Recent advances in dating and source tracing of fluvial deposits

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Abstract Establishing well-resolved chronologies is essential for any kind of environmental reconstruction. Linking responses of sedimentary systems to climate change and human impacts requires independent age information: (i) to establish response types; (ii) to identify response lags; (iii) to quantify rates of change; and (iv) to estimate magnitudes of sediment flux within the systems. Here, current technological advances in dating approaches are reviewed and their applicability for a better understanding of fluvial systems are discussed. Rapid technological development has led to recent advances in many chronometric fields. These range from fundamental innovations that allow completely new applications to advances that improve the performance of existing techniques. Also discussed is the breakthrough achieved for constructing age-models from chronometric data based on statistical techniques and taking into account data precision and stratigraphic information.

Key words U-series; cosmogenic nuclides; TCN; radiocarbon; luminescence; ESR; racemisation; dendrochronology; lichonometry; age-model

INTRODUCTION

To unravel sediment dynamics in changing environments, accurate and precise age information is essential. Linking cause and consequence in catchment systems is often difficult to achieve as different catchment processes can result in rather similar river response and causal links are often hard to establish unambiguously. Recent advances in dating techniques can offer some help to establish the coincidence of processes, to enable correlation of river response to changes in climate or human behaviour, and for hypotheses testing. Age information is also essential to determine pre-human-impact sediment fluxes that are needed as baseline information for establishing meaningful environmental management targets.

Tracing techniques allow determining of spatial links between sediment source and sink. In this volume several examples of advances in source-tracing techniques are presented. Here, the focus will be on recent advances in dating techniques and their possible use for establishing temporal sediment dynamics. The motivation is to create the awareness and provide a guide to in-depth information of how state-of-the-art chronologies for fluvial sediments can be constructed. The focus will be on techniques that, due to recent progress, are now for the first time available for dating fluvial deposits on timescales of 10^2–10^4 years.

A recent review on dating of fluvial sediments is provided by Stokes & Walling (2003). More general information on the different dating techniques can be found in Aitken, (1990), Wagner (1998), Lang *et al*. (1999) and Lowe & Walker (2005).

The main recent advances in chronometry are based on innovations in analytical techniques like Accelerator Mass Spectrometry (AMS), Thermally Ionising Mass Spectrometry (TIMS) and laser fusion, leading to significant improvements in the performance of established dating methods and, in addition, enabling new types of dating applications. In general, the technical innovations allow for both higher precision and higher accuracy, the latter often due to a reduction of sample size which, in turn, reduces the chance of contamination. Significant achievements have also been obtained in extracting age information from dating results. Baysian techniques have proved essential for establishing age-models for sequences of radiocarbon ages. Recent advances allow the combination of different types of dating results and establishing age models for discontinuous sedimentary sequences.

Dating techniques can be grouped into radiometric, dosimetric, biological, chemical, event and archaeological techniques. The focus here is on those technical advances that are relevant to dating fluvial deposits like radiometric and dosimetric techniques, as well as specific advances obtained within the fields of biological and chemical approaches. The advances achieved in the archaeological and event dating techniques are mainly of regional importance, but may offer possibilities for specific case studies.

RADIOMETRIC TECHNIQUES

The dating techniques that extract time information from radioactive decay are called radiometric dating techniques. The advantage of these is that the half-life of a specific decay is independent of environmental conditions (temperature, pressure, etc.) and the ionizing radiation (α-, β- and γ-rays) can be detected relatively easily and precisely using counting and spectrometric techniques. The time frame for which a radiometric technique can be applied depends on the half-life of the radioisotope considered and the detection limit of the technique used. In the past two decades, massive technical changes in detection systems have occurred. Most importantly different types of mass spectrometry have been developed and applied for chronometric studies. Accelerator Mass Spectrometry (AMS) is the most prominent example: it uses a particle accelerator combined with a high resolution detector system capable of counting single atoms (Elmore & Phillips, 1987). AMS resolution is $1/10^{15}$, which means that one ^{14}C-atom can be detected among 10^{15} ^{12}C-atoms. The advantage of AMS over β-counting for determining the concentration of ^{14}C is that it does not rely on radioactive decay, which is an inefficient means for measuring long-lived nuclides. For example, in ^{14}C-dating only one in every million ^{14}C-atoms in a sample is detected after three to four days of β-counting. With AMS the sample size is reduced by a factor of 1000 (1 mg instead of 1 g), the detection time is reduced from days to minutes, and the detection limit is enhanced by an order of magnitude.

Radiometric dating techniques are subdivided according to the origin of the radioactive nuclides from: (a) primordial sources; (b) cosmogenic production; and (c) anthropogenic release.

(a) Primordial source

Having formed prior to the aggregation of our solar system, long-lived radio nuclides like ^{238}U, ^{235}Th, and ^{40}K are found commonly in the Earth's crust. Most important in terms of dating Late Quaternary deposits are isotopes within the decay chain of uranium and ^{40}K, the radioactive isotope of potassium.

U-series dating: most radionuclide techniques based on the radioactive decay of U exploit the different physical and chemical behaviour of parent and daughter nuclides. Whereas the parent nuclide is bonded within a mineral's crystal lattice, the decay products are not, and may escape. Some of the decay products are soluble in water, some are gaseous and others are insoluble and attach to clay particles. These different characteristics lead to fractionation processes during weathering, erosion, transport and deposition of minerals. For the Late Quaternary, U-series techniques are especially useful for dating carbonate precipitation in alluvial sediments by pedogenic, biogenic or pure chemical processes (as in molluscs, bones, speleothems, calcrete, travertine, and even sinter crusts; for a comprehensive overview see Bourdon *et al.*, 2003). If the carbonate precipitates act as closed system, over time a radioactive equilibrium will become established. The extent to which equilibrium has been re-established is a measure of the time elapsed since the end of fractionation.

The classic techniques of U-series dating rely on α- and γ-spectrometry to determine the activity of members of the U-decay chains. More recently, Thermally Ionised Mass Spectrometry (TIMS) has become available that allows measuring the concentrations of these isotopes directly. The significance of TIMS for U-series dating is comparable to that of AMS for cosmogenic nuclide dating. TIMS allows dating of sub-gram-sized carbonate samples with very small analytical errors (Edwards *et al.*, 1986, 1987). With TIMS, highly processed sample fractions are ionised by heating. The ionised nuclei are then accelerated, separated and detected with the aid of a particle mass spectrometer. The smaller sample size allows selective sampling of the most homogenous parts of a limestone, thereby reducing the risk of detrital contamination, and better fulfilling the closed system condition for radiometric dating. TIMS results in lower systematic and statistical errors (precision <1%) and has made a range of suitable U-daughters accessible for dating. More recently, MC ICPMS (Multi Collector Inductively Coupled Plasma Mass Spectrometry) has also been used, having a precision at a comparable level to TIMS (Goldstein & Stirling, 2003). Based on the mass spectrometry techniques the age-range accessible with U-series dating stretches from less than 1 ka to more than 500 ka. Recent applications include the dating of pedogenic and groundwater calcretes (Candy *et al*., 2005), freshwater tufas (Howard *et al*., 2000), and travertine (Anders *et al*., 2005; Pederson *et al*., 2006).

During the past few years U-series dating has benefited from another innovation: laser-ablation MC ICPMS (Eggins *et al*., 2005). This has the big advantage that sample preparation (that is very laborious for most of the other mass spectrometry techniques mentioned) is greatly reduced. It has the disadvantage that it can not yet offer similar precision to the other techniques and is presently only of limited use for the Holocene.

For much shorter timescales the end members of the ^{238}U decay ^{210}Pb/^{206}Pb can be used. An intermediate daughter in the decay series is the radioactive gas radon (^{222}Rn). It escapes into the atmosphere and decays to ^{210}Pb, which is quickly washed out of the atmosphere by precipitation. In fluvial systems, lead is attached to and transported with fine grained sediments. ^{210}Pb decays into the stable ^{206}Pb with a half-life of 22.3 years. The ratio of ^{210}Pb/^{206}Pb can thus be used to determine the time of deposition in fine grained sediments up to about 150 years. This technique has been especially useful for low-energy depositional environments with quasi continuous sediment supply (lake or deep sea; Appleby *et al*., 1979). Applications to date fluvial sediments have proven more difficult due to more complex sedimentation histories. Limitations result from significant spatial and temporal variations of ^{210}Pb fluxes to the flood plain. In a sequence of overbank deposits ^{210}Pb originates from three sources: *in situ* production, direct atmospheric fallout and flood-derived inputs from the catchment. He & Walling (1996) and Walling *et al*. (2003) unravelled the age-related signal by combining the ^{210}Pb inventory with other natural and anthropogenic tracers (e. g. heavy metals, ^{137}Cs). Another approach is determining the activity of ^{210}Pb through α-spectrometry of its ^{210}Po daughter. This allows a reduced sample size and the possibility of selectively leaching ^{210}Po from the exterior of mineral grains to measure only the mobile, exogenic ^{210}Pb activity, and not the endogenic activity from within the grains (Aalto *et al*., 2008).

Another primordial radionuclide is ^{40}K, the radioactive potassium isotope. Its decay is used in K-Ar and Ar-Ar dating. From its very long half-life (1.26×10^9 years) one would not expect the possibility of dating applications for the 10^3–10^4 year timescales of the Late Quaternary. It is the high abundance of ^{40}K (of the order of 10^{-4} compared to the 10^{-6} range of U and Th), and again, the use of high resolution mass spectrometry that recently allowed the dating of potassium-rich Holocene tephra (Hu, *et al*., 1994; Scaillet & Guillou, 2004). For Late Quaternary fluvial sediments the technique is of special importance where tephra is abundant and a tephra found in a sedimentary sequence can not be matched to an already existing tephrochronology.

(b) Cosmogenic production

The Earth is continually bombarded by high-energy primary cosmic rays that originate predominantly from super nova explosions within our galaxy. Interactions between these high-energy cosmic rays and the Earth's atmosphere creates secondary cosmic rays, including neutrons and muons, that interact with atoms of the atmosphere and the Earth surface and produce new – so-called – cosmogenic isotopes.

(i) Atmospherically produced cosmogenic nuclides Clearly the most widely used dating technique for the Late Quaternary is radiocarbon dating. A current review of technical details and developments is provided by Bronk Ramsey (2008). The latest major technical innovation in radiocarbon dating (the introduction of AMS techniques) now lies several years in the past and AMS ^{14}C dating is carried out routinely. AMS has led to significant changes in the way we use radiocarbon dating and opened up many new possibilities like the use of component-specific AMS

^{14}C dating of organic compounds. This current development allows determination of molecular-specific residence times of carbon compounds (Gonia *et al*., 2005; Scheefuß, 2008) in river sediments.

Other recent improvements relate to the calibration of ^{14}C – ages. For a number of periods the new and significantly improved calibration data set INTCAL04 (Reimer *et al*., 2004) provides increased precision that translates into more precise calibrated ages with fewer ambiguities. This also illustrates the need of re-calibrating earlier radiocarbon ages using the current calibration data set before comparisons of calibrated ^{14}C ages are carried out.

(ii) *In situ* produced cosmogenic nuclides In the upper few metres of the Earth's surface cosmogenic nuclides, such as ^{3}He, ^{10}Be, ^{14}C, ^{21}Ne, ^{26}Al and ^{36}Cl, are produced by nuclear reactions *in situ* (also called: terrestrial *in situ* cosmogenic nuclides: TCN). For example, ^{36}Cl is produced by spallation reactions of ^{39}K and ^{40}Ca and by activation of ^{35}Ca. The rates of accumulation of these nuclides are proportional to the cosmic ray flux and to the concentration of target nuclides in the surface material. The concentrations of cosmogenic nuclides can thus be used to determine the length of time this material has spent at or near the Earth's surface. Before the technological innovation of AMS, the detection of cosmogenic isotopes was confined to extraterrestrial materials (meteorites and lunar rocks) that have much higher concentrations of cosmogenic nuclides due to the much higher cosmic-ray fluxes beyond the Earth's atmosphere. Today, thanks to modern high-precision AMS techniques, the datable age range for terrestrial samples stretches from <1 ka to several Ma. The choice of the most suitable nuclide for dating depends on the material to be dated, its age, and on the facilities available for processing and analysing the samples (for reviews see Lal, 1988; Gosse & Phillips, 2001; Bierman & Nichols, 2004; Cockburn & Summerfield, 2004). The increasing availability of sample preparation laboratories and AMS facilities during the last few years has led to a boost of TCN applications in geomorphic research. Recent applications to fluvial systems include: (1) dating of terrace surfaces (exposure ages); (2) determination of point denudation rates (surface lowering); and (3) determination of long-term and catchment-wide erosion rates:

(1) Exposure ages of terrace surfaces have been used to date a variety of fluvial sedimentary bodies (Repka *et al*., 1997; Hancock *et al*., 1999; Schildgen *et al*., 2002; Pratt-Sitaula *et al*., 2004; Anders *et al*., 2005) in arid to humid settings, and over a wide range of timescales. The techniques used usually assume continuous cosmic ray irradiation of surfaces, thus excluding burial by snow or erosion since deposition. Also, nuclide inheritance from periods of exposure prior deposition has to be accounted for (Brocard *et al*., 2003). Violation of these assumptions can result in inaccurate age estimates: nuclide inheritance will result in age overestimates, burial or erosion of terrace surfaces in underestimates.
(2) Several studies have determined rates of surface lowering and thus denudation rates to study fluvial incision or landscape denudation (e.g. Schaller *et al*., 2005; Ward *et al*., 2005). Other TCN applications achieved better understanding of sediment delivery to channels: sediment transport rates on desert piedmonts (Nichols *et al*., 2002), estimates of upland erosion rates and sediment delivery to arroyos (Clapp *et al*., 2001), or bedrock to soil conversion (Wilkinson & Humphreys, 2005).
(3) Another application of TCN in fluvial sediments is the determination of long-term and catchment-wide erosion rates (Bierman & Steig, 1996). This is especially helpful for establishing pre-human-impact erosion rates (see review in von Blanckenburg, 2005).

Initially, steady-state approaches were utilised and a balance between production rate and average erosion rate assumed. More recently, this has been shown to be problematic as river systems only rarely represent steady state conditions: the effects of sediment delivery by bedrock mass movements have been investigated by Niemi *et al*. (2005). An interesting study by Codilean *et al*. (2008) utilised TCN concentrations of single clasts instead of bulk samples. They were able to show that the variation in erosion in a catchment is reflected in the distribution of the cosmogenic nuclide concentrations in sediments leaving the catchment.

The application of TCN in fluvial system dynamics is a rapidly developing field with an immense potential. It is still in its early years and there are several unresolved issues. These relate to the technique itself, like the variability in past production rates or elevation shielding factors (sources of random and systematic errors are listed in Gosse & Phillips, 2001), but also in the underlying geomorphic assumptions on averaging and steady-state behaviour.

DOSIMETRIC TECHNIQUES

These dating techniques rely on increasing radiation damage with increasing exposure times to environmental radiation. The dosimetric techniques that have proven useful for the study of river systems over the Late Quaternary timescale are luminescence (especially optically stimulated luminescence dating, OSL) and Electron Spin Resonance (ESR or EPR). Both determine the time since last exposure of sediment grains to daylight and for both the main breakthrough came with the analysis of single sand-sized grains: it is now possible to determine the distribution of depositional ages of sand grains within a sediment layer and extract the part of the distribution that represents the last depositional event.

The development of luminescence techniques was recently reviewed by Wintle (2008). Issues relating to the resetting of the OSL-signal in sedimentary environments and how to ensure that the last exposure to light was sufficient to allow successful OSL dating are discussed in Thrasher *et al.* (2008) and Shen *et al.* (2008). There is a recent upsurge of OSL applications to unravel Holocene river dynamics that cover a wide variety of environmental settings and spatial scales. For example, OSL was used to determine the scroll-bar migration of the Klip River, South Africa (Rodnight *et al.*, 2005); catchment response to changes in land-use and climate in The Netherlands (de Moor *et al.*, 2008); the functioning of arroyo systems in the USA (Arnold *et al.*, 2007); the dynamics of an anabranching river in northern Australia (Tooth *et al.*, 2008); flood recurrence in Israel (Jacoby *et al.*, 2008); residence times of in-channel sediment storage of a mountain catchment in southeast Australia (Thomson *et al.*, 2007); and river response to changing monsoon activity in South India (Thomas *et al.*, 2007).

Besides sufficient bleaching during deposition, appropriate luminescence properties of the dosimeter (i.e. sufficient OSL sensitivity of the quartz) is another issue that occasionally prevents successful OSL dating. Pietsch *et al.* (2008) show that the OSL sensitivity of quartz is enhanced during fluvial transport. This explains that, in general, quartz extracted from sediments that went through many cycles of erosion and deposition are better suited for OSL dating than quartz extracted from upland systems where sediments have only recently been derived from igneous or metamorphic rocks.

Dating applications of ESR to fluvial sediments are much less frequent. Compared to OSL, ESR allows dating of longer time spans (Middle Quaternary, if not the whole Quaternary, Beerten & Stesmans, 2007) but provides less precise ages. Recent examples of ESR dating of fluvial sediments are provided by Voinchet *et al.* (2007) and Bahain *et al.* (2007).

EVENT TECHNIQUES

The sudden occurrence (or a specific occurrence-pattern) of natural or artificial substances in fluvial sediments can be used to obtain age information. Probably the most common technique is tephrochronology that is based on identifying and correlating tephra (pyroclastic ejecta from volcanic eruptions). Especially in active volcanic areas (res. during periods of volcanic activity) this is an excellent tool as tephra layers form isochrons across the landscape (Andres *et al.*, 2001; Dugmore, 2004).

Substances used as tracers also carry age information (cf. examples in this volume). Especially for the period since severe human impact, macroscopic remains like tin-cans, traces of heavy metal pollution, carbon spheres from combustion engines or artificial radionuclides can provide excellent time markers as long as dispersion of contaminants from point sources are known (e.g. Coulthard & Macklin, 2003) and reworking or other post-depositional disturbance can be ruled out

BIOLOGICAL TECHNIQUES

Age information can be derived from the growth of organisms. In fluvial environments two techniques have been used successfully: lichonometry and dendrochronology.

Especially in cold climate and upland catchments, newly exposed stone surfaces are quickly colonised by lichen. Lichen size represents a measure of time elapsed since the exposure of the stone surfaces, i.e. the deposition of pebbles and boulders. Lichenometry is a well established technique that has proven successful for dating fluvial deposits in many upland environments (Harvey *et al*., 1984; Innes, 1985). A recent example is provided by Gob *et al*. (2008). In principle, the lichen size-distribution on the fluvial deposits is compared to a lichen growth curve established from known age surfaces of similar lithologies. Lichenometry has mainly utilised species within the *Rhizocarpon* subgenus on siliceous substrates. For lichen size analysis it is important that only sufficiently sized clasts (boulder size) are used to ensure that the maximum lichen size is represented. Also, a rather large number of observations and rigorous statistical criteria are needed to correctly represent lichen size-distributions (Locke *et al*., 1979). The mean or modal lichen diameter can be used as an age-index for unimodal lichen size distributions, but more commonly the population size-frequency distribution is used. The latter requires measuring all lichens present in the studied landform. Another possibility is the largest-size-frequency distribution that involves the measurement of the largest lichen on each boulder. A significant improvement was obtained by Jomelli *et al*. (2007): using a Bayesian method based on extreme value theory, they managed to develop a technique to quantify sampling error and calibration uncertainty for the first time.

Dendrochronology is based on tree ring analysis and is the most precise and accurate method for dating river dynamics. It allows determination of the year and, under favourable conditions, also the season when a flood occurred. Living trees as well as buried tree trunks can be used. Dendrochronology involves measuring a sequence of tree rings, detrending the sequence and matching it to the regional master tree-ring chronology (recent review in Stoffel & Bollschweiler, 2008). Techniques are well developed and have been successfully used in several studies (e.g. Becker & Schirmer, 1979).

In many cases buried tree trunks allow dating of their fluvial transport and sedimentation. If the outermost tree-ring is preserved, the time when the tree died can be determined. When the outermost ring is not preserved, the youngest remaining tree-ring gives a maximum age for the deposition. The oldest ring of a living tree gives a minimum age for the underlying deposits. Depositional ages can also be inferred from sprouting of adventitious roots in response to partial burial of the stem by fluvial deposits (Strunk, 1989). Recent advances in the technique utilise tree-root exposure and anatomical changes (Gärtner, 2007) or stem burial (Friedman *et al*., 2005) for dating.

CHEMICAL TECHNIQUES

There are a number of techniques that extract age information from chemical alterations over time (cation ratio dating, hydratation rinds, etc.), none of which are of great importance for dating fluvial deposits over centennial and millennial timescales. The only exception is Amino Acid Racemization (AAR) that utilises the inter-conversion of amino acids from one form (L- laevo amino acids, the building blocks of proteins) to a mixture of L- and D- (dextro) forms following protein degradation. At equilibrium there is typically a 1:1 ratio of L to D forms and this mixture is said to be racemic. Dating applications utilize the increase in the proportion of D-amino acids as a function of time and temperature. Attempts to provide age information use a calibration technique where a sample's amount of racemization is compared to the amount of racemization of a known-age specimen. Kosnik *et al*. (2008) estimate that the compound accuracy of this approach is ±20%.

Recent improvements in sample selection and chemical treatment helped to greatly improve analytical precision and accuracy by excluding contaminants and unstable components (Penkman *et al*., 2007, 2008). This was achieved by using more robust calcitic structures (opercula of *Bithynia*, a freshwater gastropod), the isolation of an intracrystalline fraction of amino acids, a new chromatographic technique and the analysis of multiple amino acids at a time. In addition, a better knowledge of past temperatures and temperature modelling allows bypassing the calibration and direct age calculation. This technique has great potential as the freshwater gastropod *Bithynia* is quite common in fluvial deposits. The time frame that can be covered is 10^2 years to 10^6 years.

GENERIC ISSUES

Even after obtaining chronometric data, establishing a robust chronology for fluvial deposits is still difficult to achieve. Only a few techniques provide an age estimate of the depositional process (direct dating); most techniques are based on associated finds, i.e. materials incorporated in the deposits (and thus derive "younger than" age estimates) or materials developed after deposition has occurred ("older than" age estimates). Considering this "quality" of age information together with the uncertainty of chronometric data (i.e. precision and accuracy, see Scott *et al*., 2007, for an overview) is still not common practice. Here, newly developed age models offer big improvements for sequences of fluvial sediments. Statistical analysis of age information based on the probability distribution of an age estimate and Bayesian reasoning have been developed for ^{14}C data in continuous sedimentary sequences. These analyses allow us to overcome the inherent uncertainties of isolated ^{14}C ages, and to identify the leads, lags, or synchrony between different events (Blaauw & Christen, 2005a). Even more, these techniques allow the identification of outliers and their removal (for an overview see Bronk Ramsey, 2008b). The recent developments of these techniques go beyond ^{14}C-dating and now allow inclusion of other chronometic data and age information (Millard, 2004, 2006). Also, spatially and temporally discontinuous data sets can now be analysed based on their stratigraphic relationship, a technique that is especially useful for fluvial deposits (Chiverrell *et al*., 2008).

Still, even with sophisticated age-depth modelling the chronology derived may not be fully accurate. Telford *et al*. (2004) show for simple continuous sedimentary settings that model performance is especially bad when only a few data points are available. This will be especially important in fluvial deposits were erosion and reworking are commonplace.

The number of dates needed to construct a robust chronology for a sedimentary sequence will depend on the required precision and the complexity of the stratigraphic setting. In general, the utilisation of isochrons (time parallel markers such as tephra layers or geomagnetic stratigraphy), the use of many more age estimates than is currently the norm, and the use of several independent techniques will help in reducing uncertainties.

CONCLUSIONS

Continuous technological development in many chronometric fields enables new possibilities for dating in fluvial systems. Especially developments in OSL and TCN dating achieved a break-through for quantifying temporal system dynamics. But still, each technique has its limitations and will not be applicable in every situation. In the majority of cases the quality of age information is dependent on the quality of the sample supplied and the interpretation of its context in relation to the problem being addressed rather than the performance of the technique employed. Highest quality results can only be obtained if field scientists and chronometry specialists cooperate closely before sample collection.

The recent technological developments have not just led to new fields of application, but also to better performance of many techniques. Increased precision is one of the main targets of technological development but – as with all data – chronometric data will always carry a level of uncertainty. To derive age information from chronometric data this uncertainty has to be taken into account. Here, recently available statistical-age models offer big improvements. One should be aware, however, that the uncertainties involved in age determination are in most cases small compared to the uncertainties involved in, for example, establishing a sediment budget!

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