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Investigating how fundamental parameters of XRF sample preparation and analysis affect the observed elemental concentration: an experiment using fluvial sediment from Sabah, Borneo.

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X-Ray Fluorescence (XRF) is an important technique for measuring the concentrations of geochemical elements and inorganic contaminants adsorbed to sediments as an input to sediment tracing methods used to evaluate sediment transport dynamics in river catchments. In addition to traditional laboratory-based XRF instruments, the advent of increasingly advanced portable handheld XRF devices now mean that samples of fluvial sediment can be analysed in the field or in the laboratory following appropriate sample preparation procedures. There are limitations and sources of error associated with XRF sample preparation and analysis, however. It is therefore important to understand how fundamental parameters involved in sample preparation and analysis, such as sample compression and measurement exposure duration, affect observed variability in measurement results. Such considerations become important if the resulting measurement variability is high relative to the natural variability in element concentrations at a sample site.

This paper deployed a simple experimental design to assess the impacts of varying a number of sample preparation and XRF analysis parameters on recorded measurements of elemental concentrations of the fine fraction (<63um) of bed-sediment samples. Specifically the study compared observed elemental concentrations measured using a Rigaku NEX-CG laboratory machine versus a handheld Niton XL3t-900 XRF elemental analyser. Helium purging was used on both machines to enable measurement of lighter geochemical elements. Sediment sub-samples were taken from a larger homogenised sample from a sediment core taken from an in-channel lateral bench deposit of the Brantian river in Sabah, Borneo; the core site is being used for research into multi-proxy sediment fingerprinting as part of the Stability of Altered Forest Ecosystems (SAFE) project.

Some fundamental sample preparation procedures consistent with US EPA Method 6200 were applied to all sediment samples in order to explore key variables of interest. All sediment samples were air-dried to constant weight and sample quantity was sufficient to satisfy the assumption of ‘infinite thickness’ of sample. Standard plastic sample cups were used for both the Rigaku laboratory machine and the Niton portable XRF machine. A computer-controlled desktop laboratory stand was used in conjunction with the Niton handheld XRF analyser to ensure consistent repeated measurements. Parameters investigated related to sample preparation included consistent mechanical compression of samples within the sample cup and film thickness. Parameters investigated related to XRF analysis included the XRF machine selected and measurement exposure duration. As XRF is a non-destructive technique, wherever possible the same sample material was used to test different parameters, so as to reduce variations due to the heterogeneous nature of sediment.

Observed XRF measurements demonstrate how the precision and relative accuracy of elemental concentrations of sediment can be affected by the XRF analyser selected as well as fundamental parameters of sample preparation and analysis procedure. This has implications for studies where comparability and repeatability of measurements is important. Furthermore, the heterogeneous nature of sediments over small spatial scales means that it is important to understand the levels of variability in elemental concentrations resulting from variations in sample preparation and analysis procedures.