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Assessment of particle size and organic matter correction factors in sediment source fingerprinting investigations: An example of two contrasting watersheds in Canada

Alexander J. Koiter^{a,b,*}, Philip N. Owens^c, Ellen L. Petticrew^d, David A. Lobb^e

^a Geography Department, Brandon University, 270 18th St, Brandon, MB R7A 6A9, Canada

^b Natural Resources and Environmental Studies Program, University of Northern British Columbia, 3333 University Way, Prince George, BC V2N 429, Canada

^c Environmental Science Program and Quesnel River Research Centre, University of Northern British Columbia, 3333 University Way, Prince George, BC V2N 4Z9, Canada

^d Geography Program and Quesnel River Research Centre, University of Northern British Columbia, 3333 University Way, Prince George, BC V2N 429, Canada

e Department of Soil Science and Watershed Systems Research Program, University of Manitoba, 13 Freedman Crescent, Winnipeg, MB R3T 2N2, Canada

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ABSTRACT

This study investigated the conservative behaviour of geochemical fingerprints through the soil mobilization process. This was accomplished by comparing the concentrations of a broad suite of geochemical elements of soil mobilized by simulated rainfall from small plots to the source soil, in two contrasting agricultural regions in Canada. All samples were sieved to isolate the fine-grained fraction ($< 63 \,\mu m$) prior to analysis in an effort to limit the differences in both particle size and organic matter content. Despite sieving the samples, there were still particle size and organic carbon content differences between the source soil and the mobilized soil. To account for these differences, particle size, and sometimes additional organic matter, correction factors are often applied to the source soil concentrations in an attempt to make the two sample types more directly comparable. The particle size and organic matter correction factors are typically based on the ratio of specific surface area (SSA), or soil organic carbon (SOC) content, between the eroded material and source material and the same correction factors are applied to all elements. It was demonstrated that for most geochemical elements the particle size correction factor resulted in an over-correction of the source soil relative to the mobilized soil and that using the organic matter correction factor in addition to the particle size correction exacerbated this over-correction. For most geochemical elements, the uncorrected source soil concentrations showed a smaller difference in concentrations relative to the mobilized soil as compared to the corrected source soil. In addition, it was found that the magnitude, direction and interaction of the relation of SSA and SOC on concentrations were generally both element and watershed specific. This study highlights some of the potential issues with applying correction factors to account for differences in particle size and organic matter content to a broad suite of geochemical fingerprints.

1. Introduction

Information on the relative contribution from different sources of sediment within a watershed is an important part of understanding sediment dynamics and is used for the development of sediment management plans. In particular, fine-grained sediment ($< 63 \mu$ m) is of the greatest environmental concern as a significant amount of nutrients, heavy and trace metals, radionuclides, pharmaceuticals, pesticides and other industrial contaminants are transported in association with this fraction (Oliver et al., 2007; Maskaoui and Zhou, 2010; Zheng et al., 2012). This is primarily due to the higher specific surface area (SSA) and chemical reactivity of this material as compared to coarse-grained

sediment (e.g., sand and gravel) (Horowitz, 1991). However, the high spatial and temporal variability of soil erosion and sediment delivery to surface waterways can make the identification of the dominant sources of sediment at the watershed scale difficult (Smith et al., 2011).

Sediment fingerprinting is an approach that is based on the concept that one or more of the natural physical or biogeochemical properties of the sediment (i.e., fingerprints) will reflect its source, and therefore can be used diagnostically to identify the origin(s) of the sediment; see Davis and Fox (2009) and Owens et al. (2016) for reviews of the sediment fingerprinting approach. The sediment fingerprinting approach has been used for a variety of different applications including agricultural (e.g., Koiter et al., 2013a), forest harvesting (e.g., Motha et al.,

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^{*} Corresponding author at: Geography Department, Brandon University, 270 18th St, Brandon, MB R7A 6A9, Canada. *E-mail address:* koitera@brandonu.ca (A.J. Koiter).

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2003), wildfires (e.g., Owens et al., 2012) and urbanization (e.g., Carter et al., 2003). There is a wide variety of soil and sediment properties that have been utilized as fingerprints including fallout radionuclides, geochemistry, colour, mineral magnetics and stable isotopes (Foster and Lees, 2000; Haddadchi et al., 2013).

The sediment properties selected as fingerprints need to: (1) provide good discrimination between potential sources of sediment; and (2) behave conservatively (i.e., fingerprints should not be affected by the sediment generation, delivery and transport processes in a way that cannot be quantified). There are many statistical approaches that have been used to select the best suite of fingerprints that maximize the discriminatory power between the sources of sediment (e.g., discriminant function analysis, canonical analysis) (Collins and Walling, 2002). However, it is more difficult to assess the suitability of fingerprints in terms of the conservative behaviour of sediment as it is transported through a watershed.

Ideally, the physical and biogeochemical properties of the sediment do not change as it moves through a watershed and thus a direct comparison of source material and downstream sediment can be made. However, the particle size and organic matter selectivity of the soil erosion, sediment delivery and fluvial transport processes generally results in downstream fining and an increase in organic matter content due to the preferential mobilization and transport of the small and less dense soil particles (Slattery and Burt, 1997; Di Stefano and Ferro, 2002; Asadi et al., 2011; Koiter et al., 2015). There are also other processes that can alter the physical and biogeochemical properties of sediment during transport from source to sink including abrasion/ breakage, adsorption/desorption and organic matter decomposition (see Koiter et al., 2013b for a review of these processes). The latter processes have not received much attention in the literature as it can be difficult to predict the behaviour of fingerprint properties in the environment. However, changes in particle size and organic matter content are often accounted for by first limiting the analysis to the finegrained fraction (i.e., $< 63 \,\mu$ m) as the coarser mineral grains are preferentially deposited upstream. Secondly, particle size, and sometimes organic matter, correction factors are then applied to the fingerprint data set to further correct for any remaining differences in particle size and organic matter.

Correction factors are used in many cases as the geochemical concentrations within soil and sediment samples have been shown to be strongly correlated with both the particle size distribution and organic matter content (Horowitz, 1991). Therefore, correction factors are used to account for this enrichment of fine-grained and organic-rich particles that typically occurs as sediment moves through a watershed. The commonly used correction factors are based on the ratio of specific surface area (SSA) and soil organic carbon (SOC) between each collected sediment sample and the mean for each source group (e.g., Collins et al., 1997). The correction factor value(s) are then multiplied by the concentration in the source material for each fingerprint so as to make the resultant values more comparable to those of the collected sediment. This allows any differences in fingerprint concentrations to be attributed to the relative contribution from sediment sources as opposed to the influence of downstream fining and organic matter enrichment.

There has been an increase in the number of sediment fingerprinting studies that use the geochemical composition of the sediment as potential fingerprints, as a single standard laboratory procedure (i.e., ICP-MS following a digestion with acid) can yield information on a broad suite of elements (> 50) that can each be used as a potential fingerprint. The basis of using particle size and organic matter correction factors in conjunction with geochemical fingerprints is that the concentrations of many geochemical elements are dependent on the particle size and organic matter content (Horowitz, 1991). The commonly used correction factors assume a linear and positive relation between both SSA and SOC and the geochemical concentration. In addition, applying both a particle size and organic matter correction factor

assumes that the effects of SSA and SOC on the geochemical concentration are multiplicative. However, some researchers do not use both, citing that SSA and SOC are often correlated which can result in the over-correction of fingerprint values (e.g., Dutton et al., 2013). The use of correction factors also assumes that the relations are the same for each element; however, there is a growing amount of evidence to suggest that the direction and magnitude of these relations are element specific and that they vary between watersheds (Russell et al., 2001; Smith and Blake, 2014).

The use of correction factors has been shown to affect both the fingerprint selection process and ultimately the apportionment results (Laceby et al., 2017). For example, Smith and Blake (2014) found that through the combination of both particle size and organic matter correction factors the relative differences in the values of excess ²¹⁰Pb between sources were altered, with the corrected channel bank source having higher values as compared to cultivated and pasture surface soils. This is inconsistent with the atmospheric deposition of ²¹⁰Pb which has been shown to effectively label topsoil and the fact that channel banks are primary composed of subsoil and thus should exhibit lower values (Mabit et al., 2014). Furthermore, Smith and Blake (2014) demonstrated that the use of the combined correction factors for the Tamar Gunnislake watershed, UK, resulted in a change to the source apportionment results by 45%, as compared to uncorrected values, and subsequently came to a different conclusion as to the dominate source of sediment. Therefore, without an adequate assessment of the relations between SSA and SOC with geochemical concentrations prior to using correction factors there is an unknown amount of uncertainty and error being incorporated into the sediment fingerprinting approach (Motha et al., 2002; Laceby et al., 2017).

In this study, the particle size and organic matter selectivity, and its subsequent effect on the geochemical composition of mobilized soil, was investigated in two contrasting agricultural watersheds in Canada, using a rainfall simulator to mobilize soil. The two contrasting agricultural regions in which this study took place are locations where there has been, and continues to be, a large effort being made to understand the influence of both land use and geomorphology on sediment dynamics using fingerprinting techniques (e.g., Koiter et al., 2013a; Barthod et al., 2015). Furthermore, as part of this effort research is also being undertaken to further develop and test both conventional and new methodologies and fingerprints. Previous research by Koiter et al. (2017) using the same source soil and mobilized soil samples as the current study, focused on investigating the role of soil surface properties (e.g., vegetative cover) on the particle size and carbon selectivity of interrill erosion. As part of that research, the differences in both the particle size distribution and organic carbon content between the source and mobilized soil was quantified. The conclusions of that study demonstrated the mobilized soil was comprised of a greater proportion of fine-grained and organic-rich particles as compared to the source soil. However, Koiter et al. (2017) did not evaluate the implications of these differences in particle size and organic matter content on the geochemical concentrations of the mobilized soil, if such differences suggested that corrections factors were in fact required, and what effect this may have on the use of geochemical properties as fingerprints. The objectives of this research were to investigate: (1) the physical and biogeochemical properties of source soils and mobilized soil; (2) the relations between SSA and SOC and their interactions on the geochemical concentration for a broad suite of elements; and (3) the suitability of commonly used correction factors to account for differences in particle size and organic carbon content between source and mobilized soil.

2. Methods

2.1. Watershed descriptions

The South Tobacco Creek (STC) watershed is located in south-

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