water (Barker et al., 1999). Samples for rock-magnetic analyses were collected every 10-cm from Hole 1098A. The magnetic mineralogy of this core has been examined in detail, and consists of magnetite and titanomagnetite (Brachfeld et al., 2002). Quantitative opaline silica measurements were made at a 2.5-cm interval down the entire length of Hole 1098B (Anderson and Ravelo, 2001; Dunbar et al., 2000, 2001, 2002, 2003, 2004). The second site, NBP99-03 JPC28 from the Schollaert Drift ($64^{\circ}38.647'$ S, $62^{\circ}52.407'$ W, 673 m water depth), was cored with a single jumbo piston core during cruise 99-03 of the *R/VIB Nathaniel B. Palmer* (Domack et al., 2003). The middle-Holocene to late-Holocene low-field susceptibility profiles of the two sites are very similar (Fig. 2). However, the magnetic mineral assemblages have key differences. The base of JPC28 contains magnetite and a high-coercivity phase, identified as hematite using X-ray diffraction (Manley et al., 2002). Opaline silica measurements from core JPC28 are the purview of another project and are not presented here. However, the presence of hematite in JPC28 provides an opportunity to investigate and quantify the potential complicating factors in the χ_{HF} method and test techniques for removing these complications.

Magnetic measurements were performed at the Institute for Rock Magnetism, University of Minnesota, using freeze-dried sediment samples of 100–200 mg. Magnetic hysteresis measurements were made on a Prince-

ton Applied Research Vibrating Sample Magnetometer (VSM) and on a Princeton Measurements Corp. Micro Vibrating Sample Magnetometer (μ VSM-3900). A peak field of 1 T was used on both instruments. Samples from Ocean Drilling Program Site 1098 were measured primarily on the VSM, whereas samples from core NBP99-03 JPC28 were measured primarily on the μ VSM. Selected samples from both cores were measured on both instruments for cross calibration, and were also measured on a Quantum Design Magnetic Properties Measurement System (MPMS) in applied fields up to 2.5-T. Low-temperature remanence measurements and frequency dependence of susceptibility measurements (10, 31, 100, 310, and 1000 Hz, from 20 to 300 K) were made on the Quantum Design MPMS.

Quantitative opaline silica measurements were made at the University of Santa Cruz Institute of Marine Sciences and the Stanford University Stable Isotope Laboratory (Anderson and Ravelo, 2001; Dunbar et al., 2000, 2001, 2002, 2003, 2004). Sediment samples were freeze-dried, lightly ground, and sieved to obtain a uniform particle size distribution. Samples were dissolved in hot sodium hydroxide for several hours, and aliquots were periodically removed to measure the Si concentration via spectrophotometry. Weight percent silica was determined by calibration with standards of known Si concentration. The concentration versus time trend was extrapolated back to time zero to determine the initial weight percent of biogenic silica (Fig. 3).

3. High-field magnetic susceptibility

χ_{HF} is the high-field slope of magnetization (M) versus applied field (H) curves, commonly referred to as hysteresis loops, which are measured using a steady

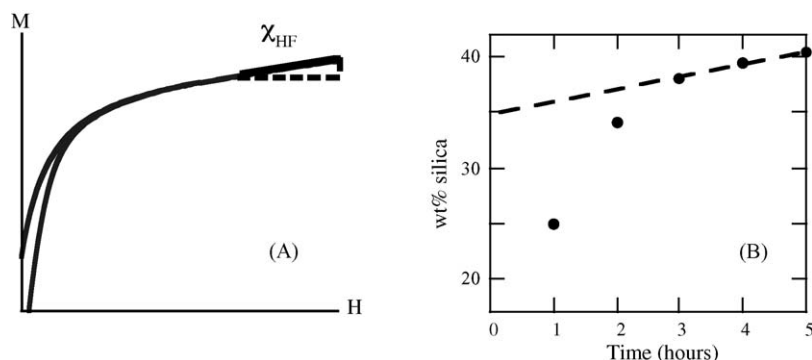


Fig. 3. (A) The + M + H quadrant of a hysteresis loop. χ_{HF} is calculated as the slope of the M - H curve at applied fields that are higher than those required to saturate ferro- and ferrimagnetic minerals, i.e., field strengths above where the loop's upper and lower branches converge. (B). A schematic example of the selective dissolution method to determine % opaline silica. Aliquots of sample dissolving in NaOH are collected and measured every hour via spectrophotometry. A trend-line is fit to the data as the solution approaches Si saturation, and extrapolated back to time zero to determine the initial concentration of Si. Absorbance is converted to weight % silica using a series of standard solutions with known concentration of Si.

field generated by electromagnets. χ_{HF} is calculated at field strengths higher than those required to saturate the ferrimagnetic minerals (Fig. 3). When a purely ferrimagnetic material is saturated, increasing the strength of the applied field will cause no further change in the intensity of the induced magnetization; $dM/dH=0$ and ferrimagnetic susceptibility is zero. Therefore, χ_{HF} reflects only the contributions of paramagnetic and diamagnetic minerals in the sample, i.e., the biogenic sediment and the “non-magnetic” detrital sediment. Magnetite and titanomagnetite saturate in fields of 100–300 mT. High-coercivity antiferromagnetic minerals such as hematite and goethite require saturation fields >0.3 to >2 T (Dunlop and Özdemir, 1997).

The robustness of χ_{HF} as a proxy of biogenic sediment content is examined here by calculating χ_{HF} using several different subsets of the high-field M – H data. The term H_{max} is used to indicate the maximum field strength applied to the sample during the hysteresis loop measurement ($H_{\text{max}} = 1$ T for this study). The term “% field” is used to indicate the applied field range used to calculate χ_{HF} . For example, a % field value of 70% denotes that χ_{HF} was calculated over the interval $0.7 \times H_{\text{max}}$ to H_{max} , which corresponds to 0.7–1 T for this study. The calculation of χ_{HF} is made using all four high-field limbs of the hysteresis curve. Using 70% as an example, the four limbs are $+0.7H_{\text{max}}$ ramped up to $+H_{\text{max}}$, $+H_{\text{max}}$ ramped down to $0.7H_{\text{max}}$, $-0.7H_{\text{max}}$ ramped up to $-H_{\text{max}}$, and $-H_{\text{max}}$ ramped down to $-0.7H_{\text{max}}$. A linear regression was applied to each of the four limbs to determine the slope of the M – H data, and the four slopes were then averaged to calculate χ_{HF} . This calculation was made for the intervals 0.7–1 T (% field = 70%), 0.75–1 T (% field = 75%), 0.8–1 T (% field = 80%), 0.85–1 T (% field = 85%), and 0.90–1 T (% field = 90%). The number of data points used in the regression for each of these intervals is approximately 60, 50, 40, 30, and 20, respectively.

4. Results

The use of χ_{HF} to monitor biogenic sedimentation is predicated on the saturation of the magnetic mineral assemblage. Saturation may not be achieved if high-coercivity minerals are present (e.g., hematite, goethite), or if superparamagnetic particles (SP) are present. The presence or absence of these phases was assessed using the saturation field observed in hysteresis loops, and using diagnostic temperature-dependent order/disorder transitions (see Dunlop and Özdemir, 1997).

Examples of hysteresis loops from the late Holocene interval (strong but variable k) and middle Holocene

interval (uniformly weak k) of ODP Site 1098 and JPC28 are shown in Fig. 4. The Palmer Deep sediments reach saturation in fields of 200–300 mT. Late Holocene samples from core JPC28 reach saturation in fields of 200–300 mT. Middle Holocene samples from JPC28 required fields of 500–600 mT to saturate, indicating the presence of a high-coercivity phase. Samples from this interval display the magnetite Verwey transition, but not the hematite Morin transition. The observed low-temperature remanence behavior, with greatly separated field-cooled and zero-field-cooled curves, is consistent with, although not diagnostic of the mineral goethite (see goethite low-temperature curves available online at www.irm.umn.edu/bestiar/index.html). However, the presence of hematite was confirmed via X-ray diffraction measurements (Manley et al., 2002). Whether the high-coercivity behavior in JPC28 is due solely to hematite or to a hematite–goethite mixture has yet to be determined. Nevertheless, the samples do reach saturation, and this interval provides an opportunity to test the limitations of χ_{HF} when a high-coercivity mineral is present.

χ_{HF} values may be skewed by the presence of unsaturated superparamagnetic (SP) particles. Very fine SP particles can require fields up to 800 mT to achieve saturation (Fig. 5) (Carter-Stiglitz et al., 2001). The presence of SP particles in Antarctic sediment was assessed using the frequency-dependence of magnetic susceptibility at low temperatures (20–300 K) (see Worm, 1998). Frequency-dependence refers to the frequency of the applied magnetic field, which controls the amount of time that the field “observes” the particles. SP particles are detected by varying time and temperature conditions to force SP particles to behave like stable single domain grains (SSD), which reduces their contribution to k . Frequency and temperature dependence measurements are shown in Fig. 6. Neither Site 1098 nor JPC28 exhibits significant frequency dependence of magnetic susceptibility in samples from the late Holocene interval (Fig. 6). However, magnetic extracts from the middle Holocene interval of Site 1098 display frequency dependence below ~ 100 K (a property of titanium-rich titanomagnetite – M. Jackson, unpublished data) and above ~ 250 K, which suggests the presence of SP titanomagnetite particles (Brachfeld and Banerjee, 2000). Hysteresis loops from this interval are clearly saturated by 300 mT, and there is no systematic variation in χ_{HF} as a function of % field (Fig. 7). Therefore, it is believed that the SP particles present at Site 1098 are coarse SP and saturated below 0.7 T. Middle Holocene samples from JPC28 do not display frequency dependence of susceptibility (Fig. 6). However, the JPC28 measurements were

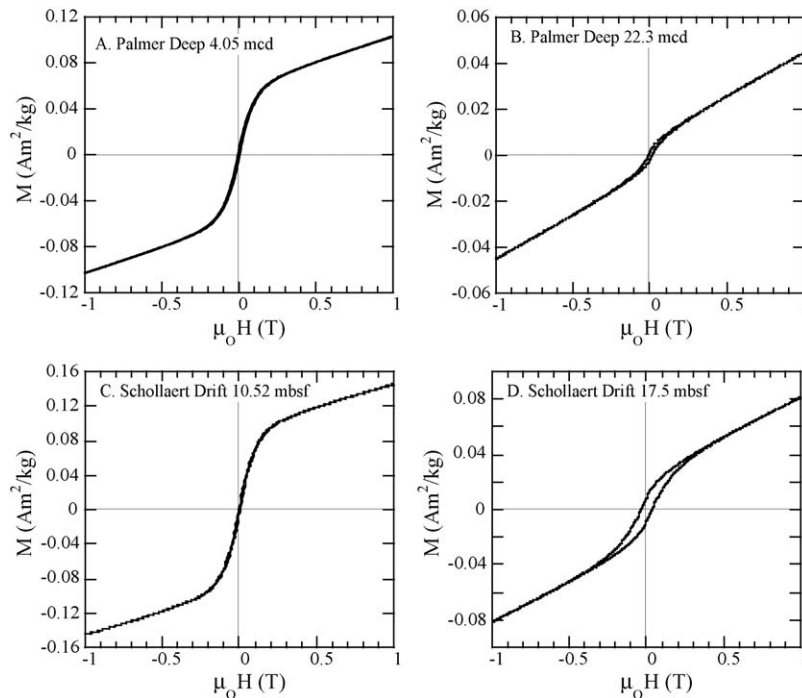


Fig. 4. Hysteresis loops from (A) the Palmer Deep late Holocene sediment, (B) the Palmer Deep middle Holocene sediment, (C) Schollaert Drift late Holocene sediment, and (D) Schollaert Drift middle Holocene sediment. All samples reach saturation below 0.7 T.

made on bulk sediment, and SP particles would be difficult to detect in bulk sediment if they are present in very low concentrations.

χ_{HF} as a function of % field is shown in Fig. 7. Nearly all samples from Site 1098 display 2–4% variation in χ_{HF} . In contrast, the late Holocene interval of JPC28 samples show more sensitivity to the % field. Late Holocene (0–10.5 mbsf) samples from JPC28 display a 2–14% variation in χ_{HF} as a function of % field. Generally, χ_{HF} decreases as higher applied fields are

used in the calculation of the parameter, which suggests incomplete saturation. Middle Holocene samples (10.5–20 mbsf) from JPC28 display only 2% variation in χ_{HF} as a function of % field. It is important to note that the hematite/goethite indicators were observed in the middle Holocene samples, and these are samples with very stable values of χ_{HF} . The JPC28 late Holocene χ_{HF} behavior can potentially be explained by the presence of unsaturated SP particles, which may be magnetite, hematite, or perhaps maghemite. Work is in progress

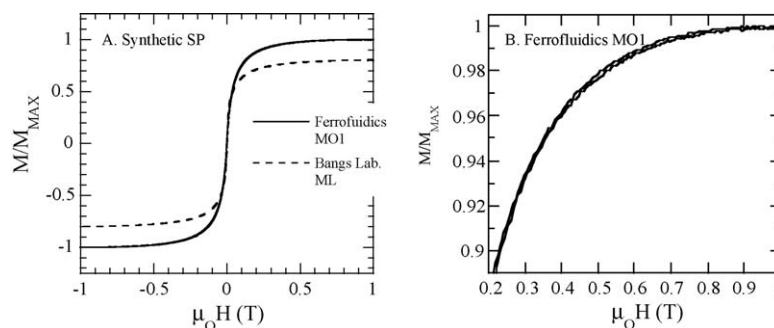


Fig. 5. (A) Room temperature M – H curves for SP particles in ferrofluid (Ferrofluidics MO1, solid line) with a mean grain-size of 10-nm (Carter-Stiglitz et al., 2001), and SP particles embedded in magnetic latex (ML) microspheres (Bangs Laboratories Inc., dashed line) with a mean grain size of 11-nm (Relle and Grant, 1998). Grain size was estimated from best-fit Langevin functions (Relle and Grant, 1998). M is plotted as M/M_{max} , and the ML microsphere data has been multiplied by a factor of 0.8 for the purpose of distinguishing the two curves on the same plot. (B) Close-up of the high-field data for MO1. The saturation field is ~ 800 mT.

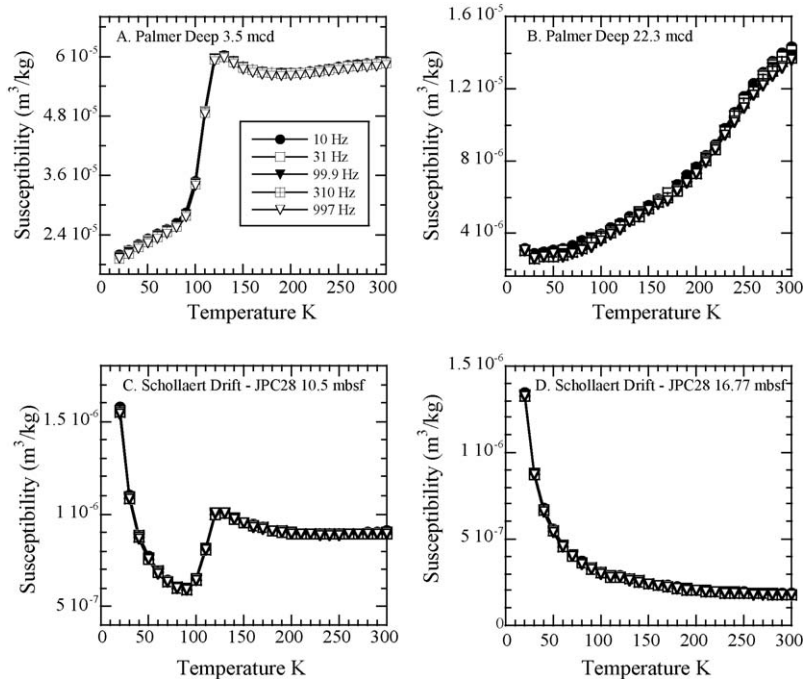


Fig. 6. (A–D) Susceptibility as a function of temperature and applied field frequency [$\chi(fT)$] for (A) the Palmer Deep late Holocene interval, (B) the Palmer Deep middle Holocene interval, (C) Schollaert Drift late Holocene interval, and (D) Schollaert Drift middle Holocene interval. There is no evidence of frequency dependence in the late Holocene samples. Schollaert Drift measurements were made on bulk samples, which may mask the frequency–temperature behavior of SP particles if they are present in very low concentrations. Magnetic extracts made from the Palmer Deep middle Holocene samples do show frequency dependence below 100 K (a property of titanomagnetites) and above ~ 250 K. A detailed discussion of $\chi(fT)$ behavior observed in these samples is given in Brachfeld and Banerjee (2000).

to extract and concentrate the magnetic particles from the JPC28 sediments in order to improve the chances of detecting the presence of, and identifying the mineralogy of the SP particles.

The stability of χ_{HF} in the middle Holocene interval of JPC28 may also be linked to the measurement conditions, specifically the measurement time constant.

Weakly magnetized samples from the middle Holocene interval of JPC28 were measured using a longer time-constant in order to reduce noise. The time constant is the time taken by the VSM to step the applied field intensity from H to $H + \Delta H$, allow the field to “settle” at the new intensity, and then measure M . The Princeton Applied Research VSM uses a field settling time

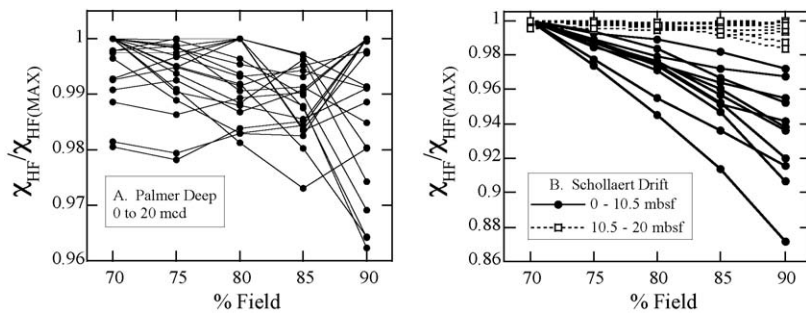


Fig. 7. χ_{HF} as a function of % high-field slope used in the calculation. Values are normalized by the maximum value calculated. Twenty samples are displayed from each core, taken from 1, 2, up to 20 mcd. (A) Samples from Site 1098 show a consistent 2–4% variation, and there is no systematic pattern in the location of $\chi_{\text{HF(max)}}$. (B) Samples from the late Holocene interval of JPC28 display 2–14% variation as a function of % field, with $\chi_{\text{HF(max)}}$ typically occurring at the 70% calculation and $\chi_{\text{HF}}/\chi_{\text{HF(max)}}$ decreasing at higher % field values. This may indicate the presence of unsaturated SP particles. Middle Holocene samples from JPC28 contain high-coercivity hematite and possibly goethite, and yet the samples show <2% variability in χ_{HF} . Longer averaging times and the absence of SP particles are likely responsible for the stability of χ_{HF} in this interval.

of approximately 0.4 s, which translates into 15 min to measure a complete hysteresis loop (J. Marvin, personal communication). Increasing the time constant greatly improves the steadiness of the applied field and reduces the uncertainty when measuring the induced moment. The Princeton Measurements Corp. μ VSM allows the user much more flexibility in setting the time constant than the VSM. Time constants of 1–1.5 s were selected for weakly magnetized samples from JPC28, which resulted in 30–45 min to measure the complete hysteresis loop on the μ VSM. The longer time constant was initially employed to improve the accuracy of the saturation remanence (M_R) and the measurement of coercivity (H_C). However, an additional benefit was greatly improved stability in the high-field slope.

The combined results from both cores suggest that χ_{HF} derived from “normal speed” hysteresis loops ($t=0.1$ – 0.5 s and complete loops measured in <15 min) will be sensitive to the range of H values used in the calculations. The data presented here suggest an inherent uncertainty of approximately 2–4% in χ_{HF} from normal speed loops. The uncertainty can be reduced by using a longer time constant, however, this increases the length of time required to measure the hysteresis loop. It should be noted that measurement of χ_{HF} does not require the complete hysteresis loop, and routines can be easily written to measure only the four high-field limbs and thus reduce the duration of the experiment, if desired. Further, although using 70% of the high-field data means more data points contribute to the calculation of χ_{HF} , the data presented here point to using 90% of the high-field data to ensure that the condition of saturation is met.

Downcore profiles of χ_{HF} are shown in Fig. 8. Whereas k profiles from Site 1098 and JPC28 appear relatively featureless in the middle Holocene, χ_{HF} reveals the continuation of regularly spaced highs and lows that are also seen in the late Holocene k records. The utility of χ_{HF} as a biogenic sedimentation tracer was assessed by comparing χ_{HF} from Site 1098 with down-core measurements of % opaline silica (Anderson and Ravelo, 2001; Dunbar et al., 2000, 2001, 2002, 2003, 2004). Low values of χ_{HF} correspond to high values of % opaline silica, with a correlation coefficient of -0.638 (Fig. 8). The correlation coefficient is likely lowered by the two samples sets coming from different cores at Site 1098; Hole A for rock-magnetism samples and Hole B for geochemistry samples. The three holes from Site 1098 were placed on a meters composite depth (mcd) scale using the program SPLICER (Acton and Borton, 2001). However, a fundamental tenet of SPLICER is that 9.5-m cores may only adjust stratigraphically up or down, and are for-

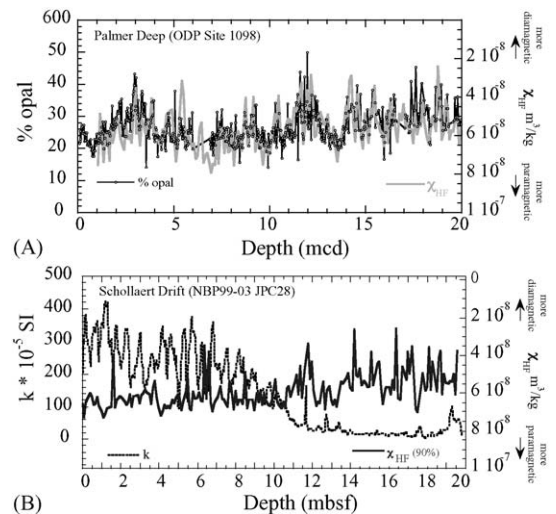


Fig. 8. (A) χ_{HF} and quantitative opaline silica from ODP Site 1098. χ_{HF} is presented as the average of the 70, 75, 80, 85, and 90% high-field slopes. Highs in % opaline silica correspond to lows in χ_{HF} , suggesting that diamagnetic silica is driving χ_{HF} values. The correlation coefficient between the two parameters is -0.638 , which is lowered by the imperfections of the mcd correlation at the centimeter scale. (B) A comparison of k and 90% χ_{HF} in JPC28. Note the absence of a discontinuity at 10.5 mbsf, which suggests that hematite and goethite are not contaminating the χ_{HF} measurement.

bidden to expand or contract. The mcd scale therefore maximizes the correlation over the entire length of the records being compared, approximately 50-m for Site 1098. The inability to expand or contract a core, and hence account for genuine differences in sedimentation rates between holes, means that the mcd correlation is imperfect at the centimeter scale. Therefore, the features seen here, with wavelengths on the order of 20–30 cm and smaller, are not perfectly aligned on the mcd scale, which almost certainly lowers the correlation coefficient between χ_{HF} (Hole A) and % opaline silica (Hole B). Towards that end, an adjusted mcd scale is in progress, along with new radiocarbon calibrations (from Hole C), that will improve the age model of Site 1098 for spectral analysis in progress at Stanford University and the University of Santa Cruz (R. Dunbar and A. Ravelo, email communications).

One additional large-scale trend is evident. The large shift in the k profile of the Palmer Deep occurs at ~ 8.3 mcd, but there is no discontinuity in χ_{HF} at that depth (Fig. 8). Similarly, there is no discontinuity in the JPC28 χ_{HF} record at 10.5 mbsf, suggesting that the high-coercivity mineralogy of the middle Holocene interval has not contaminated the high-field measurements of either record (Fig. 8). Finally, it should be noted that χ_{HF} must be calibrated using % opal data, since terrige-

nous detrital minerals (quartz, feldspars, clay minerals, etc.) contribute to the χ_{HF} measurement. Therefore, χ_{HF} cannot be directly substituted for % opaline silica in calculations of opal mass accumulation rates, primary production and export production. However, the preliminary results presented here demonstrate the promise and potential for using closely spaced χ_{HF} measurements as a proxy for biogenic sedimentation trends.

5. Conclusions

High-field mass-normalized magnetic susceptibility (χ_{HF}) is a parameter that enables direct examination of the paramagnetic and diamagnetic fraction of sediment assemblages, and is well suited to monitoring downcore variations in biogenic silica. Similarly, it is expected that χ_{HF} would track biogenic calcite in carbonate-rich sediments. Comparison of χ_{HF} with quantitative biogenic silica measurements made on two sediment cores from the western Antarctic Peninsula indicate that χ_{HF} tracks % opaline silica very well. In addition, χ_{HF} profiles reveal the presence of regularly spaced highs and lows in biogenic sediment content in intervals where low-field susceptibility (k) is featureless. Measurement of χ_{HF} can be complicated by the presence of unsaturated high-coercivity minerals and ultra-fine superparamagnetic particles. Therefore, appropriate rock magnetic analyses are needed to characterize the magnetic mineralogy and determine the field strength required to saturate the sample. In the sediment cores analyzed here, a high-field subset of $0.9H_{\text{max}}$ to H_{max} appears to ensure that the condition of saturation is met. When the appropriate field strengths are utilized, χ_{HF} has the potential to serve as a rapid, inexpensive tool to detect temporal trends in biogenic sedimentation.

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