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Detrital magnetization of laboratory-redeposited sediments

Jean-Pierre Valet, Cyrielle Tanty and Julie Carlut

Institut de Physique du Globe de Paris, Université Paris Diderot, Sorbonne Paris-Cité, UMR 7154 CNRS, 1 rue Jussieu, F-75238 Paris Cedex 05, France. E-mail: jpvalet@me.com

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SUMMARY

We conducted several redeposition experiments in laboratory using natural and artificial sediments in order to investigate the role of grain size and lithology on sedimentary remanence acquisition. The role of grain size was investigated by using sorted sediment from natural turbidites. Taking advantage of the magnetic grain size distribution within turbidites, we compared redeposition experiments performed with coarse magnetic grains taken from the bottom layers of a turbidite with fine grains from the upper layers of the same turbidite. In order to document the magnetization acquired for increasing sediment concentrations that is analogous to increasing depth in the sediment column, the samples were frozen at temperatures between -5 and -10 °C. Magnetization acquisition behaved similarly in both situations, so that little smearing of the palaeomagnetic signal should be linked to grain size variability within this context. Other series of experiments were aimed at investigating the influence of lithology. We used clay or carbonated sediments that were combined with magnetic separates from basaltic rocks or with single-domain biogenic magnetite. The experiments revealed that the magnetization responded differently with clay and carbonates. Clay rapidly inhibited alignment of magnetic grains at low concentrations and, therefore, significant magnetization lock-in occurred despite large water contents, perhaps even within the bioturbated layer. Extension of the process over a deeper interval contributes to smear the geomagnetic signal and therefore to alter the palaeomagnetic record. In carbonates, the magnetization was acquired within a narrow window of 45–50 per cent sediment concentration, therefore, little smearing of the geomagnetic signal can be expected. Finally, experiments on carbonate sediments and biogenic magnetite with increasing field intensities indicate that magnetization acquisition is linear with respect to field intensity. Altogether, the results suggest that sediments with dominant carbonate content should be favoured for records of geomagnetic field changes provided that the minor clay fraction does not vary excessively. They confirm the advantage of using cultures of magnetotactic bacteria for redeposition experiments.

Key words: Biogenic magnetic minerals; Palaeointensity; Rock and mineral magnetism.

INTRODUCTION

Sediments containing iron-titanium magnetic oxides acquire a stable remanent magnetization through complex processes that involve sedimentary as well as magnetic processes. Soon after deposition, compaction and diagenesis can interfere with magnetic particle alignment. Unfortunately, the uppermost centimetres of deep-sea or lacustrine sediments cannot be sampled without introducing physical disturbances due to relatively high water contents. Therefore, our knowledge of the mechanisms governing the acquisition of remanence can only be improved by indirect approaches. These problems were addressed by studying the magnetic characteristics of sedimentary deposits (Granar 1958; Griffiths *et al.* 1960; Irving & Majo 1964; Kent 1973; Verosub 1977; Blow & Hamilton 1978; Tauxe & Kent 1984; Lund &

Keigwin 1994; Channell & Guyodo 2004), by redeposition experiments of sediments in the laboratory (King 1955; Levi & Banerjee 1975, 1990; Barton & McElhinny 1979; Barton *et al.* 1980; Hamano 1980; Yoshida & Katsura 1985; Anson & Kodama 1987; Deamer & Kodama 1990; Løvlie 1974, 1976; Hamano 1980; Løvlie & Torsvik 1984; Lu *et al.* 1990; Kodama & Sun 1990; Van Vreumingen 1993a,b; Quidelleur *et al.* 1995; Katari & Tauxe 2000; Katari *et al.* 2000; Carter-Stiglitz *et al.* 2006; Spassov & Valet 2012), as well as by theoretical modeling (Tucker 1980; Shcherbakov & Shcherbakova 1983, 1987; Jackson *et al.* 1991; Meynadier & Valet 1996; Katari & Bloxham 2001; Roberts & Winklhofer 2004; Jezek & Gilder 2006; Shcherbakov & Sycheva 2008, 2010; Jezek *et al.* 2012; Mao *et al.* 2014; Egli & Zhao 2015) and numerical simulations (Tauxe *et al.* 2006; Heslop 2007; Bilardello *et al.* 2011; Heslop *et al.* 2014).

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Laboratory experiments cannot duplicate the depositional processes of sediments with accumulation rates as low as those of most marine sequences. In some ways, redeposition experiments in laboratory are similar to turbiditic/homogenite deposition events. This convergence suggests that comparing natural and artificial deposits at various levels within turbidites could also constrain processes that govern the depositional remanence (Tanty *et al.* 2016). Most experiments have been performed so far with natural sediments and therefore did not fully investigate the role played by the nature of magnetic material. Despite chemical and mechanical pre-treatments (sieving, ultrasonic treatment, deflocculation, etc.), the sediment cannot be completely reset to its pristine condition when falling in the water column so that experiments most likely fail to duplicate the exact processes that generated partial alignment of the magnetic grains by the ambient geomagnetic field.

Artificial sediments have been used in a few studies (Irving & Majo 1964; Hamano 1980; Deamer & Kodama 1990; Van Vreumingen 1993a,b). This approach has the advantage of dealing with simple mixtures that are restrained to a few known constituents. It is thus possible to investigate the influence of specific parameters such as magnetic grain size, sediment granulometry, sedimentary constituents or other factors such as salinity, density, etc. Another uncertainty resides in the arrangement of the magnetic grains within the sediment. Are they dispersed or do they form aggregates with other particles? These questions relate also to the importance of flocculation in deposited sediments. In this study, we attempt first to investigate the role of grain size on magnetic alignment by redeposition of coarse and fine sediments taken from the lower and upper layers of a turbidite. In a second step, we focus on the role of carbonate versus clay content for two different types of magnetic particles. Stable remanent magnetizations are carried by monodomain or pseudo-monodomain magnetite, so we preferably used single-domain magnetite from cultures of magnetotactic bacteria (MTB) but also magnetic extracts from volcanic material. In recent years, it has been shown (e.g. Roberts et al. 2012, 2013; Yamazaki & Shimono 2013) that inorganic remains of MTB can contribute significantly to the magnetization of sediments that have not undergone extensive diagenetic alteration. This makes it important to develop an improved understanding of the magnetic recording of fossil magnetosomes, which can be simulated by redeposition of fresh biogenic magnetite.

EXPERIMENTAL PROTOCOL

All experiments were performed within $2 \times 2 \times 2$ cm palaeomagnetic plastic cubes. A first set of redeposition experiments was conducted with natural sediments that were gently crushed and mixed to remove aggregates or clusters of particles. Sediment of known concentration by mass was deposited above 20 °C within a magnetic coil surrounded by U-metal cylinders in order to avoid interactions with the laboratory field. Gelatin was used to consolidate the samples with low sediment concentration by rapid cooling at 0 °C in order to generate solidification. All experiments were performed with deionized water. We did not use saline solutions as recent experiments (Spassov & Valet 2012) showed that this parameter had no significant effect in contrast to results from previous studies (Van Vreumingen 1993a; Katari & Tauxe 2000). In support of this observation, we mention that magnetizations of lake and marine sediments present similar characteristics.

The second approach involved artificial sediments. Different mixtures were obtained by combining constituents such as clay or car-



Figure 1. (a) SEM image of a magnetic extract taken from La Palma basalts used for redeposition experiments (TiMt: titanomagnetite; Pl: plagioclase and Pyr: pyroxene) and (b) TEM image of MSR-1 magnetotactic bacteria (after Orlando *et al.* 2015).

bonate with magnetic material. We checked that the anhysteretic and saturation remanences of the carbonate and kaolin powders used for the experiments were several orders of magnitude smaller than the magnetic fractions. In contrast to previous experiments (Carter-Stiglitz et al. 2006; Spassov & Valet 2012) that used gelatin for consolidation, the samples were frozen at temperatures between -5 and -10 °C. This technique has the advantage of being as rapid as the gelatin but of removing uncertainties concerning a possible influence of gelatin on the lock-in process despite its water-like physical properties. A first set of experiments was performed using magnetic separates of basaltic rocks from La Palma (Canaries, Spain). The resulting powder was ground and sieved to obtain the finest fraction (63 µm) that was subsequently magnetically separated. Microscopic examination using a scanning electronic microscope (SEM Zeiss EVO) in backscattered mode (Fig. 1a) revealed that the magnetic separates were sometimes enclosed within a nonmagnetic matrix that could not be completely removed. This observation is considered when analysing magnetic particle alignment by low magnetic fields.

About half of the experiments were conducted with MTB. We initially used Magnetospirillummagneticum (AMB-1) and then changed to the gram-negative MSR-1 MTB. A major reason for using this second species is that the bacteria are cultured in large amounts within a 70 l semi-automated fermenter by the 'Nanobacterie' Company. A typical MSR-1 magnetotactic bacterium containing a long chain of magnetite magnetosomes is shown in Fig. 1(b). Sample preparation with bacteria was conducted by



Figure 2. (a) Magnetic hysteresis loop for MSR-1 magnetotactic bacteria used in this study. (b) Day plot (Day *et al.* 1977) of MSR-1 magnetotactic bacteria (MTB) used in this study (red diamonds), AMB-1 (green diamonds) and other species have been analysed so far (grey circles, Denham *et al.* 1980; Moskowitz *et al.* 1993; Pan *et al.* 2005; Li *et al.* 2009, 2010, 2012; Lin & Pan 2009; Paterson *et al.* 2013). Also shown are the magnetic extracts from the basalts used in this study. The dotted line represents a theoretical curve drawn from Dunlop (2002) for SD+MD mixtures.

pipetting 100 μL of solution of MSR-1 bacteria containing 1.7×10^6 cells mL $^{-1}$ that was subsequently diluted to reach the required concentrations. We followed the same protocol as for the basalt powders and incorporated the magnetosomes (with their membranes) within an artificial matrix composed of carbonate and/or kaolinite powders.

MSR-1 magnetosomes remain poorly described in the literature. We tested whether their magnetic characteristics are similar to those of AMB-1. In Fig. 2(a), we show a typical hysteresis loop measured from a solution of bacteria injected within a straw sample holder that was used for measurement in a vibrating magnetometer and was then frozen with liquid nitrogen. As expected, the curve is typical of single-domain magnetite (Fig. 2b) which is consistent with published results for cultivated MTB and those obtained from species of MTB extracted with their membranes from various natural environments. Data from MSR-1 and AMB-1 lie close to each other and the results are consistent with those for single-domain magnetite. Overall, magnetic parameters for natural MTB appear to be more scattered than for the cultured species. The difference can be explained by the presence of non-biogenic material that remained aggregated within sediment particles. Also shown in Fig. 2(b) are magnetic hysteresis parameters from the basalt magnetic extracts that lie within the range of values expected for the pseudo-singledomain state (Day et al. 1977).

REDEPOSITION OF NATURAL SEDIMENTS WITH CONTROLLED GRANULOMETRY

Following previous experiments (Spassov & Valet 2012), we investigated first to what extent magnetic grain size can affect the magnetization process. A basic assumption is that the orientation of coarse magnetic grains is locked within the sediment prior to that of fine particles. In a recent magnetic study of turbidites (Tanty *et al.* 2016), downcore magnetic grain size profiles indicated a systematic and significant coarsening at the bottom of turbidites, similar to the trend of sediment grain size. The magnetic remanence of the lower layers is significant, but it is not oriented along the field

direction. During the early stage of the process, a large amount of particles were in suspension under turbulent conditions. We suspect that tiny magnetic grains were incorporated within clusters, but their alignment with the field was inhibited by friction forces. Due to rapid sediment accumulation, magnetization lock-in was fast without post-depositional reorientation.

Turbidites have the advantage of sorting naturally the sedimentary and magnetic grains, and therefore provide naturally calibrated samples that can be used for redeposition experiments. In addition, the fast discharge of natural sediments during turbiditic events can be compared with the timescale of laboratory sediment redeposition experiments.

We sampled sediment from the bottom (coarser grains) and upper levels (finer grains) of a turbidite from core MD12-3418 from the Bay of Bengal. Magnetic granulometry and sediment properties were characterized by Tanty *et al.* (2016). Redeposition experiments were performed in plastic cubes for sediment concentrations by mass increasing from 10 to 55 per cent. Beyond 55 per cent, the mud is compact and magnetic grains are embedded within the sediment matrix. At least 4 or 5 experiments were performed for each sediment concentration. Sediment was poured into plastic cubes and was subjected to a 50 µT horizontal field while cooling below 0 °C. We subsequently imparted an anhysteretic remanent magnetization (ARM) and an isothermal saturation remanence (SIRM) to normalize the detrital remanent magnetization (DRM) to the amount of remanence carrying magnetic material.

The evolution of both data sets can be compared in Fig. 3. The magnetic moments tend to be aligned by the field as long as viscosity does not inhibit grain rotation. Magnetization acquisition for bottom as well as for top layers remains at a high level up to 45 per cent sediment concentration and then drops rapidly. The comparison between turbidite and laboratory experiments can be developed further by considering the NRM/ARM or the NRM/IRM values (Tanty *et al.* 2016). Because the same 50 μ T laboratory field intensity was used for all experiments, these ratios represent the relative percentage of magnetic moments aligned by the field. The NRM/ARM ratio primarily deals with the single-domain grains, while the NRM/SIRM involves the whole distribution of grain sizes. The horizontal lines



Figure 3. Detrital magnetization (shown as DRM/ARM and DRM/SIRM ratios) for redeposition experiments with increasing sediment concentrations for sediments obtained from the bottom and top of a turbidite from core MD12-3418 (Bay of Bengal). Brown diamonds: bottom of turbidite and green circles: top of turbidite. Horizontal lines indicate the values measured in the original sediment. The original and experimental values intersect between 45 and 50 per cent sediment concentration.

in Fig. 3 indicate the mean NRM/ARM and NRM/SIRM values for the turbidite. There are some differences between the patterns of the bottom and top lines in both plots, but they are smaller than the experimental uncertainties. In all cases, they intersect the DRM/ARM and DRM/SIRM profiles of the present experiments at a sediment concentration of 48-50 per cent. The turbidite is estimated to be younger than 1 ka. The field used for the experiments does not differ much from the 43 µT present geomagnetic field at the sampling site and should thus be even closer to the field contemporaneous of the turbidite estimated age (between 0.5 and 1 ka) (Korte et al. 2011), but, regardless the overall pattern of the curves does not depend on field intensity. Indeed, the magnetic alignment is constrained by field intensity, while the lock-in depth depends on sediment physical parameters (grains sizes, magnetic concentration, interstititial voids, etc.). Therefore, complete magnetization lock-in was likely reached for similar sediment concentrations within the turbidite and in the laboratory experiments.

Complete remanence acquisition occurred for similar sediment concentrations in the upper and lower levels, likely because a small fraction of tiny magnetic grains from the bottom layers plays a significant role in the remanence and obeys the same laws as in the upper layers. Water concentration decreases with depth within the sedimentary column, thus increasing the sediment concentration is a way to simulate burial at greater depth. Assuming that these observations remain valid for slowly accumulated sediments, they imply that lock-in of NRM primarily depends on sediment concentration.



Figure 4. Magnetization acquisition measured (NRM/ARM and NRM/ SIRM) after deposition of artificial slurries of sand and basalt powder as a function of sediment concentration. For both indicators, the same amount of magnetic particles was aligned by the field at all sand concentrations.

The 45 per cent concentration obtained in the present experiments is consistent with the value obtained for natural sediments by Carter-Stiglitz *et al.* (2006). The absence of differential lock-in between coarse and fine grains suggests little smearing of magnetic records of field variations.

REDEPOSITION OF ARTIFICIAL SEDIMENTS

Test experiments with a sand matrix

The first experiment was conducted for different concentrations of Fontainebleau sand (white non-magnetic sand) that was mixed with magnetic extracts from basalts of from La Palma island. Similar experimental results were obtained for all sand concentrations (Fig. 4). Clearly, the sand matrix never inhibited orientation of the magnetic grains. High NRM NRM/SIRM values (0.25) indicate further that a large proportion of magnetic grains were involved in the experimentally acquired magnetization, far above values that are typically observed for natural sediments that carry a stable magnetization. The interstitial large voids between the coarser sand grains explain this behaviour. The results confirm also that the sediment matrix plays a significant role in the magnetization lock-in processes.

Comparative influence of CaCO₃ and kaolin

Previous redeposition experiments (Spassov & Valet 2012) of natural sediments with various carbonate concentrations suggested a possible influence of lithology on remanence acquisition. In order to investigate this point further, we documented DRM acquisition in



Figure 5. Linear relationship between ARM values and magnetotactic bacteria concentration, which validates the method used to evaluate the number of biogenic cells in solution.

artificial sediments with increasing amounts of carbonate and kaolin. Two sets of experiments were conducted using either magnetic separates from basalts that incorporate a large range of magnetic grain sizes or single-domain magnetite from bacteria that were preserved with magnetosome membranes in order to avoid magnetic chain collapse. The interest of using biogenic material was to restrain the magnetic fraction to single-domain magnetite grains that are stable remanent magnetization carriers in sediments.

The total amount of magnetite can be estimated from the SIRM. However, we are mostly dealing with single-domain magnetite grains, so we used ARM as a normalizer to estimate the amount of magnetite. We tested that the ARM values are linearly related to increasing cell concentrations of 8.5×10^5 , 3.2×10^5 and 1.7×10^5 cells mL⁻¹ (Fig. 5). We inferred that magnetic interactions did not change significantly at these concentration levels and therefore even less when adding other constituents.

Results from experiments with magnetic extracts and biogenic magnetite are plotted in Fig. 6, where we illustrate the evolution of magnetization for increasing carbonates (Fig. 6a) and kaolin concentrations (Fig. 6b). The patterns derived from both experiments are globally similar when using magnetic extracts or biogenic bacteria if we exclude fluctuations linked to experimental uncertainties that are quantified by the error bars. For carbonate (Fig. 6a), a roughly constant magnetization is acquired at concentrations lower than 35–40 per cent. Beyond this value, the magnetization decreases rapidly and becomes negligible above 45–50 per cent for the samples that contain MTB. Results from the magnetic extracts could suggest some remanence acquisition above 45 per cent, but results obtained with other normalizers (SIRM and K) indicate no acquisition at these levels.

In all cases, the basalt magnetic extracts have a stronger magnetization than the MTBs. A relevant difference between the two sets of magnetic particles is their size distribution. MTBs are characterized by a narrow range of single-domain grains, while the basalt powders have a wide grain size distribution. Therefore, tiny MTBs embedded within the sediment have little ability at align with the field due to their weak magnetic moment, while coarser magnetic



Figure 6. Magnetization acquisition for artificial slurries with (a) carbonates or (b) clay for MSR-1 bacteria or basalt magnetic extract, respectively. The ability of magnetic particles to align with the field decreases for increasing amounts of kaolin. In contrast, there is no significant change below 45 per cent for carbonates.

particles have a stronger magnetic torque and therefore acquire a stronger magnetization.

The experiments performed with a kaolinite matrix reveal a uniform decrease in magnetization acquisition (Fig. 6b) for increasing sediment concentration. DRM/ARM at low sediment concentrations is also lower for the basalt powders than for carbonates, while they are similar in both situations with biogenic magnetite and closer to the values of natural sediments with similar carbonate contents (Spassov & Valet 2012). This observation is most likely related to the large difference between the grain size distributions of both artificial sediments. We suspect that the large magnetic grains were rapidly aggregated within clay or other particles and therefore not free to align with the field, even for large water contents. This process evidently yields a lower magnetization. With increasing sediment concentration, kaolinite interacts further with the aggregated magnetic particles and restrains further their ability at align with the field (Katari & Tauxe 2000). Therefore, floc formation and/or other factors such as those linked to Van der Waals forces are efficient for kaolinite concentrations as low as ≤ 10 per cent. In contrast, carbonate powder does not really inhibit magnetic grains alignment below 40-45 per cent sediment concentration (Fig. 6b). Beyond this limit, magnetization acquisition drops sharply.

As for the experiments conducted with natural sediments, increasing sediment concentration is analogous to increasing depth in the sediment column. In this case, lock-in profiles obtained with kaolin indicate that a proportion of magnetic grains is already locked for high water contents and, therefore, will not be reoriented further. This process increases with depth, that is, for decreasing water contents. We cannot exclude that a proportion of these grains can be magnetized within the bioturbated layer and, therefore, partly randomized by biological activity. This could explain why clav-rich sediments can be associated with complex palaeomagnetic directions and large directional dispersion. It is also difficult to envision subsequent realignment because the interstitial water content rapidly decreases with depth. We must, thus, expect smearing of the geomagnetic record due to progressive lock-in as a function of depth. The magnetization profile is strikingly different for carbonates. In this case, magnetic grains remain free to reorient at sediment concentrations up to 40 per cent. Therefore, no significant lock-in occurs above the depth that fits with this concentration, but then most of the magnetization is acquired over a narrow depth window, corresponding to 40-45 per cent of sediment concentration, which implies rapid timing and lock-in and, therefore, little signal smearing.

Response of magnetization to field intensity

The absence of a linear response between the remanent magnetization and field intensity has been pointed out in a few redeposition experiments (Katari & Tauxe 2000; Katari & Bloxham 2001; Tauxe et al. 2006; Mitra & Tauxe 2009) with natural or composite sediments has been linked to either aggregation of particles or to the effects of pH and salinity. To our knowledge, redeposition experiments that have been carried out with MTBs were performed by Paterson et al. (2013), and more recently by Zhao et al. (2016). In the first study, the authors injected solutions of AMB-1 bacteria within plastic cubes and let them dry in a varying applied field for a period of 5-6 d. The NRM/ARM and NRM/SIRM values obtained at increasing field strengths were fitted by a linear model, but NRM/ARM values above 100 µT underestimate the expected value by 10 per cent. This behaviour was likely caused by magnetic interactions in stronger applied fields. Saturation of magnetic remanence is expected in the absence of any component that inhibits alignment with the field. In principle, in the absence of interactions, deviation from linearity would be expected close to saturation, which was clearly not attained at 100 µT.

In order to constrain further the relationship between magnetization and field intensity for dispersed MTB with sedimentary constituents, we followed the same protocol as in the previous sections with MTB with 20 per cent carbonate content. The same amount of bacteria was used for each experiment. The samples were stored in an ambient field for 12 hr at -8 °C. In Fig. 7, we report NRM/SIRM results as a function of field strength between 5 and 100 μ T. Each data point represents the average of 4–8 samples. The magnetization is linear with field intensity. The results could suggest a tendency toward saturation by fields higher than 80 mT, but we must take into account that the error bars are relatively large and that only 3 per cent of the magnetic grains were aligned at 100 μ T. We infer that there is no significant departure from perfect linearity (as indicated by the correlation coefficient of the linear fit).

CONCLUSIONS

Taking advantage of the deposition rates inherent to turbidites and laboratory redeposition, we have demonstrated that the magnetization lock-in profiles of the coarse magnetic grains from the lower turbidite layers is similar to those of the finer magnetic grains from



Figure 7. Linear correlation between the remanent magnetization acquired and the field applied during redeposition experiments. Each data point represents the average of at least four distinct experiments and a maximum of eight. The equation for the linear fit through the origin is $y = 3.2 \times 10^{-4}$ with a correlation coefficient R^2 of 0.97.

the upper layers. We infer that grain size distribution does not generate significant smearing of geomagnetic signal in natural environments. However, our experiments were carried out using specific lithologies and we cannot exclude smearing in other conditions. Other experiments confirmed that sand sediments are not capable of retaining a magnetic orientation, similar to natural sandy environments.

Keeping in mind that processes that govern laboratory remanence acquisition cannot be compared easily with those of slowly deposited sediments, we investigated the role of specific parameters such as carbonate and clay content on magnetization acquisition using artificial slurries at increasing sediment concentrations. We observed that magnetic moments alignment of single-domain biogenic magnetite was locked between 40-50 per cent carbonate concentrations. If we interpret these concentrations in terms of depth within the sedimentary column, we should not expect significant geomagnetic signal smearing. For artificial clay sediments, the amount of magnetization decreases as a function of sediment concentration and a large magnetization contribution can be acquired at high water contents. This situation suggests that a large fraction of magnetic grains is locked early and perhaps within the bioturbated layer yielding complex orientations. Assuming that the process extends over a large depth interval down to the critical depth of full lock-in, we must expect smearing of the geomagnetic signal. Therefore, the present observations suggest that smearing could be linked to the amount of clay and its variability within sediments rather than to magnetic granulometry. This could explain why significant smearing is observed only in a few records of geomagnetic polarity reversals and excursions that meet specific conditions (Valet et al. 2016). Finally, a series of successive redeposition experiments in field intensities up to 100 µT confirm the linear response of magnetic remanence to field intensity.

Our results indicate that redeposition experiments remain pertinent to document the alignment of magnetic particles within sediments. New technical aspects developed in this study include experiments with artificial sediments that were frozen during redeposition, which have proven to be appropriate for assessing sedimentary remanence acquisition. Our results also reveal that cultures of MTB are ideal for future experimental studies which should include a wide range of investigations involving different sediment compositions and mixtures of bacteria with other magnetic material.

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