# Chapter 2
# Geochemical Fingerprinting

**Abstract** Use of geochemical fingerprinting methods to determine sediment provenance has progressively increased since the late 1990s, and is now considered by many investigators as the method of choice to quantify sediment source contributions at the catchment scale. Application of geochemical fingerprinting largely rests on four factors: (1) the inability of other techniques (e.g., sediment load monitoring, photogrammetric methods, and mathematical modeling approaches) to effectively determine sediment provenance at the required spatial scales, (2) improvements in analytical methods that allow for the analysis of large numbers of samples for a wide range of elements, (3) the modification of the utilized statistical methods (e.g., inverse/unmixing models) to more effectively account for uncertainty in the modeled results, and (4) the ability to apply the methods to historic sedimentary deposits retrospectively to determine changes in sediment provenance at a site through time. In this chapter, we focus on the application of geochemical fingerprinting to contemporary river sediments as well as alluvial deposits that are less than about 150 years old. Our intent is not simply to summarize the voluminous and growing body of literature on the subject, but to document the strengths, weaknesses, and uncertainty inherent in the approach.

**Keywords** Geochemical fingerprinting · Sediment provenance · Unmixing models · Model uncertainty

## 2.1 Introduction

In order to mitigate the impacts of sediment and sediment-associated contaminants on aquatic ecosystems, one must first determine from where the sediment is derived. Once identified, the predominate sediment sources can be targeted using the often limited financial resources available. While conceptually simple, identifying sediment sources is not as easy as you might think. For example, the use of site specific monitoring of sediment loads to determine the source of sediments to a water body has proven to be a costly, labor intensive, long-term process with a spatial resolution limited by the number of monitoring sites that can be effectively maintained for significant periods of time. An alternative approach is to identify upland areas that are being eroded and then quantify the rate at which sediment is being removed. Such



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methods have been aided in recent years by technological advances in surveying, remote sensing, and photogrammetric techniques that have improved our ability to document temporal and spatial patterns in erosion (Collins and Walling 2004). Collins and Walling (2004) point out, however, that these methods fail to determine the degree to which sediment sources are connected to the river and the inherent uncertainty in routing sediment from the source to the channel. To overcome the problems inherent in the direct measurement of sediment loads or upland erosion rates, distributed modeling routines have been used, but these complex algorithms require the collection and compilation of significant input and validation data, and often have difficulties apportioning riverine sediments to individual sources (Collins and Walling 2004). As a result, investigators have turned in recent years to the use of physical and geochemical tracers, which can be applied relatively rapidly to gain insights into the source of sediment and sediment-associated contaminants within a catchment.

The specifics of the fingerprinting approach vary widely, as do the parameters that have been used as tracers to determine the source of sediments contained within a river or its associated features (e.g., floodplain, reservoir, riparian wetland, etc.) (for a review, see D'Haen et al. 2012). Table 2.1, while far from exhaustive, shows the most commonly utilized parameters with regards to riverine systems. The applicability of these methods varies according to (1) the grain size fractions to which they can be applied (i.e., gravel, sand, or silt and clay-sized material), and (2) the temporal and spatial scale for which they can be used (D'Haen et al. 2012). To date, an overwhelming majority of source ascription studies at the catchment scale have focused on fine-grained sediments ($<\sim63\,\mu m$ in size) eroded from diffuse upland areas in response to either natural or anthropogenic disturbances (e.g., wildfires, deforestation or timber harvests, agricultural practices, and urban/exurban development). The focus on fine sediment, as noted in Chap. 1, reflects both its direct impacts on riverine ecosystems (Wood and Armitage 1997; Armstrong et al. 2003; Syvitski et al. 2005; Bo et al. 2007; Kemp et al. 2011) and its chemically reactive nature, which allows for a wide range of contaminants (e.g., nutrients, agricultural chemicals, and trace metals and metalloids) to be carried from upland areas to the drainage network (Horowitz 1991; Collins et al. 2005; Miller and Orbock Miller 2007). The movement of nutrients from agricultural lands to rivers, reservoirs, and lakes, for example, is often a significant issue in rural areas, and can lead to severe cases of eutrophication (Fig. 2.1). With regards to fine-grained sediments, geochemical tracers, fallout radionuclides (FRNs), and mineral magnetic properties have been most extensively utilized in provenance studies of both historical (50–10,000 ybp) and contemporary (<50 ybp) sediments (D'Haen et al. 2012) (Fig. 2.2).

In this chapter, we focus on a specific methodological approach often referred to as geochemical fingerprinting to determine the provenance of sediments suspended within the water column or contained within alluvial deposits that are less than about 150 years old. The catchment-scale approach involves two primary components: (1) the identification of a set of sediment-associated geochemical parameters (i.e., a fingerprint) that can be used to discriminate between the sediments of variously defined sediment sources, and (2) the estimation of the relative proportion of sediment from each of the individual sources that comprise suspended sediments (or other type

**Table 2.1** Tracer types and representative studies that have utilized them (adapted from D'Haen et al. 2012 and Guzmán et al. 2013)

| Tracer | Representative references |
|---|---|
| *Physical tracers* | |
| Particle color | Grimshaw and Lewin (1980), Giosan et al. (2002), Krein et al. (2003), Croft and Pye (2004), Martínez-Carreras et al. (2010) |
| Grain size distribution | Dudley and Smalldon (1978), Kurashige and Fusejima (1997), Stuut et al. (2002), Weltje and Prins (2003), Weltje and Prins (2007), Weltje (2012) |
| Grain morphology and texture | de Boer and Crosby (1995), de Boer et al. (2000), Cardona et al. (2005), Madhavaraju et al. (2009) |
| Magnetic properties ($\chi_{Lf}$, $\chi_{Hf}$, $\chi_{Fdep}$, ARM, IRM, HIRM)[a] | Yu and Oldfield (1993), Caitcheon (1998), Oldfield et al. (1999), Slattery et al. (2000), Dearing et al. (2001), Jenkins et al. (2002), Morton and Hallsworth (1994), Oldfield (2007), Zhang et al. (2008), Maher et al. (2009), Hatfield and Maher (2009), Armstrong et al. (2010), Guzmán et al. (2010) |
| *Mineralogical tracers* | |
| Mineralogy | Abu-Zeid et al. (2001), Arribas et al. (2000), Pirrie et al. (2004), Pye (2004), Benedetti et al. (2006) |
| Heavy minerals | Basu and Molinaroli (1991), Damiani and Giorgetti (2008), Oszczypko and Salata (2005), Vologina et al. (2007), Hardy et al. (2010) |
| Clay minerals | Eberl (2004), Gingele and De Deckker (2005) |
| Cathodo-luminescence quartz | Gotze et al. (2001), Bernet and Bassett (2005), Gotte and Richter (2006) |
| *Geochemical and biogeochemical tracers* | |
| Major elements | Rollinson (1993), Douglas et al. (2003) |
| Rare earth elements | Mahler et al. (1998), Zhang et al. (2008), Polyakov and Nearing (2004), Polyakov et al. (2009), Kimoto et al. (2006), Lee et al. (2008), Deasy and Quinton (2010), Yang et al. (2008), Wude et al. (2008), Singh (2009), Xu et al. (2009), Collins et al. (2013), Miller et al. (2013) |
| Trace metals metalloids (Cd, Cu, Pb, Zn, As) | Collins et al. (1997a), Collins et al. (1998), Collins et al. (2010a), Collins et al. (2012), Collins et al. (2013), Miller et al. (2005), Hallsworth and Chisholm (2008), Decou et al. (2009), Grimes et al. (2007), Rowan et al. (2012), Massoudieh et al. (2013), Zhang et al. (2012) |
| Elemental ratios (e.g., Cu/Pb; Si/Al; Pb/Al) | Wang et al. (2009), Rowan et al. (2012) |
| Fallout radionuclides ($^{137}$Cs, $^{210}$Pb, $^{7}$Be, $^{239,240}$Pu) | Wallbrink and Murray (1993), Walling and He (1999), Walling et al. (1999), Walling et al. (2009), Wallbrink et al. (2002), Nagle et al. (2007), Mabit et al. (2008), Ritchie and Ritchie (2008), Wilkinson et al. (2009), Evrard et al. (2010), Parsons and Foster (2011), Taylor et al. (2012), Gaspar et al. (2013), Golosov et al. (2013), Walling (2013), Wilkinson et al. (2013) |
| Isotopic ratios ($\delta^{13}$N, $\delta^{13}$C, $\delta^{87}$Sr, $^{204}$Pb/$^{206}$Pb, etc.) | Douglas et al. (1995), Douglas et al. (2003), Gingele and De Deckker (2005), Lee et al. (2008), Yang et al. (2007), Fox and Papanicolaou (2008a), Fox and Papanicolaou (2008b), Alt-Epping et al. (2009), Mukundan et al. (2010) |
| Biogeochemical (N, C, P) | Hasholt (1988), Hillier (2001), Fox and Papanicolaou (2008b), Alt-Epping et al. (2009), Hancock and Revill (2013) |
| Mineral ages (zircon, monazite, muscovite) | Gleason et al. (2007), Kirkland et al. (2009), Veevers and Saeed (2007), Reynolds et al. (2009), Amidon et al. (2005), Morton et al. (2008) |

[a] $\chi$ Magnetic susceptibility (low, high frequency and frequency dependent); *ARM* Anhysteretic remanent magnetization; *IRM* isothermal remanent magnetization; *HIRM* derived remanence parameters

**Fig. 2.1** Eutrophication in a reservoir within the KwaZulu-Natal region of South Africa

of alluvial material) within the river. The latter is accomplished by comparing the geochemical parameters that make up the fingerprint in the source sediments to that of the riverine material. The use of such geochemical fingerprinting techniques has increased dramatically since the late 1990s. In fact, many investigators now consider geochemical fingerprinting the method of choice with respect to diffuse sediment sources. The increased use of geochemical fingerprinting is due, in part, to recent advances in analytical instrumentation that allow for large numbers of elements to be analyzed in a large number of samples in a relatively short period of time. These analytical advances have been accompanied by the enhancement of source ascription methods that provide for a more detailed and quantitative understanding of the uncertainty inherent in the derived results. The intent of our analysis herein is not simply to summarize the voluminous and growing body of literature on the subject, but to document the strengths, weaknesses, and uncertainty inherent in the approach in general, and specific methods in particular.

## 2.2 Conceptual Model and Inherent Assumptions

Upstream portions of the riverine sediment-dispersal system are characterized by a network of channels and their associated hillslopes, both of which serve as zones of sediment production (Figs. 1.2 and 2.3). Hillslope areas can be geographically

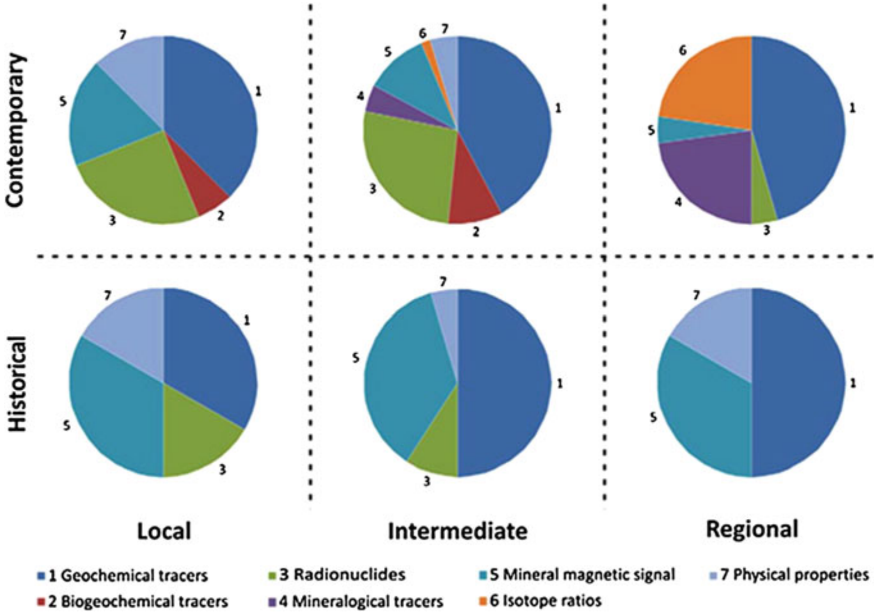


**Fig. 2.2** Relative use of tracer types identified and categorized on the basis of temporal and spatial scale by D'Haen et al. (2012) for determining the provenance of fine-grained sediment in alluvial deposits. Spatial scales defined as local ($<10\,km^2$), intermediate ($10–10{,}000\,km^2$) and Regional ($>10{,}000\,km^2$); temporal scales subdivided into contemporary (<50 ybp), and historical (50–10,000 ypb) (from D'Haen et al. 2012). Examples of tracers associated with each category of tracer provided in Table 2.1

subdivided into units on the basis of the underlying geology, soil type, etc., each unit defining a distinct sediment source. Sediment sources can also be defined according the processes of sediment generation (e.g., whether the sediment was derived from sheet, rill, gully or bank erosion). Source areas defined according to the generating process are often referred to as source types. Particles eroded from these defined source areas or types are transported, often intermittently, through a channel/valley network to a downstream depositional basin that serves as a long-term sediment repository (Weltje 2012). In the process, particles from all of the source areas are combined such that the sediments within the channel represent a mixture of particles from all of the source areas in the basin. The physical and geochemical composition of this sediment mixture (which we will refer to as river sediment) is a function of the composition of the source area sediments and the relative amount of sediment that each source area contributes; if both are known, then it is possible to predict the composition of the mixture. Mathematically, this predictive calculation is considered a linear forward problem (Weltje 2012). More commonly, however, the objective is to determine the relative volume of material supplied to a particular type of river sediment (suspended load, channel bed material, floodplain deposit, etc.) from each sediment source. This type of calculation represents a linear inverse

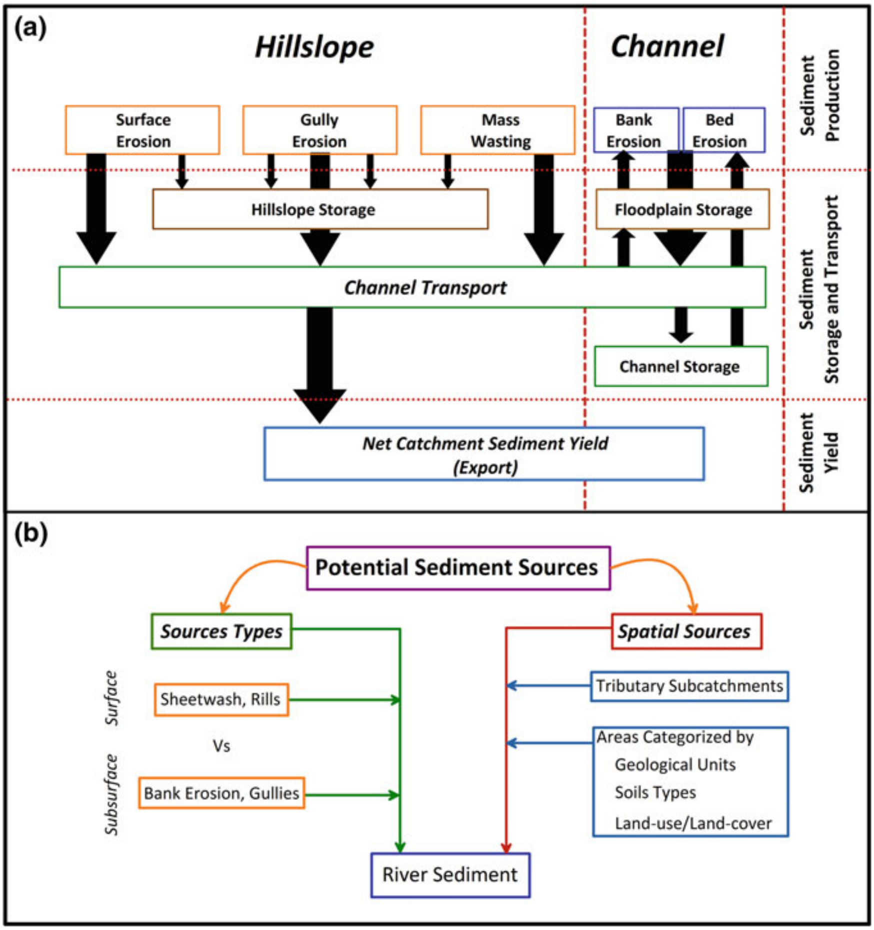


**Fig. 2.3** **a** Flow diagram showing typical sources and pathways of sediment movement within upland areas (modified from USEPA 1999). **b** Classification of the potential sources of riverine sediments commonly defined for geochemical fingerprinting studies

problem when the number of sources and their characteristics are known (Weltje 2012); it is typically solved using a statistically based inverse or unmixing model. In essence, inverse modeling requires that the composition of the source materials be known for a selected set of physical and/or chemical properties, and then defines the mixing proportions from each source that best fits the observed composition of the studied river sediment (Weltje 2012).

The application of inverse modeling to riverine sediments is complicated by the fact that the composition of the alluvial sediment reflects a wide range of physical and geochemical processes in addition to the simple mixing of particles from the source areas (Johnson 1993; Weltje and von Eynatten 2004). Of particular significance are hydraulic sorting processes in which the original population of particles from a source area is modified by the selective entrainment (erosion), transport, and deposition of grains as they are dispersed through the system according to their size, density, and shape (Knighton 1998; Miller and Orbock Miller 2007). Mechanical and chemical weathering processes also lead to modifications in the initial grain population. The net effect of these processes is that sediment of different size, shape, and density within the river is transported downstream at different rates, often producing a downstream fining in particle size. Sediment also may be transported by different methods (e.g., by suspended and bedload process) (Weltje 2012), or be partitioned by the flow into distinct depositional units at a given site (e.g., pools, riffles, point bars, floodplains, etc.) (Miller and Orbock Miller 2007). The river and source area sediments, then, may represent two very different populations of particles. Suspended sediments, for example, may only represent the finest materials within the source areas, and their mineralogy would be expected to differ from that of the bulk material. Moreover, the geochemical properties of the river and source area sediments are likely to differ as fine-grained particles characterized by large surface areas and high surface charge tend to be more reactive and have a greater potential to collect, concentrate, and retain ions (e.g., trace metals).

Modification of the source area sediments during dispersal by physical and chemical processes is important because an assumption inherent in inverse modeling is that the physical and geochemical composition of river sediment differs from a specific source area only because it has been mixed with sediment from another source area(s). Thus, physical and chemical modifications of the source area sediments during erosion, transport and/or deposition must be eliminated, or at least limited, to effectively use inverse modeling. Provenance studies, particularly those aimed at determining the provenance of sediments in stratified rocks, often deal with these modifications using a concept that Weltje (2004) referred to as transport invariance. The concept assumes that particles with similar sizes, shapes, and densities will be entrained, transported and deposited under similar conditions. Thus, by comparing particles from the source areas and the river that fall within a narrowly defined range of sediment size, density or shape, the potential, transport-related modifications can be reduced, and the composition of the river sediment will primarily reflect the relative mixing of sediment from the various source areas. As we will see below, approaches other than the analysis of transport invariant populations have also been proposed to account for the physical and geochemical modification of the source sediments. The point to be made here is that a determination of the provenance of the bulk sediment (consisting of a wide range of particle sizes) may require the combined analysis of multiple size ranges. In fact, it is quite possible that the predominant source(s) of sediment found within the river may vary as a function of particle size (Miller et al. 2013). Sandstone strata, or the soils developed within it, for example, are likely to contribute more sand-sized sediment to a river than a shale and its associated soils. Determining the provenance of multiple size fractions can be time consuming and expensive. Thus, most studies of sediment provenance focus on the particle size fraction that is of importance to the question at hand. For the majority of the geochemical fingerprinting studies, the focus has been on relatively fine-grained sediment ($<63\,\mu m$) as it is this size fraction that forms a significant portion of the suspended load, is largely responsible for decreasing water quality, and is chemically reactive, thereby serving as an important conveyor of hydrophobic contaminants. Sand-sized sediment, however, may also be of importance. Within many of the gravel-bed rivers of the Southern Appalachian Mountains of the southeastern U.S., for instance, aquatic habitats are predominantly affected by the deposition of sand-sized sediments on the channel bed, and their infiltration into the interstitial spaces between gravel sized clasts.

Another fundamental problem inherent in inverse mixing models is the potential for sediments to be eroded from a defined sediment source, transported downvalley and temporarily deposited within the channel (or some other unit) before being 'remobilized'. These reworked sediments are often difficult to recognize (Weltje 2012); thus, it is generally assumed that the source area sediment travels directly from its point of detachment to its point of sampling. The degree to which this assumption is violated depends largely on the size of the basin and the degree of physical connectivity that exists along the drainage network; the chances of determining the ultimate source of sediment, and not its proximal one, decreases with increasing catchment size and decreasing connectivity (Miller et al. 2013).

Inverse modeling, as defined above, is aimed at determining the relative contribution of sediment from defined source areas to a specific type of river sediment. Emphasis is placed on the composition of the river sediment and the origin of the particles contained within it. Some geochemical fingerprinting studies, however, propose a slightly different objective: to assess the relative amount of sediment eroded from the defined source areas or source types. The difference between these two objectives is subtle, but important. When the goal is to determine the amount of sediment eroded from each of the sediment sources, an additional assumption is applied to geochemical fingerprinting. It must be assumed that the sediment leaves all sources at the same time and is transported downstream at an equal rate so that it arrives at the sampling point simultaneously. This assumption is often violated by differences in the proximity of a source to the sampled depositional area, or by differences in the rate at which particles of differing size or shape are transported downstream (the transport variance problem). Take, for example, a 2 cm thick sample collected from the surface of a floodplain that received sediment from two upstream sources. One source is located immediately adjacent to the sampling site, whereas the other is located a considerable distance upstream. Also assume that equal amounts of material are eroded from both sediment sources, and the rate of sediment deposition from both sources is the same. At the onset of the runoff event sediment from the closest source will reach the site first; thus, the lower portions of the 2 cm thick sampling interval will be composed of material from only this source. As the event continues, material from the other source reaches the site, and equal proportions of sediment from both sources will be deposited at the site. If the entire 2 cm of sediment is not composed of a single event, the other events will follow the same pattern until 2 cm of sediment has been accumulated. When the inverse/mixing model is applied to the sample, it will correctly indicate that a larger relative percent of sediment was derived from the closest site over the timeframe represented by the 2 cm increment (this is the objective of the inverse modeling as defined earlier). Thus, sediment provenance with respect to the deposit has been correctly assessed within the errors inherent in the statistical analysis. However, if the intent is determine the relative amount of sediment eroded from the two source areas, the results will be biased such that the model will overestimate the amount of material eroded from the closest source. Differences in particle transport rates produced by varying particle sizes can lead to similarly biased results.

In the case where elemental concentrations are used as geochemical fingerprints, there is also an assumption that the elements exhibit conservative behavior. That is, the elements selected as a fingerprint move with the sediment and are not lost from the system. This follows because inverse/mixing models represent a form of mass balance analysis. Thus, elements that tend to be mobile within aquatic systems and possess lower affinities for particulate matter generally serve as poor fingerprints.

## 2.3 Methodological Approach

While the specific methods used in geochemical fingerprinting varies from one investigator to the next, the general approach involves the completion of five key steps (after Zhang et al. 2012): (1) delineation and characterization of sediment sources within the catchment, (2) determination of the fingerprinting properties that most effectively identify and discriminate between sediments of the defined sources, (3) collection and characterization of river sediment, selected on the basis of the timeframe under consideration, (4) determination of sediment provenance using numerical modeling procedures, and (5) assessment of the uncertainty inherent in the modeling results. These steps are discussed in detail below.

### *2.3.1 Source Delineation*

The first step in any fingerprinting analysis is to define the primary sediment sources within the catchment that may be of interest. Historically, sediment sources have been subdivided into two main categories: upland (hillslope) sediments, and channel bed and banks sediments (Fig. 2.3a). Both types of sediment may be eroded and transported to the water body by one or more geomorphic processes.

For fingerprinting analyses, upland sources are often subdivided further on the basis of the spatial extent and location of mapped geological units (Collins et al. 1997a; Walling et al. 1999; Douglas et al. 2003; Miller et al. 2005), soil types (Miller et al. 2013), land-use/land-cover categories (Collins 1995; Walling and Woodward 1995; Russell et al. 2001; Miller et al. 2013), or contributing tributary areas (Klages and Hsieh 1975; Collins et al. 1997b, 2009, 2010a) (Fig. 2.3b).

This spatially defined source approach is plagued by several problems. First, soil erosion is not only a function of soil type, land-use, or the underlying geology, but varies as a function of factors such as topography and process. Agricultural pastures, for example, may be eroded in steep upland areas by sheet and rill processes and on low-relief floodplains adjacent to the channel by advancing headcuts associated with gullies (Fox and Papanicolaou 2008b). Thus, spatially defined sources fail to directly identify the geomorphic processes responsible for sediment generation. Second, soil types and land-use/land-cover categories are often transitional to one another, confounding their spatial delineation within the catchment as well as the geochemical differences in their sediments (Rowan et al. 2012). Differences in the underlying geological units may also complicate the issue. Third, recent changes from one land-use/land cover type to another may limit the ability of geochemical parameters to distinguish between sediment source areas (Miller et al. 2013). In other words, the geochemistry of the sediment sources may reflect both its current and past land cover history, making it difficult to distinguish between sediments associated with the various land-use/land-cover categories.

In light of the above, an alternative method of defining sediment sources is by the erosional process through which the sediments are generated and delivered to the river. Referred to as the 'source type', a distinction is most often made between sediments generated near or at the ground surface in upland (hillslope) areas by sheet or rill erosion and sediment derived from the 'subsurface' by means of gully or bank erosion (Fig. 2.3) (Walling and Peart 1979; Gellis et al. 2009; Gellis and Walling 2011; Massoudieh et al. 2013). Differentiation between surface and subsurface sediments requires the use of geochemical parameters that differ as a function of depth below the ground surface, such as organic matter or short-lived radionuclides (described in the next chapter).

As neither the spatial or process approach to defining sediment sources is ideal on its own, it is not uncommon for investigators to combined the two methods, thereby defining sediment sources on the basis of both spatial and type categories (Russell et al. 2001; Juracek and Ziegler 2009; Wilkinson et al. 2009), particularly for catchments less than about 200 $km^2$ (Mukundan et al. 2012). Within larger catchments, the heterogeneity of sediment source properties defined by land-use, soil type, or geomorphic process is likely to increase, making it more difficult to distinguish between the sources and hindering source ascription (Collins et al. 1998). In addition, sediment contributions from relatively minor sources, which may still cover large areas, could be underestimated (Mukundan et al. 2012). As a result, the application of geochemical fingerprinting methods to large basins ($>$500 $km^2$) is more difficult, although a number of studies have shown that sediment sources may be effectively defined according to the underlying geological units within the catchment (Walling et al. 1999; Bottrill et al. 2000; Douglas et al. 2003) or by tributary catchment areas (Collins et al. 1996; Walling et al. 1999), both of which tend to exhibit less property heterogeneity than sediments defined according to land-use, soil type, or erosion process (Collins et al. 2012; Mukundan et al. 2012; Wilkinson et al. 2013).

### *2.3.2 Collection and Characterization of River Sediment*

A wide range of river sediments have been targeted for geochemical fingerprinting (Fig. 2.4). The sediments which are selected dictate the timeframe under

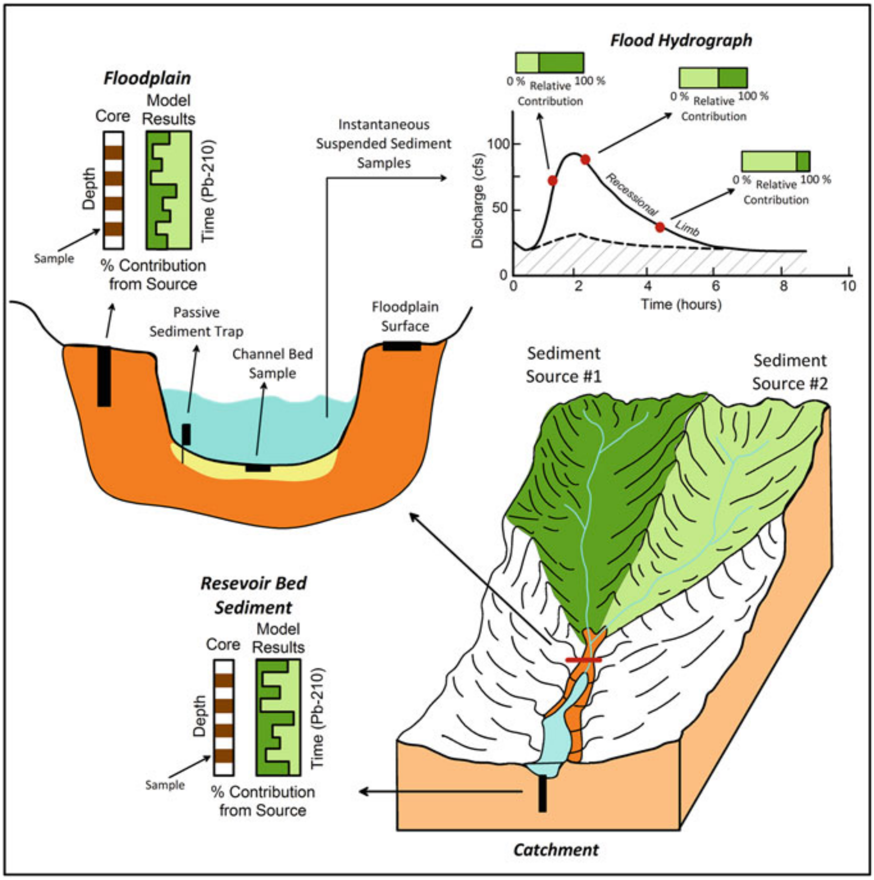


**Fig. 2.4** Illustration of the types of river sediments that are collected for the geochemical fingerprinting of contemporary and historical sediments. Each type of sample is associated with a specific timeframe of analysis, ranging from instantaneous samples collected during a specific part of a flood hydrograph (*red*, *filled circles*) to passive and channel bed sediments (periods of months to a few years, *horizontal rectangles*) to floodplain and reservoir sediments (representing periods of years or decades, *vertical rectangles*)

consideration, and may range from recent, instantaneous suspended sediment samples collected during a specific portion of a flood hydrograph to river sediments deposited within floodplains or riparian wetlands during an entire flood and that may be hundreds or even thousands of years old.

Historically, suspended sediments were sampled to assess the contributions of contemporary sediments to the channel from key sources during flood events (e.g., Collins et al. 1997a, 1998, 2001; Peart and Walling 1986; Walling and Woodward 1992, 1995). There has been a growing realization, however, that the fingerprinting of instantaneously collected samples may not be the most time and cost effective method of determining sediment provenance for an entire flood. At issue is the fact that sediment loads typically vary throughout the event, with larger loads tending to be associated with higher flows. The relationships between sediment load and discharge is not always perfect, however, as larger sediment loads may be associated with the rising, peak, or falling stages of a hydrograph. For example, the episodic erosion of easily eroded sediment during the onset of a runoff event often leads to larger loads during the rising stage of the flood, in comparison to the same discharge conditions during the falling stage, producing a phenomena referred to as the first flush (Miller and Orbock Miller 2007). This 'first flush' phenomenon not only demonstrates that the rates of soil erosion vary through the flood, but that erosion varies from one sediment source to another over the landscape at any one time. Thus, the source contributions determined for an instantaneously collected sample will apply only to the portion of the flood that was sampled, rather than for the entire storm (Collins et al. 2001; Massoudieh et al. 2013). To address this issue it is now common to collect an integrated sample in which sediments are obtained continuously or semi-continuously over a longer time span, such as the entire flood. The sediments within these samples can be expected to reflect the averaged contribution from each sediment source within the watershed (Fox and Papanicolaou 2008b). While such integrated samples may be collected using automated, pump-type sampling devices, the need for relatively large sediment sample sizes for geochemical analysis has led to the use of passive samplers or sediment traps (e.g., Phillips et al. 2000; Russell et al. 2001) that collect materials representing the entire storm hydrograph (Massoudieh et al. 2013).

An alternative to the use of these time-integrated sediment traps is to sample the channel bed material (Evrard et al. 2013; Collins et al. 2013) as recent studies have shown that such bed sediments serve as an effective surrogate of continuously collected material over multiple flood events (Miller and Orbock Miller 2007; Horowitz et al. 2012; Collins et al. 2013). Two additional advantages of sampling the channel bed sediment is that it is not necessary to wait for a flood event to conduct the sampling, nor does one have to sample over an extended period of time (Mukundan et al. 2012). The sediment stored in the channel bed may change, however, over time and at an unknown rate. Thus, bed sediment may need to be sampled on more than a single occasion to assess the relative contributions from key sources over, say, an entire year (Collins et al. 2013).

Some investigators have sampled the surface of floodplain deposits (e.g., Collins et al. 2010a, b, 2012). This particular sampling scheme does not assess the sediment loads during low to moderate flood events contained within the channel banks, but rather is used to assess sediment provenance during events capable of inundating the floodplain. The assumption inherent in this approach is that these overbank events transport a majority of the sediment within the catchment, a conclusion reached by studies dating back to the 1960s (e.g., Wolman and Miller 1960). Thus, the results provide a reasonable assessment of sediment source contributions within the catchment by flows that transport, on average, the most sediment (Collins et al. 2012).

While most early studies were aimed at documenting contemporary sediment sources, Mukundan et al. (2012) point out that the same basic approach has been applied to floodplain, reservoir, wetland, and lake deposits to determine the changes in sediment source to a river through time (Fig. 2.4) (Foster et al. 1998; Owens et al. 1999; Walling et al. 2003a, b; Miller et al. 2005, 2013; Pittam et al. 2009; Collins et al. 2010c). Essentially, it is assumed that the sampled deposits represent an historical record of sediment transport within the basin, where the age of the sediment varies as a function of depth below the ground surface. Thus, fingerprinting can be carried out on samples collected at differing depths to reconstruct the changes in sediment provenance to the depositional site through time. The method is useful in that it allows an understanding of the contemporary sediment sources to be placed into an historical framework. It also illustrates that fingerprinting can be used to retrospectively determine the primary sources of sediment to the channel, something that cannot be done using monitoring data.

### *2.3.3 Identifying Effective Geochemical Fingerprints*

Studies of sediment provenance in the 1980s and 1990s often relied on a single parameter, many of which were based on the physical characteristics of the sediment, such as it grain size distribution, mineralogy, or magnetic properties (Table 2.1). Later investigations, beginning in the late 1990s, showed that erroneous sediment-source area associations were common when only a single fingerprinting parameter was utilized (Collins and Walling 2002). Thus, there was a move to use multiple parameters to fingerprint source area sediments (Collins et al. 1997a, b; Miller et al. 2005; Mukundan et al. 2012; Collins et al. 2010a, 2013; Miller et al. 2013). This composite fingerprinting approach was aided by (1) advances in analytical chemistry that greatly expanded the number and rate for which samples that could be analyzed for a large number of constituents (Walling et al. 2013), and (2) the increased use of multivariate statistical methods to manipulate the composite fingerprinting data, thereby allowing for the quantification of the results. Both factors also increased the use of geochemical parameters as fingerprints, particularly the elemental concentrations of trace metals (Lewin and Wolfenden 1978; Macklin 1985; Knox 1987, 1989; Passmore and Macklin 1994; Miller et al. 2005, 2013), rare earth elements (Morton 1991; Miller et al. 2013), organic substances (Hasholt 1988), fallout radionuclides (Peart and Walling 1986; Walling and Woodward 1992; Wallbrink and Murray 1993), and various radiogenic or stable isotopes (Douglas et al. 1995). In many cases, the utilized geochemical constituents are natural, but in others, investigators have made use of anthropogenic pollutants, such as heavy metals, pesticides and fertilizers (Bravo-Espinoza et al. 2009; Takeda et al. 2004). Takeda et al. (2004), for example, found that while phosphate fertilizers contained 10–200 times more U than soils, they contained lower Th concentrations than the soils. Thus, the U/Th ratio proved to be an effective fingerprinting tool (Evrard et al. 2013).

In general, the type of tracer used for a given study depends on how the sediment sources are defined. For example, if the intent is to determine the relative contributions of sediment on the basis of source type (e.g., sheet, rill, gully, and bank erosion), then it will be important to consider constituents that are elevated in surface materials eroded by sheet and rill erosion and low in subsurface materials eroded by gully and bank erosion (or vice versa). Agricultural pesticides or fertilizers may be useful in separating agriculturally related soils from other types of land-use/land-cover. The number of parameters to select also depends on the defined sediment sources because, in general, inverse/unmixing models require $n$ number of parameters to discriminate $n + 1$ sediment sources (Mukundan et al. 2012). However, it is not uncommon to utilize a fingerprint containing more parameters than source areas or types, as described below.

The most common approach at the present time for determining an effective fingerprint is to analyze the source and river sediments for a wide range of constituents and then select the fingerprinting parameters using a multi-step, empirically based process (Fig. 2.5). The nature of these statistical methods is important as they heavily influence the reliability of the fingerprinting results (Walling et al. 2013). The current trend is to use a three step process that eliminates parameters that do not meet certain assumptions inherent in the use of inverse/unmixing models, while identifying the parameters that most effectively discriminate between sediment from the defined sediment sources or source types.

The initial step in this three-part process is to eliminate geochemical parameters from further consideration that do not behave conservatively. Conservative behavior is often determined using simple range tests that ensure that the range of parameter values measured within the sampled river sediment(s) fall within the observed range of values measured for the sampled sediment sources (Billheimer 2001; Phillips and Gregg 2003; Fox and Papanicolaou 2008a; Collins et al. 2012; Wilkinson et al. 2013). This requirement often eliminates relatively soluble elements (e.g., Na, Cl, and P), and those primarily associated with organic matter.

Conservative behavior also requires that there be no enrichment or depletion in parameter values as a result of physical or chemical processes that modify the source area sediment during their dispersal (e.g., by hydraulic sorting or grain weathering) (Mukundan et al. 2012). In essence, the question is whether the sedimentological characteristics of the sediment (e.g., grain size, shape, density, mineralogy) within the source areas and the river can be directly compared as is assumed. Three different approaches have been used to address the issue. Perhaps the most commonly used approach is to analyze and focus on a narrowly defined grain size fraction. This approach is essentially analogous to the transport invariant approach often used to assess the provenance of sediments within lithified strata as described earlier (Weltje 2012). It must be remembered, however, that the results of such a fingerprinting analysis apply only to that grain size fraction. Determining the source of the bulk sample (or other size fractions) will require additional analyses, increasing analytical costs and effort. In addition, the analyses do not provide for an understanding of the actual concentrations in the bulk sample which may be required for other types of environmental assessments (e.g., a pollutant's potential impact on biota).

An alternative approach is to mathematically manipulate the geochemical data obtained from the bulk sediment sample using information collected from a separate subsample of the analyzed sediment. The most common form of manipulation

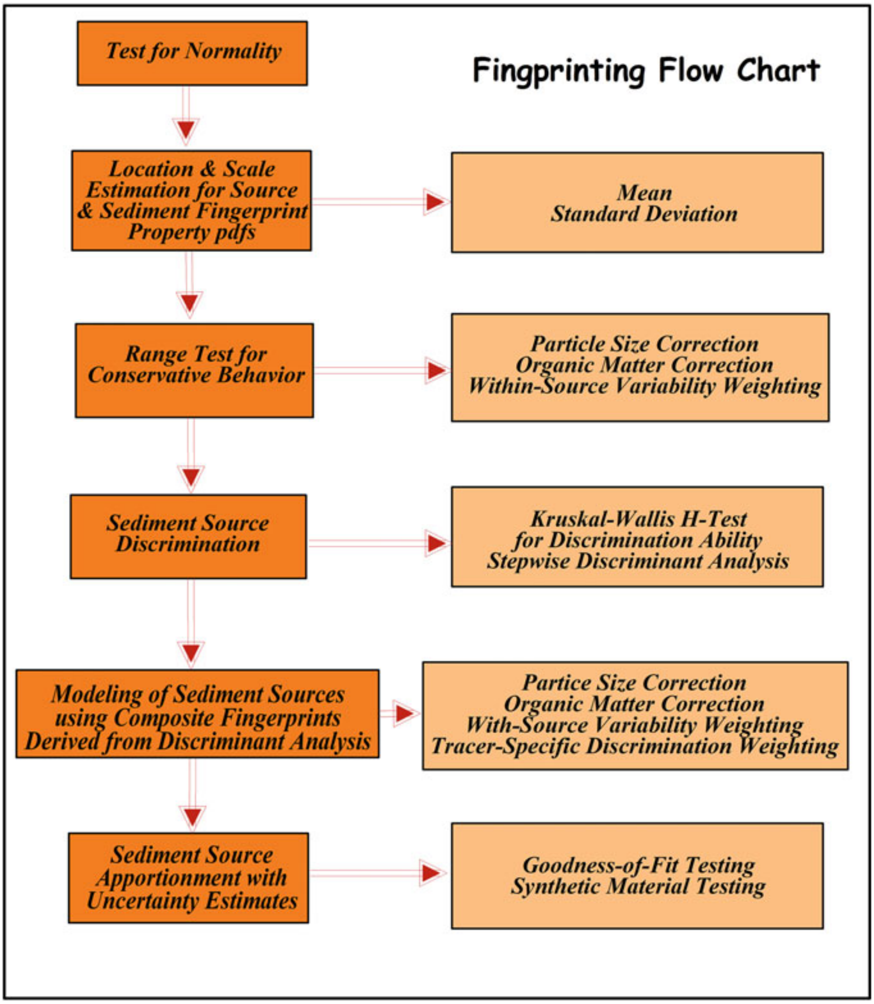


**Fig. 2.5** Summary of inverse/mixing model procedure utilized by Collins et al. (2012, 2013) and others

involves the normalization of bulk concentrations (i.e., the concentration measured on the total sediment sample) to account for differences in sediment geochemistry related to particle size and mineralogy. The typical assumption is that certain constituents (e.g., sand-sized sediment or quartz and feldspar grains) act to dilute the concentration of elements associated with the more reactive materials (e.g., fine-grained particles enriched in clay minerals, Fe and Mn oxides and hydroxides, and organic matter). Thus, normalization removes the diluting effects of the non-reactive constituents. Normalized concentration (*NC*) with respect to grain size, for example, estimates the concentration of the sediment if it was entirely composed of fine-grained, chemically reactive material, and is performed using the following equation:

$$NC = DF \cdot BC \tag{2.1}$$

where *BC* is the bulk concentration and the dilution factor, *DF*, is calculated as:

$$DF = \frac{100}{RS} \tag{2.2}$$

where *RS* is the percentage of reactive sediment of a given size range.

A significant disadvantage of the approach is that the normalized data do not necessarily reflect the actual chemical concentrations within the sampled sediments for the selected size range, particularly when the samples contain <50 % silt and clay (Horowitz 1991).

A slightly different approach is to normalize bulk elemental concentrations by the concentration of a conservative element such as Al, Ti, or Li. In contrast to the methods used for grain size, normalization is performed by dividing the concentration of the potential tracer by the concentration of the conservative element.

The third method commonly used to deal with the transport invariant problem is to incorporate a correction factor into the mixing model (Collins et al. 1998; He and Owens 1995; Russell et al. 2001; Motha et al. 2003, 2004; Juracek and Ziegler 2009). This approach has been widely used to account for the effects of both grain size and organic matter. However, Mukundan et al. (2012) point out that the relationship between a specific fingerprinting parameter and grain size and/or organic matter content may vary between the other parameters used in the composite fingerprint; thus, the incorporation of a single, universally applicable correction factor into the model may not be appropriate. In addition, it has been argued that the use of multiple correction factors, such as one for grain size and one for organic matter, may result in over correction of the parameter values, a problem which is difficult to test (Mukundan et al. 2012).

Once the geochemical parameters that exhibit non-conservative behavior have been removed from the list of potential fingerprints, a statistical test is generally used to identify geochemical properties that are good at discriminating between sediment from various sources. The most commonly used statistical method is the Kruskal-Wallis H-test (Collins et al. 1998, 2001; Walling et al. 1999), but a wide range of other methods have also been applied, including the Mann-Whitney U-test (Carter et al. 2003; Porto et al. 2005), the Wilcoxon rank-sum test (Juracek and Ziegler 2009), and the Tukey test (Motha et al. 2003). A subset of parameters identified during this step is then selected to define the fingerprint that is assumed to represent the optimum combination of parameters for discriminating between the sediment sources or source types (Walling and Woodward 1995; Collins et al. 1998; Mukundan et al. 2012). This last step often relies on the use of a step-wise discriminant function analysis (e.g., Evrard et al. 2013; Miller et al. 2013), although other data reduction techniques (e.g., Principle Component Analysis) have also been used.

## 2.3.4 *Inverse/Unmixing Models*

### 2.3.4.1 Derivation of the Inverse/Unmixing Models

Early work by Yu and Oldfield (1989, 1993) and Collins et al. (1997a, b) was particularly instrumental in defining the general fingerprinting approach most often used today. It can be viewed as a process in which the composite fingerprint created for the sediment sources is compared to the river sediments using an inverse/unmixing model to unravel the relative amount of sediment from each source that comprises the river sediment of interest.

Mathematically, constraints on the mixing model require that (1) each source type contributes some sediment to the mixture, and thus the proportions ($x_j$, $j = 1, 2, \ldots, n$), derived from $n$ individual source areas must be non-negative ($0 \leq x_j$), and (2) the contributions from all source areas must equal unity, i.e.:

$$\sum_{j=1}^{n} x_j = x_1 + x_2 + \cdots + x_n = 1. \tag{2.3}$$

Three significant factors may lead to situations where this latter assumption of linear additivity in property values is not fully achieved. First, analytical errors may be associated with the characterization of the measured geochemical parameters. These errors are typically on the order of ±5 %, and in most instances do not pose a significant issue. Second, an important sediment source may not have been recognized or sampled. The failure to characterize a significant source primarily occurs when dealing with large basins composed of a large number of sediment sources (geological units, soils types, or land-use/land-cover categories). Third, the tracer(s) may have exhibited non-conservative behavior either during transport, or as a result of diagenetic alterations following deposition (Walden et al. 1997; Rowan et al. 2012).

Assuming that a particular tracer has been established as comprising part of a fingerprint for the $n$ sources, the downstream mixture of this particular tracer is represented by

$$\sum_{j=1}^{n} a_j x_j = a_1 x_1 + a_2 x_2 + \cdots + a_n x_n \tag{2.4}$$

where $a_j$ represents the measurement of the tracer within the $j$th source area.

Initial studies used the mean or median of the data points from each source in the fingerprint. Collins et al. (2010a), for example, noted that the "use of the mean concentration value to represent a particular source can be justified as being physically realistic since the sediment collected from the catchment outlet inevitably represents a mixture of material mobilized and delivered from numerous locations upstream. As a result, the collection of representative source material samples from a range of locations throughout the catchment and the use of the sample to derive the mean fingerprint property concentrations can be assumed to be analogous to natural sediment mixing during the sediment mobilization and delivery process."

Since the fingerprint will typically be comprised of $m$ tracers, the mixing model results in an $m \times n$ system of linear equations. Each equation represents the contributions from each of the $n$ sources determined on the basis of the measured amount of a tracer in the sediment:

$$\begin{array}{l} a_{1,1} * x_1 + a_{1,2} * x_2 + \cdots + a_{1,j} * x_j + \cdots + a_{1,n} * x_n = b_1 \\ a_{2,1} * x_1 + a_{2,2} * x_2 + \cdots + a_{2,j} * x_j + \cdots + a_{2,n} * x_n = b_2 \\ \vdots \qquad\qquad \vdots \qquad\qquad \vdots \\ a_{i,1} * x_1 + a_{i,2} * x_2 + \cdots + a_{i,j} * x_j + \cdots + a_{i,n} * x_n = b_i \\ \vdots \qquad\qquad \vdots \qquad\qquad \vdots \\ a_{m,1} * x_1 + a_{m,2} * x_2 + \cdots + a_{m,j} * x_j + \cdots + a_{m,n} * x_n = b_m \end{array} \tag{2.5}$$

In theory, the mixture is exact, but in reality, there will exist some differences (error) between the values of the $m$ measured tracers in the source area, $a_{i,j}$ ($i = 1, 2, \ldots, m, j = 1, 2, \ldots, n$), and the downstream mixture (river sediment), $b_i$ ($i = 1, 2, \ldots, m$). The residual error corresponding to the $i$th tracer can be determined as follows:

$$\varepsilon_i = b_i - \sum_{j=1}^{n} a_{i,j} * x_j \tag{2.6}$$

for $i = 1, 2, \ldots, m$, where $a_{i,j}$ ($i = 1, 2, \ldots, m, j = 1, 2, \ldots, n$) are measurements of the corresponding $i$th tracer within the $j$th source area and $b_i$ is the measurement of the tracer of the $i$th tracer in the river sediment (mixture).

When the number of utilized tracers exceeds the number of source areas or types within the catchment (as is often the case when using geochemical data), the system of equation (2.5) is over-determined, and a 'solution' is typically obtained using a computational method that optimizes an objective function. This function, subject to the previously noted constraints, estimates a best fit solution to the entire data set (Yu and Oldfield 1989).

There are several ways to obtain a best fit, but in previous studies, the objective function, $f$, has taken the form of the sum of the relative errors where

$$f(x_1, \ldots, x_n) = \sum_{i=1}^{m} \left| \frac{\varepsilon_i}{b_i} \right| \tag{2.7}$$

(as defined by Yu and Oldfield 1989) or the sum of the squares of the errors (Collins et al. 1997a),

$$f(x_1, \ldots, x_n) = \sum_{i=1}^{m} \left( \frac{\varepsilon_i}{b_i} \right)^2 = \sum_{i=1}^{m} \left( \frac{b_i - \sum_{j=1}^{n} a_{i,j} x_j}{b_i} \right)^2 \tag{2.8}$$

Note that the measurements of different tracers are often magnitudes apart; for instance, Cu concentrations ranged from 2.78 to 823 ppm, while Cd ranged from

0.200 to 6.30 ppm within sediments of the Mkabela Basin of South Africa studied by Miller et al. (2013). Thus, the error terms for each tracer in equations (2.7) and (2.8) are normalized by dividing by the amount of the tracer found in the sediment mixture. This insures that the error term of any one tracer does not dominate the objective function.

Ultimately, it is necessary to minimize the function $f$, (either 2.7 or 2.8), while satisfying the non-negativity constraints on $x_j$ and the unity constraint (2.3). While the error function (2.7) used by Yu and Oldfield (1989) may seem, at first, more intuitive, the mathematical techniques for solving this constrained minimization problem are more arduous than those using the error function given by Eq. 2.8. Since Eq. 2.8 is a quadratic in $(x_1, x_2, \ldots, x_n)$ on a closed convex subset of $R^n$, the constrained minimization problem is mathematically guaranteed to have a solution. Mathematically, the mixing model would be considered as follows:

$$\begin{aligned} &\textit{Minimize} \;\; f(x_1, \ldots, x_n) = \sum_{i=1}^{m} \left(\frac{\varepsilon_i}{b_i}\right)^2 \\ &\textit{Subject to} \qquad \sum_{j=1}^{n} x_j = 1 \\ &\qquad\qquad\qquad\quad x_j \geq 0 \end{aligned} \tag{2.9}$$

Such problems, known as quadratic programming problems, are well understood. Many mathematical programs, such as MATLAB, *Mathematica*, even Excel, have built in programs to solve these problems. There are other packages that solve the quadratic programming problem as well.

Rowan et al. (2000) solved a different form of objective function based on a variation of the R-value used in regression. They referred to the objective function as an efficiency function, $E$. This efficiency function, $E$, is defined as:

$$E(x_1, \ldots, x_n) = 1 - \frac{\sum_{i=1}^{m} (b_i - \sum_{j=1}^{n} a_{i,j} x_j)^2}{\sum_{i=1}^{m} (b_i - \frac{1}{n} \sum_{j=1}^{n} a_{i,j})^2} \tag{2.10}$$

Instead of being minimized, $E$ was maximized subject to the non-negativity and unity (2.3) constraints.

#### 2.3.4.2 Solving the Optimization Problem

A criticism of using mixing models to determine the relative contribution of sediments from a source is that there may be a number of solutions that are statistically equivalent, particularly where contributions from a given source approach 0 or 100 % (often referred to as the equifinality problem). In other words, similar levels of model performance as measured by an error or efficiency function can be produced by differing sets of source contributions (Collins et al. 2010a; Rowan et al. 2012). In addition, uncertainty in the modeling results may be associated with (1) the inherent variability of the fingerprint within the source materials, (2) the source material sampling density, (3) analytical errors, and (4) changes in sediment characteristics during particle entrainment, transport and deposition which may significantly influence the chemical and physical nature of the sampled deposits, such as grain size and organic matter content (Small et al. 2004).

Without the benefit of formalized numerical optimization techniques, initial attempts at solving the constrained optimization problems (e.g., by Rowan et al. 2000; Jenkins et al. 2002; Phillips and Gregg 2003) involved pushing various combinations of $(x_0, x_1, \ldots, x_n)$ that satisfied the non-negativity function and the unity condition through the objective function to find the optimal solutions. All values of the objective function were then recorded and compared to determine the optimum value and those combinations of proportions which yielded this optimal value.

As an example, to solve the mixing problem using the efficiency function (2.10), Rowan et al. (2000) created all possible combinations of proportions, $(x_1, x_2, \ldots, x_n)$, by generating all $n$-tuples (over 300,000 in all for 5 sources) differing by increments of $\Delta x = 0.02$. They then substituted them into the efficiency function $E$. By plotting $E$ vs $x_i$, Rowan et al. (2000) were able to determine for each source a range of proportions that would generate an efficiency above a certain tolerance.

Jenkins et al. (2002) applied the same method to terrestrial and marine sediments to determine sediment provenance using mineral magnetic properties as a fingerprint. The approach has also been used in disciplines other than geomorphology. Phillips and Gregg (2003), for example, applied the method to determine the structure of food-webs using stable isotopes as a fingerprint of food sources.

While, this technique allows one to generate numerous values of the objective function, it is severely limited by the size of memory necessary to record all possible values of $f$ with $(x_1, x_2, \ldots, x_n)$ for large numbers of sources beyond increments of $\Delta x \leq 0.01$. Later attempts at solving the mixing model recognized the constrained optimization problem as a standard quadratic programming problem and used available packages to solve it, as mention in Sect. 2.3.4.1.

#### 2.3.4.3 Modifications to the Mixing Model

Since the late 1990s, a number of modifications have been made to mixing models to improve upon their overall effectiveness. One of the first modified the objective function in the mixing model (2.8) to account for differences in grain size and organic matter content between the source area sediments and the river sediments (Collins et al. 1997a, 2001). Later, Collins et al. (2010a) made two additional modifications to the objective function. First, they added a ‘within source variability’ weight. They found the use of this weighting parameter gave smaller ranges of possible source contributions when calculated using a Monte Carlo method (discussed below). Second, they introduced a tracer discriminatory weight to ‘reflect the tracer discriminatory power.’ This weighting factor takes into account the relative ability of a specific fingerprinting parameter to differentiate between the various sediment sources.

When the above factors are incorporated into the objective function (2.8), it exhibits the following form:

$$f(x_1, \ldots, x_n) = \sum_{i=1}^{m} \left( \frac{b_i - \sum_{j=1}^{n} a_{i,j} * ps_j * om_j * ws_{i,j} * x_j}{b_i} \right)^2 * W_i \qquad (2.11)$$

where $ps_j$ and $om_j$ are the particle size and organic matter weights, respectively, for the $j$th source; $ws_{i,j}$ is the within source weight for the $i$th tracer within the $j$th source; and $W_i$ is the discriminatory weight for the $i$th tracer. Specific definitions for the correction factors that have been added by Collins and his colleagues to the model are provided Table 2.2.

Another form of modification that has recently received considerable attention is related to Bayesian statistics. Walling and Collins (2005), and Collins et al. (2013), for example, modified the approach to incorporate prior knowledge about river processes into the model. More specifically, they limited the potential contribution from one of the sources within the model by noting, in this case, that the contribution of sediment to the channel by bank erosion could be no more than 50 %. Thus, they imposed an additional constraint on the model. That is, in addition to the unity constraint (2.3), when $x_1$ represents the proportion of the mixture due to channel bank erosion, it must adhere to the following constraints: $0 \leq x_1 \leq 0.5$ and $0 \leq x_j$ for $j = 2, 3, \ldots, n$.

#### 2.3.4.4 Handling Uncertainty of Tracer Data: The Monte Carlo Method

A significant source of uncertainty in the fingerprinting approach is the inherent variability of a fingerprinting parameter within the source and river sediments. Take, for example, a source area defined by its land-cover such as forest vegetation. Forested areas may cover multiple geological units or soil types, each possessing a unique mineralogical and geochemical set of characteristics. Thus, the variability inherent in the collected geochemical data is likely to be relatively high. When combined with

**Table 2.2** Definition of correction/weighting factors used in inverse models (Collins et al. 1997a, 2001, 2010a)

| Correction/weighting factor | Definition |
|---|---|
| Particle size correction factor | Ratio of specific surface area measured for a given source to the average of the surface area from all the sources |
| Organic matter correction factor | Ratio of the organic carbon content within a given source to the average organic carbon content of all the sources |
| Within source variability weight | The inverse of the standard deviation of a parameter for all samples from the source |
| Tracer discriminatory weight | Calculated for each tracer as the ratio of the percentage of source type samples classified correctly for that tracer to the minimum of all such measurements determined during the discriminant analysis used to identify the fingerprint |

the fact that the number of samples collected to characterize the sediment sources are generally limited because of financial and time constraints, the mean value of the fingerprinting parameter calculated from the sample data from any source may not necessarily represent the true mean of each tracer within the source. Thus, using the average of the data points of all samples collected within the source for each tracer elicits an error of unknown magnitude. This error is exacerbated by the fact that the fingerprint is comprised of several tracers (Collins et al. 2010a).

In order to reduce and quantify the uncertainty in mixing/unmixing models related to this inherent variability in the source area data, recent studies have explored the use of a Monte Carlo sampling framework (Small et al. 2004; Collins et al. 2010a, 2012). Validation of these approaches using constructed laboratory mixtures of source materials and synthetic data show the methods hold considerable promise (Small et al. 2004).

As an example, Small et al. (2002, 2004) used the sample data from each source to create a probability distribution to estimate the mean value for each tracer within the source. The estimated means of all tracers within all sources are then used as parameters in the objective function which is minimized to solve for the proportions. This process is repeated numerous times (on order of several thousands) until there are sufficient results to estimate confidence intervals (e.g., 95 %). As described in more detail below, Rowan et al. (2012) used this method to determine the effects land use management practices have on algae blooms.

Collins et al. (2010a) used a similar approach in which a goodness of fit function, which they refer to as a relative mean error (RME), was assumed to measure the robustness of the optimized solutions of the mixing model:

$$RME = 1 - \left[ \frac{1}{m} \sum_{i=1}^{m} \left( \frac{b_i - \sum_{j=1}^{n} a_{i,j} * ps_j * om_j * ws_{i,j} * x_j}{b_i} \right)^2 * W_i \right] \quad (2.12)$$

Subsequent studies by Collins et al. (2012, 2013) modified the approach by using the median values instead of mean values for the tracer parameter within each source. The median values were estimated on the basis of a probability density function. They also use an estimated frequency-weighted average median, initially introduced by Collins et al. (2012), in which $R = \sum_{i=1}^{n} v_i Fr_i$ where $n$ is the number of intervals for the predicted deviate relative contribution, scaled between 0 and 1, and $v_i$ and $Fr_i$ are the mid-value and the frequency for the $i$th interval, respectively.

A question that arises in the use of the fingerprinting approach is how results obtained from using mean source and river sediment values compare to the output generated using the Monte Carlo approach. While additional investigations are needed to answer this question, Fig. 2.6 compares the result generated using only mean values (arrows) to the results obtained using the objective function (2-norm error squared) and confidence interval of 95 % using the RME defined above (2.12) for six samples collected from a wetland core within the Mkabela catchment of South Africa. Grain size and organic matter correction factors were not included in the analysis. While differences exist, the results for these specific samples are

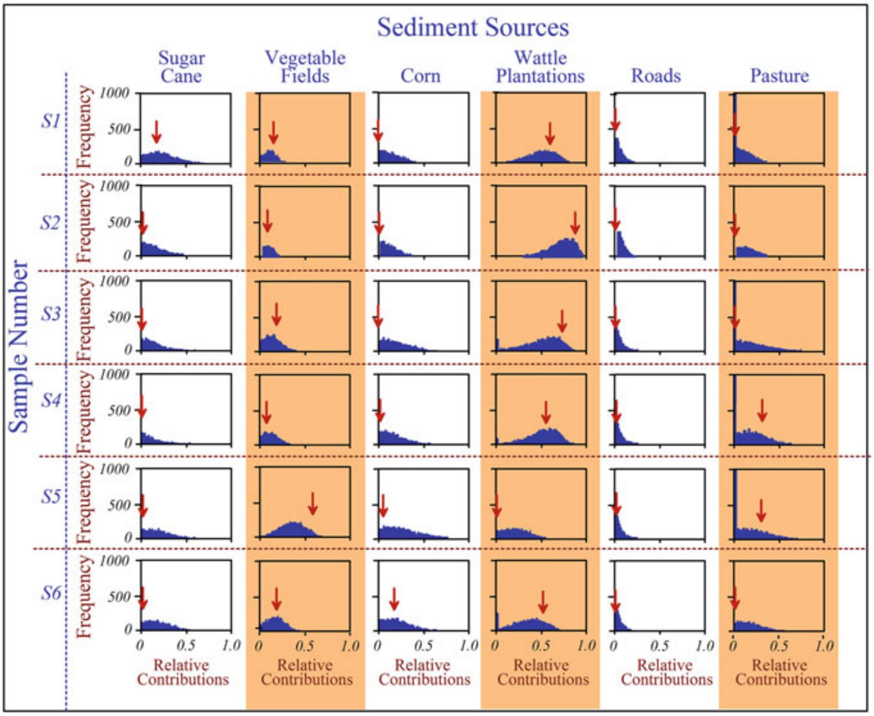


**Fig. 2.6** Comparison of relative contributions of sediment estimated using a Monte Carlo approach (frequency diagrams) versus using only source means for wetland core samples collected within the Mkabela Basin, South Africa. *Red arrows* represent average contribution derived using only source means. In general, results are similar for these data, but the Monte Carlo method allows for an assessment of result uncertainty

generally comparable. The primary difference is, of course, that uncertainty can be accessed using the Monte Carlo method.

## 2.4 Applications

Thus far we have focused on the methods of using geochemical fingerprinting to determine the provenance of river sediments, and the assumptions and uncertainties inherent in the produced results. We will now examine a few individual studies in more detail. The review is not intended to be exhaustive of the wide range of investigations that have utilized geochemical fingerprinting, but rather to provide an overview of some the types of problems that may be addressed using the fingerprinting approach.

A number of early geochemical fingerprinting studies were aimed at determining the source of contemporary sediments in rivers by sampling suspended sediments during flood events (e.g., Peart and Walling 1986; Walling and Woodward 1992, 1995; Walling et al. 1993; Wood 1978; Collins et al. 1997a, b, 1998, 2001). Collins et al. (2001), for example, collected 65 samples from 13 floods during two wet periods between 1997 and 1999 within the upper Kaleya catchment of southern Zambia. The objective was to determine the predominant contributions of sediment from four sources including bush grazed lands, commercially cultivated lands, communally cultivated lands, and channel banks and gullies. As is now common, the collected sediment samples were analyzed for a range of properties that respond to different environmental controls, an approach that is expected to lead to better source area sediment discrimination because the individual tracers will exhibit a higher degree of independence (Walling et al. 1993; Collins et al. 1997b). Collins et al. (2001) chose to analyze sediments for pyrophosphate-dithionite extractable trace metals (Al, Fe, Mn), acid extractable metals and metalloids (As. As, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Sn, Sr, Zn), base cations (C, K, Mg, Na), organic matter (C, N), radionuclides ($^{137}Cs$, $^{210}Pb_{ex}$, $^{226}Ra$) and total P. Source contributions were determined using the unmixing model described by Collins et al. (1997a), which included correction factors for grain size and organic matter as well as a weighting factor to account for differing degrees of analytical precision between the tracers. They found that surface soils in communally cultivated lands were the predominant sediment source. However, there were minor variations in source contributions between the samples collected during an individual flood (Fig. 2.7a). More importantly, because the samples were collected during different discharge and sediment load conditions, attempts to decipher source contributions during an entire flood (or longer time periods) needed to consider the sediment load at the time the suspended sediment samples were collected. By considering load, the contributions associated with samples characterized by higher sediment loads are given more weight than samples characterized by lower sediment loads. Mathematically, a load-weighted mean source contribution ($P_{sw}$) can be calculated for any source(s) for any given time period (Walling et al. 1999) using the following equation:

$$P_{sw} = \sum_{s=1}^{n} P_{sx} \frac{L_x}{L_t} \tag{2.13}$$

where $L_x$ is the instantaneous suspended sediment load at the time of sample collection, $L_t$ is the sum of the instantaneous sediment loads for all samples collected during the time period of interest (e.g., a flood or season), and $P_{sx}$ is the percentage contribution from a specific sediment source, $s$, to the sediment sample, $x$. When applied to an individual flood, the weighted mean source contribution provides information on the source of the sediments transported past the monitored site during the event. In the case of the upper Kaleya catchment, Collins et al. (2001) found that in addition to the noted intra-flood variability in source contributions, inter-flood and seasonal variations in contributions occurred (Fig. 2.7b).

The use of suspended sediment samples to determine the primary sources of sediment to a channel has decreased in recent years as it is plagued by several difficulties, including (1) the fact that estimates of sediment source within suspended

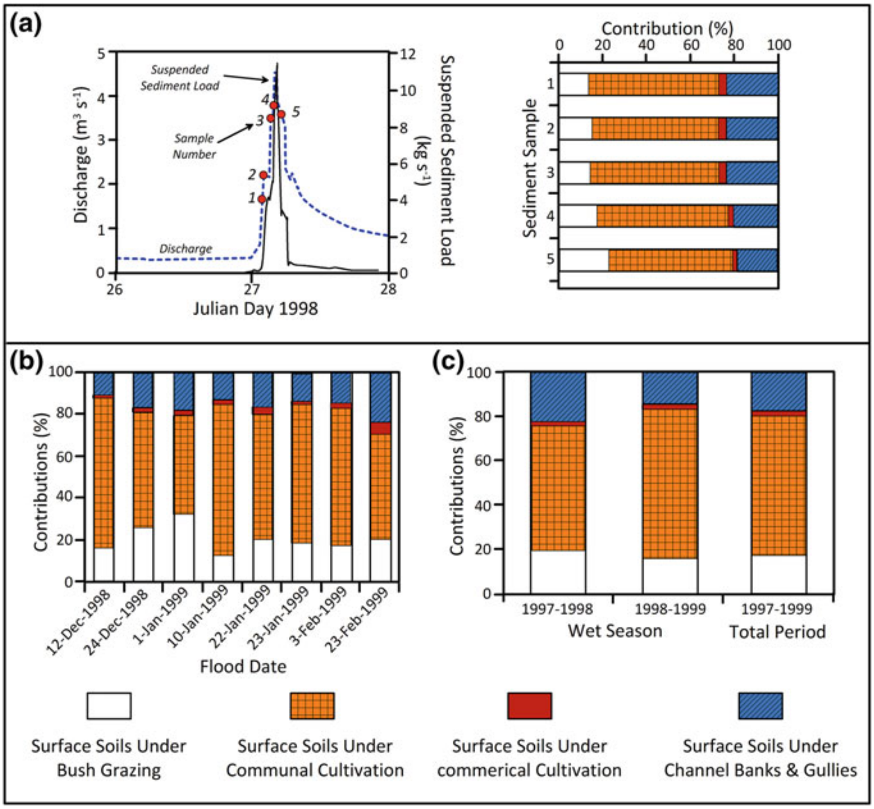


**Fig. 2.7** **a** Estimated relative source contributions to suspended sediment sampled during a flood event in 1998 within the upper Kaleya Catchment. Note intra-flood variability in sediment provenance. **b** Load-weighted sediment source contributions for runoff events sampled in 1998 and 1999 (*left*), and calculated for the entire wet periods between 1997 and 1999 (*right*) within the upper Kaleya Catchment (adapted from Collins et al. 2001)

samples are sensitive to the timing of sample collection, (2) the need to collect samples during flood events; (3) the need to collect and process significant volumes of water to obtain enough sediment for geochemical analysis, and (4) the costs and time required for processing a large number of samples to adequately characterize contributions during a protracted time period (e.g., season or year). More recent studies have focused on sediment collected using passive sediment traps/samplers, channel bed sediments, or sediments located at the surface of floodplains.

Collins et al. (2010a), for example, applied geochemical fingerprinting to river sediments obtained from the floodplain surface near the mouth of seven subcatchments of the Somerset Levels in the UK (Fig. 2.8). These samples were assumed to represent sediments eroded during moderate floods that transported the majority of the sediment in the basins. The study area is part of the England Catchment Sensitive Farming Delivery Initiative where Catchment Sensitive Farming Officers are

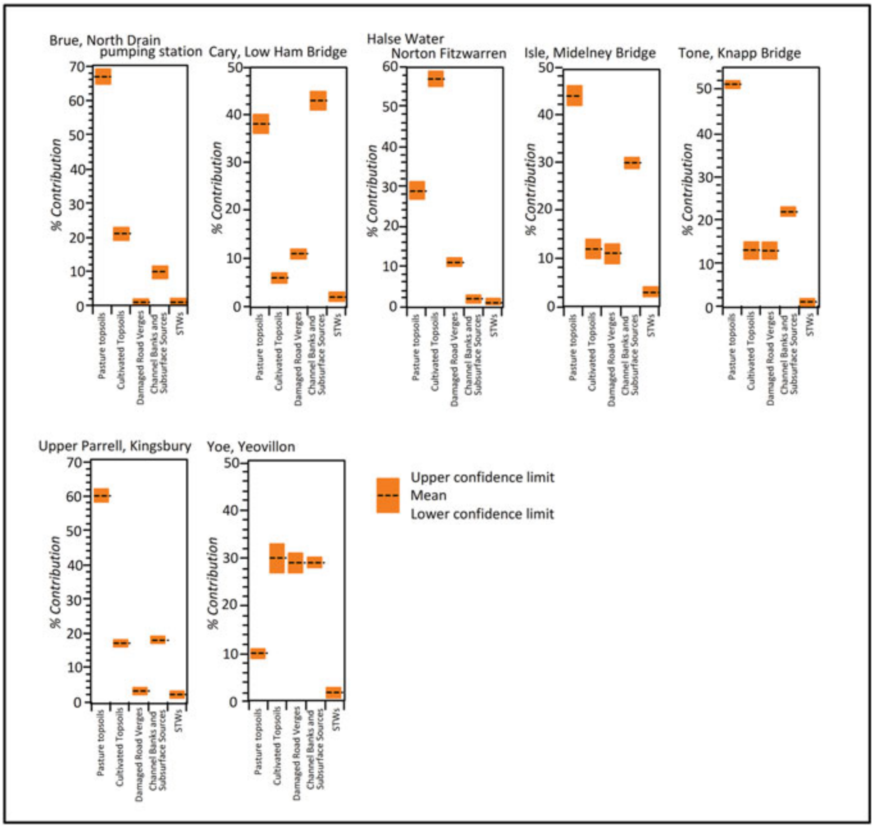


**Fig. 2.8** Proportional contribution of sediment to floodplain surface samples collected at the mouth of seven subcatchments in the Somerset Levels, south west UK. Note variability in source contributions between catchments (from Collins et al. 2010a)

responsible for assessing the potential sources and impacts of pollutants on aquatic resources, and for providing advice to stakeholders who are in need of assistance in protecting aquatic environments (Collins et al. 2010b). The goal of the project was to determine the relative contribution of sediment from five sediment sources: pasture lands, cultivated topsoil, damaged road verges, channel banks and other subsurface sources (e.g., gullies), and sewage treatment wastes (a point source). The fingerprint was developed using the procedures outlined by Collins et al. (1997a) from a suite of 40 geochemical parameters analyzed for each of the source area samples. Inverse modeling relied on a Monte Carlo approach and the modified optimization method descibed above which included correction factors for grain size and organic matter content as well as weightings for tracer discriminatory ability and within-source variability of the individual tracers. The generated results were provided in terms of the mean contribution from the defined sources and their 95 % confidence limits. Figure 2.8 shows that the contribution from each of the sediment sources varied significantly between the seven subcatchments. The observed variations in source area inputs to the river not only reflected the proportion of the basins covered by the sediment source, but also a host of other factors that controlled the erosion of the source sediments. Collins et al. (2010a) argued, for instance, that sediment inputs from bank erosion were probably influenced by such factors as “channel morphology and density, river bank dimensions, and riparian land use pressures”. Regardless of the controls, the data clearly indicated that sediment mitigation strategies will need to be tailored to individual subcatchments, rather than using a one-method fits all approach.

Evrard et al. (2013) utilized geochemical fingerprinting (and an alternative, diffuse reflectance infrared Fourier Transform spectroscopy method) to assess the predominant source of sediments to tropical rivers in central Mexico underlain by different soil types. In this case, the goal was to determine whether the sediments were primarily produced by gully erosion or sheet erosion. As they were attempting to decipher sediments eroded from surface and subsurface sites, they relied on fallout radionuclides ($^{137}Cs$ and $^{210}Pb_{ex}$) and biogenic elements (C, N) as fingerprints. These parameters are known to differ significantly between surface and subsurface sources (as will be discussed in the next chapter). The analyzed river sediments were obtained in 2009 from the channel bed, and were assumed to represent average contributions to the river during the rainy season. Evrard et al. (2013) found that within the Huertitas subcatchment dominated by Acrisols the majority of the sediment (between 88 and 98 %) was derived from gullies. The amount of sediment derived from croplands by sheet erosion decreased during the rainy season, possibly as a result of increased vegetation cover that helped stabilize the soil surface. In contrast, the majority of the sediment (50–85 %) within the Andisol dominated La Cortina catchment was derived from the surface of croplands. Within the Potrerillos catchment, characterized by both Acrisols and Andisols, contributions of sediment from gullies and croplands were highly variable between storms. Gullies, for example, generated between 5 and 86 % of the sediment, while the sheet erosion of rangelands generated between 14 and 95 % of the sediment. However, when combined with other data, it appears that fine-sediment delivered to the Cointzio reservoir was primarily derived from gullies developed in Acrisols, even where Acrisols covered small areas of the basin (<0.5 %). Thus, they suggested that sediment mitigation efforts should focus on stabilizing gully networks.

While the three analyses described above focused on contemporary sediments, other studies have combined their investigation of contemporary sediment sources with an analysis of the changes in sediment provenance through time. These studies typically focus on cores taken of semi-continuously accumulated sediments on floodplains (e.g., overbank deposits) or within reservoirs, lakes, or wetlands that receive sediments from the upstream drainage network. Such fingerprinting studies can be extremely useful from a management perspective in that the sediment cores can be dated, allowing changes in sediment provenance to be linked to (1) an analysis of sediment accumulation rates and the changes in those rates through time, (2) temporal changes in the concentration of various contaminants including trace metals and nutrients within the system, and (3) alterations in land-use/land-cover and other human activities within the catchment. Thus, a link can be made between sediment-contaminant source, sediment influx rates to the river, and anthropogenic activities.

Rowan et al. (2012), for example, applied the geochemical fingerprinting approach to cores extracted from a lake—Llyn Tegid located in the headwaters of the River Dee, Wales—to assess the potential cause(s) of recent cyanobacteria blooms. They found by dating the cores (using $^{210}$Pb and $^{137}$Cs methods) that the sedimentation rates within the lake were generally constant during the first half of the 20th century (Fig. 2.9). However, sediment accumulation rates began to progressively increase

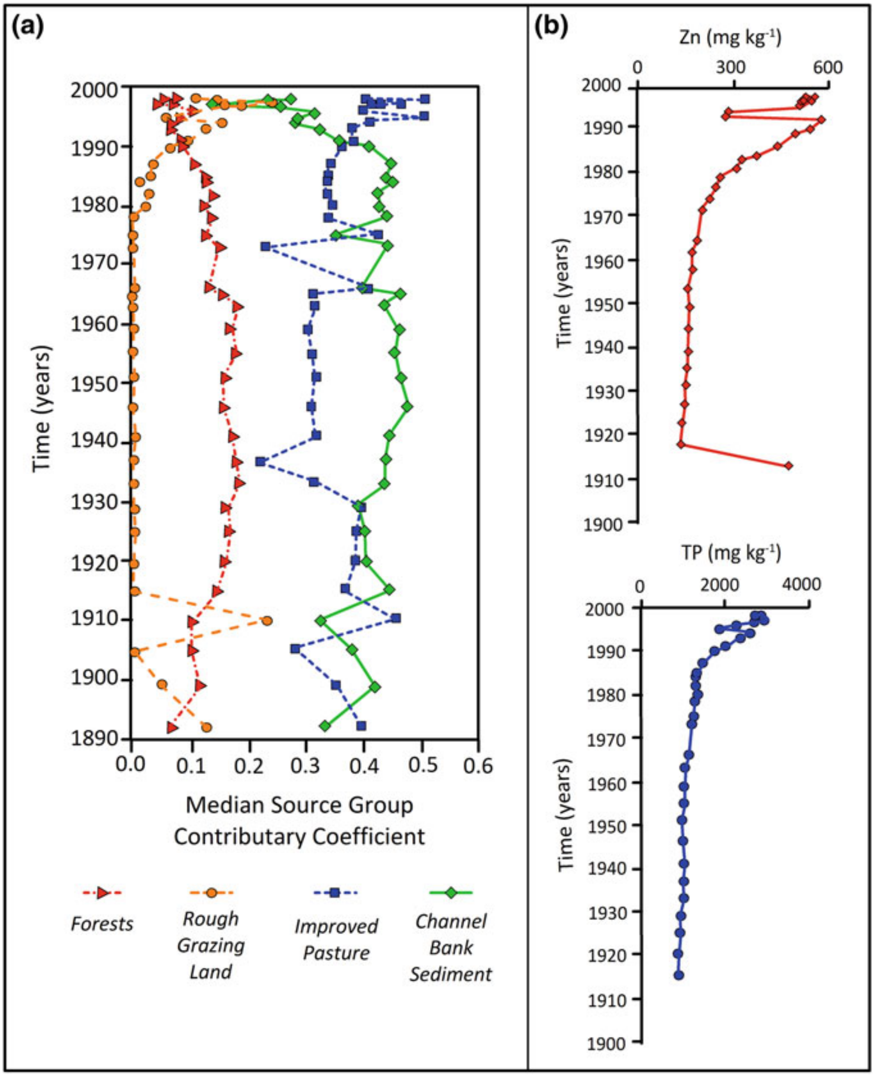


**Fig. 2.9** **a** Variations in sediment source contributions through time as determined from the incremental sampling and analysis of a core from Llyn Tegid, a lake in the Snowdonia National Park, Wales. **b** Downcore trends in total phosphorus and Zn (modified from Rowan et al. 2012)

after about 1950, with the most significant increases occurring in the 1980 s and 1990s. The largest increases in sedimentation in 1995 were correlated with increases in sediment-associated trace metal and nutrient (primarily total P) concentrations and blue-green algae blooms. In addition, Rowan et al. (2012) were able to show using an unmixing model described by Franks and Rowan (2000) that the majority of the sediments were derived from improved grazing lands (i.e., lands subjected to improved management practices) and channel bank erosion throughout the period of record. However, the observed increase in sediment accumulation rates was closely linked to an increased supply of sediment from improved pastures in the late 1980s. Improved grazing lands subsequently became the predominant source throughout the 1990's during which they supplied about 50 % of the total sediment to the lake. Increases in sedimentation rates were also associated with increased sediment yields from rough grazing areas (Fig. 2.9). The accelerated flux of sediment from both the improved and rough grazing areas was consistent with an intensification of agricultural activity in the basin in the 1980s. Rowan et al. (2012) concluded that the algae blooms were strongly influenced by the increased influx of sediment-associated nutrients from grazing lands, particularly improved grazing lands which were actively managed. Following the establishment of algal populations in the lake, simulation modeling of phytoplankton dynamics suggested that even a 50 % reduction in total P input would not eradicate the algae problem because several factors will work to maintain them, including a pool of P in the lake bed sediments, flow augmentation from the Trywryn and impoverished littoral macrophyte assemblages.

By analyzing multiple cores from wetlands and reservoirs located along the axial drainage of the Mkabela Basin within KwaZulu-Natal Midlands of eastern South Africa, Miller et al. (2013) demonstrated that it was possible to decipher both the temporal changes in provenance to the drainage network at any given site and spatial changes in sediment provenance along the valley system at any given time. Their study design also differed from many previous efforts in that source areas were defined and analyzed using two spatial classification systems. One system examined sediment source by soil type, whereas the other defined sediment source according to five land-use categories including pastures, pine forests, sugar cane fields, vegetable plots, and wattle groves. Geochemical fingerprints for the sediment sources were determined for both classification systems using the same set of sample data, but which had been stratified differently. Results from an unmixing model, based on the optimization method described by Rowan et al. (2000), showed that silt- and clay-rich layers found within wetland and reservoir deposits of the upper and upper to mid-portions of the basin were derived from the erosion of fine-grained, valley bottom soils frequently utilized as vegetable fields (Fig. 2.10). These sediments also exhibited elevated concentrations of Cu and Zn, presumably from the use of fertilizers. In contrast, coarser-grained sediments were primarily derived from the erosion of sandier hillslope soils, extensively utilized for sugar cane, during relatively high magnitude runoff events that were capable of transporting sand-sized sediment off the slopes. Thus, the combined data showed that the complex interactions between runoff, soil type, and land use (among other factors) created temporal and spatial variations in sediment provenance. Moreover, downstream contrasts in sediment

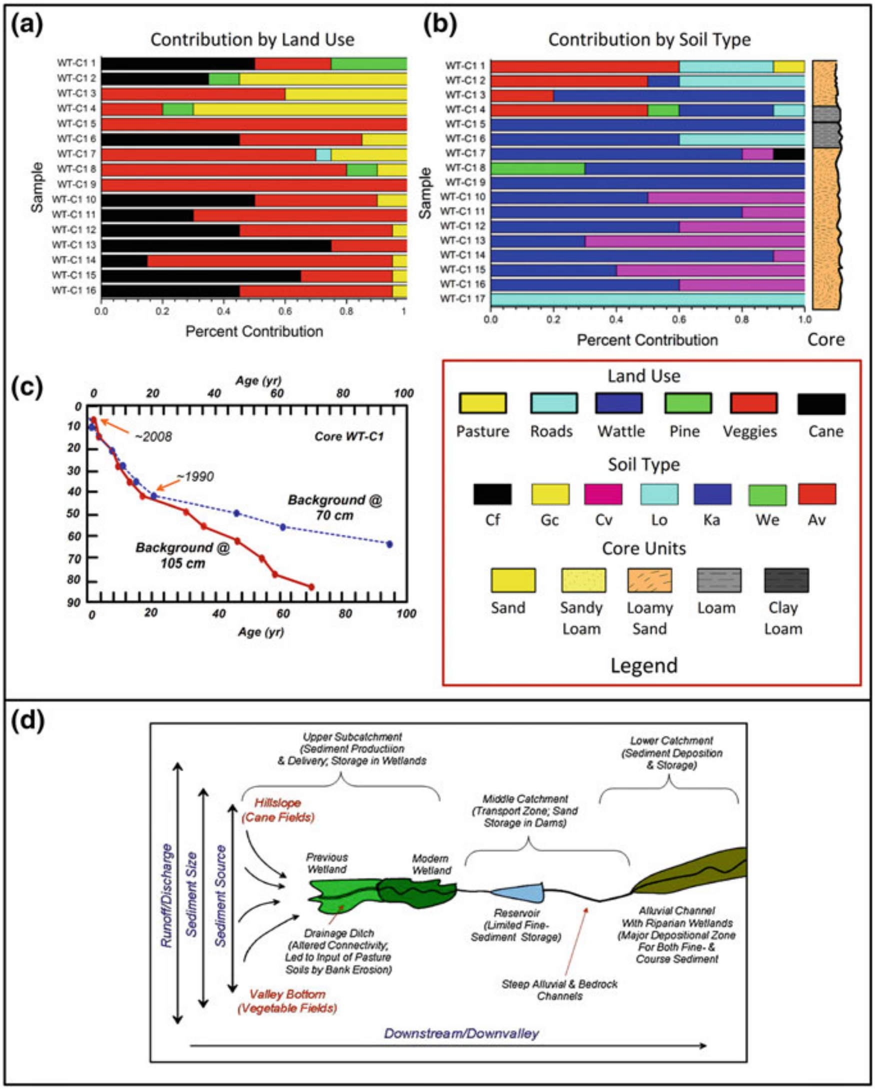


**Fig. 2.10** Estimated contributions of sediment to a wetland from **a** delineated land-use categories and **b** soil types. **c** Estimated age of wetland sediments using two different models. Sedimentation rates increase after 1990 as a results of a constructed drainage ditch that allowed sediment to be transported to the wetland. **d** Schematic diagram of predominant sediment sources and the variations in sediment size and source with runoff magnitude (adapted from Miller et al. 2013)

source contributions, combined with observed changes in Cu and Zn within the cores, suggested that sediment export from upper to lower catchment areas was limited until the early 1990s, in part because the lower parts of the watershed were hydrologically disconnected from the upper catchment wetlands during low- to moderate flood events (Fig. 2.10). The construction of a drainage ditch through an upstream wetland altered the hydrologic connectivity of the catchment, allowing sediment and sediment-associated nutrients to be transported from the headwaters to the lower basin, a process that also increased downstream sedimentation rates as determined by dating a sediment core. From an applied perspective, the results of the study showed that the positive benefits of controlling sediment/nutrient exports from the catchment by means of upland based best management practices were partly negated by modifying the axial drainage system.

The above discussion may suggest that geochemical fingerprinting to assess sediment provenance is a straightforward process that can be utilized by a relatively well-defined methodology. In reality, each catchment exhibits a unique set of characteristics requiring investigators to modify the approach to fit the catchments hydrologic, geomorphic and climatic setting. Nonetheless, the rapidly increasing volume of literature on the topic shows that fingerprinting can provide important insights into the temporal and spatial changes in sediment provenance for specific grain size fractions. Moreover, it is apparent that geochemical fingerprinting can be combined with other forms of geomorphic, hydrologic, and geochemical tracing to more fully address a wide range of sediment related problems within a watershed.

## 2.5 Use of Geochemical Fingerprinting as a Management Tool

There is little debate that sediment source identification is a fundamental requirement for the effective mitigation of diffuse sediment and sediment-born contaminant inflows to rivers and other aquatic environments. However, the use of fingerprinting techniques to decipher the source and dispersal of non-point source contaminants, including sediment, at the catchment scale has yet to be extensively utilized by land-use managers or regulatory agencies. In fact, Mukundan et al. (2012) found that only one state in the U.S. (Minnesota) was using geochemical fingerprinting as part of a defined management strategy, and, to the best of our knowledge, it remains the only state as of 2014. Given the nature of recent legislation in many developed countries, and the need for a sound understanding of the predominant sources of sediment to rivers, it seems likely that geochemical fingerprinting will more extensively be used in the future for management or regulatory purposes. Actually, the potential benefits and difficulties of transforming geochemical fingerprinting from a research to a management tool is currently being explored. Mukundan et al. (2012), for example, examined the use of geochemical fingerprinting for the establishment of total maximum daily loads (TMDLs) in the U.S. TMDLs, which states must define for impaired waters as part of a management strategy, represent the maximum amount of a given pollutant, in this case sediment, that the water body may receive without violating water-quality standards. A key component of the USEPA's organizational framework for establishing TMDLs is contaminant source assessment; thus, it would seem that geochemical fingerprinting could (and perhaps should) be incorporated into the framework for establishing TMDLs. The analysis by

Mukundan et al. (2012) shows, however, that the transformation of geochemical fingerprinting from the research to the management/regulatory realm is not as straight forward as one might think, and has been slowed by several factors.

1. *Many land-use managers and regulatory personal are unfamiliar with the specifics of the approach.*
   In comparison with many traditional approaches, land-use managers/regulators may have a relatively poor understanding of geochemical fingerprinting techniques, and therefore do not necessarily understand the benefits of incorporating the methods into their management framework. As Mukundan et al. (2012) point out, they are likely to question the practicality of applying the approach, especially with regards to the cost and time required for the analysis, the spatial scale to which it can be applied, the type of geochemical analyses that must be carried out, and the likelihood of obtaining meaningful results. Moreover, the benefits of applying geochemical fingerprinting methods as part of a larger management strategy may be unclear. Sediment budgeting, for example, is a relatively well known method of conceptualizing the sediment dispersal system. However, defining the terms within a sediment budget by monitoring sediment loads, quantifying upland erosion rates, or through the use of empirically or physically based models have proven problematic. In contrast, fingerprinting has been shown to be an effective approach to estimate the relative contributions of sediment from defined sources at the catchment scale, and can be applied within a relatively short time frame. It allows, then, for the targeting of the primary source areas or types to reduce sediments loads within the catchment. In addition, mixing model results may be combined with upland erosion and downstream sediment load data to determine: (1) the fraction of the sediment load generated from each sediment source that exists within the basin, and (2) the fraction of sediment eroded from upland areas that is deposited and stored within channels and floodplains (Mukundan et al. 2012). Similarly, geochemical fingerprinting can be linked to the results obtained from watershed models to determine the export and storage of sediment from individual sources. Thus, fingerprinting can be incorporated into the traditional budgeting or modeling approach to refine and greatly improve upon its overall results, a fact that is not always recognized. It is also important to note that the conversion of relative source contributions to estimates of mass sediment transport may allow for a comparison with the more traditional methods of measuring sediment inputs to rivers from the defined sources. For example, rates of bank erosion determined through repeated channel surveys may be compared to estimates of bank erosion influx made by fingerprinting techniques. In doing so, fingerprinting serves as a way to assess the uncertainty inherent in the outcomes of the traditional methods (Mukundan et al. 2012).
2. *Currently, a well-defined set of procedures are lacking.*
   Most analyses performed within a regulatory framework are governed by a well-defined set of operating procedures that are accepted by the 'scientific community' and that lead to accurate and reproducible results. The intent is to produce results that can withstand the rigors of both scientific and legal review. At the

present time, standardized procedures that can be used on a routine basis for geochemical fingerprinting are generally lacking (Mukundan et al. 2012). Particular aspects of the approach that require some form of guidance and/or standardization are many, including the number of samples required for source area characterization and the methods used to collect them, the type of river sediments that should be sampled and analyzed to assess sediment source contributions for a given timeframe, the approach(es) that should be used to alleviate the problems of hydraulic sorting and other processes that modify the sediment as it is dispersed through the system, the quantitative approach that should be followed to define the most discriminating fingerprints, the nature of the mixing models to be utilized, and the methods through which uncertainty in the modeling results can be characterized and assessed, to mentioned just a few. While it can be argued that such procedures should be developed by the agency(ies) that intend on using the approach, the transformation of fingerprinting methods from research to management tool will require significant input from the scientific community.

3. *Geochemical fingerprinting of diffuse sources at the catchment scale has yet to be completely accepted by the scientific community*.
   The results from geochemical fingerprint studies conducted on diffuse pollution sources continues to be met with skepticism by some highly-respected geomorphologists and other environmental scientists. A primary issue for some is the inability of the conventional mixing models to quantitatively assess the uncertainty of the generated results. Recent refinements in the models to reduce, assess, and quantify the uncertainty in their results is likely to alleviate much of this concern in the future. For other scientists, the concern rests on the assumptions that form the foundation for inverse modeling. For example, in many instances geochemical fingerprinting documents the ultimate source of the sediment, but not necessarily the most recent source as the sediment may have been eroded and transported intermittently along the drainage network before being sampled. Additional studies will need to be performed to assess the degree to which the assumptions inherent in fingerprinting are met.

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