

## 1.4.1. Environmental Magnetism: Sediment Source Tracing

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**ABSTRACT:** Environmental magnetism is a technique used to analyse the natural characteristics of materials. The mineral magnetic array exhibited by a sample e.g. a single rock-based or composite soil, sediment or core material, are differentiated according to the iron oxide assemblage. Such accumulations of iron oxides principally reflect the parent material characteristics, but also any subsequent environmental processes e.g. erosion, deposition, and biogenic processes. Consequently, environmental magnetism has been utilised in sediment source tracing investigations. Sediment accumulations in impacted locations, i.e. ‘*targets*’ such as rivers and lakes, are assumed to possess the characteristics of the soils within the contributing catchment area. Mineral magnetic measurements, including magnetic concentration, mineralogical composition and magnetic grain size, can therefore be used as sediment tracers to indicate subtle differences between potential source samples. The contribution of sources to the target is determined using multivariate statistical analysis or un-mixing models. The results of source tracing studies are essential to inform cost-effective management strategies targeted at reducing soil erosion and improving water quality.

**KEYWORDS:** environmental magnetism, remanence, susceptibility, sediment sources

### Introduction

Environmental magnetism utilises the mineral magnetic behaviour of a material to interpret the environmental processes acting upon it. The magnetic character is dependent upon the assemblage of minerals within a material. Although all materials will respond to magnetic fields, iron oxides are particularly sensitive. The near-universal occurrence of iron oxides in the environment e.g. haematite, magnetite and goethite, provides many opportunities to apply the technique. The assemblage of magnetic minerals is primarily dependent upon the parent material, specifically rock-formation, and any subsequent modification due to geomorphic processes e.g. transportation, deposition and post-depositional processes (Liu *et al.*, 2012). Due to the range of electron structures within different oxides, responses from each type will differ when exposed to externally applied magnetic fields, and this enables

discrimination between contrasting sources and processes.

The principles of environmental magnetism, initially introduced as a research area by Thompson and Oldfield (1986), were founded on rock magnetic techniques. Since then, the approach has been applied to a variety of applications (Table 1), including: estuarine and fluvial sediment source tracing (Caitcheon, 1993; Jenkins *et al.*, 2002); reconstruction of ice extent in glacial environments (Walden and Ballantyne, 2002); climatic reconstruction of loess sequences (Heller and Evans, 1995); reconstruction of paleo-environmental geomorphology (Sun *et al.*, 2011); classification of soils (Booth *et al.*, 2005); and, pollution studies to identify spatial patterns of vehicle pollution (Maher *et al.*, 2008).

*Table 1: Summary of environmental magnetism studies for multiple applications*

Application	
Glacial	Nolan <i>et al.</i> (1999), Walden and Ballantyne (2002), Gurney and White (2005), Ojala <i>et al.</i> (2011)
Palaeoclimatic	Sagnotti <i>et al.</i> (1998), Dearing <i>et al.</i> (2001), Gathorne-Hardy <i>et al.</i> (2009), Bradák <i>et al.</i> (2011)
Paleomagnetism	Zijderveld <i>et al.</i> (1991), Dupont-Nivet <i>et al.</i> (2002), Lisé-Pronovost <i>et al.</i> (2009), Sun <i>et al.</i> (2011)
Pollutants	Maher <i>et al.</i> (2008), Blundell <i>et al.</i> (2009), Zhang <i>et al.</i> (2008a), Wang (2013), Crosby <i>et al.</i> (2014)
Sediment sources	Caitcheon (1993), Foster and Walling (1994), Caitcheon (1998), Jenkins <i>et al.</i> (2002), Hounslow and Morton (2004), Blake <i>et al.</i> (2006)
Soil	Maher (1998), Booth <i>et al.</i> (2005), Neff <i>et al.</i> (2005), Hannam and Dearing (2008), Lourenço <i>et al.</i> (2014)

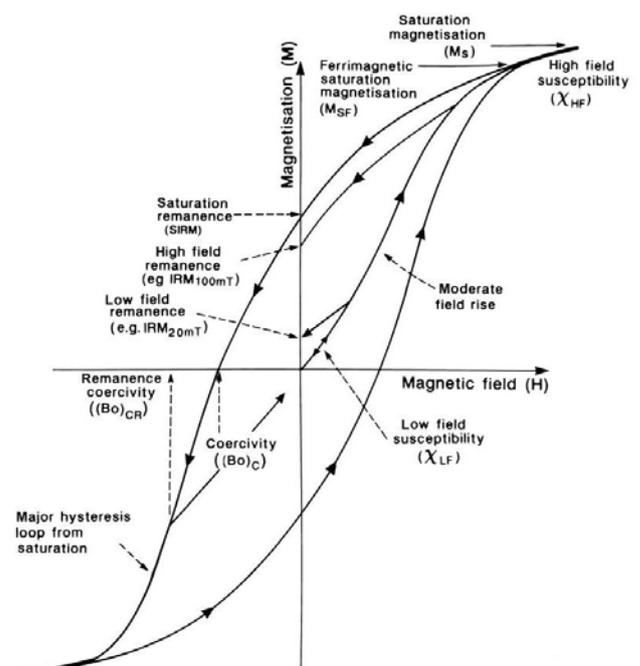
containing iron or iron-based substances, e.g. iron-oxides and iron-sulphides. When exposed to a small magnetic field, the concentration of magnetic material termed the magnetic susceptibility ( $\chi$ ) is measured. Characteristics of  $\chi$  for different magnetic behaviours are summarised in Table 2, however, composite samples such as soils and sediments will vary according to the constituent minerals (Hatfield and Maher, 2008).

On application of stronger magnetic field, ferromagnetic minerals additionally respond such that they hold a permanent magnetic moment or natural remanent magnetisation. A sample cannot return to the original pre-field magnetisation measurement (Dekkers, 1997). As field strength increases, remanent magnetisation also increases until it reaches 'saturation', i.e. the maximum magnetisation possible which is known as the Saturation Isothermal Remanent Magnetisation (SIRM). Once the material reaches the saturation magnetisation, an 'in-field' hysteresis loop (Figure 1) is established. To remove the remanence, de-magnetisation must be conducted (Walden, 1999a). The magnetic field required to return a sample to full demagnetisation is defined as the coercivity.

Mineral magnetism provides great utility for techniques such as source tracing studies. Identification of sources of fluvial sediment is crucial to inform targeted and cost-effective soil and water quality mitigation programmes. Characteristics of sediments collected from a catchment outlet, e.g. lake or river, are compared to the soils within the contributing catchment area and the source provenance un-mixed. By determining the principal sources, these areas may be targeted for management. This chapter summarises the principles of magnetic behaviour and application of the technique to fluvial source tracing.

## Mineral magnetism

Three main types of magnetic behaviour exist; diamagnetism, paramagnetism and ferromagnetism (Smith, 1999). Diamagnetic minerals include those lacking in iron such as calcite, quartz and halite (Booth *et al.*, 2005), organic substances and water (Maher *et al.*, 2009). Paramagnetic minerals, for example olivine, biotite and pyrite may contain iron in their structures (Dearing, 1999). Ferromagnetic behaviour occurs in minerals



*Figure 1: Theoretical remanence acquisition and hysteresis of a ferrimagnetic mineral denoting key susceptibility and iso-thermal remanence terminology. (From Maher *et al.*, 2009).*

Table 2: Response of magnetic mineral sub-classes to susceptibility and remanence measurements. (Summarised from Dearing, 1999; and Smith, 1999).

	Susceptibility ( $10^{-6} \text{ m}^3 \text{ kg}^{-1}$ )	Remanence
Diamagnetism	Weak negative magnetisation ( $<0$ )	No remanence
Paramagnetism	Weak positive magnetisation ( $0.01-1$ )	No remanence
Canted anti-ferromagnetism (magnetically hard)	Moderate positive magnetisation ( $0.27-1.19$ )	Low fields: little or no remanent magnetisation High fields: 300 mT required to obtain response
Ferrimagnetism (magnetically soft)	Strong positive magnetisation ( $50-1116$ )	Remanent magnetisation exhibited at lower applied fields e.g. $<100$ mT
Ferromagnetism	Very strong positive magnetisation ( $68850-276000$ )	Abundance limited in natural samples

The character of remanence acquisition in a ferromagnetic material alters according to the mineralogy and the domain arrangement within it. In general, magnetically 'softer' minerals known as ferrimagnets (e.g. magnetite) exhibit a large proportion of the SIRM in lower applied fields. Magnetically 'harder' materials known as canted anti-ferromagnets (e.g. haematite) require much higher applied fields to achieve a substantial remanent response (Table 2). As such, multiple fields of measurement are investigated to determine the mineralogical composition.

Magnetic domains or grain size additionally affects remanence measurements. Five grain size classes are defined (from the finest): super-paramagnetic, viscous magnetism, stable single domain, pseudo-single domain, and multi-domain (Smith, 1999). For a single mineral larger, multi-domain grains will respond to remanence measurements differently to smaller, single domain grains which are more resistant to external current (Peters and Dekkers, 2003; Maher and Thompson, 2009). An hysteretic remanent magnetisation (ARM) is used to detect ultra-fine magnetic mineral grains (Banerjee *et al.*, 1981) and has been utilised in multiple investigations.

## Application of technique

### Tracing sediment sources

Sediment source tracing is a geomorphological technique to determine the provenance of sediments within a catchment. The method has been applied on a series of scales, from catchments less than  $15 \text{ km}^2$  (Gruszowski *et al.*, 2003) to those greater than  $100,000 \text{ km}^2$  (Maher *et al.*, 2009). Sediment sources are determined according to their particulate characteristics, therefore the range of parameters or 'tracers' measured by mineral magnetics provides a suitable technique for source tracing.

A number of assumptions underlie sediment tracing techniques and must be considered: (1) a tracer must distinguish at least two sources; (2) a tracer is transported with sediment and not altered by selective erosion; (3) source properties are stable over time; and, (4) no post-depositional tracer modification (Foster and Lees, 2000).

### Desktop study

Source tracing programmes require subdivision of a contributing catchment area into definable and contrasting source areas. The specific research hypothesis will dominate source area definition, e.g. to target sub-catchment basins, erosion processes or using a pilot study to determine appropriate source areas. In order to effectively sub-divide, it is essential to understand the sources and generation of magnetic minerals in the environment.

Naturally, the variety of abundance and characteristics of magnetic materials occurs due to the combined effect of parent materials and secondary processes. The formation of crystals in igneous rocks is dependent on the speed of magma cooling, subsequently determining the exact components of metallic compounds. In metamorphic and sedimentary rocks however, the materials available for formation, in addition to the nature and intensity of formation, will determine the magnetic character (Liu *et al.*, 2012). Consequently, contrasting catchment geology will create a primary magnetic 'imprint'.

### Field assessment

In some cases, parent signatures are sufficient to distinguish between soil types or lithologies (Booth *et al.*, 2005; Hatfield and Maher, 2008). However, a range of secondary processes may affect the consistency of the magnetic signature (e.g. ex-situ material delivery), bio-mineralisation, in-situ authigenesis, and anthropogenic additions (Thompson *et al.*, 1980; Hilton *et al.*, 1986; Dekkers, 1997).

Bio-mineralisation, for example, occurs in topsoils which enhance the fine ferrimagnetic concentration in water-logged soils or those frequently exposed to wetting and drying cycles (Maher and Taylor, 1988; Fassbinder *et al.*, 1990). Burning of vegetation for vegetation control or by wildfire increases the fine magnetic component in topsoils due to extreme soil temperature (Blake *et al.*, 2006). Such secondary signatures modify the primary, parent material signature, providing either an opportunity to define a source according to such characteristics, or adding complication to source interpretations. Additionally, small quantities of strong magnetic material may overpower the signal of weaker minerals, in some cases this will make the technique unsuitable for the particular study (Lees, 1999). It is therefore essential to complete a visual field assessment and a preliminary magnetic susceptibility study of the monitoring area, to determine which areas may be dominated by secondary magnetic processes (Lees, 1999).

### Sample collection

Collection of source samples must be carefully conducted to prevent contamination

of the natural mineral magnetic signal. Non-metallic equipment must be used where possible, and where unavoidable, potentially contaminated particles in contact with a metallic surface must be removed. Relevant site specific information such as, in the case of soils, the sampled horizon, which may explain primary or secondary mineral forming processes, should be recorded in order to aid later interpretation. Where surface measurements are made with a field-sensor, high vegetation cover and soil moisture may cause fluctuations in results due to the addition of diamagnetic materials. Source areas must be adequately sampled in order to fully determine the characteristics of each area. Where intra- and inter- unit variability overlap, the definitive capabilities of the final result are compromised (Lees, 1999).

Target sample collection for un-mixing is dependent on the depositional environment. Suspended sediments, river-bed, lake, estuarine and sea-shelf deposits have all been collected in source provenance studies (Collins *et al.*, 1998; Jenkins *et al.*, 2002; Collins and Walling, 2007; Maher *et al.*, 2009).

Signature modification may also occur in the post-depositional environment due to the bacterial formation of magnetite. Jelinowska *et al.* (1997) discovered that bacteria in anoxic lake sediments reduced iron-oxides such that the magnetic domains were irreversibly altered. Maher *et al.* (2009) used acid dissolution to remove secondary magnetic material to expose particles within host grains of target samples, i.e. the primary magnetic signature for source tracing. Measurements performed on treated and un-treated samples were found to be distinctly different, supporting the concept that the impact of secondary magnetic processes is significant.

Post-collection, samples must be made airtight and refrigerated to prevent biological activity altering the mineral magnetic composition. Depending on ambient temperature during field sampling, it may be sensible to store samples in a cool box to prevent temperature related modifications of the magnetic signature.

### Sample preparation

Samples can be processed as a composite sample or core. Where samples need to be

dried: air-drying, freeze-drying, or oven drying below 40°C is advised. Temperatures exceeding this threshold may modify the natural magnetic assemblage (Walden, 1999b). Manual grinding is recommended as mechanical grinders pose greater risk of cross contamination and mineral breakdown. Sink samples, particularly those in suspension require freeze-drying, centrifuging or sedimentation methods to extract particulates for analysis.

Particle size separation should also be considered for target samples. Hatfield and Maher (2008) sub-divided suspended sediment samples into four particle size classes (<2 µm, 2-8 µm, 8-31 µm, 31-63 µm) to determine inconsistencies in magnetic imprint. The 8-31 µm and 31-63 µm classes were found to be most effective for source tracing, whereas <2 µm and 2-8 µm reflected post-depositional bacterial magnetite formation.

The majority of magnetic analysis instruments, (such as the MS2 laboratory sensor, pulse magnetiser, and magnetometer) accept samples in 10 cc sample pots. It should be ensured that the sample completely fills the pot to prevent movement of particles during analysis, which may produce spurious results.

Where sample quantity is limited, the sample can be immobilised in cling film and packed using cotton wool. Some instruments cannot accommodate sample lids and tape can be used to seal the pot. For core based samples, specific equipment attachments can be used for magnetic susceptibility measurements or non-metallic blades used to split cores into sub-sections.

Where mass specific measurements are intended, each sample must be weighed and recorded. Once the sample pot is filled and clearly labelled, it is helpful to draw an axis direction on each pot. This will aid Advanced Remanence Measurements (ARM) and Iso-thermal Remanence (IRM) measurements which are made on, or reversed to, a single axis.

## Measurement

Mineral magnetic measurements must be approached systematically with increasing field strength (Figure 2). Units are generally reported as volume specific magnetisation, however conversion to mass specific measurements are beneficial for interpretation of results and comparison with the wider literature.

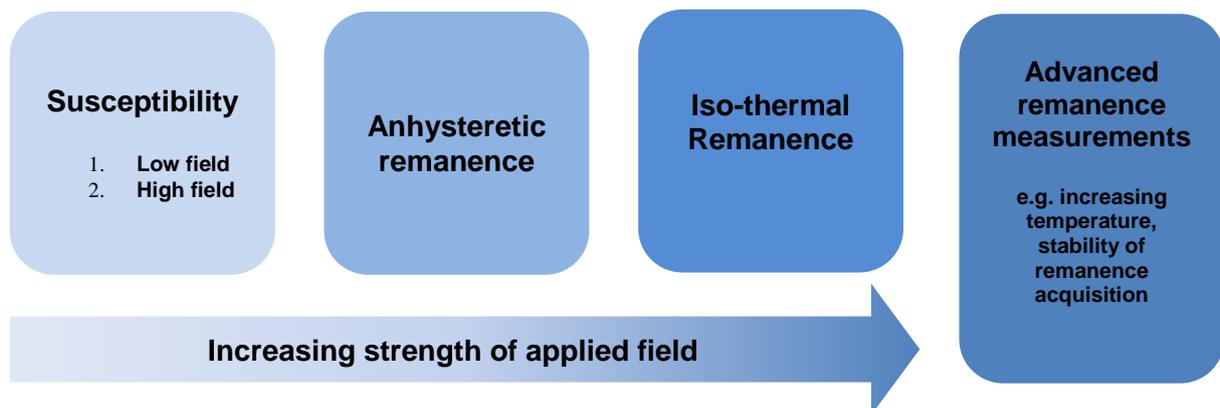


Figure 2: Schematic diagram showing order of magnetic measurements and relative field strength

### Magnetic susceptibility

Magnetic susceptibility meters (e.g. MS2/MS3 Bartington Instruments Ltd shown in Figure 3) are used to collect lab- and field- based susceptibility data. Magnetisation is reported as volume specific (dimensionless  $\kappa$ ), mass specific susceptibility,  $\chi$ , in SI units of

$10^{-6} \text{ m}^3 \text{ kg}^{-1}$  are converted, shown by Equation 1:

$$\text{Mass specific susceptibility} = \frac{\kappa \times 10}{\text{Sample mass}} \quad (\text{Eq. 1})$$

Where the sample mass is in grams. The laboratory based sensor features both low- ( $\chi_{\text{LF}}=0.465 \text{ kHz}$ ) and high- ( $\chi_{\text{HF}}=4.65 \text{ kHz}$ )

susceptibility frequencies and two sensitivity categories. The frequency dependent susceptibility ( $\% \chi_{FD}$ ) is a ratio between  $\chi_{HF}$  and  $\chi_{LF}$  measurements that reflects the super-paramagnetic grain size component (Blundell *et al.*, 2009) and is calculated by Equation 2:

$$\left( \frac{\chi_{LF} - \chi_{HF}}{\chi_{LF}} \right) \times 100 \quad (\text{Eq.2})$$

Magnetic susceptibility equipment must be situated in a magnetically stable environment. Readings are sensitive, therefore, care must be taken to remove metallic or electronic components in the vicinity of the sensor. To an extent, background interference can be accounted for by 'zeroing' the sensor before a sample is measured and taking a subsequent air-measurement to monitor and account for drift (Dearing, 1999).

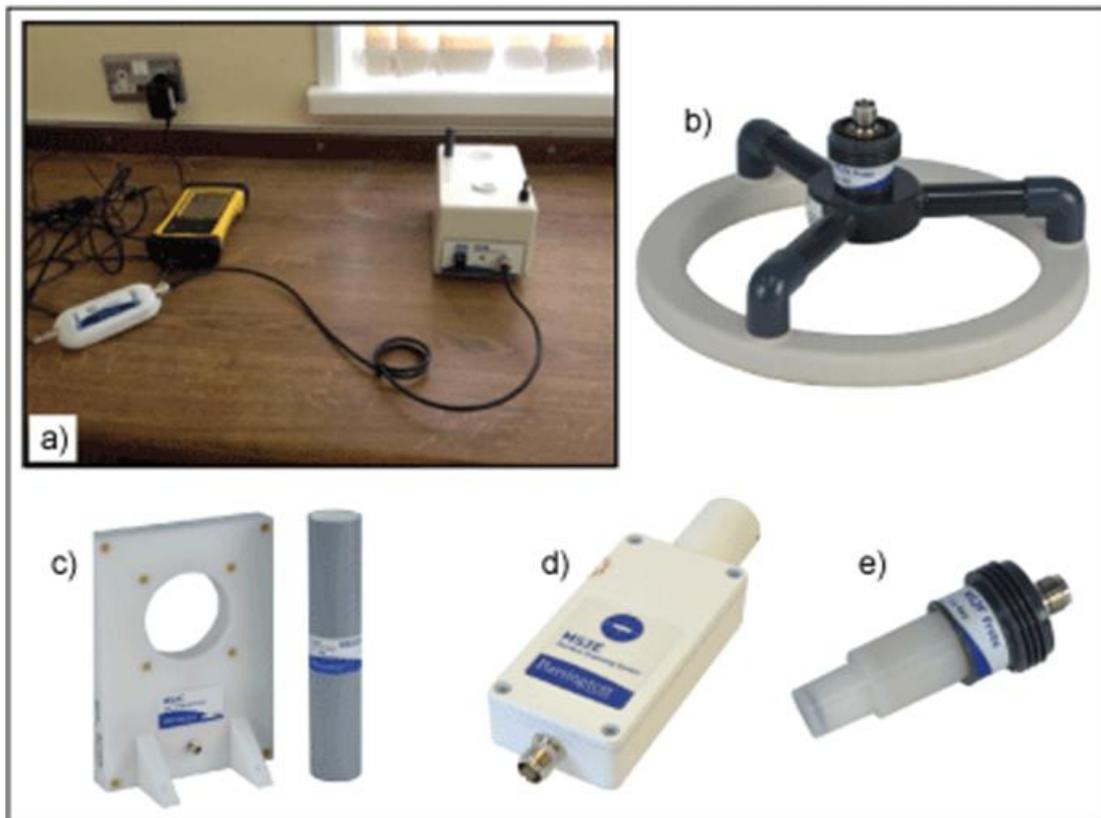


Figure 3: Bartington MS3 magnetic susceptibility meter with attachments: a) laboratory sensor, b) surface sensor, c) core scanning sensor, d) core logging sensor, e) surface point probe (The images for b-e are taken from [www.bartington.com](http://www.bartington.com)).

### Remanence acquisition

Isothermal remanence measurements are made using two instruments: (1) a magnetiser or electromagnet to apply a magnetic field; and, (2) a magnetometer to measure the intensity of magnetisation (Walden, 1999a). The SIRM is commonly defined as 1 Tesla. It is sufficient to distinguish mineralogical differences between soft and hard materials, however, if full saturation of hard minerals is required, a larger field may be required. Values are most commonly reported in  $10^{-3} \text{ Am}^{-1}$ . Conversion to mass specific measurements uses Equation 3 below:

$$\text{Mass specific magnetisation} = \frac{\text{Magnetisation} \times 1.29}{\text{Sample weight}} \quad (\text{Eq. 3})$$

Where sample weight is in grams and the mass specific magnetisation is reported in units of  $10^{-5} \text{ Am}^2 \text{ kg}^{-1}$ .

Measurement of multiple fields is recommended between zero and SIRM, to characterise the remanence acquisition curve. The exact number is dependent upon study requirements, access to facilities and time (Walden, 1999a). These intermediate fields can be performed as forward or backward fields. Forward fields are repeated at increasing strength from low ( $\sim 20 \text{ mT}$ ) through medium ( $\sim 100 \text{ mT}$ ) to high ( $\sim 300 \text{ mT}$ ) denoted as  $\text{IRM}_{20\text{mT}}$ ,  $\text{IRM}_{100\text{mT}}$  and  $\text{IRM}_{300\text{mT}}$  respectively.

Table 3: Summary of common interpretations of mineral magnetic parameters (Summarised from: Blake et al., 2006; Blundell et al., 2009; Booth et al., 2005; Dekkers, 1997; Liu et al., 2012; Maher, 1998; Maher and Taylor, 1988; Walden, 1999a; Walden and Ballantyne, 2002).

Parameter	Description	Examples
<b>Concentration</b>		
Magnetic susceptibility Mass specific $\chi$	Concentration of magnetic material	a,b,c,d,e,f
SIRM Mass specific SIRM	Concentration of remanence holding material (ferromagnetic grains)	a,b,c,d,e,f
<b>Domain state</b>		
Frequency dependent susceptibility $\% \chi_{FD}$	Proportion of super-paramagnetic magnetite	a,b,d,f
Susceptibility of anhysteretic remanence $\chi_{ARM}$	Ultra-fine magnetite particles near to the super- paramagnetic/single domain boundary	a,b,c,f
$\chi_{ARM}/SIRM$	Relative grain size of magnetite, high values suggest superparamagnetic grains whereas low values indicate coarser multi domain minerals	b,e,f
$\chi_{ARM}/\chi_{LF}$	Stable single domain ferromagnetic grains- super paramagnetic and fine viscous grains can affect parameter	b,f
<b>Mineralogical composition</b>		
Forward IRM fields Mass specific (IRM <sub>xmT</sub> )	Abundance of minerals capable of remanence acquisition at the denoted field Low fields (<100 mT) magnetically 'soft' material High fields (>300 mT) magnetically 'hard' material	b,c,f
IRM ratios (%) Forward field/SIRM	Percentage of SIRM acquired at the specified field. Large values at low fields indicate 'soft' minerals. Increases at higher fields indicated 'hard' minerals.	e
Hard IRM (HIRM) SIRM-IRM <sub>300mT</sub>	Mass specific indication of hard magnetic minerals	d
Backfield ratios	Demagnetisation of remanence, magnetically 'soft' minerals demagnetise at low backfield fields, 'hard' minerals demagnetise at higher applied backfields	a,d
S-ratio IRM-100 <sub>mT</sub> /SIRM	Ratio of saturated to non-saturated minerals at - 100mT. Range of values -1 to +1; values close to -1 are dominated by 'soft' minerals, higher values reflect an increasing 'hard' component.	b,d,f
SIRM/ $\chi_{LF}$	Proportion of ferromagnetic material in a sample, lower ratio suggests a greater proportion of diamagnetic and paramagnetic minerals	b

\* Examples of application in source tracing studies: a. Russell et al. (2001); b. Jenkins et al. (2002); c. Hatfield and Maher (2008); d. Zhang et al. (2008b); e. Maher et al. (2009); f. Blake et al. (2006)

Conversely, backfield remanent magnetism can be investigated by initially imparting a forward saturating field to obtain the SIRM, and subsequent applications of fields in the opposite direction, i.e.  $180^\circ$  of the primary axis (Walden, 1999a).

Anhyseretic remanence uses a demagnetiser, with ARM attachment to impart the measurement and a magnetometer to determine the magnetisation intensity (Walden, 1999a). Equation 3 is similarly used to determine the mass specific ARM ( $10^{-5} \text{ Am}^2 \text{ kg}^{-1}$ ) and when normalised by the biasing field strength (A/m), is termed the anhyseretic susceptibility ( $\chi_{ARM}$ ) reported in  $\text{m}^3/\text{kg}$ .

The suite of parameters described can be measured quickly with little sample preparation. The parameters measured, and ratios between them, are used to interpret the characteristics of each sample (Table 3).

### Statistical analysis

Analysis methods vary from bi-plot and multivariate techniques such as cluster, factor and principal components analysis to determine source area grouping (Booth *et al.*, 2005; Hatfield and Maher, 2008). Contributions from source areas are frequently determined using un-mixing models (Collins *et al.*, 1998; Franks and Rowan, 2000). Lees (1999) summarises statistical techniques in source tracing exercises.

### Case study

Mineral magnetic source tracing was carried out in a small ( $10 \text{ km}^2$ ) intensively agricultural fluvial catchment in Ireland. Potential source areas were selected due to their agricultural or non-agricultural significance, consequently seven sources were sampled; field topsoils, field subsoils, on-farm tracks, road verges, channel banks, sub-surface drains outlets, and open field drains. Channel bank, field drain, track, drain outlet and road verge samples were taken using a plastic trowel to collect a surface scrape. Field topsoils used an auger to consistently sample the top 5 cm of soil and a second screw corer to sample at 40 cm for subsoil samples. Target samples were collected at the catchment outlet using time integrated suspended sediment samplers (TISS) (Phillips *et al.*, 2000), which were emptied and re-installed at 6-12 week intervals.

Source samples were returned to the laboratory and dried at  $<40^\circ\text{C}$  in plastic trays. Once dried, samples were sieved to  $125 \mu\text{m}$  to replicate the particle size distribution of TISS samples. TISS samples were refrigerated for a minimum of three days to allow sedimentation of particles. Water was siphoned without disturbing the deposited sediment. The remaining sediment and water mixture were dried at  $<40^\circ\text{C}$  and manually ground using a mortar and pestle once dry.

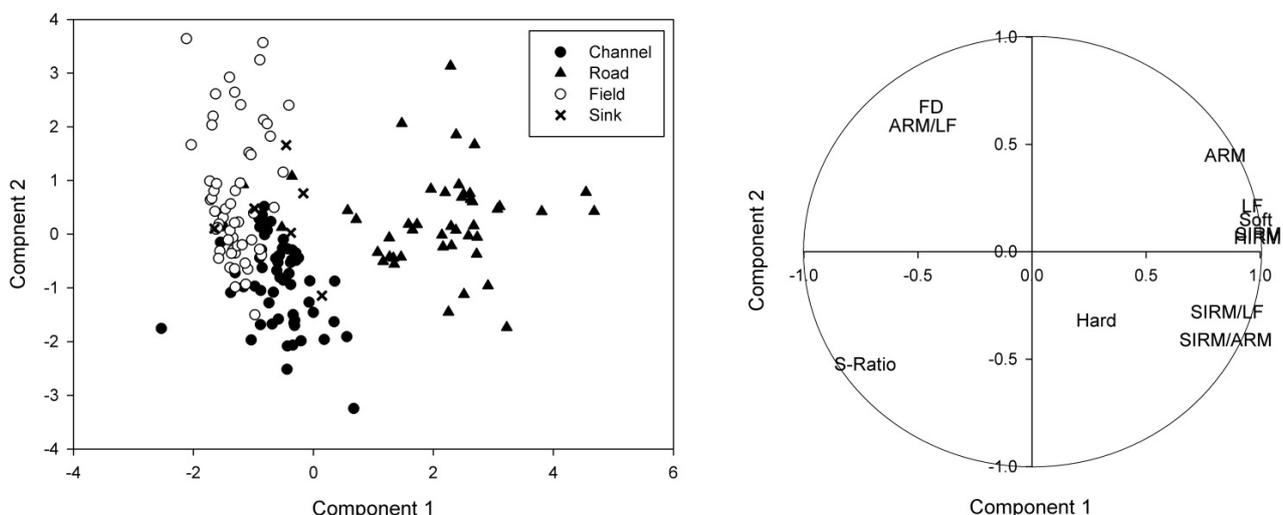


Figure 4: Principal components analysis of soil and sediment samples for source area ascription of the case study: a) score plot, and b) loading plot.

All samples were weighed and packed into plastic 10 cc pots either full of the sample, or where quantities were low, immobilised in cling film and cotton wool. Samples were analysed for  $\chi_{LF}$ ,  $\chi_{HF}$ ,  $\chi_{ARM}$ , SIRM, bIRM<sub>40mT</sub>, bIRM<sub>100mT</sub>, and bIRM<sub>300mT</sub>. The parameters  $\chi_{LF}$ , % $\chi_{FD}$ ,  $\chi_{ARM}$ , SIRM, SIRM/  $\chi_{LF}$ , SIRM/  $\chi_{ARM}$ ,  $\chi_{ARM} / \chi_{LF}$ , bIRM<sub>40mT</sub>, bIRM<sub>300mT</sub>, S-ratio and HIRM were calculated and used for statistical analysis.

Multivariate analysis showed that the seven source areas could not be satisfactorily distinguish and therefore three parent groups were identified; fields (topsoil, subsoil and sub-surface drains), roads (road verges and tracks) and channel (channel banks and open drains). Multiple discriminant analysis showed 96.3% discrimination capability with all eleven tracers (SPSS v18). Principal components analysis performed on JMP 9.0 showed that the three parent source groups are well defined, and the loading diagram showing the arrangement of parameters to produce the score plot (Figure 4a). Target sink values were enclosed within the spatial limits of the source areas, indicating that post-depositional or transportation effects were not apparent, or significant enough to affect sample composition. We can therefore conclude that suspended sediments are predominantly comprised of material from field and channel bank sources.

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