

Silt and phosphorus availability in unpaved road materials from forested watersheds in Canada,
and implications for drinking water resources

by

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Abstract

Many communities in Canada rely on surface drinking water supplies that are located within or downstream of forests. The typically clean, clear waters flowing from forests are sensitive to disturbances that can degrade source water quality. Unpaved access roads are one of the most widespread disturbances in forested watersheds, and they are known to increase the amount of sediment and nutrients delivered to streams. The goal of this thesis was to improve understanding, in a drinking water source quality and treatment context, of the hazards associated with unpaved access roads in forested watersheds. This study focused on quantifying two potential hazards to drinking water resources: silt (particles $<63 \mu\text{m}$, in this thesis) and phosphorus (P), specifically bioaccessible P. Silt and P availability, along with the mobilization of silt from road-related sediment sources, reflects what could be transported in runoff either directly to surface water supplies or to their contributing areas upstream.

Watershed-scale median silt contents of road surface materials ranged from 7–55%. Cutslope sample sizes were limited; however, cutslope silt contents were typically higher than those of road surface materials from the same site ($p = 0.01$). The silt contents of road surface materials were variable, with ranges as high as 50–74% within watersheds, and “hotspots” of high silt availability were common. The median silt content of sediments mobilized in runoff from unpaved road surfaces was 95%, and sediments contained, on average, 3.6 times more silt than road surface materials. These results were consistent with selective erosion. Phosphorus was ubiquitous in the road-related materials sampled, with concentrations ranging from 97–6016 mg/kg. However, across all road surface material and cutslope samples, bioaccessible P comprised only 12% to 17% of total P. Rather, most of the P in road-related materials (median = 82% and 72% for road surface materials and cutslopes, respectively) consisted of apatite P, which is not

bioaccessible. These results indicate that, in many cases, total P is not a reliable indicator of the water quality hazard associated with P inputs from unpaved access roads. As with silt contents, bioaccessible P concentrations in road surface materials varied among watersheds (by up to ca. 200 mg/kg) and within watersheds (by up to 200–300 mg/kg). “Hotspots” of high P availability also occurred within some watersheds. The occurrence of “hotspots” of high silt and P availability represent elevated water quality hazards, particularly where they overlap, due to the increased potential for bioaccessible P transport associated with the selective erosion of silt.

This study shows that unpaved access roads are potential source of silt and P (particularly bioaccessible P), provides new information on the amount and types of P in unpaved access road-related materials, and highlights the opportunity to preventively manage hazards associated with unpaved access roads.

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Chapter 1. Thesis introduction

1.1 Forests and drinking water

Forests are important drinking water source areas globally (Bladon et al., 2014; Emelko et al., 2011), including Canada, where many communities rely on surface drinking water supplies that are either located within or downstream of forests (e.g., Anderson et al., 2009; APEL, 2014; Barlak, 2011; Dobson and Cook, 2013; Halifax Water, 2009; Hébert, 2007; Metro Vancouver, 2011; Robinne et al., 2019; Shoal Lake Watershed Working Group, 2002; van Meerveld et al., 2014). Forests typically yield high-quality source waters (Anderson and Lockaby, 2011; Bladon et al., 2014; Emelko et al., 2011; Pike et al., 2010), which require less intensive treatment and pose fewer human health risks (Davies and Mazumder, 2003). However, land disturbances—whether natural or human-caused—can degrade source water quality, resulting in higher treatment costs (Anderson and Lockaby, 2011; Bladon et al., 2014; Emelko et al., 2011; Ryan, 2000). Managing source watersheds to protect drinking water quality is often viewed as an effective strategy to keep costs low, ensure long-term sustainability, and protect human health (Davies and Mazumder, 2003; Ryan, 2000). To do this effectively, watershed managers need to understand the hazards associated with different types of disturbances.

1.2 Unpaved access roads in forested watersheds

In addition to being important drinking water source areas, forests support a variety of social, cultural, and economic uses such as natural resource extraction, wildfire and pest management, residential use, grazing, subsistence (i.e., hunting, fishing, trapping, and foraging), research and monitoring, and recreation (Al-Chokhachy et al., 2016; Brown et al., 2013; Elliot, 2013; Gucinski et al., 2001; Luce and Wemple, 2001). Vast networks of access roads have been

developed to provide access for these activities (Luce and Wemple, 2001). Although common, unpaved roads usually comprise a relatively small proportion of watershed area—however, their impacts on water quality are often disproportionately high relative to their footprint (Motha et al., 2003; Tiecher et al., 2019).

Extensive research conducted over more than five decades across a variety of ecophysiological environments has shown that unpaved access roads alter hydrologic (e.g., infiltration, surface runoff) and geomorphic processes (e.g., mass wasting) in ways that increase the amount of sediment delivered to streams (Gucinski et al., 2001; Wemple et al., 2017). Where unpaved access roads are highly connected to streams, such as at watercourse crossings or culverts (Al-Chokhachy et al., 2016; Brown et al., 2013), estimated sediment delivery rates at individual drain points can be up to thousands of kilograms annually (Al-Chokhachy et al., 2016; Cissel et al., 2014). The impacts of increased erosion and sedimentation associated with unpaved access roads in forested watersheds have typically been examined in an aquatic ecology context—for example, benthic invertebrate abundance (e.g., Kreuzweiser et al., 2005; Tebo, 1955), and spawning and rearing habitat quality for salmonid species (e.g., Al-Chokhachy et al., 2016; Bilby, 1985; Cederholm and Reid, 1987; Platts et al., 1989). However, as one of the most prominent disturbances in forested watersheds (Al-Chokhachy et al., 2016; Baird et al., 2012; MacDonald and Coe, 2008) and well-established sources of sediment, the potential impacts of unpaved access roads warrant further examination through a source water quality lens.

1.3 Implications of sediment for drinking water source quality and treatment

Considered “the greatest pollutant of forest streams” (Elliot et al., 2009), sediment can adversely impact drinking water supplies in different ways. For instance, higher sediment loads

can lead to increased treatment and maintenance needs, and therefore higher costs (Emelko et al., 2011; Health Canada, 2012; Holmes, 1988; Marquis, 2005; Scatena, 2000). In some cases, high suspended sediment levels could even make it challenging to achieve drinking water quality guidelines for turbidity, or to supply water at all if drinking water intake shutdowns are required to avoid damaging pumps (e.g., Dobson Engineering, 2007). Additionally, inorganic particles—such as those that erode from unpaved access roads—can interfere with coagulation, flocculation, disinfection, and pathogen detection (Health Canada, 2012; LeChevallier et al., 1981). Notably, sediment is a vector for several potential contaminants, such as heavy metals, pesticides, organic compounds (e.g., polycyclic aromatic hydrocarbons, or PAHs), microbial pathogens, and nutrients (Droppo et al., 2006; Emelko et al., 2011; Falconer, 2004; Health Canada, 2012; Ongley et al., 1992; Ritter et al., 2002; Ritter, 1988).

Many of the human health risks associated with sediment-bound contaminants can be managed by removing suspended particulates (Health Canada, 2012). However, some sediment-borne contaminants are not effectively mitigated by drinking water treatment processes, especially where utilities rely solely on disinfection. Phosphorus (P), particularly, remains a concern for drinking water supplies. Though P is not itself a human health hazard (Ritter et al., 2002; Scatena, 2000), nutrient enrichment can indirectly lead to human health risks, higher treatment costs, and diminished treated water aesthetics (Bladon et al., 2014; Cooke and Kennedy, 2001; Davies and Mazumder, 2003; Dodds et al., 2009; Emelko et al., 2011; Falconer, 2004; Ritter et al., 2002; Smith, 2003; Smith and Schindler, 2009; Yuan et al., 2018). Due to its role in promoting algal growth in freshwaters (Cooke and Kennedy, 2001; House, 2003; Pick, 2016; Reddy et al., 1999), excess P could cause foul taste and odour and increase the potential for disinfection by-product formation (Cooke and Kennedy, 2001; Davies and Mazumder, 2003; Davies et al., 2004; Dodds

et al., 2009). Phosphorus can also influence cyanobacterial predominance in phytoplankton communities (Downing et al., 2001; Pick, 2016). Since some species can produce neuro- or hepatotoxins (Kotak and Zurawell, 2007), such cyanobacterial proliferations are a threat to drinking water supplies (Cooke and Kennedy, 2001; Dodds et al., 2009; Downing et al., 2001; Falconer, 2004; Health Canada, 2017; Kotak and Zurawell, 2007; Ritter et al., 2002). In drinking water distribution systems, P poses an additional human health risk as it can support bacterial growth (Emelko et al., 2011; Miettinen et al., 1997).

The impact of unpaved access road-derived sediment on water quality is a product of both the amount and the properties of the sediment delivered to surface waters (Bilby et al., 1989). Specifically, silt and clay, which comprise the particle size fraction $<63 \mu\text{m}$ (Wentworth, 1922) and hereafter referred to collectively as silt, pose the greatest hazard to source water quality. Firstly, silt often erodes preferentially from unpaved road surfaces (e.g., Bilby, 1985; Bilby et al., 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016). Secondly, due to low settling velocities, silt is readily suspended and mobilized in runoff (Marquis, 2005; Proffitt et al., 1991). This means that silt is more likely to be delivered to surface waters and transported downstream (Baird et al., 2012; MacDonald and Coe, 2007), where it could impact water supplies. Finally, the highest sediment P concentrations generally occur in the $<63 \mu\text{m}$ size fraction (e.g., Kerr et al., 2011; Stone and Mudroch, 1989), and the same is likely true for road sediments (e.g., Dudley et al., 1997). Thus, when it comes to sediment sources, there is a particular need to better understand silt availability associated with unpaved access roads.

1.4 Unpaved access roads as a source of phosphorus

Unpaved access roads are likely sources of P in forested watersheds. Firstly, P is naturally-occurring in geological materials, including the surficial materials (e.g., Hartmann et al., 2012; Yang et al., 2013) and rocks (Filippelli, 2002; Porder and Ramachandran, 2013) that commonly make up road surfaces and exposed cut- and fillslopes and ditches on unpaved access roads. Secondly, unpaved access roads are often the predominant source of sediment—which is the principal vector for P transport to surface waters (Allin et al., 2012)—in forested watersheds (Elliot, 2013; MacDonald and Coe, 2008). Previous studies have revealed that unpaved access road materials and sediments contain P (e.g., Dudley et al., 1997; Forsyth et al., 2006; Sheridan and Noske, 2007; Sheridan et al., 2008; Tiecher et al., 2019; Wemple et al., 2017; Yousefi et al., 2016). Not surprisingly, strong relationships between total P in unpaved road runoff and total suspended sediment have also been reported (e.g., Croke et al., 2000; Sheridan and Noske, 2007; Sheridan et al., 2008).

Despite the water quality concerns related to P and the relationships between unpaved access roads, sediment, and P, studies that examine unpaved access roads as P sources in forested watersheds are limited. Notably, most of the results published to date are from Australia (e.g., Croke et al., 2000; Forsyth et al., 2006; Sheridan and Noske, 2007; Sheridan et al., 2008), where P concentrations are predictably lower than would be expected in North America because soils are older and more weathered (Kerr et al., 2011). For example, mean P concentrations in road-related materials (e.g., road surface materials, cutslopes, roadside soils, ditches, road-derived sediments) from a state forest in Queensland, Australia were ca. 44 mg/kg (Forsyth et al., 2006), compared to 396 mg/kg in materials from the Lake Champlain watershed in Vermont (Wemple et al., 2017). Since the distribution of P in geological materials is geographically variable (e.g., Hartmann et al.,

2012; Yang et al., 2013), data from Canadian watersheds are needed to understand how much P could be mobilized from unpaved access roads through erosion and sedimentation.

1.5 Forms of phosphorus and bioaccessibility

The potential effects of sediment-related P on the aquatic environment cannot be adequately assessed based on total P alone (Kaiserli et al., 2002; Kerr et al., 2011). Rather, the implications for water quality are associated with the forms of P present, since their mobility in the aquatic environment varies (Emelko et al., 2016; Kaiserli et al., 2002; Kozerski and Kleeberg, 1998; Pardo et al., 2004; Ruban et al., 1999)—in other words, not all types of P are capable of supporting primary productivity (Boström et al., 1988). Primary mineral P, which consists mainly of apatite minerals (i.e., calcium phosphates; Filippelli, 2008; Yang and Post, 2011; Yang et al., 2013) is not biologically available (Yang et al., 2013). However, apatite P undergoes transformations as it weathers, producing bioaccessible forms of P (Filippelli, 2008; Smeck, 1985; Walker and Syers, 1976; Yang and Post, 2011; Yang et al., 2013). Bioaccessible refers to what is immediately available for biological uptake (i.e., what is bioavailable) and what could become bioavailable (Semple et al., 2004). Thus, bioavailable P consists of reactive P (e.g., ionic species such as H_2PO_4^- , HPO_4^{2-} ; Orihel et al., 2017) and, following Semple et al. (2004), also includes P that is loosely bound to particle surfaces and easily desorbed. Less bioaccessible forms of P include P bound to particle surfaces that releases more slowly or only under certain conditions—for example, P adsorbed to secondary mineral surfaces (e.g., Al and Fe oxides or oxyhydroxides) or encapsulated in secondary minerals (typically Fe oxides; Smeck, 1985) may become bioavailable in the aquatic environment depending on factors such as pH, redox conditions, and the presence of competitor ions (Boström et al., 1988; Orihel et al., 2017).

Chemical sequential extractions can be used to examine the composition of sediment-P in terms of the mechanisms of P release in the environment. Since operationally-defined fractions are based on the ecological significance or bioaccessibility of different geochemical forms of P (e.g., Emelko et al., 2016; Hawthorn, 2014; Kaiserli et al., 2002; Pettersson et al., 1988; Psenner et al., 1984; Ruban et al., 1999; Stone and English, 1993; Tiecher et al., 2019; and as reviewed by Wang et al., 2013), these methods can provide better insight into road-related P hazards in source watersheds. However, with few exceptions (e.g., Dudley et al., 1997; Tiecher et al., 2019), P concentrations in unpaved access road-related studies conducted to date have been measured and reported in terms of total P.

1.6 Research objectives

The goal of this thesis is to increase awareness and understanding of the hazards associated with unpaved access roads in forested source watersheds. Unpaved access road erosion and sediment transport processes have been well-studied; however, there has been comparatively little emphasis on the characteristics of the materials being eroded from roads and delivered to surface waters. Further, while it is generally understood that sediment delivery from unpaved access roads will increase nutrient delivery (e.g., Gucinski et al., 2001; Hawthorn, 2014), information on how much P is in road-related materials or sediments is lacking. Existing studies are limited to a few geographic areas and they report almost exclusively on total sediment loads or total P, whereas potential water quality impacts are related primarily to silt and to bioaccessible P.

To address these knowledge gaps, this study employs methods to examine silt and bioaccessible P content in unpaved access road-related materials at a broad spatial scale. The study includes forested source watersheds that represent a range of ecophysiological settings across

Canada, including the Pacific Maritime, Montane Cordillera, Boreal Plains, Boreal Shield, and Atlantic Maritime ecozones. Using particle size analysis and a chemical sequential extraction procedure, this study aims to answer the following questions:

- How much silt is available in unpaved access road-related materials?
- How much of that silt is mobilized in runoff?
- How much P is in unpaved access road-related materials?
- How much of that P is bioaccessible?

The potential for unpaved access roads to produce and mobilize silt is examined in Chapter 2. This chapter focuses on road surfaces and cutslopes, as they are typically the most significant road-related sediment sources (Arnáez et al., 2004; Baird et al., 2012; Croke et al., 2006; Fahey and Coker, 1992; MacDonald and Coe, 2008; MacDonald et al., 2001; Megahan et al., 2001; Ramos-Scharrón and MacDonald, 2007; Reid and Dunne, 1984) and the materials are representative of other potential road-related sources (e.g., ditches, fill slopes). The specific objectives of Chapter 2 are to:

- 1) Assess silt availability in road surface materials and cutslopes, and
- 2) Assess silt mobilization from road surfaces and cutslopes.

Phosphorus content in unpaved access road-related materials, focusing on the same sediment sources and watersheds as in Chapter 2, is examined in Chapter 3. The objectives of Chapter 3 are to:

- 1) Quantify the total amount of P, and
- 2) Estimate the amount of bioaccessible P.

Chapter 3 also examines the relative composition (i.e., operationally-defined fractions) of P, which provides context for P bioaccessibility. In addition, this chapter includes a discussion on the implications of road-related P for source water quality.

Chapter 4 includes a synthesis of results from Chapters 2 and 3, identifies management opportunities and challenges, and offers recommendations for further study. This chapter also highlights the key contributions of this work towards understanding water quality hazards associated with unpaved access roads.

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Chapter 2. Silt availability in unpaved access road surface materials and cutslopes in forested source watersheds

2.1 Introduction

Unpaved roads provide access for resource extraction, forest management, grazing, and recreation (Al-Chokhachy et al., 2016; Brown et al., 2013; Elliot, 2013; Gucinski et al., 2001; Luce and Wemple, 2001), and they are one of the most widespread human-caused disturbances in forested watersheds (Al-Chokhachy et al., 2016; Baird et al., 2012; MacDonald and Coe, 2008). In such watersheds, unpaved access roads are often the predominant source of sediment (e.g., Anderson and Potts, 1987; Beschta, 1978; Christie and Fletcher, 1999; Elliot, 2013; Forsyth et al., 2006; Grayson et al., 1993; MacDonald and Coe, 2008; Reid and Dunne, 1984; Reid et al., 1981; Reiter et al., 2009; Sidle et al., 2004; Swank et al., 2001; Zemke 2016). Estimates from road erosion studies show that they can produce hundreds of tonnes of sediment per kilometre of road (e.g., Reid and Dunne, 1984; Reid et al., 1981) or tens of tonnes of sediment per hectare of road surface (e.g., Grayson et al., 1993) annually. Unpaved access roads can also contribute a disproportionate amount of sediment relative to their surface area in a watershed (e.g., 6–25% vs. 0.4–0.8%, respectively; Motha et al., 2003).

Of the road prism elements (Figure 2.1), road surfaces typically produce the most sediment (e.g., Croke et al., 2006; Fahey and Coker, 1992; MacDonald et al., 2001; Ramos-Scharrón and MacDonald, 2007; Reid and Dunne, 1984). The loose materials detached during road construction, use, and maintenance are easily mobilized in runoff, which is readily produced on these characteristically bare, compacted surfaces (La Marche and Lettenmaier, 2001; Luce and Wemple, 2001; MacDonald and Coe, 2008). Yet, cutslopes can be an important sediment source in watersheds where they are highly weathered, or where mass wasting or freeze-thaw cycles supply

loose material that is readily entrained in runoff (Arnáez et al., 2004; Baird et al., 2012; MacDonald and Coe, 2008; Megahan et al., 2001; Ramos-Scharrón and MacDonald, 2007). In some cases, cutslopes can yield sediments of the same order of magnitude as road surfaces (e.g., hundreds of tonnes per hectare annually; Megahan et al., 2001), and they can comprise significant proportions of road-related sediments. Ramos-Scharrón and MacDonald (2007), for instance, showed that up to 50% of road sediments originated from cutslopes, and Arnáez et al. (2004) found that cutslopes produced, on average, eleven times more sediment than unpaved road surfaces. If there is no direct connection between roads and surface waters via overland flow, channelling, or gullying, sediment can settle out before reaching the water body (Gomi et al., 2005). However, sediments can be delivered to surface waters at points where roads and water are highly connected, such as at watercourse crossings and drainage outlets (Al-Chokhachy et al., 2016; Brown et al., 2013; La Marche and Lettenmaier, 2001).

Silt (particles $<63 \mu\text{m}$, in this thesis) is often preferentially mobilized in road runoff (e.g., Bilby, 1985; Bilby et al., 1989), and especially in runoff from road surfaces (e.g., Bilby et al., 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016). Preferential transport typically results in sediments that have high silt contents as a result of enrichment. For example, Costantini et al. (1999) found that particles $<50 \mu\text{m}$ made up approximately 86% of sediments derived from road surfaces even though road surface materials contained only 8% of this size fraction. Due to low settling velocities, silt is readily suspended and mobilized in runoff (Marquis, 2005; Proffitt et al., 1991), which increases the likelihood that it will be delivered to surface waters and transported downstream (Baird et al., 2012; MacDonald and Coe, 2007). So, of the sediments produced by roads, silt has the greatest potential to impact drinking water supplies.

The most apparent impact of unpaved access roads on drinking water source quality and treatability is related to turbidity. Silt is often implicated in increased turbidity (Health Canada 2012; Marquis, 2005). High turbidity can interfere with disinfection and pathogen detection (Health Canada, 2012; LeChevallier et al., 1981). In some watersheds, there is concern that road-derived sediments might be compounding high natural turbidity. High turbidity and suspended sediment levels may make it difficult to achieve drinking water quality guidelines for turbidity in treated water, to adequately disinfect drinking water, or to supply water when drinking water intake shutdowns are required in order to avoid damaging pumps (e.g., Dobson Engineering, 2007). High turbidity may increase operating costs depending on the processes and capacity of the current treatment system (Emelko et al., 2011). However, sediment is also a vector for microbial pathogens (Droppo et al., 2006; Health Canada, 2012) and for potential contaminants such as nutrients, pesticides, and metals (Emelko et al., 2011; Falconer, 2004; Health Canada, 2012; Ritter et al., 2002; Ritter, 1988). Phosphorus, in particular, is a concern for drinking water utilities as it can have consequences for both drinking water quality and treatment costs (Emelko et al., 2011; Emelko et al., 2016). Studies have shown that unpaved access road sediments can be a source of P in watersheds (e.g., Forsyth et al., 2006; Sheridan and Noske, 2007; Sheridan et al., 2008; Tiecher et al., 2019; Wemple et al., 2017; see Chapter 3). In general, the highest sediment P concentrations occur in the <63 μm particle size fraction (e.g., Kerr et al., 2011; Stone and Mudroch, 1989), and the same is likely true for road sediments (e.g., Dudley et al., 1997).

Because it can cause or contribute to turbidity and transport P from roads to surface waters, the silt in road-related sediments has important implications for drinking water treatment. The general objective of this chapter is to understand the potential for unpaved access roads—particularly road surfaces and cutslopes—to generate silt in forested watersheds that either

constitute, or are connected to, drinking water supplies across a range of ecophysiological settings in Canada. This information is currently lacking, and where available, it is limited to a few individual watersheds. More specifically, this chapter examines the potential for road surfaces and cutslopes to yield silt in two ways: 1) by assessing silt availability in road surface materials and cutslopes, and 2) by assessing silt mobilization from road surfaces and cutslopes. These objectives are addressed using a combination of empirical methods and literature review.

2.2 Methods

2.2.1 Sample locations

Road surfaces and cutslopes were sampled in 27 watersheds located within five of the major forest ecozones of Canada (Figure 2.2). Watersheds were selected to represent a diversity of managed forests within these ecozones. The proximity of the sampling sites to drinking water supply intakes varied; some were located only a few metres from a stream, lake, or reservoir, while others were located in the headwaters that supply utilities located over 100 km downstream. Drinking water usage also varied from domestic consumers sourcing water from tributaries (e.g., in the UB and BG watersheds) to large municipal utilities that draw from reservoirs (e.g., in the SG, UW, and PT watersheds). Note that data for separate watersheds serving the same water utility were summarized together (i.e., CB, SG, DC, UW, and PT watersheds). Road access and management values varied along a spectrum: many of the watersheds sampled had public access and were managed for multiple uses (e.g., natural resource extraction, grazing, recreation), others had controlled access and were managed for values including natural resource extraction, research, and recreation, and some were restricted access and managed solely as drinking water source areas.

Road surfaces and cutslopes were selected after scouting the watersheds to assess the range of road types, surface materials, drainage types (natural and engineered), and watercourse crossings. Once on site, samples were easy to collect so over 200 road surfaces and cutslopes were sampled from road segments that represented low, moderate, and high erosion hazard (hazard was assessed for a companion study by J. Hall, unpublished). The samples were catalogued and a subset was selected for analysis. The subset was chosen to represent the variety of road classes (e.g., main roads, branch roads, and operational roads) and surface materials present in each watershed. It favoured road segments with obvious erosion, sedimentation, or water quality hazard (e.g., rilling or gullying present on road surfaces or cutslopes, sediment plumes or deposits in ditches or below drainage structures, where road-surface connectivity was comparatively high, or where algal growth was observed in ditch waters, settling pits, or at watercourse crossings). The subset also included road segments that had large (long or high) unvegetated cutslopes.

2.2.2 Road surface and cutslope sampling

The sampling protocol was intended to collect materials within the potential depth of erosion. For road surfaces, that comprised the float (loose material on the road surface; Skorseth and Selim, 2000) and the top of the compacted running surface immediately below. For cutslopes, the top 2–3 cm of material was sampled. This is within the range of annual losses from cutslopes (<1–7 cm) reviewed by Megahan et al. (2001). Road surface materials were collected using a sweep sampling method derived from the U.S. Environmental Protection Agency's (EPA) standard operating procedure for Chip, Wipe, and Sweep Sampling (1994; Figure 2.3). Each road surface sample consisted of materials swept from two 30 cm long by 30 cm wide plots (one on each side of the running surface). The plots were swept with a plastic scrub brush for 30 seconds and the

materials were collected into a plastic dust pan. Materials from high-yielding plots were subdivided in the field and a portion was discarded in order to accommodate limited storage space in the sample cooler.

Where an unvegetated cutslope was present at the road segment being sampled, the surface (i.e., the top 2–3 cm of material) was sampled at two different locations along the cutslope. Samples were initially collected using a plastic scraper (cutslope samples from the SG and DC watersheds) and was subsequently swapped for a stainless-steel trowel as the plastic scraper showed signs of wear.

The composite samples were stored in labeled, 4 mil polyethylene, resealable soil bags or Ziploc freezer bags (HR samples). The samples were also being analyzed for non-apatite inorganic P, apatite P, and organic P (see Chapter 3), so the sampling tools were thoroughly cleaned between sites and the samples were stored in a cooler with ice during travel until they could be refrigerated.

2.2.3 *Sediment sampling*

A portable rainfall simulator was used to generate and collect runoff from road surfaces.

2.2.3.1 *Portable rainfall simulator design and operation*

The rainfall simulator used during the 2018 field season consisted of a 3 m tall, freestanding steel frame with a pivoting nozzle clamp to which a brass full cone spray nozzle (FullJet 1/4HH-14WSQ, Spraying Systems Co., Wheaton, IL, USA) was attached (Figure 2.4a). A fall height of 3 m allows simulated rain drops to approximate the velocity of natural rain (Covert and Jordan; 2009; Humphry et al., 2002). Locally-sourced water was pumped from a 273 L collapsible bladder through a 1 μ m particulate filter (Rainfresh model FC005 housing with HP1 filter, Envirogard

Products Ltd., Richmond Hill, ON, CAN) and an acrylic rotameter (FL7302, Omega Environmental Inc., Laval, QC, CAN) to the nozzle by a battery-powered 12 V pump. A glycerin-filled pressure gauge was placed parallel to the acrylic rotameter. The flow rate and water pressure were controlled with ball valves in order to achieve a consistent drop size and precipitation depth across rainfall simulations. A flow rate and pressure of 2.6 L/min (0.7 gpm) and 69–72 kPa (10–10.5 p.s.i.), respectively, produced a plot-average rain depth of 18 mm in 30 minutes and a mean raindrop size of 0.3 mm (refer to calibrations in 2.2.3.2). An overflow hose was connected back to the collapsible bladder in order to recycle excess water during the experiment. A tarp was secured over the top of the frame for some simulations to keep natural rainfall off the plot. For the 2019 field season, the frame was reconfigured to mount to a truck hitch (Figure 2.4b). Additionally, the collapsible bladder was swapped for a polyethylene tank. Tarps were wrapped around the frame (2018) or hung from the frame (2019) to block the wind (e.g., Battany and Grismer, 2000; Cerdà et al., 1997; Covert and Jordan, 2009; Humphry et al., 2002) and prevent rain from being blown away from or across the plot area (Battany and Grismer, 2000; Howard, 2018).

The 1 m wide by 1.5 m long plot frame consisted of steel C-channel on three sides (shown in Figure 2.4). Short plots may not be completely representative of road-scale erosion because, for example, the higher overland flow velocities associated with longer flow paths increases erosivity and particle transport (Sharpley and Kleinman, 2003) and the short flow path precludes rill formation (Foltz et al., 2009). However, this was the longest plot that would fit in the vehicle. After centering the plot frame under the nozzle using a plumb bob (Covert and Jordan, 2009), each side of the plot frame was sealed to the road surface by tucking refrigeration insulation alongside and underneath the C-channel. Plot slope was measured (for 2019 plots) using a level and an angle square. The road surface materials were sampled to approximately 5 cm depth outside of the plot

area; samples were stored in 4 mil polyethylene, resealable soil bags and gravimetric moisture content (Topp et al., 2006) was determined in the laboratory on 10 g subsamples. The collection pan, which can be seen in Figure 2.4, was installed a few centimeters below the road grade on the open (downslope) side of the plot. Installing the collection pan slightly below the road grade allowed runoff to flow onto the pan instead of becoming dammed by the seal, and allowed the pan to be sealed to the compacted road surface below the float. The collection pan was sealed to the road using modelling compound (e.g., Play-Doh) and spreading petroleum jelly along the leading edge of the seal. A hole, large enough to fit a 1 L high-density polyethylene (HDPE) bottle, was dug below the spout of the collection pan. The collection pan was covered with a rigid plastic sheet to exclude rain from outside of the plot area (Howard, 2018).

Rainfall simulation sites were selected, in consultation with local road managers, based on proximity to a water source and traffic (safety) considerations. Each rainfall simulation lasted 30 minutes. During the simulations, runoff samples were collected in 500–600 mL HDPE bottles every 2 minutes and the time to fill the bottles was recorded (e.g., Howard, 2018; van Meerveld et al., 2014). Runoff generated in between timed samples was collected into 1 L HDPE bottles and transferred to 20 L buckets. After the end of the run, runoff was collected until it slowed to a drip. Any sediment remaining on the collection pan was rinsed into sample bottles. Runoff sample and bucket volumes were measured in the field using a 1 L graduated cylinder. The buckets were emptied on site and runoff samples were packaged into cardboard boxes or dark plastic bins to prevent algal growth.

2.2.3.2 Rainfall simulator calibrations

The rain rate at a given pressure and flow rate was determined by placing twelve cylindrical containers in a grid within the 1.5 m² plot area. Rainfall depth was determined by measuring the volume in each of the cups using a 25 mL graduated cylinder and calculating the depth using the volume of water collected (where 1 mL water = 1 cm³) and the container surface area (in cm²). The initial calibration consisted of ten 5-minute runs conducted in April and August 2018. These calibration runs produced a calculated plot-average rain depth of 13 mm in 30 minutes at a pressure of 72.4 kPa (10.5 p.s.i.) and a flow rate of 2.6 L/min (0.7 gpm). This rainfall intensity was consistent with the estimated median 2-year return period event for the forested region of Canada (minus British Columbia) based on the maps of Bruce (1968). However, the calibration result was later questioned as measurements of total runoff volume exceeded the theoretical rain volume. Thus, a new calibration was completed in the fall of 2019 with three 10-minute runs and one 15-minute run. The calculated average rain depth was 18 mm in 30 minutes. This rainfall intensity corresponds to a 5–10-year return period event (FL watershed) to less than a 2-year return period event (northern Boreal Plains and eastern Canada) across the rainfall simulation sites (Simonovic et al., 2018).

Spatial uniformity of the simulated rainfall was measured using Christiansen's uniformity coefficient (C_u ; Christiansen, 1942). This was determined by placing 135 cups in a grid to cover the 1.5 m² plot area and generating rainfall for 10 min at pressure of 72.4 kPa (10.5 p.s.i.) and a flow rate of 2.6 L/min (0.7 gpm). Three 10-minute runs were completed in August 2018 and one run was completed in the fall of 2019. The average C_u of the four runs was 59% (s.d. = 2%). The C_u obtained with this nozzle and plot size is poor compared to values achieved by others (e.g.,

Cerdà et al., 1997; Humphry et al., 2002; Sharpley and Kleinman, 2003; van Meerveld et al., 2014; Zemke, 2016), which are over the commonly accepted C_u of 80% (Liu et al., 2017).

Raindrop size was determined using a stain method (Lowe, 1892; also described by Kathiravelu et al., 2016). Sheets of cardstock were shaken with potassium permanganate then exposed to simulated rain and allowed to dry. A correction factor was required to account for the splattering that occurred when the drops hit the paper; this was calculated as the ratio between drops of a known size and the diameter of their imprint on treated cardstock when dropped from a height of 3 m. First, drops were produced using a transfer pipette and their diameters were measured, using calipers with a precision of 0.05 mm, from photographs (which included a ruler in the background for scale) of the drops in mid-air. The mean diameter of these drops was 4.7 mm (s.d. = 0.7 mm; n = 26 drops). Then, the same pipette was used to drop water onto potassium permanganate-covered cardstock from a height of 3 m. The mean diameter of these drops was 23.5 mm (s.d. = 2.6 mm; n = 33 drops). Thus, the calculated correction factor was 0.2.

To measure simulated raindrops, the twenty sheets of cardstock (ca. 23 x 15 cm) exposed to simulated rain were divided into six roughly equal, numbered cells. One cell was randomly selected from each sheet, and the drops in each of these cells were measured. Corrected drop diameters ranged from 0.1–2.2 mm, and the majority (90%) of drops were between 0.1–0.6 mm. The corrected, median simulated raindrop size was 0.3 mm (n = 3843 drops measured). Drop diameter was small compared to other Canadian studies, in which median simulated raindrop sizes ranged from 0.9–1.1 mm (depending on the intensity; van Meerveld et al., 2014) up to 1.4 mm (Covert and Jordan, 2009). With the same nozzle used in this study, Howard (2018) produced a drop size distribution between 0.8–2 mm, with most drops in the range of 1.0–1.4 mm. The difference in the predominant drop size range is likely due to changes in the flow and pressure

used to produce different rainfall intensities and coverage for different plot sizes: Howard's (2018) rainfall simulations were conducted at a lower intensity (approximately 22.5 mm/hr) on 1 m² plots, versus an intensity of 36 mm/hr and a plot size of 1.5 m² used in this study. Nonetheless, the drop size distribution produced in this study falls within the expected range for natural rainfall, i.e., 0–7 mm (Meyer, 2017). It is also consistent with low-intensity (<40 mm/hr) rainfall simulations, in which drop sizes tend to be below 1 mm (Grismer, 2012).

2.2.4 Sample processing and analysis

2.2.4.1 Road surface materials and cutslopes

Samples were dried in aluminum tins lined with parchment paper for 72 hours at 22–36°C. The dried road surface and cutslope samples were sieved, weighed, and partitioned as per Figure 2.5. Stainless steel sieves, receivers, and covers (0488110G—#10 sieve, 0488110CC—#230 sieve, 04887B—receiver, 04887A—cover, Fisher Scientific Company, Toronto, ON, CAN) were washed between samples with phosphate-free liquid dish soap and water. Dry fractions were transferred to labeled, resealable polyethylene bags and stored in boxes at room temperature.

2.2.4.2 Road surface sediment samples

Suspended sediment mass was determined using an evaporation method based on ASTM D3977-97–A (2013). After settling, the supernatant was transferred from sample bottles into a 20 L bucket. Supernatants of all samples from individual plots were mixed in the same bucket to determine a pooled total dissolved solids (TDS) value for each site using the average of three 15 mL subsamples. The remaining sediment-water mixtures were rinsed into separate pre-weighed aluminum tins and evaporated either on a rack or benchtop, in a fume hood, or in a drying oven below 90°C. Once all visible water was evaporated, samples were placed in an oven at 105°C for

2 hours, allowed to cool to room temperature on a benchtop, and weighed. The net dry sediment mass was calculated as the difference between the 2-hour dry mass and the dish mass. The average pooled TDS value was $\leq 0.1\%$ of the measured net dry sediment mass for all sites, therefore no TDS corrections were applied.

After determining net dry sediment masses, the sediments from each site were pooled, homogenized with a mortar and pestle, sieved to < 2 mm, and transferred to labeled polyethylene bags.

2.2.4.3 Particle size analysis

Sediment, road surface material, and cutslope samples were subsampled (ca. 1 g) using a spinning riffler (SP-230, Gilson Co., Lewis Center, OH, USA). The subsamples were analyzed by the Natural Resources Analytical Laboratory (NRAL) at the University of Alberta using a laser diffraction particle size analyzer (LS 13 320, Beckman Coulter, Indianapolis, IN, USA). As such, the silt (particles < 63 μm) contents are reported on a percent by volume basis. All samples were riffled and analyzed in duplicate. Three re-runs were analyzed in triplicate. The subsample replicates were averaged to calculate the percentage of silt. Repeatability was evaluated using the relative standard deviations (RSDs) provided by NRAL for each particle size fraction (classified according to Soil Classification Working Group, 1998). Generally, when the RSD of at least one fraction exceeded 15%, the sample was re-run. However, since some RSDs were inflated due to small values (e.g., very little clay or sand), the RSDs were evaluated on a sample-by-sample basis. Eleven samples from the 2018 field season were re-run, and four samples were re-run twice. For samples with re-runs, results from the run with the best RSD were reported.

2.2.5 *Statistical analysis*

Differences in the distribution of silt were described at the ecozone scale using Kruskal-Wallis tests followed by pairwise Wilcoxon rank-sum tests with a Benjamini-Hochberg (BH) correction (stats package; R Core Team, 2020). The difference in silt content between road surfaces and cutslopes was assessed using a two-sided paired t-test, and the difference in silt content between sediments and road surface materials with a one-sided paired t-test (stats package; R Core Team, 2020). The results were evaluated using a significance level of $\alpha = 0.05$.

2.3 **Results**

2.3.1 *Silt availability in road surface materials and cutslopes*

2.3.1.1 *Silt availability in road surface materials*

Median silt content, as a percent of the <2mm particle size fraction, in road surface materials for the five ecozones sampled is summarized in Table 2.1. Silt distributions varied across ecozones ($p < 0.001$). Higher silt contents occurred most frequently in the western ecozones (Pacific Maritime, Montane Cordillera, and Boreal Plains). Although the pairwise tests indicated that the silt distributions of the Pacific and Atlantic Maritime ecozones were similar, the p -value was marginal ($p = 0.08$). Likewise, the result indicating that the silt distributions of the Boreal Shield and Atlantic Maritime regions were different was borderline ($p = 0.03$). Across watersheds, median silt content in road surface materials ranged from 7% (KH and RM) to 55% (MC) (Figure 2.6).

Silt content varied markedly—by up to 72%—within the western ecozones, in which the sample ranges were 2–3 times higher than in the eastern ecozones (Table 2.1). While substantial variability may be expected at broad (i.e., ecozone) scales, local variability could be equally

extensive: silt contents within the SG, UP, FC, UW, and KR watersheds differed by as much as ca. 50–74%.

2.3.1.2 Silt availability in cutslopes

Across ecozones, the silt contents in the <2mm particle size fraction of the cutslope samples analyzed were approximately 3–3.5 times higher in western interior ecozones (Montane Cordillera and Boreal Plains) than in the Pacific Maritime and Boreal Shield (Table 2.1). The highest cutslope silt contents were observed in the lower East Kootenay region of British Columbia (MC and KC; Montane Cordillera), and the lowest cutslope silt contents occurred in the Victoria, British Columbia area (SG) in the Pacific Maritime ecozone and north of Québec City (RM) on the Boreal Shield (Figure 2.6). Cutslope silt was similar in some regions—for example, in the MC and KC watersheds, which are adjacent each other, or in the RD watershed (Figure 2.6)—but could differ by up to ca. 30% within a watershed (e.g., UW; Figure 2.6).

In road-cutslope pairs (i.e., road surfaces and cutslopes from the same road segment; Figure 2.7), cutslope silt content was higher than that of road surface materials ($p = 0.01$). The mean difference in silt content between road surface materials and cutslopes was 39%.

2.3.2 Road surface sediment silt content

Sixteen rainfall simulations were conducted. Two plots were excluded from the following analyses and figures: one experiment did not produce runoff, and the other had a poor seal between the road and the plot frame and the flow and pressure fluctuated during the experiment.

Sediments generated during rainfall simulations were predominantly silt (median = 95% by volume), though silt content varied by up to 87% compared to the median (Figure 2.8). Despite the wide range of silt contents observed across the rainfall simulation plots, the majority (10 of 14)

of sediments contained approximately 75% silt or higher (Figure 2.8). Across all samples, silt content was significantly greater in sediments than in the road surface materials from which they originated ($p < 0.001$; Figure 2.9); on average, sediments contained 3.6 times more silt than road surface materials (s.d. = 2.3).

There was no clear trend between the percentage of silt in road surface materials and sediment silt content. Sediments generated by road surface materials across the typical range of silt contents (ca. 10–40%) all produced sediments with silt contents between approximately 75–100%, and road surface materials with silt contents of approximately 10% silt yielded sediments with 22–98% silt (Figure 2.10). The RM plot did not generate any sediment—and therefore no silt—even though silt content in road surface materials was similar to other sites (i.e., KH and PT; 6–7%) that produced sediments with 8–9% silt. Sediment silt content was generally lowest (<60%, which corresponds to the first quartile) when road surface materials had less than 10–11% silt (with the UW plot being the exception; Figure 2.10). However, sediments generated from these road surface materials could still be enriched in silt by up to five times (e.g., TC; Figure 2.10).

Plot slope and moisture content in road surface materials did not have a discernable effect on silt content. Of the five plots with slope measurements, three plots with slopes between 7° and 15° produced sediments with anywhere from ca. 10% to 75% silt, while two plots with a slope of approximately 30° produced sediments with <10% to ca. 55% silt. Additionally, only one plot had distinct rutting (RE) and it still produced sediments with over 70% silt. Moisture content of road surface materials adjacent plots ($n = 14$ samples) ranged from 1.7–7.5%. Plots with the lowest and highest moisture contents produced two of the lowest sediment silt contents. Furthermore, plots across a range of moisture contents (2.6–6.8%) yielded sediments with over 90% silt.

2.4 Discussion

2.4.1 *Silt availability in road surface materials and cutslopes*

Since texture is indicative of the range of particle sizes that are available to be eroded (Grismer et al., 2008), silt content in road surface materials and cutslopes can provide a qualitative measure of road-related silt supply in watersheds. Silt (particles $<63\ \mu\text{m}$) is of primary interest in this study because of 1) its potential to increase turbidity, as low settling velocities increase the likelihood that silt will remain entrained in runoff (Proffitt et al., 1991) and subsequently be delivered to surface waters, and 2) its role in sediment-bound P transport, as the $<63\ \mu\text{m}$ particle size fraction generally contains the highest P concentrations (e.g., Dudley et al., 1997; Kerr et al., 2011; Stone and Mudroch, 1989). The silt content results demonstrate that road surfaces and cutslopes are likely sources of silt in many forested watersheds across Canada. The drinking water hazards associated with these pools of available silt—namely turbidity and P loading to surface waters—appear to be higher in the western ecozones, and within ecozones, in certain watersheds. One of the most prominent characteristics of the silt content data is the degree to which silt content can vary across road segments within watersheds. However, silt content can also change over time, so low silt availability at a single point in time may not be representative of the long-term supply; this is especially the case for road surface materials.

2.4.1.1 *Silt availability in road surface materials*

Silt is an important constituent of road surface materials because it provides cohesion, which creates a smooth running surface and allows the road to shed water (Skorseth and Selim, 2004), and adds strength (Fannin and Lorbach, 2007). So, all road surfaces are expected to contain

silt. However, for reasons already discussed, high silt contents¹ may pose a risk to water quality. At the regional scale, silt availability appears to be a greater issue in western Canada compared to the east (Table 2.1, Figure 2.6). Nonetheless, the variability within watersheds (Figure 2.6) indicates that, even where silt contents are predominantly low, there are likely road segments with comparatively high proportions of silt that can be mobilized in runoff.

Determining the causes of variation in the measured silt contents is beyond the scope of this study; however, the distribution of silt in road surface materials could be the product of several spatial and temporal factors or processes (Table 2.2). For instance, the high median silt content in the MC watershed (Figure 2.6) might reflect road surface materials derived from the fine-textured surficial geology in the watershed (i.e., siltstone and wacke; B.C. Geological Survey, 2011; Cui et al., 2017; Hoadley, 1947). Road managers may choose to source higher quality materials for major roads, while temporary road surfaces consist of local parent materials that contain higher proportions of silt. This could explain the elevated silt contents of operational roads in the EL, RM, and PT watersheds, which had silt contents at least twice as high as the second-highest road in the watershed (Figure 2.6). Differences in traffic volumes could also explain some of the variation in silt content, as road use pushes coarse fragments into the running surface which forces fine particles upward (known as dynamic pumping or subgrade mixing), breaks down aggregate, and abrades particles from road surfaces (Baird et al., 2012; Bilby et al., 1989; Croke et al., 2005; Elliot et al., 2009; Foltz, 1996; Ramos-Scharrón and MacDonald, 2007; Reid and Dunne, 1984; Reid et al., 1981; Rhee et al., 2018; Ziegler et al., 2000; Ziegler et al., 2001). For example, six samples from a single road in the UB watershed had silt contents ranging from 14–17% at sites ca. 40 km up the road to 27–45% at the lower end of the road. In addition, road surface silt content is

¹ Légère and Mercier (2004) concluded that surface materials for forest roads should contain 4–15% fines (defined as particles passing through a #200, or 75 µm, sieve) by weight.

expected to change over time as the loose material built up on road surfaces under the influence of traffic washes off during rain events (e.g., Bilby et al., 1989; Ziegler et al., 2000; Ziegler et al., 2001), and as new material is exposed and subsequently eroded (Maloney et al., 2018). These processes might explain the difference in silt content on the same road (HR mainline) measured in this study (25–27%) versus another study (ca. 50%; van Meerveld et al., 2014). Given that the roads sampled represent a broad range of material types, construction methods, traffic volumes, and precipitation regimes, the distributions of silt at both the ecozone and watershed scales likely reflect a combination of the factors listed in Table 2.2.

The factors and processes that determine silt content in road surface materials have important implications for 1) managing the potential hazards for water quality, and 2) drawing conclusions about silt availability in the watersheds sampled in this study. Firstly, some of the factors that determine silt content can be managed in order to reduce the amount of silt produced by road surfaces: surfacing roads with materials that are more resistant to crushing and limiting traffic volumes may help to minimize silt production (Table 2.2). However, access to good quality materials can be limited by geography or by cost (Foltz, 1996); for example, friable siltstone aggregate was used on the HR mainline because better quality materials were not available on Graham Island (van Meerveld et al., 2014). Limiting traffic in watersheds might also be challenging due to pressure to maintain public access or shared use between resource sectors (e.g., forestry, energy, mining). Secondly, the continual buildup and wash-off of silt from road surfaces introduces an unknown amount of uncertainty into silt content results. Estimating long-term (e.g., seasonal or annual) silt availability will likely require several measurements over multiple years in order to capture the temporal variability in road surface silt content. The results presented in Table 2.1 and Figure 2.6 should be interpreted with caution given that samples were only collected

once during the study. Also, materials can be redistributed across road surfaces via sweeping (Rhee et al., 2018). Two subsamples were collected from each road segment in this study, but this may have been inadequate to capture the variability in particle size across road surfaces with a high degree of redistribution (e.g., Figure 2.11).

2.4.1.2 Silt availability in cutslopes

Cutslope samples were limited because they were not ubiquitous among or within watersheds. Also, although silt availability was generally higher in cutslopes than in corresponding road surfaces (Figure 2.7), cutslopes likely present less of a hazard than road surfaces as the latter tend to yield higher amounts of sediment (e.g., Croke et al., 2006; Fahey and Coker, 1992; MacDonald et al., 2001; Ramos-Scharrón and MacDonald, 2007; Reid and Dunne, 1984). Their steep slopes make them prone to erosion (Arnáez et al., 2014; Jordán and Martínez-Zavala 2008; MacDonald and Coe, 2008; Megahan et al., 2001), but they have short slope lengths which prevents flow from accumulating, thereby limiting erosion potential (MacDonald and Coe, 2008). Additionally, whereas coarse fragments in road surface aggregate must often be below a specified size (e.g., 20–40 mm; Alberta Transportation, 2019), the large rock fragments or rock debris on cutslope surfaces can shield fine materials from erosion (Jordán and Martínez-Zavala, 2008; Reid and Dunne, 1984). Thus, the focus of the study was on road surfaces.

Nonetheless, despite the small sample sizes, the results indicate that cutslopes are potential—and sometimes substantial—sources of silt (Figure 2.6). Cutslope silt contents are likely most strongly controlled by geology. The influence of geology was evident in some cutslope samples: for example, surficial geology in the MC and KC watersheds, in which the cutslopes sampled contained over 90% silt (Figure 2.6), is predominantly fine-textured sedimentary rock

(e.g., argillite, wacke, siltstone; Cui et al., 2017; Province of British Columbia, 2015). The texture of the local surficial materials could also explain the high silt content (70%) of the cutslope sampled in the FL watershed, as the area is predominantly underlain by till with a sandy silt matrix (Plouffe, 1996). However, weathering and erosion processes also affect silt content. After loose or unstable materials are depleted from cutslope surfaces in the year or so after construction (Megahan et al., 2001), silt can be generated through freeze-thaw or wetting-drying cycles (Megahan et al., 2001). This could explain, for instance, why cutslopes in the RD watershed have higher silt content (76–86%; Figure 2.6) than might be expected based on the texture (ca. 50% silt) of the surficial materials' matrix (Bayrock and Reimchen, 1975). Also, in steep terrain, a fresh supply of silt can be created when cutslopes fail and new material is exposed (MacDonald and Coe, 2008).

Since the main factors and processes controlling cutslope silt content are natural (i.e., geology and weathering), there are fewer opportunities to control silt availability compared to road surfaces. However, the potential for silt—and potentially P-bearing sediments—from reaching surface waters can still be minimized by reducing erosion. This can be achieved by, for example, revegetating cutslopes (Baird et al., 2012; Burroughs and King, 1989; Elliot et al., 2009; Luce and Black, 1999; MacDonald and Coe, 2008; Megahan et al., 2001; Reid and Dunne, 1984; Reid et al., 1981; Sheridan and Noske, 2007) or applying surface treatments like wood chips or fine organic material (e.g., compost; Grismer et al., 2008). Burroughs and King (1989) review several cutslope erosion control strategies, and also note that terracing can be effective at containing sediment by promoting deposition.

2.4.2 Road surface material and cutslope sediment silt content

Although silt content was intended to provide a qualitative measure of road-related silt supply in watersheds, silt availability may not be indicative of how much silt is transported in runoff relative to sand-sized particles (63 μm –2 mm; Wentworth, 1922). This is especially true for road surfaces, as evidence suggests that silt is preferentially mobilized in runoff (e.g., Bilby et al. 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016). The predominantly high silt contents of sediments (ca. $\geq 75\%$; Figure 2.8) and enrichment compared to road surface materials (Figure 2.9, Figure 2.10) are consistent with selective transport. Sediment silt content and enrichment varied though (Figure 2.8, Figure 2.10); this could reflect differences in plot characteristics such as slope (e.g., Bilby et al., 1989; MacDonald et al., 2001), erosion processes (Proffitt and Rose, 1991), or material properties (e.g., organic matter content; Guerra, 1994; Parsakhoo et al., 2014). Also, the results indicate that silt production from road surfaces with low silt contents (ca. 10% or less) can be limited. Cutslope sediment silt content was not examined in this study, and results from studies on cutslope or hillslope sediments are mixed. Road surfaces and cutslopes that produce relatively small proportions of silt are less likely to cause turbidity issues. However, they can still contribute large amounts of potentially P-bearing silt to surface waters over the long term because roads are a chronic sediment source (Al-Chokhachy et al., 2016; Brown et al., 2013; Carson and Younie, 2003; Elliot, 2013; Gucinski et al., 2001; Karr et al., 2004; MacDonald and Coe, 2008).

2.4.2.1 Road surface sediment silt content

Costantini et al. (1999) attributed the elevated percentage of particles $< 50 \mu\text{m}$ that they observed in sediments to “road surfaces operating as a high detachment/high deposition

environment where selective transport is extremely high”. In other words, high percentages of silt in road-derived surface sediments reflect erosion, deposition, and entrainment processes in which particles are detached non-selectively by splash erosion, and coarser particles with high settling velocities (e.g., sand) are deposited at a much higher rate than the silt that remains entrained in runoff (Proffitt et al., 1991). While high silt contents in sediments (Figure 2.8) suggest that silt was selectively mobilized from most of the road surfaces, silt enrichment (Figure 2.9, Figure 2.10) provides direct evidence that selective transport occurred. These findings are consistent with other studies that showed that road-derived sediments are enriched in silt. For example, Costantini et al. (1999) showed that while road surface materials consisted of 8% particles <50 μm , the same size fraction made up 86% of the particles in sediments. Zemke (2016) found similar results: road surface materials consisting of approximately 15–30% silt produced sediments that contained ca. 65% silt. Although high sediment silt contents resulting from selective transport prevailed across most of the rainfall simulation plots, silt content varied considerably (Figure 2.8).

The conditions that limit silt mobilization are of interest to road managers, since silt-limiting characteristics can be leveraged in road construction or material selection to minimize silt transport from road surfaces. There were no obvious trends among the high silt- and low silt-yielding plots, except that silt production was often limited in sediments originating from road surface materials with low silt contents (ca. 10% or less; Figure 2.10). However, sediments from a road surface with similarly low silt content (i.e., UW) contained between ca. 2–12 times as much silt (Figure 2.10). The disparity in sediment silt content among the plots—particularly those with similar silt contents—could be due to differences in the amount of loose silt accumulated on road surfaces rather than the initial silt content. Surfaces with less loose material should become armoured more quickly, thereby favoring coarser particles. Surface armouring occurs when small

particles with low settling velocities that are suspended in runoff (i.e., silt) become depleted while coarser particles with higher settling velocities (sands and gravels) are deposited forming a protective layer that prevents further particle detachment through splash erosion (Proffitt and Rose, 1991; Zemke, 2016; Ziegler et al., 2001). As the deposited particles are re-entrained in runoff and particle detachment from the original surface is limited—so little to no additional silt is mobilized—the sediment becomes coarser (Proffitt and Rose, 1991).

Other plot-scale or material characteristics can also influence sediment particle size distributions, and therefore silt content. Steeper slopes produce sediments with coarser particle size distributions (e.g., Bilby et al., 1989; Kiani-Harchegani et al., 2018; MacDonald et al., 2001) because rills develop more quickly (Shi et al., 2012), and rill erosion results in higher flow velocities capable of entraining coarser particles compared to interrill erosion (Kiani-Harchegani et al., 2018; Proffitt and Rose, 1991; Shi et al., 2012; Wang and Shi, 2015). Similarly, ruts can produce coarser sediments (e.g., Costantini et al., 1999) because flow velocities increase in concentrated flows (Foltz, 1996; Ramos-Scharrón and MacDonald, 2007; van Meerveld et al., 2014). Material characteristics that influence aggregate stability and particle detachment by splash erosion could include: organic matter content (Guerra, 1994; Parsakhoo et al., 2014), Ca or CaCO₃ content (Paige-Green, 2008; Parsakhoo et al., 2014), moisture content (Parsakhoo et al., 2014), the amount and type of clay (Briaud, 2008; Burroughs et al., 1992; Guerra, 1994), and the presence of dispersive clays or silts (Paige-Green, 2008). That sediment silt content from the rainfall simulations did not follow expected trends related to slope and moisture content could indicate complex interactions between material characteristics, plot characteristics, and antecedent conditions across the diversity of roads sampled.

Although the sample set was small ($n = 14$ road surfaces), it included a broad range of road and surface material types across a variety of ecophysiological settings. The high relative silt contents and silt enrichment in sediments from the majority of plots show the potential for silt transport from diverse road types and surface materials. Since sediment particle size distributions are the product of a combination of erosion processes (e.g., splash erosion vs. runoff detachment), type of flow (sheet vs. rill), and material type that affect the sediment particle size distribution (Proffitt and Rose, 1991), the differences in silt content among the rainfall simulation plots reflect a variety of factors. Under natural conditions, rainfall intensity and time will also affect road sediment silt contents: sediment particle size distributions become coarser at higher intensity (e.g., Croke et al., 2006), and it has been shown that sediment particle size distributions change over the course of a rainfall event (e.g., Asadi et al., 2011; Ghardiri and Rose, 1991; Proffitt et al., 1991; Proffitt and Rose, 1991; Shi et al., 2012; Wang and Shi, 2015). A constant rainfall intensity and duration were used in this study, so the silt contents are only representative of a single event size and duration. Given the significance of silt and the need to minimize silt mobilization from road surfaces, further study is warranted to determine 1) the relative influences of surface material characteristics and processes that control sediment particle size distributions, and 2) silt production associated with different road surface materials under natural conditions.

2.4.2.2 Cutslope sediment silt content

The general consensus in erosion research is that interrill erosion is selective for particles $<63 \mu\text{m}$, while rill erosion is less selective and becomes non-selective once flow exceeds a shear stress threshold (Govers, 1985; Kiani-Harchegani et al., 2018; Proffitt and Rose, 1991; Wang and Shi, 2015). Thus, surfaces predominantly affected by interrill erosion should produce sediments

that are enriched in silt, and surfaces on which rill erosion predominates should yield coarser sediments. Cutslopes are expected to yield coarser sediments with lower proportions of silt than road surfaces (e.g., Lane and Sheridan, 2002; Ramos-Scharrón and MacDonald, 2007) because rill erosion is prevalent on cutslopes (provided that they are tall enough for rills to develop; MacDonald and Coe, 2008). Also, larger particles are more easily mobilized from higher slopes due to gravity and inertia, and rolling (or bedload) transport becomes more important with increased slope (Kiani-Harchegani, 2018; Shi et al., 2012; Wang and Shi, 2015). These processes can yield sediments with silt contents that resemble those of cutslope materials. For example, Guo et al. (2020) showed that, in the absence of mass movement, sediments from steep ($60\text{--}80^\circ$, or $173\text{--}567\%^2$) hillslopes had nearly identical silt (defined as particles $\leq 50\ \mu\text{m}$) contents to those of the soils. Govers (1985) demonstrated that once flow exceeded a threshold shear velocity ($3.0\text{--}3.5\ \text{cm/s}$ in the soils that were investigated), erosion became non-selective with respect to particle size.

Parity between the particle size distributions of materials and sediments from hillslopes is a key assumption of the Water Quality Effectiveness Evaluation (WQEE; Maloney et al., 2018), which is commonly used in western Canada to assess road erosion to determine how much silt will be produced by cutslopes. However, there is evidence to suggest that texture may not be a good indicator of the relative amount of silt in hillslope or cutslope sediments (e.g., Grismer et al., 2008). For instance, Shi et al. (2012) showed that, over a range of slopes ($10\text{--}25\%$) and at two different times during the experiments ($3\text{--}6$ minutes and $51\text{--}54$ minutes), hillslope sediments were enriched in particles $\leq 54\ \mu\text{m}$. Kiani-Harchegani et al. (2018) demonstrated that sediments generated from hillslopes with $15\text{--}25\%$ grades could still be enriched in particles < 2 to $\leq 16\ \mu\text{m}$ by up to 1.5 times

² A slope of $45^\circ = 100\%$. Slopes greater than 100% indicate that change in height (rise) $>$ run (distance over which change in height is measured). For instance, a slope of 567% represents a $5.67\ \text{m}$ gain in height over a horizontal distance of $1\ \text{m}$.

compared to soils, but the results were dependent on rainfall intensity: parity (or near parity) in particle sizes with soils was observed at high rainfall intensity (60–90 mm/hr). Additionally, Grismer et al. (2008) found that silt (in this case, particles $\leq 50 \mu\text{m}$) was consistently higher in sediments from disturbed areas on hillslopes (e.g., road cuts, ski runs) than in soils. Proffitt and Rose (1991) suspected that the conflicting results of studies on the particle sizes of sediments from interrill and rill erosion may be due to differences in detachment and transport occurring in interrill areas. Interestingly, Grismer et al. (2008) also found that slope was not a good predictor of sediment coarsening—in fact, runoff contained greater percentages of clay (particles $< 2 \mu\text{m}$) and silt (particles 2–50 μm) as slope increased.

Although there is a basis for assuming that cutslope sediments will be coarser than road surfaces—even to the extent that sediment silt contents may resemble those of the exposed materials on cutslope faces—the results of cutslope and hillslope erosion studies on sediment silt contents are inconclusive. The discrepancies in the findings among studies could be attributed to the fact that rill and interrill erosion occur concurrently (Wang and Shi, 2015). Both rill erosion and interrill erosion occur on cutslopes (MacDonald and Coe, 2008), so particle size distributions could vary depending on which process predominates. Furthermore, silt content may depend on characteristics like slope length and gradient because of how they affect rill development (MacDonald and Coe, 2008; Shi et al., 2012) and downslope rolling of coarser particles under the influence of gravity (Kiani-Harchegani, 2018; Shi et al., 2012; Wang and Shi, 2015). But higher slope gradients may not be synonymous with increases in rill erosion and sediments with lower proportions of silt (e.g., Grismer et al., 2008). Silt content in cutslope or hillslope sediments can vary depending on material type (e.g., Wang and Shi, 2015), cover type (e.g., Grismer et al., 2008), and rainfall intensity (e.g., Kiani-Harchegani et al., 2018). Erosion research has typically focused

on slopes <25% (Shi et al., 2012), and there have been few studies that directly assess particle size distributions—and silt content in particular—in sediments from cutslopes. Additional research on steep slopes that are within the expected range of cutslope gradients (ca. 33–275%; Alberta Infrastructure and Transportation, 2007; Arnáez et al., 2004; B.C. Ministry of Forests, 2002; Bilby et al., 1989; Fahey and Coker, 1992; Fannin and Lorbach, 2007; Megahan et al., 2001; Reid et al., 1981) will provide a better understanding of silt content in cutslope sediments.

2.5 Conclusion

Road surface materials and cutslopes are sources of silt in forested watersheds. Road surface silt appears to be higher in western Canadian ecozones (Pacific Maritime, Montane Cordillera, and Boreal Plains). Silt availability is particularly high in some watersheds; in these areas, roads should be managed with the goal of minimizing silt production (i.e., on road surfaces) and erosion in order to limit possible impacts to surface waters. However, silt content can change over time, so one-time measurements cannot adequately predict long-term silt availability. Silt availability in road surface materials can likely be reduced by leveraging characteristics such as material type and traffic volumes. Since cutslope silt contents are mainly controlled by natural factors or processes (e.g., geology, weathering), erosion control measures should be put in place to limit silt transport. Silt is generally preferentially transported from road surfaces, which results in sediments that are enriched in silt and that have high silt contents. This means that silt is transported in higher proportions than the silt content of the original material would indicate—in other words, road surface material silt content is a poor predictor of sediment silt content. Cutslope sediment silt contents were not examined directly and results vary widely among studies.

More information on which characteristics most strongly influence silt availability and production from road surfaces is needed. This will help road managers select better materials and construct roads that minimize the hazards to water quality and treatability (e.g., turbidity, possible P loading) posed by road-derived silt. Finally, silt content in sediments from steep slopes should be studied further to better understand silt mobilization potential from cutslopes.

Tables and Figures

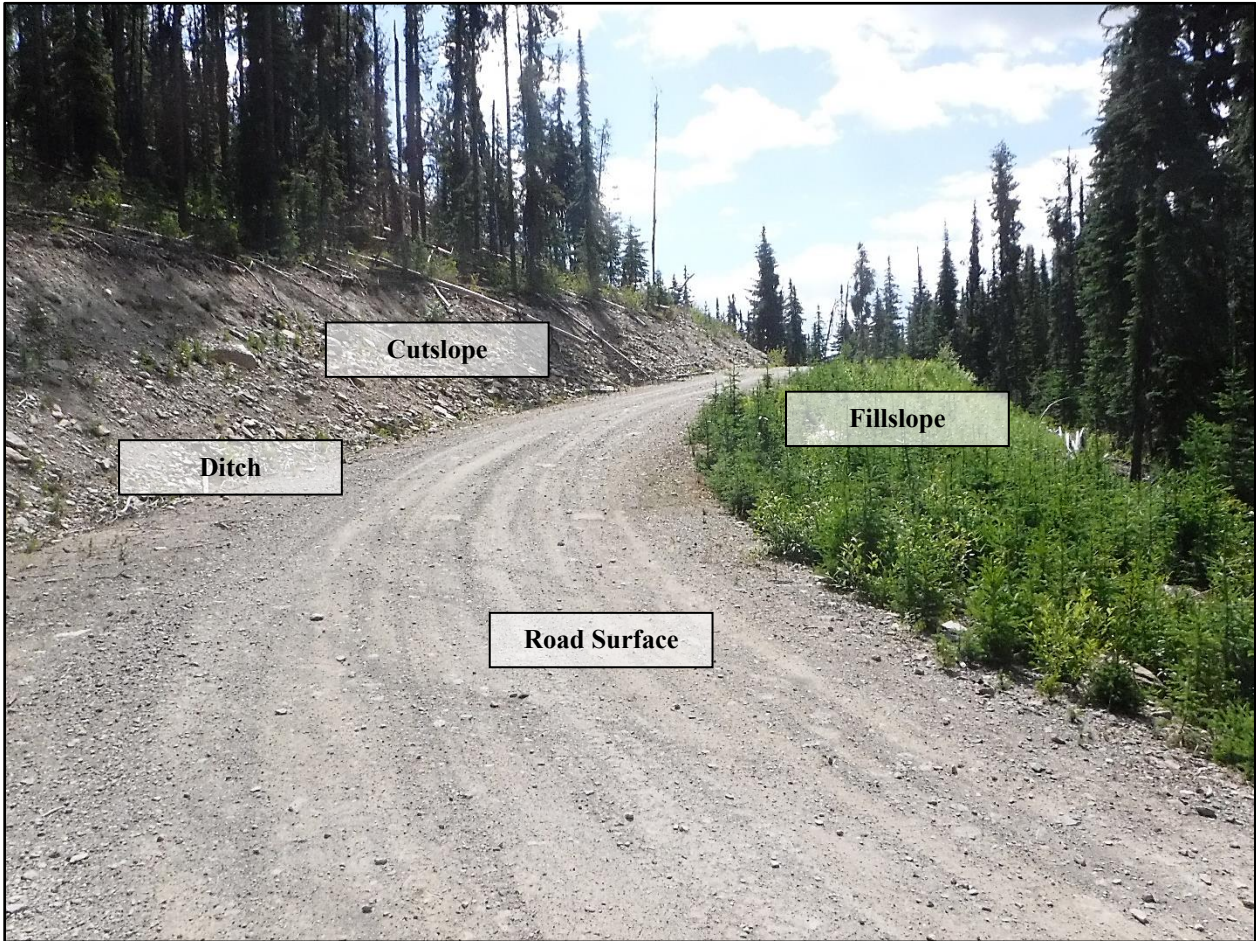


Figure 2.1. Forest road prism (cut and fill road).

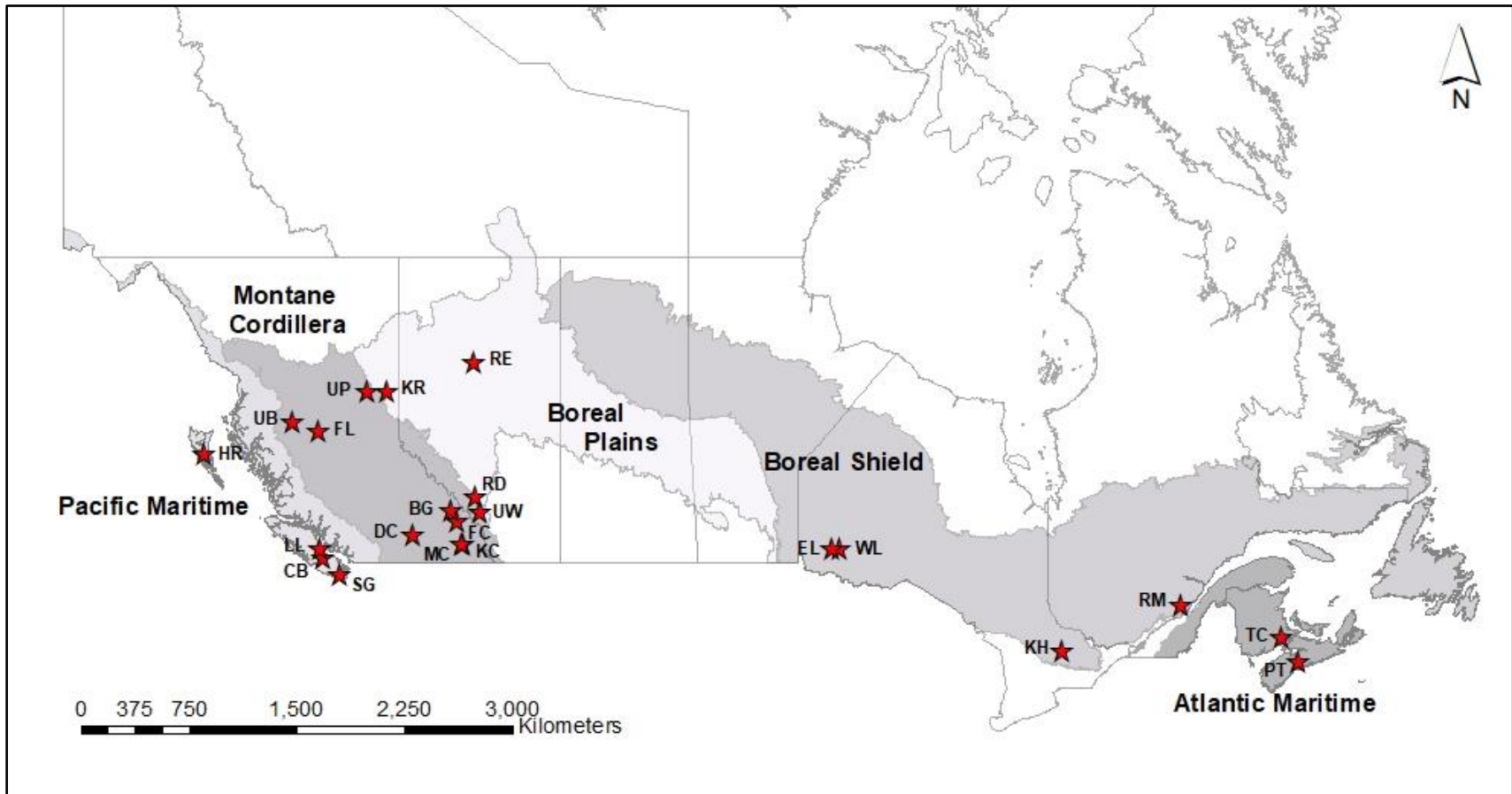


Figure 2.2. Locations of forested ecoregions and watersheds sampled. Note that data for separate watersheds serving the same water utility were summarized as single groups (i.e., CB, SG, DC, UW, and PT).



Figure 2.3. Sweep sampling on a road surface.



Figure 2.4. Freestanding rainfall simulator used in 2018 (a) and hitch-mounted rainfall simulator used in 2019 (b) with plot frame and collection pan; windbreak is shown in (b).

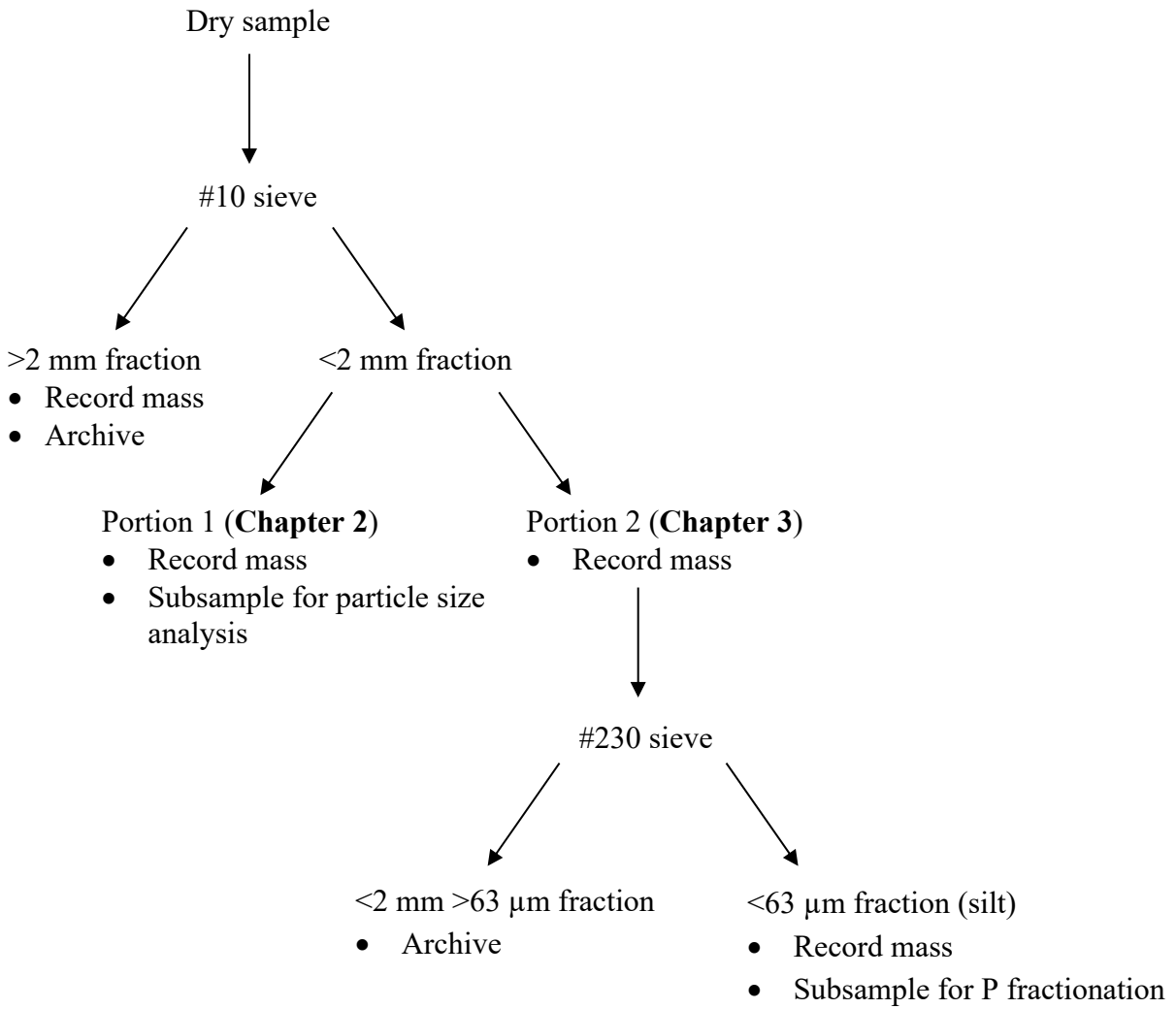


Figure 2.5. Partitioning scheme for road surface material and cutslope samples.

Table 2.1. Median silt content (% by volume of the <2 mm fraction) in road surface materials and cutslopes by ecozone. Letters represent similar silt distributions ($\alpha = 0.05$); ranges (minimum, maximum) are in brackets. NA = not available (no samples collected).

Terrestrial Ecozone					
	Pacific Maritime	Montane Cordillera	Boreal Plains	Boreal Shield	Atlantic Maritime
Roads	22ab (9, 61)	25a (7, 70)	27a (9, 81)	7c (5, 31)	11bc (6, 30)
n	15	37	20	17	11
Cutslopes	22 (14, 29)	79 (33, 96)	76 (48, 86)	24 –	NA –
n	2	9	3	1	NA

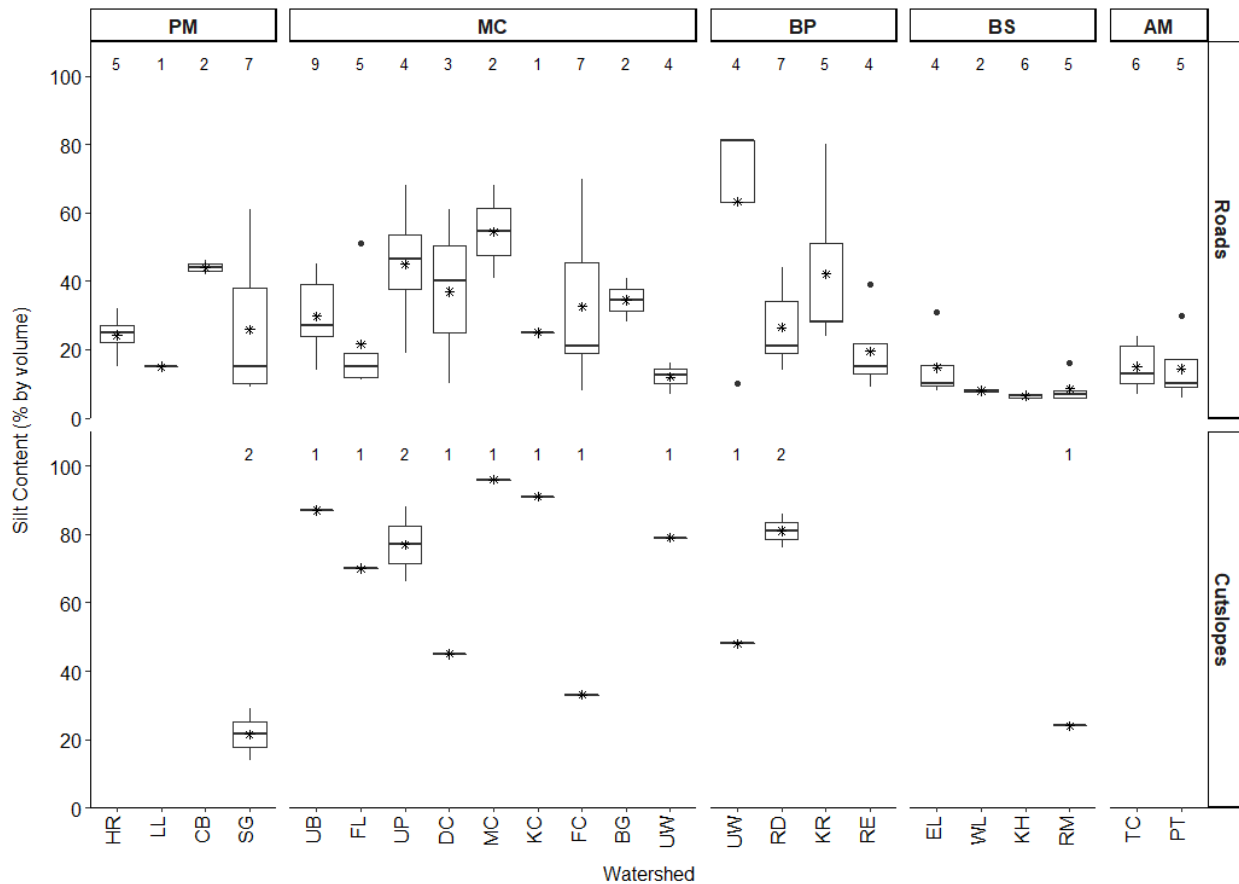


Figure 2.6. Silt content (% by volume of the <2 mm fraction) in road surface materials and cutslopes by watershed. Note that the UW watershed spans two ecozones; median watershed silt content for road surfaces is 15%. Values within plots denote (n). Bold bars indicate medians and asterisks (*) denote means. Data for separate watersheds serving the same water utility were summarized as single groups (i.e., CB, SG, DC, UW, and PT).

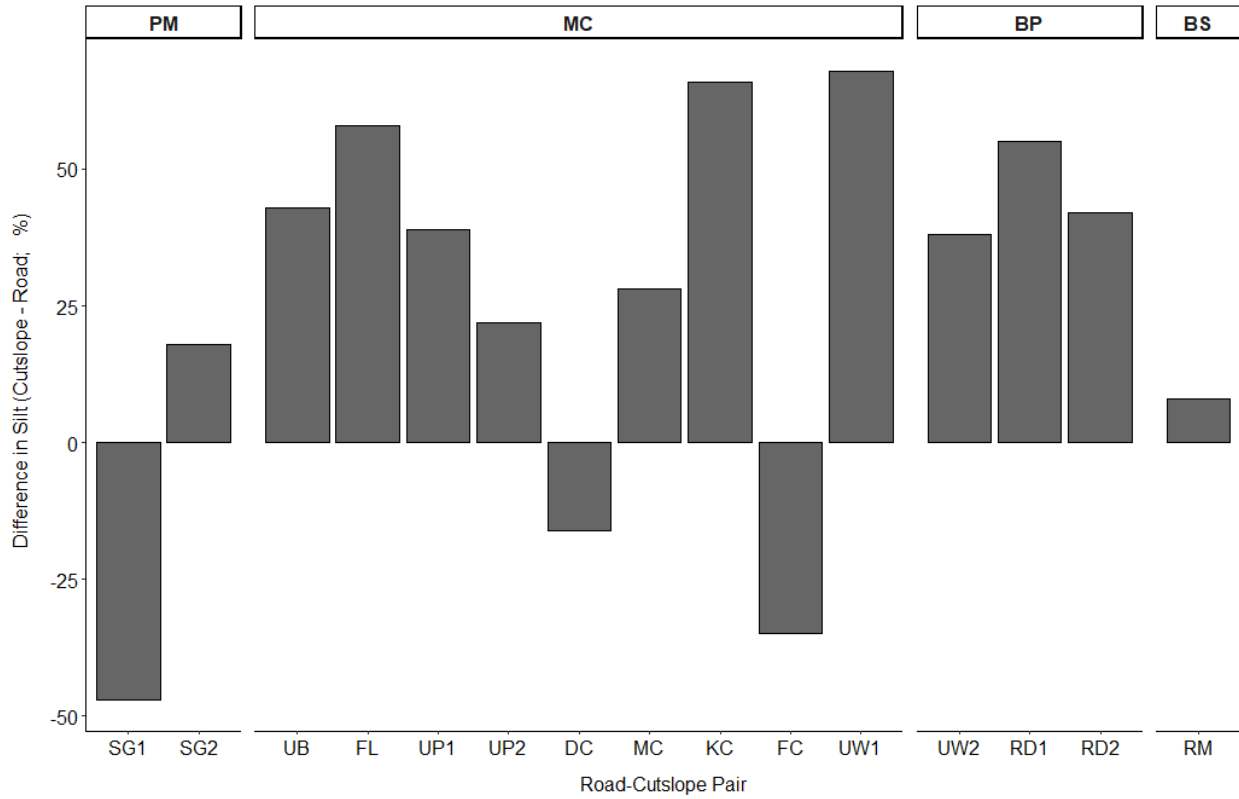


Figure 2.7. Difference in silt content (%) between road surface materials and cutslopes from the same road segment. Positive values indicate cutslope silt content > road surface silt content.

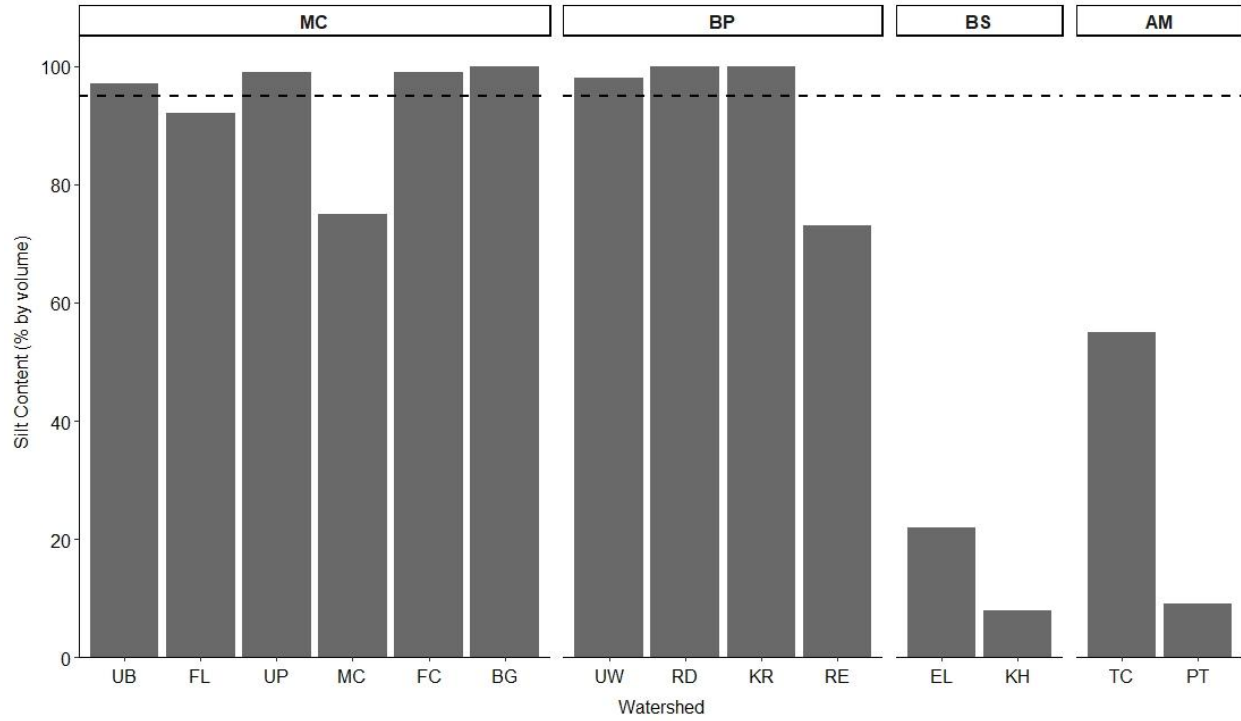


Figure 2.8. Silt content of sediments generated under simulated rainfall. The dashed line denotes the median silt content across the dataset (95%).

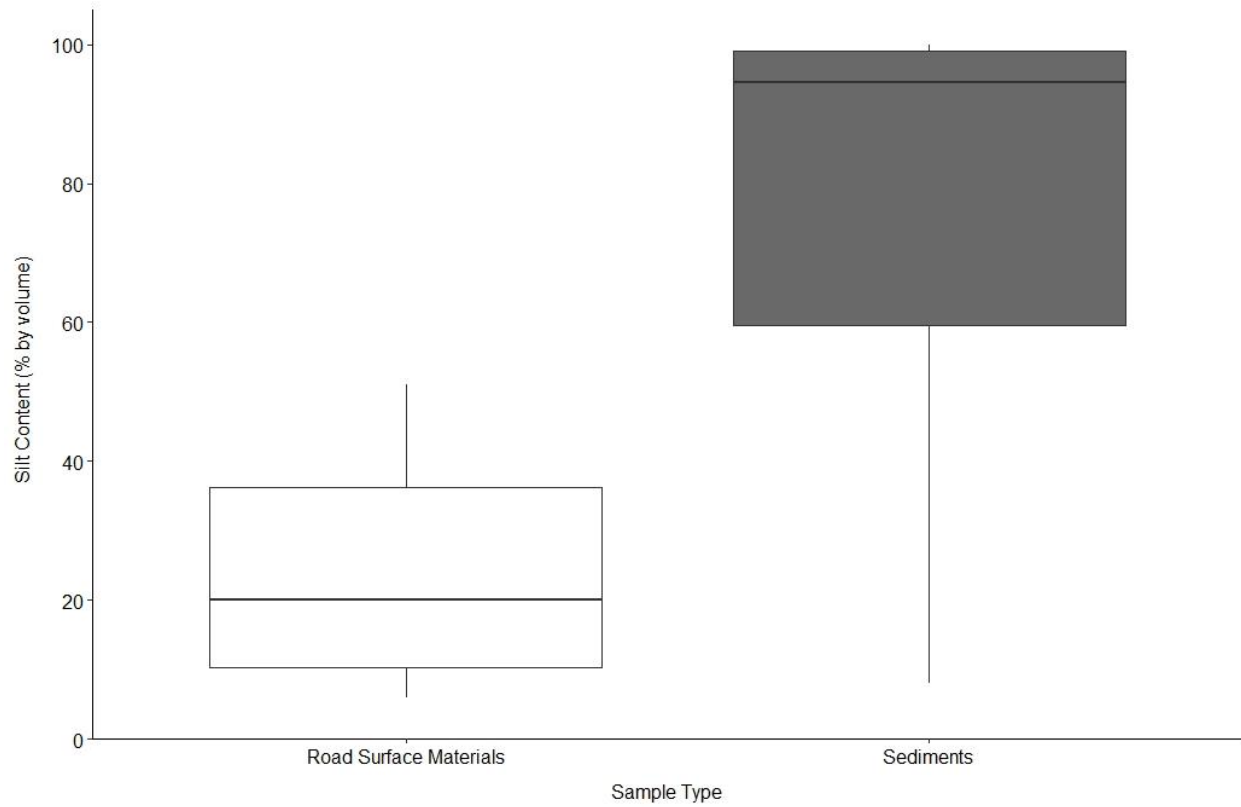


Figure 2.9. Sediment silt content is significantly higher ($p < 0.001$; paired t-test) than that of the road surface materials from which they originated.

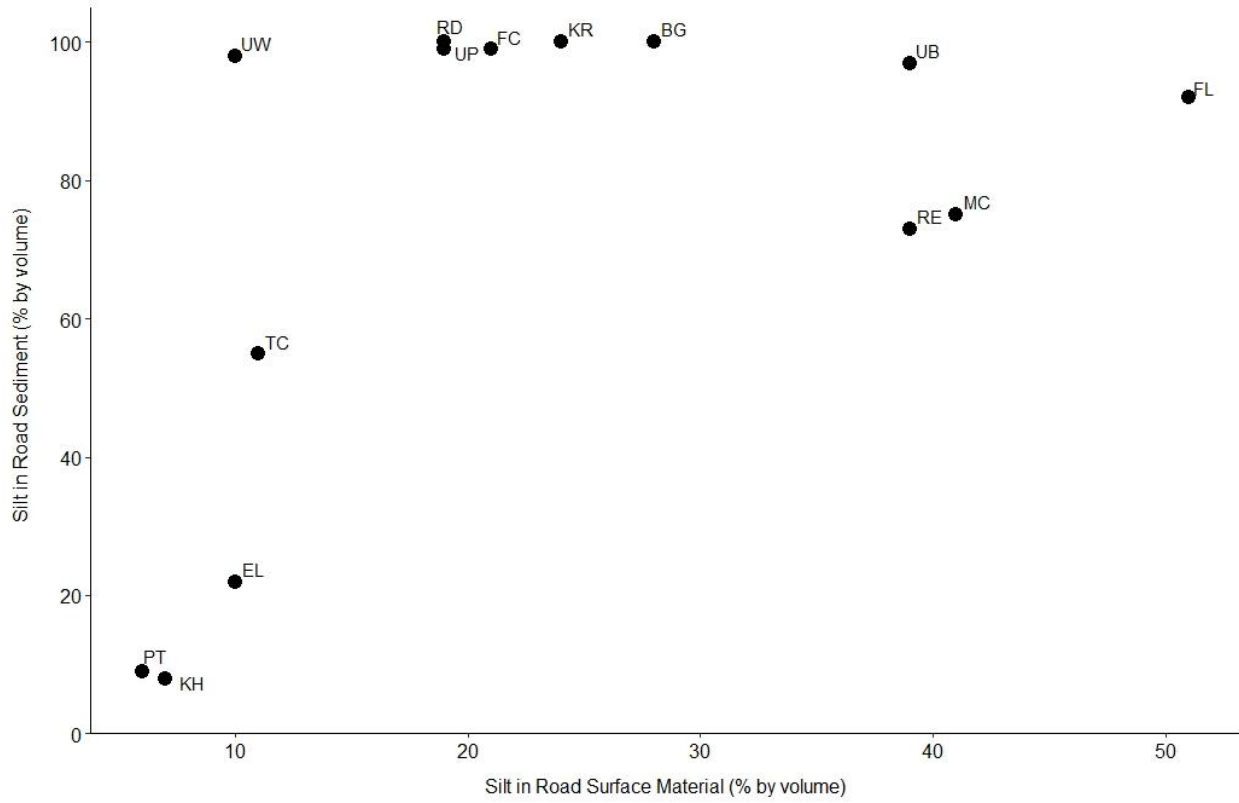


Figure 2.10. Sediment silt content as a function of silt in road surface materials. Note that sediments are typically enriched in silt compared to road surface materials.

Table 2.2. Controls on silt content in road surface materials.

Factor/Process	Explanation	Type of Variation
Type of surface material	Variability may reflect surficial geology within the watershed, or imported materials from outside the watershed. Some road capping materials (e.g., pit run gravel) naturally contain higher proportions of silt than other types (e.g., shot rock).	Spatial
	Texture of weathering products is related to parent rock type: for example, volcanic lavas (e.g., basalt) break down into clay (Ryder, 1986).	Spatial, Temporal
	Lower quality aggregate (less resistant to crushing) generates more silt as it wears (Foltz, 1996).	Spatial, Temporal
Detachment and aggregate breakdown due to traffic and maintenance ¹	For a given road surface material, the float on high use roads or road segments may contain higher proportions of silt.	Spatial, Temporal
Dynamic pumping (subgrade mixing)	Silt availability increases as surface gravel is pushed into underlying substrate and finer particles are forced upward ² , so older or more highly-used roads may have higher proportions of silt.	Temporal, Spatial
	Dynamic pumping may be prevented or minimized using an adequate depth of base course (Bilby et al., 1989), therefore road construction methods can affect the amount of silt produced on road surfaces.	Spatial
Sweeping	Materials can be redistributed horizontally across the road surface through use (Rhee et al., 2018), so the particle size distribution will depend on where the samples are collected.	Spatial
	The degree of redistribution may reflect both the amount of traffic and road age or time since grading.	Temporal
Rainfall and runoff	Silt content may fluctuate over time as the loose material accumulated on road surfaces under the influence of traffic is eroded by rainfall and runoff (e.g., Bilby et al., 1989; Ziegler et al., 2000; Ziegler et al., 2001). Silt may become depleted as it can be selectively transported in runoff ³ .	Temporal

¹ Bilby et al., 1989; Croke et al., 2005; Elliot et al., 2009; Foltz, 1996; Maloney et al., 2018; Ramos-Scharrón and MacDonald, 2007; Reid and Dunne, 1984; Reid et al., 1981; Rhee et al., 2018; Skorseth and Selim, 2000; Ziegler et al., 2000; Ziegler et al., 2001

² Baird et al., 2012; Bilby et al., 1989; Maloney et al., 2018; Reid and Dunne, 1984; Reid et al., 1981; Rhee et al., 2018

³ Bilby 1985, Bilby et al. 1989, Commandeur, 1992; Costantini et al., 1999; Croke et al., 2005; Fahey and Coker, 1992; Foltz, 1996; Zemke, 2016



Figure 2.11. Redistribution of coarse materials across a road surface (sweeping).

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Chapter 3. Phosphorus availability in unpaved access road surface materials and cutslopes in forested source watersheds

3.1 Introduction

Forests are important drinking water source areas globally (Bladon et al., 2014; Emelko et al., 2011), including Canada, where many communities rely on surface drinking water supplies that are either located within or downstream of forests (e.g., Anderson et al., 2009; APEL, 2014; Barlak, 2011; Dobson and Cook, 2013; Halifax Water, 2009; Hébert, 2007; Metro Vancouver, 2011; Robinne et al., 2019; Shoal Lake Watershed Working Group, 2002; van Meerveld et al., 2014). In addition to being important drinking water source areas, forests support a variety of social, cultural, and economic uses such as natural resource extraction, wildfire and pest management, residential use, grazing, subsistence (i.e., hunting, fishing, trapping, and foraging), research and monitoring, and recreation (Al-Chokhachy et al., 2016; Brown et al., 2013; Elliot, 2013; Gucinski et al., 2001; Luce and Wemple, 2001). Extensive networks of unpaved access roads have been developed as a result (Luce and Wemple, 2001). For example, in British Columbia alone there are over 600,000 km of unpaved roads (Forest Practices Board, 2015)—enough to circle the globe 15 times.

Despite being one of the most widespread disturbances (Al-Chokhachy et al., 2016; Baird et al., 2012; MacDonald and Coe, 2008) and frequently the predominant source of sediment in forested watersheds (Elliot, 2013; MacDonald and Coe, 2008), the potential impacts of access roads on water quality are rarely examined in a drinking water context. Yet, increased sediment loading to streams can be detrimental to drinking water supplies, treatment, and costs (Emelko et al., 2011; Health Canada, 2012; Holmes, 1988; Marquis, 2005). For instance, inorganic particles—such as the silt (particles <63 µm, in this thesis) that often erodes preferentially from unpaved road

surfaces (e.g., Bilby, 1985; Bilby et al., 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016)—can affect coagulation and flocculation, shield microorganisms during disinfection, and impact the taste of drinking water (Health Canada, 2012). Sediment is also a vector for many potential contaminants, including heavy metals, pesticides, organic compounds (e.g., polycyclic aromatic hydrocarbons, or PAHs), and nutrients (Health Canada, 2012; Ongley et al., 1992; Ritter et al., 2002; Ritter, 1988). Although many of the risks associated with sediment-bound contaminants are mitigated through the removal of suspended particulates during drinking water treatment (Health Canada, 2012), some risks remain.

The potential impacts of phosphorus (P), in particular, are not effectively mitigated by typical treatment processes. Though P itself is not considered a human health hazard (Ritter et al., 2002; Scatena, 2000), it is a concern for drinking water managers because nutrient enrichment can lead to changes in water quality that pose human health risks, reduce drinking water palatability, and increase treatment costs (Bladon et al., 2014; Cooke and Kennedy, 2001; Davies and Mazumder, 2003; Dodds et al., 2009; Emelko et al., 2011; Falconer, 2004; Ritter et al., 2002; Smith, 2003; Smith and Schindler, 2009; Yuan et al., 2018). Generally regarded as the main nutrient that limits algal productivity in freshwater ecosystems (Emelko et al., 2016; e.g., Cooke and Kennedy, 2001; House, 2003; Pick, 2016; Reddy et al., 1999), excess P in drinking water sources could indirectly cause problems like foul taste and odour or increased potential for disinfection by-product formation due to high algal biomass (Cooke and Kennedy, 2001; Davies and Mazumder, 2003; Davies et al., 2004; Dodds et al., 2009). Phosphorus also plays a role in controlling the predominance of cyanobacteria in phytoplankton communities (Downing et al., 2001; Pick, 2016). With some species able to produce neuro- or hepatotoxins (Kotak and Zurawell, 2007), cyanobacterial proliferations present a threat to drinking water supplies (Cooke and

Kennedy, 2001; Dodds et al., 2009; Downing et al., 2001; Falconer, 2004; Health Canada, 2017; Kotak and Zurawell, 2007; Ritter et al., 2002). Additionally, P can promote bacterial growth in drinking water distribution systems (Emelko et al., 2011; Miettinen et al., 1997), which is a risk to consumers.

Sediment is the main vector for P transport to surface waters (Allin et al., 2012). Unsurprisingly, studies have shown a strong relationship between total P in unpaved road runoff and total suspended sediment (e.g., Croke et al., 2000; Sheridan and Noske, 2007; Sheridan et al., 2008). Although these findings demonstrate that unpaved access roads are likely sources of P, information on how much P is in road-related materials—which can provide a basis for estimating P exports from roads (e.g., Wemple et al., 2017)—or sediments is lacking. Currently, the majority of published data are from Australia (e.g., Croke et al., 2000; Forsyth et al., 2006; Sheridan and Noske, 2007; Sheridan et al., 2008), where P concentrations are expected to be low because soils are older and more weathered (Kerr et al., 2011). Phosphorus concentrations in parent materials, which commonly make up road surfaces and become exposed on cut- and fillslopes and ditches of unpaved access roads, vary geographically (Yang et al., 2013). Thus, results from studies conducted in Australia and watersheds in Maine (Dudley et al., 1997), Vermont (Wemple et al., 2017), Brazil (Tiecher et al., 2019), or Iran (Yousefi et al., 2016) are not broadly applicable. For example, the P mean concentration that Forsyth et al. (2006) measured in road surface materials from the Beerburrum State Forest in Queensland (ca. 44 mg/kg)—and the correspondingly low estimated P export from roads (0.2–0.57 kg/km)—are much lower than those (396 mg/kg and 6.1 kg/km) that Wemple et al. (2017) observed in roadside soils and ditches in a Vermont watershed.

Another notable trend in unpaved access road-related P studies is that, with few exceptions (e.g., Dudley et al., 1997; Tiecher et al., 2019), P concentrations are measured and reported in

terms of total P. Total P, and the corresponding estimates of P export from roads, may not effectively represent the potential hazard to drinking water supplies because the implications for water quality are related to specific forms of P and their mobility in the aquatic environment (Emelko et al., 2016; Kaiserli et al., 2002; Pardo et al., 2004; Ruban et al., 1999). From a water quality perspective, the focus should instead be on bioaccessibility. Bioaccessible refers to what is immediately available for biological uptake (i.e., what is bioavailable) and what could become bioavailable (Semple et al., 2004). Thus, bioavailable P consists of reactive P (e.g., H_2PO_4^- , HPO_4^{2-} ; Orihel et al., 2017) and, following Semple et al. (2004), also includes loosely-bound P that can rapidly desorb from particle surfaces. Less bioaccessible forms include P bound to particle surfaces that releases more slowly or only under certain conditions, such as P adsorbed to secondary mineral surfaces (e.g., Al and Fe oxides or oxyhydroxides), or P encapsulated in secondary minerals (typically Fe oxides; Smeck, 1985). These forms of P can become bioavailable in the aquatic environment depending on factors such as pH, redox conditions, and the presence of competitor ions (Boström et al., 1988; Orihel et al., 2017). Using chemical sequential extractions, the composition of sediment-P can be examined in terms of the mechanisms of P release in the environment. Since operationally-defined fractions are based on the ecological significance or bioaccessibility of different geochemical forms of P (e.g., Emelko et al., 2016; Hawthorn, 2014; Kaiserli et al., 2002; Pettersson et al., 1988; Psenner et al., 1984; Ruban et al., 1999; Stone and English, 1993; Tiecher et al., 2019; and as reviewed by Wang et al., 2013), they can provide better insight into road-related P hazards in source watersheds.

Given Canada's reliance on drinking water resources derived from forests and the links between unpaved access roads, sediment, P, and the implications for drinking water supplies, there is a need to understand the potential for unpaved access roads to contribute P in source watersheds.

The objectives of this chapter address two key knowledge gaps related to unpaved access roads and P. Specifically, there are no known studies that directly examine P content and P availability in road-related materials in Canadian source watersheds. Thus, the objectives of this chapter are to quantify the total, bioaccessible, and dominant fractions of P in road surface materials and cutslopes from source watersheds across Canada. Improved understanding of P sources in forested source watersheds will help watershed managers design and implement targeted strategies and best management practices (BMPs) to mitigate risks associated with nutrient enrichment in water supplies and ensure drinking water sustainability.

3.2 Methods

3.2.1 Sample locations

Sampling locations are described in more detail in Chapter 2. In summary, over 200 road surfaces and cutslopes were sampled across a variety of watersheds ($n = 27$) in five of the major forested ecozones of Canada. Watersheds were selected to include a variety of drinking water source types (e.g., lake, reservoir, or stream), watershed management regimes (e.g., public vs. closed access, with land use activities ranging from natural resource extraction, grazing, and recreation to research to drinking water source management only), and water users (e.g., large municipal utilities serving over one million residents to domestic users). For simplicity, results for separate watersheds serving the same water utility were summarized as single groups (i.e., CB, SG, DC, UW, and PT watersheds). All of the watersheds sampled either served as, or were connected to (up to ca. 100 km upstream), drinking water supplies. Only a subset of road surface and cutslope samples were analyzed; these were selected to represent a variety of road classes (e.g., main, branch, and operational roads).

3.2.2 *Road surface and cutslope sampling*

The sampling, handling, and storage procedures are also described in Chapter 2. Briefly, the sampling protocol was intended to capture surface materials within the potential depth of erosion and P-runoff interaction. On road surfaces, the zone of influence was assumed to consist of the loose material on the road surface and the top of the compacted running surface immediately below. Samples from cutslope faces were collected from the top 2–3 cm of material. This depth is conservative, but within the range of annual losses from cutslopes (<1–7 cm) reviewed by Megahan et al. (2001) and the sampling depths recommended by Coale (2000; 2–4 cm) to capture the potential for P to be mobilized in runoff. Composite samples of road surface materials were collected using a sweep sampling method derived from the U.S. EPA’s standard operating procedure for Chip, Wipe, and Sweep Sampling (1994). Cutslopes were sampled at two points along the face using a plastic scraper (initially) or a stainless-steel trowel. In addition to the procedures described in Chapter 2, the sampling tools were washed with phosphate-free liquid dish soap and allowed to air-dry between samples to prevent cross-contamination. Samples were stored in a cooler with ice for the duration of the sampling trip and subsequently refrigerated, as recommended by Coale (2000) and Sheppard and Addison (2006) for P analysis and chemical speciation, respectively. Multiple refrigerators were required to store the large number of samples; storage temperatures ranged from 4–7°C.

3.2.3 *Sample processing and analysis*

3.2.3.1 *Sample processing*

Samples were dried in aluminum tins lined with parchment paper for 72 hours. The targeted drying temperature was 50°C (e.g., Christophoridis and Fytianos, 2006; Hawthorn, 2014;

Wuenschel et al., 2015). At this temperature, drying is faster (compared to air-drying) but avoids the potentially detrimental effects (e.g., oxidation of inorganic compounds or alteration of organic compounds) that occur during oven-drying at 105°C (Sheppard and Addison, 2006). However, the temperature of the oven used in 2018 could not be set accurately and it fluctuated unpredictably, so the temperature was set below 50°C as a precaution. For 2018 samples, the drying temperature ranged from 22–36°C (verified using a spirit thermometer). In 2019, samples were dried using a different oven. The temperature was set at 30°C so as to be consistent with samples from 2018, but occasional temperature measurements still varied between 26–34°C. Although the drying temperature was lower than intended, it was consistent with suggested drying temperatures (e.g., 25–30°C, Coale, 2000; 30–40°C, Sheppard and Addison, 2006) and other studies in which soils or sediments were air-dried prior to analysis (e.g., DeLuca et al., 2015; Stone and Mudroch, 1989). Air-drying is reasonable for materials that naturally go through wetting and drying cycles (Lajtha et al., 1999).

The dried road surface and cutslope samples were sieved, weighed, and partitioned for particle size analysis and P fractionation (Chapter 2). Stainless steel sieves, receivers, and covers (0488110G—#10 sieve, 0488110CC—#230 sieve, 04887B—receiver, 04887A—cover, Fisher Scientific Company, Toronto, ON, CAN) were washed with phosphate-free liquid dish soap and dried between samples to prevent cross-contamination. The dry particle size fractions were transferred to labeled, resealable polyethylene bags and stored in boxes at room temperature.

3.2.3.2 *Phosphorus fractionation*

Unlike speciation methods—which are used to measure the amount of one or more individual chemical species (e.g., isotope, oxidation state, or molecular structure) in a sample

(Templeton et al., 2000)—fractionation procedures differentiate analytes (such as P) into operationally-defined fractions based on physical or chemical (e.g., reactivity) properties (Pettersson et al., 1988; Templeton et al., 2000). According to the International Union of Pure and Applied Chemistry (IUPAC) recommended definitions (Templeton et al., 2000, as above), the modified Psenner method used to characterize P in this study is classified as a fractionation method and it will be referred to as such throughout this chapter.

The <63 μm size fraction (silt, in this thesis) was analyzed because it tends to be preferentially mobilized from unpaved access roads (e.g., Bilby, 1985; Bilby et al., 1989), particularly from road surfaces (e.g., Bilby et al., 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016). Furthermore, this size fraction typically contains the highest P concentrations (e.g., Kerr et al., 2011). In short, P in silt is expected to be more representative of P associated with road-derived sediments that could be transported to surface waters.

3.2.3.3 Modified Psenner method—forms of P

The P fractionation scheme used in this study (Figure 3.1) was based on the modified Psenner method (Psenner et al. 1984), as outlined in Pettersson and Istvanovics (1988) and Stone and English (1993).

Phosphorus extracted with 1 M ammonium chloride (NH_4Cl -RP) represents labile or loosely-bound P (Christophoridis and Fytianos, 2006; Emelko et al., 2016; Hieltjes and Lijklema, 1980; Hupfer et al., 2009; Kaiserli et al., 2002; Kozerski and Kleeberg, 1998; Pettersson et al., 1988; Pettersson and Istvanovics, 1988; Stone and English, 1993). This fraction is considered bioavailable, according to the definition of Semple et al. (2004). Reductant-soluble P (BD-RP) was extracted with 0.11 M bicarbonate-dithionite ($\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$). Reductant-soluble forms

include P adsorbed to or bound within Fe and Mn oxides and hydroxides (Christophoridis and Fytianos, 2006; Emelko et al., 2016; Hupfer et al., 2009; Kaiserli et al., 2002; Kozerski and Kleeberg, 1998; Psenner et al., 1984); however, Mn-P contributions are likely minimal (e.g., Jones et al., 1983; Psenner, 1984). The composition of the 1 M sodium hydroxide-extractable (NaOH-RP) fraction varies (Boström et al., 1988), but includes surface-sorbed P that is exchangeable for OH⁻ (Christophoridis and Fytianos, 2006; Hupfer et al., 2009; Kozerski and Kleeberg, 1998). According to Psenner et al. (1984), this fraction consists of Al-P, ligand exchangeable P, and P bound in humic acids. Non-apatite inorganic P (NAIP) is the sum of the NH₄Cl-RP, BD-RP, and NaOH-RP fractions, and represents the amount of bioaccessible P.

The 0.5 M hydrochloric acid (HCl-RP) and hot (85°C) 1 M sodium hydroxide (NaOH₈₅-RP) fractions represent apatite P (AP) and organic P (OP). The former comprises apatite- and carbonate-bound P, and possibly trace hydrolysable OP (Christophoridis and Fytianos, 2006; Emelko et al., 2016; Hupfer et al., 2009; Kaiserli et al., 2002; Kozerski and Kleeberg, 1998; Psenner et al., 1984; Stone and English, 1993). However, Psenner et al. (1984) found that OP in this fraction was negligible as any available OP was removed in the preceding extractions. The OP fraction may consist of biomolecules such as nucleic acids (e.g., deoxyribonucleic acid (DNA)), mononucleotides, phosphate sugars, phospholipids, phosphonates, and polyphosphates (e.g., adenosine di- and triphosphate (ADP and ATP), and phytin (Orihel et al., 2017; Reddy et al., 1999)).

Total P was calculated as the sum of P in the AP, NAIP, and OP fractions.

3.2.3.4 Application of the modified Psenner method

The <63 μm fractions were coned and quartered to obtain subsamples for P analysis, based on the method of Gerlach et al. (2002). Briefly, the samples were mounded on a fresh piece of parchment paper and divided into quarters by splitting the mound in half, then splitting each half into two parts. The two opposite quarters were combined and returned to the bag, while the other two quarters were combined and retained. This process was repeated until approximately 0.5–1 g remained. From this, 0.50xx g of material was weighed out, the mass recorded, and the subsample transferred to a labeled 50 mL polypropylene centrifuge tube.

The extractant concentrations, extraction times, and temperatures followed the steps shown in Pettersson and Istvanovics (1988) and Stone and English (1993). The procedure differed in that:

- the samples were dried at approximately 30°C prior to extraction,
- the soil:solution ratio was 1:50,
- extracts were centrifuged at 8950 g for 20 minutes, and
- residues were refrigerated overnight following the NH_4Cl extractions and between the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ and NaOH extractions. This allowed all filtrates to be run immediately after preparation.

Steps added to the procedure (but not specifically mentioned in Pettersson and Istvanovics, 1988 or Stone and English, 1993) included: ensuring that the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ solution was made fresh, air-sparging the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ extracts, adding ethylenediaminetetraacetic acid (EDTA) to the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ and NaOH filtrates, and filtering the extracts. The 0.11 M $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ solution was made fresh (i.e., the morning of the extraction), as per Psenner et al. (1984), because the pH of this solution was unstable. Air-sparging the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ extractions prior to analysis prevents interference when measuring P photometrically, and this step

was consistent with the original Psenner method (Psenner et al. 1984). Laboratory trials showed that it was necessary to remove dithionite by air sparging prior to analysis. Adding EDTA prevents phosphate from precipitating with Fe (Psenner et al. 1984); this added step was also consistent with the original Psenner method (Psenner et al. 1984). One drop of 1 mM EDTA was added to the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$ and NaOH filtrates. Lastly, and also in accordance with the original Psenner method (Psenner et al., 1984), the extracts were passed through 0.45 μm filters prior to analysis.

The filtrates were analyzed for molybdate-reactive P using a Thermo Gallery Plus Beermaster Autoanalyzer (Thermo Fisher Scientific, Vantaa, Finland), according to U.S. EPA Method 365.1 (1993), by the Natural Resources Analytical Laboratory (NRAL) at the University of Alberta. The reported limit of quantitation (LOQ³) was 18 $\mu\text{g/L}$ for the NH_4Cl extracts and 0.1 mg/L for the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$, NaOH, and HCl extracts. These concentrations corresponded to 1 mg/kg in the solid samples for the NH_4Cl extracts and 5 mg/kg for the $\text{NaHCO}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$, NaOH, and HCl extracts. Values of re-runs and replicates were averaged, with the exception of samples 18, 19, and 72. If results for any fraction were below the LOQ in one run and above the LOQ in another, the run with the reportable value was used.

3.2.4 Phosphorus fractionation quality assurance and quality control

A standard operating procedure (Appendix A) ensured consistent sample handling and extraction. Method blanks, within-batch replicates, and reference materials provided a measure of method consistency and performance. Total P (i.e., the sum of all P fractions) was compared to P

³ The lowest concentration that can be “quantitatively determined with suitable precision and accuracy” (International Conference on Harmonisation, 2005; Shoari and Dubé, 2018; Thermo Fisher Scientific, 2015); calculated as the blank response plus 10x the standard deviation or the lowest calibration standard.

extracted using a standard method to assess the effectiveness of the fractionation procedure at extracting P. Details and results are presented in Appendix B.

3.2.5 *Statistical analysis*

Where sample sizes permitted (i.e., at the ecozone scale), differences in the distribution of total P and NAIP were described using Kruskal-Wallis tests followed by pairwise Wilcoxon rank-sum tests with a Benjamini-Hochberg (BH) correction (stats package; R Core Team, 2020). Differences in NAIP between road surfaces and cutslopes were assessed using a two-sided paired Wilcoxon rank-sum test (stats package; R Core Team, 2020). The results were evaluated using a significance level of $\alpha = 0.05$.

In one road surface sample, all of the NAIP fractions were below the applicable LOQ (Appendix C). Based on the analytical measurements, which were above the detection limit (LOD⁴) but below the LOQ, a value of 8 mg/kg was estimated for NAIP and was substituted for the censored data point. Although the substitution approach has no statistical basis (Helsel, 1990; Helsel, 2006; Shoari and Dubé, 2018; Slymen et al., 1994), it is justified in this case because the approximation is not arbitrary. Moreover, substitution can be a reasonable approach provided that the conclusions are not undermined by assuming a constant for censored data (Ogden, 2010)—this is true for the median NAIP concentrations (at both the ecozone and watershed scales) reported in this study.

⁴ The concentration below which the measurement cannot be reliably differentiated from zero (International Conference on Harmonisation, 2005; Shoari and Dubé, 2018; Thermo Fisher Scientific, 2015); calculated as the blank response plus 3x the standard deviation.

3.3 Results

3.3.1 Total phosphorus in road surface materials and cutslopes

Phosphorus was ubiquitous in road surface materials and cutslopes. Total P concentrations in road surface materials ranged from 97 mg/kg to 6016 mg/kg (Figure 3.2, Figure 3.3), and median total P across all road surface materials was 728 mg/kg. The distribution of total P in road surface materials varied across major Canadian forested ecozones ($p < 0.001$), with higher concentrations occurring more frequently in the Pacific Maritime and Boreal Shield regions compared to the Boreal Plains and Atlantic Maritime regions (Figure 3.2, Table 3.1). However, P was not randomly distributed within ecozones, and geographic trends were apparent across watersheds (Figure 3.3). Consistently high total P concentrations occurred in the Laurentian Mountains of Québec in the Boreal Shield ecozone (RM), and total P in road surface materials from the Haliburton Highlands of Central Ontario (KH) was elevated compared to other Boreal Shield watersheds. Elevated total P concentrations (e.g., watershed median > 1000 mg/kg) in road surface materials also occurred in the Montane Cordillera of northern British Columbia (i.e., UB, FL, UP) and in the north Okanagan region (DC). However, these higher concentrations were offset at the ecozone scale by lower concentrations in watersheds located in the Kootenays (MC, KC, FC, BG) and Rocky Mountain Eastern Slopes (UW).

Total P concentrations in cutslopes ranged from 128 mg/kg to 5158 mg/kg (Figure 3.2, Figure 3.3). At 708 mg/kg, median total P across cutslope samples was similar to that of road surface materials. The distribution of total P in cutslopes was hard to assess due to limited sample sizes: cutslopes were not ubiquitous, particularly bare cutslope faces that would be prone to erosion.

3.3.2 Bioaccessible phosphorus in road surface materials and cutslopes

Unlike total P, NAIP concentrations were similar across ecozones ($p = 0.53$; Table 3.2). However, as with total P, differences in NAIP were evident at the watershed scale (Figure 3.4). Notably, median NAIP was higher—by approximately 160 to 200 mg/kg—in some watersheds (e.g., LL, MC) than others within the same ecozone. Nearby or neighboring watersheds sometimes had similar median NAIP concentrations (e.g., UB and FL, FC and BG), whereas in other instances median NAIP was two to six times higher in one (e.g., MC, RD, EL) than the other (e.g., KC, UW, WL, respectively). Road surface NAIP concentrations varied by ca. 200 to 300 mg/kg in some watersheds (e.g., DC, UW, RM, TC), whereas others exhibited much narrower ranges (e.g., SG, FL, UP).

Non-apatite inorganic P concentrations in cutslope samples ranged from 17 mg/kg to 1089 mg/kg (Table 3.2, Figure 3.4). Cutslopes had the highest NAIP concentrations observed in the dataset (i.e., SG, RM). Despite instances of high cutslope NAIP concentrations compared to road surfaces (ca. 2.5–12.5 times higher, or 115–1000 mg/kg difference), cutslopes were, overall, equally likely to have either higher or lower NAIP concentrations than road surface materials at corresponding locations ($p = 0.09$; Figure 3.5).

The reported NAIP concentrations typically represented 12–17% (median percentage) of total P in road surface materials and cutslopes, respectively (Figure 3.6). The NAIP fraction predominated—that is, it made up over half of total P—in only 13% of samples, so it was generally a minor component of total P. The different forms of NAIP were distributed similarly between road surface materials and cutslopes (Figure 3.6). Very little of the NAIP present in road surface materials and cutslopes was bioavailable: the NH_4Cl -RP fraction was below the LOQ (reported as 0% of total P) in 93% of samples analyzed. This fraction never exceeded 2% of total P in either

sample type. Rather, the NAIP fraction comprised mainly less reactive forms of bioaccessible P (Figure 3.6), i.e., BD-RP and NaOH-RP. Median BD-RP in road surface materials and cutslopes was 3% and 4%, respectively. Median NaOH-RP was 8% of total P in road surface materials and 13% of total P in cutslopes. Sodium hydroxide-reactive P (NaOH-RP) was the most abundant form of NAIP across all samples; it exceeded BD-RP in 84% of samples.

3.3.3 *Non-bioaccessible phosphorus in road surface materials and cutslopes*

Phosphorus in road surface materials and cutslopes consisted primarily of AP (Figure 3.6). Across all samples, AP content was marginally higher in road surface materials (median = 82%) than in cutslopes (median = 72%). Of the ten samples with the lowest AP content (less than ca. 25%, Figure 3.6), the majority occurred within a few watersheds (SG, MC, TC). Organic P was generally a minor component of total P in the materials sampled (Figure 3.6): OP made up only 7–10% (median values) of total P in road surface materials and cutslopes, respectively.

3.4 Discussion

Since sediment is the main vector for P transport (Allin et al., 2012) and unpaved access roads are a significant (Elliot, 2013; Elliot et al., 2009; Gucinski et al., 2001)—if not predominant (MacDonald and Coe, 2008)—sediment source in forested watersheds, it follows that unpaved access roads may be significant sources of P. This study provides new information on the amount and types of P—particularly bioaccessible P—in road-related materials. The different forms of bioaccessible P indicate how much P is potentially bioavailable and under what conditions less reactive forms of bioaccessible P in road sediments might be liberated in the aquatic environment. This exploratory study raises questions about the factors driving bioaccessible P concentrations in road-related materials. Specifically, how do road-related processes influence concentrations, how

rapidly do concentrations change, and are bioaccessible P concentrations in the reported ranges relevant from a source water quality perspective? Improved understanding of P sources will help watershed managers quantify the hazard associated with P loading from unpaved access roads and inform the implementation of BMPs to minimize nutrient enrichment of water supplies.

3.4.1 Total phosphorus in road surface materials and cutslopes

Phosphorus is a relatively minor component of the Earth's upper crust (0.09–0.15%; Filippelli, 2008; Rudnick and Gao, 2014) and terrestrial surface materials (0.063%; Hartmann et al., 2012). Nevertheless, P is common: it is globally widespread in surficial materials (Hartmann et al., 2012; Yang et al., 2013), and P minerals can occur in all major types of rocks, i.e., igneous, sedimentary, metamorphic, and biogenic (Filippelli, 2002; Porder and Ramachandran, 2013). Phosphorus was ubiquitous in road surface materials and cutslope samples collected from different road types and forested source watersheds across diverse ecophysiological settings, and this indicates how prevalent the hazard associated with road-related P loading may be if sedimentation is not effectively controlled.

Local geology could explain comparatively higher total P and “hotspots” in road surface materials and cutslopes across watersheds. For example, elevated median P in the UB watershed agrees with values for P-rich basalt and andesite rocks (916 mg/kg and 1000 mg/kg, respectively; Porder and Ramachandran, 2013), which, along with other alkaline volcanic rocks, make up the underlying bedrock (Cui et al., 2017). The watershed with the highest total P concentrations (RM) is underlain by charnockite rocks of the Grenville Province (Thériault and Beauséjour, 2012; Wheeler et al., 1997). Charnockite rocks are characterized, in part, by high P₂O₅ (Kilpatrick and

Ellis, 1992) and have apatite (primary mineral P) among their primary constituents (Beard et al., 1994).

Despite relationships between geology and total P, the former may not always be a good indicator of total P in road-related materials. First, P can vary widely (by hundreds or thousands of mg/kg), even within the same type of rock (Porder and Ramachandran, 2012). Total P in road surface materials, in particular, may not always be consistent with the local geology because they are sometimes imported from outside the watershed, or from other areas within the watershed that have different geology. This could explain why median total P in the FL watershed was among the highest (Figure 3.3), even though local geology consists primarily of granites and diorite (Cui et al., 2017). These coarser-grained rocks are higher in Si and tend to be lower in P (median P for granite is 440 mg/kg; Porder and Ramachandran, 2013). Phosphorus in road-related materials could also differ from expected concentrations given their origin (i.e., in rocks or surficial materials; Hartmann et al., 2012; Porder and Ramachandran, 2013) from being exposed at the surface, sometimes for several decades (the oldest roads in the dataset are ca. 100 years old). Weathering will result in a loss of P over time (Filippelli, 2008; Smeck, 1985; Walker and Syers, 1976; Yang and Post, 2011; Yang et al., 2013; Figure 3.7).

3.4.2 Phosphorus availability in road surface materials and cutslopes

Phosphorus transport from unpaved access roads has traditionally been assessed using total P (e.g., Croke et al., 2000; Forsyth et al., 2006; Sheridan and Noske, 2007; Sheridan et al., 2008; Wemple et al., 2017). However, the results show that total P is not especially useful for evaluating the hazard associated with road-related sedimentation in a water quality context because, in

general, only a relatively small portion of P in road surface materials and cutslopes is bioaccessible (i.e., the NAIP fraction; Figure 3.6).

Bioaccessible forms of P—i.e., loosely sorbed, adsorbed to inorganic secondary mineral (e.g., Al and Fe oxides) surfaces, or encapsulated in inorganic secondary minerals (e.g., in crystal lattices, coatings, or concretions)—are produced as primary mineral P (e.g., AP, calcium phosphate) weathers (Filippelli, 2002; Filippelli, 2008; Smeck, 1985; Walker and Syers, 1976; Yang and Post, 2011; Yang et al., 2013; Figure 3.7). The NH_4Cl -RP fraction—that is, bioavailable P (i.e., easily desorbed P; Semple et al., 2004)—in road surface materials and cutslopes (Figure 3.6) is expected to be low as soluble P is readily incorporated into biomass, adsorbed to secondary mineral surfaces, or leached (Smeck, 1985). The negligible amounts of loosely-bound P could explain the low levels of dissolved P in road runoff observed by Dudley et al. (1997; median dissolved orthophosphate = 6 $\mu\text{g/L}$ vs. median total P = 1060 $\mu\text{g/L}$).

Though further geochemical analyses were not conducted to investigate the P species present in the different NAIP fractions, the relative amounts of BD-RP and NaOH-RP (Figure 3.6) appear to reflect the abundance of Fe and Al in the Earth's upper crust (estimated at 5.04% and 15.4% by weight, respectively; Rudnick and Gao, 2014). This suggests that Mn-P compounds are not a significant component of BD-RP, as indicated by Psenner (1984) and Jones et al. (1983), and that Al-P is the primary component of NaOH-RP. Reductant-soluble P (BD-RP) is released from benthic sediments when ferric oxides and hydroxides undergo reductive dissolution (Boström et al., 1988; Christophoridis and Fytianos, 2006). Microbial activity plays a significant role in the mobility of reductant-soluble P because it depletes oxygen at the sediment-water interface, which promotes reductive dissolution resulting in P release (Boström et al., 1988; Christophoridis and Fytianos, 2006). Surface-bound Al-P (NaOH-RP) is typically released when OH^- is exchanged for

PO₄ (Christophoridis and Fytianos, 2006; Hupfer et al., 2009; Kozerski and Kleeberg, 1998). This occurs at high pH—for instance, during periods of high primary productivity (Christophoridis and Fytianos, 2006). However, surface-bound Al-P can also be exchanged for other competitor ions, e.g., sulfate, arsenate, citrate, bicarbonate, and oxalate (as reviewed by Orihel et al., 2017). Exchangeable P can become rapidly bioavailable throughout the water column (Psenner, 1984), and results from prior studies have demonstrated that this P pool can be a predominant source of algal-available P (as reviewed by Boström et al., 1988).

3.4.3 *Variability of bioaccessible P in road surface materials and cutslopes*

Since NAIP is produced through weathering, it follows that the spatial distribution of NAIP in road surface materials and cutslopes (Figure 3.4) is determined primarily by parent material P (initial P), which limits the amount of NAIP that can be produced (Yang et al., 2013), and by the multitude of local factors (e.g., geology, biological activity, climate; Table 3.3) that influence weathering rates. Walker and Syers' (1976) conceptual model of P transformations (Figure 3.7) shows that NAIP concentrations in road surface materials and cutslopes are expected to change over time. Natural changes in NAIP concentrations introduce uncertainty into the results, and call into question the reliability of one-time measurements to assess P availability in sediment sources. Given the median percentages of AP—particularly that of road surface materials (Figure 3.6)—the conceptual model suggests that many of the materials sampled are in the phase of rapidly increasing NAIP. However, it is unclear by how much the reported NAIP concentrations could change over timescales that are relevant to unpaved access roads (e.g., months, years, decades). Phosphorus transformations are expected to be slow—for instance, complete weathering of AP generally takes hundreds to thousands of years (Smeck, 1985; Walker and Syers, 1976). Yet, there

is evidence to suggest that P transformations could happen much faster: highly weathered, P-depleted samples occurred in the dataset (indicated by AP content less than ca. 25%, Figure 3.6), despite that the oldest roads sampled are believed to be only ca. 100 to 110 years old. It is possible that these materials were pre-weathered (Walker and Syers, 1976), but they could also reflect enhanced weathering occurring within a few watersheds due to some local factor (Table 3.3).

Erosion processes might also introduce uncertainty into the reported NAIP concentrations. Road surface materials are subject to processes known to increase silt availability, such as crushing (from vehicle traffic) and dynamic pumping (or subgrade mixing; Rhee et al., 2018). The resulting increase in particle surface area increases the potential for chemical weathering of AP (Guidry and MacKenzie, 2003; Newman, 1995; Table 3.3). As such, vehicle traffic would be expected to create conditions conducive to NAIP production, which should lead to higher NAIP concentrations on road surfaces compared to cutslopes. This trend did not occur across road-cutslope pairs (Figure 3.5), and this could be attributed to the use of imported materials, which are sometimes used to cap road surfaces (especially on permanent roads). It is also possible that, even if road and cutslope materials are of the same or similar origin, that they are of a different age—for example, if fresh surface course has been applied during routine road maintenance.

Another explanation, however, can be gleaned by examining differences between road surfaces and cutslopes on temporary roads. Imported materials are rarely used on temporary roads, so running surfaces and cutslope faces typically consist of the same materials that have been exposed for the same amount of time (e.g., RM, Figure 3.5). The large difference in NAIP concentrations in the RM road-cutslope pair suggests that the cyclical buildup and washoff process that causes silt content to vary in road surface materials (e.g., Bilby et al., 1989; Ziegler et al., 2000; Ziegler et al., 2001) could be limiting NAIP concentrations on road surfaces. In other words,

NAIP concentrations may fluctuate over rather short timescales on road surfaces as the surface is “renewed”, i.e., as less weathered material becomes exposed when the more highly weathered float is periodically removed in runoff. Such large differences in NAIP did not occur on in road-cutslope pairs from other temporary roads (e.g., UP1, RD2), but light road use would minimize this process.

3.4.4 Implications of road-related bioaccessible P for source water quality

While prior studies have indicated that P contributions from unpaved access roads are typically a small part of the total P budget (e.g., Sheridan and Noske, 2007; Wemple et al., 2017), it is conceivable that bioaccessible P inputs from unpaved access roads could impact water quality. Hawthorn (2014), for example, observed increased amounts of fine (<250 μm , in this case) streambed sediments, P, and benthic periphyton growth in a logged watershed compared to an undisturbed watershed, and suggested that unpaved roads were the likely source. Even where bioaccessible P concentrations in source materials are similar to—if not lower—than natural sources (e.g., stream channels), unpaved access roads can contribute a disproportionate amount of sediment—and therefore P—compared to their surface area in a watershed (e.g., 25% vs. 2%; Tiecher et al., 2019). However, the impact of road-associated P loading on water quality depends on myriad traits and processes related to both unpaved roads and the receiving environment. Thus, the impact of road-related P is expected to be watershed-specific (e.g., Sheridan and Noske, 2007) and includes a lot of uncertainty.

Since sediment is the primary pathway for P transport (Allin et al., 2012), silt production, erosion, and sediment delivery ultimately control the supply of bioaccessible P to source waters. The amount of bioaccessible P delivered from roads to surface waters depends on factors such as: the type and quality of road surface material, rainfall amount and intensity, road use intensity and

the type of vehicle traffic, the distance between roads and streams (connectivity), and the spacing between drains (Al-Chokhachy et al., 2016; Brown et al., 2013; Foltz, 1996; MacDonald and Coe, 2008; van Meerveld et al., 2014). Once P is delivered to surface waters, it can be stored (e.g., through deposition), transformed (either chemically or biologically, into more or less bioavailable forms), diluted, or transported downstream (MacDonald and Coe, 2007; Withers and Jarvie, 2008). In turn, sediment—and therefore P—transport depends on characteristics that vary among watersheds, such as stream morphology, stream flow (capacity), and large woody debris (Hawthorn, 2014; as reviewed by MacDonald and Coe, 2007).

Next, whether the bioaccessible P delivered to surface waters is released into the water column and made available for uptake by algae depends on the chemical and physical characteristics, and biological processes of the receiving water body (as reviewed by Boström et al., 1988; Orihel et al., 2017; Reddy et al., 1999; Withers and Jarvie, 2008). For example, dissolved P can precipitate with calcium (Withers and Jarvie, 2008), rendering it unavailable for algal uptake. Conversely, anoxic conditions at the sediment-water interface, mediated by bacterial activity, promote the release of reductant-soluble P (Boström et al., 1988; Christophoridis and Fytianos, 2006; Orihel et al., 2017; Withers and Jarvie, 2008). Warmer water temperatures could increase P mobility by promoting the release of iron-bound (reductant-soluble) P as dissolved oxygen levels decrease (Jeppesen et al., 2009).

Furthermore, whether algae are able to proliferate as a result of increased P supply and availability depends again on various environmental, chemical, and biological factors (Cooke and Kennedy, 2001; Pick, 2016; Smith, 2003). Herbivory, water residence time, light availability (shading), and mixing regime can all influence algal productivity in lakes and streams (Lewis and Wurtsbaugh, 2008; Withers and Jarvie, 2008). Increased lake and stream temperatures, caused by

lower water levels associated with climate change, are expected to promote algal growth (Takaro et al., 2022). The type of drinking water supply is also important. For instance, P is readily flushed through when water residence times are low (Withers and Jarvie, 2008), making lakes and reservoirs more sensitive to the potential effects of P loading than drinking water supplies from high-energy systems.

While there is still no direct evidence linking water quality issues in the study watersheds (or elsewhere) to unpaved access roads, enhanced P mobilization from the terrestrial to the aquatic environment caused by weathering, erosion, and sedimentation from unpaved access roads poses a threat to source water quality. Anthropogenically-driven increases in P loading are recognized as the cause of eutrophication and subsequent degradation of freshwaters worldwide (Yuan et al., 2018). Fortunately, the risks to drinking water supplies associated with P loading from unpaved access roads can be effectively mitigated by implementing BMPs to control erosion, runoff, and sedimentation. Using high quality aggregate on road surfaces, for instance, can minimize silt production (Foltz, 1996) and revegetating cutslopes can reduce erosion (Baird et al., 2012; Burroughs and King, 1989; Elliot et al., 2009; Luce and Black, 1999; MacDonald and Coe, 2008; Megahan et al., 2001; Reid and Dunne, 1984; Reid et al., 1981; Sheridan and Noske, 2007). In particular, minimizing connectivity (the length of road that drains directly to surface waters; MacDonald and Coe, 2008)—by reducing the length of stream crossing approaches (Brown et al., 2013), or by dispersing road runoff by increasing the number of cross-drains (Anderson and Lockaby, 2011; MacDonald and Coe, 2008)—will effectively reduce the amount of sediment, and therefore P, delivered from roads to surface waters. Significant reductions in sedimentation could be achieved economically by selectively implementing BMPs at the relatively small proportion of

sites within a watershed that produce and deliver the most sediment (Al-Chokhachy et al., 2016; Luce and Black, 1999).

3.4.5 Limitations

Since the geologic origin of most road surface materials was unknown, the same materials may be represented more than once within a watershed. Also, sample sizes were small compared to the length and potential diversity of roads in most watersheds. The source watershed for the City of Dawson Creek (KR), for example, covers nearly 285,000 ha and has an estimated 1,400 km of access roads (Dobson Engineering and Urban Systems, 2003). In addition, the results could be biased towards higher concentrations because the subset of samples analyzed favoured higher-hazard road segments (e.g., where algal growth was observed in ditches or at watercourse crossings). An ideal sampling design would be representative of different types of roads, ages of roads, and road materials across each watershed. However, road maps could not always be obtained *a priori*, and such detailed information on roads was generally not available. Still, the results provide an indication of the range and order of magnitude of NAIP concentrations in road-related materials from source watersheds across a variety of ecophysiographic and geological settings in Canada.

The concentrations of bioaccessible P presented in this study are only estimates. Many fractionation methods have been developed to examine P mobility in soils and sediments. Each has advantages and limitations, and results can vary depending on the operational definitions used, the extractants used, and the characteristics of the materials being analyzed (Hieltjes and Lijklema, 1980; Hupfer et al., 2009; Pettersson et al., 1988; Pettersson and Istvanovics, 1988; Ruban et al., 1999; Wang et al., 2013). The estimates also do not account for all of the bioaccessible P generated

by road-related materials over time. According to the conceptual model of P transformations (Walker and Syers, 1976; Figure 3.7), some P has already been lost as dissolved P (e.g., in runoff; Smeck, 1985) and bioaccessible P concentrations change over time.

Notably, OP can be mobilized as molecules are decomposed and mineralized by bacteria (Boström et al., 1988; Orihel et al., 2017; Reddy et al., 1999). While typically considered non-reactive (e.g., Psenner and Pucsko, 1988; Psenner et al., 1984; Stone and English, 1993)—and thus not bioaccessible—the mobility of organic species in the aquatic environment varies as some species are more labile than others (Boström et al., 1988; Orihel et al., 2017; Reddy et al., 1999). Some OP species may require decades to decompose, yet others can be hydrolysed over the course of a season (Orihel et al., 2017). Depending on the nature of OP in road-related materials or sediments, bioaccessible P may be higher than the reported NAIP concentrations would indicate.

3.5 Conclusion

This study is believed to be the first in Canada to assess the range of concentrations and types of P in materials from unpaved access roads. In most cases, total P in road surface materials and cutslopes is a poor indicator of the amount of P that could be mobilized in the aquatic environment. Very little P is bioavailable and the majority of bioaccessible P is bound in less reactive fractions, with the exchangeable (or Al-P) fraction being the most abundant.

Bioaccessible P concentrations can vary among, and often within, watersheds. Previous studies on P transformations in terrestrial environments indicate that the distribution of bioaccessible P is primarily dependent on factors that influence weathering and that bioaccessible P varies temporally. Future studies could explore the influence of different factors on the variability of bioaccessible P in road-related materials. Of particular interest are the effects of

physical breakdown of materials due to vehicle traffic and silt buildup and washoff—specifically, the extent to which these characteristic processes might enhance natural weathering rates and control bioaccessible P variability on road surfaces. In addition, understanding how much P concentrations can change over timescales that are relevant to roads (e.g., months, years) could help watershed managers quantify the long-term hazard to drinking water supplies across unpaved access road networks, as well as the level of uncertainty, associated with P loading from unpaved access roads.

The hazard to source water quality appears to be widespread; however, it is not known whether or to what extent P transport from unpaved access roads through erosion and sedimentation are impacting source water quality. Nonetheless, the results highlight an opportunity to reduce this particular threat to drinking water supplies by implementing BMPs to control road erosion and runoff, thereby reducing road-related P inputs.

Tables and Figures

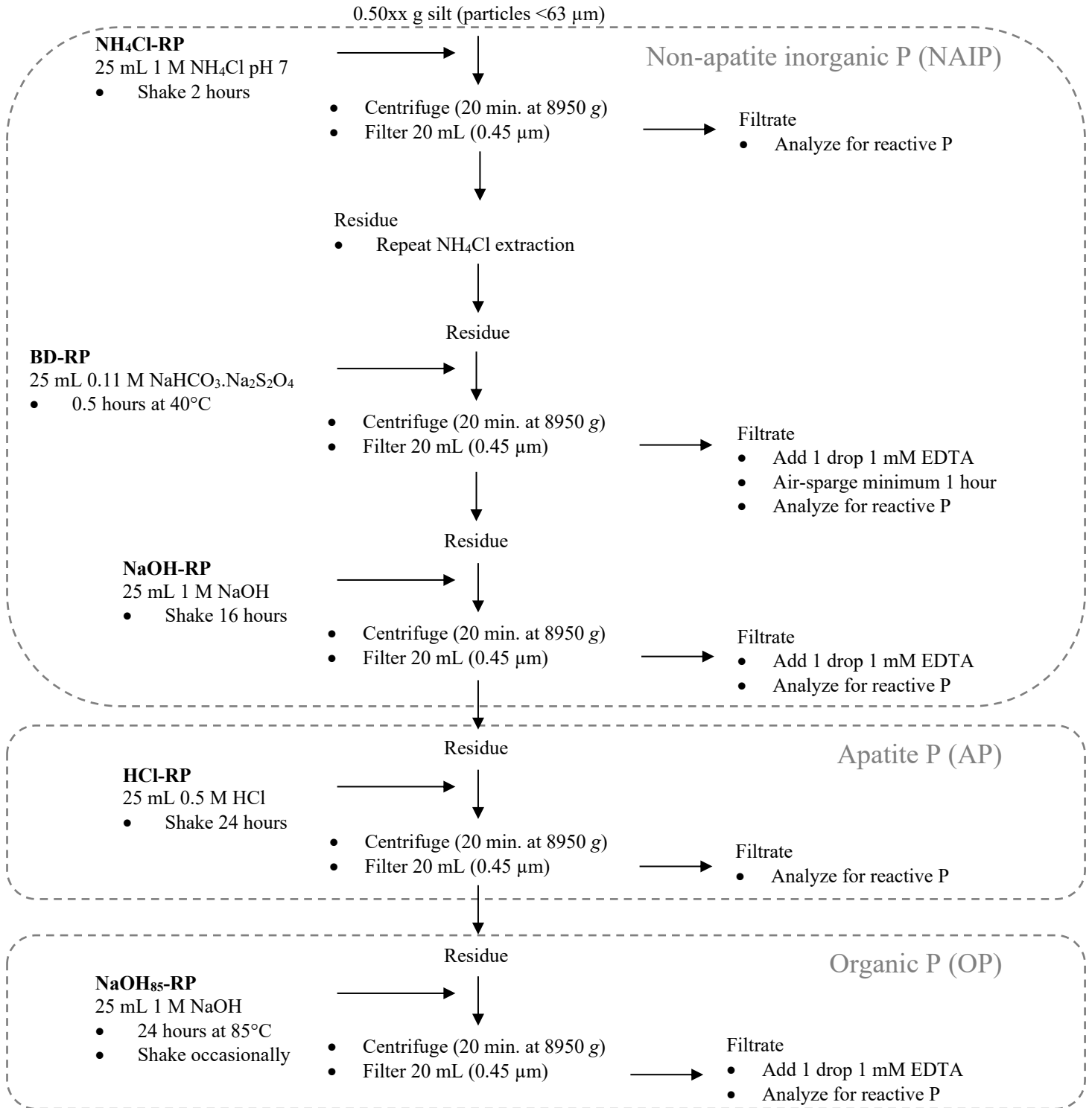


Figure 3.1. Phosphorus fractionation procedure (adapted from Pettersson and Istvanovics, 1988; Psenner et al., 1984; and Stone and English, 1993).

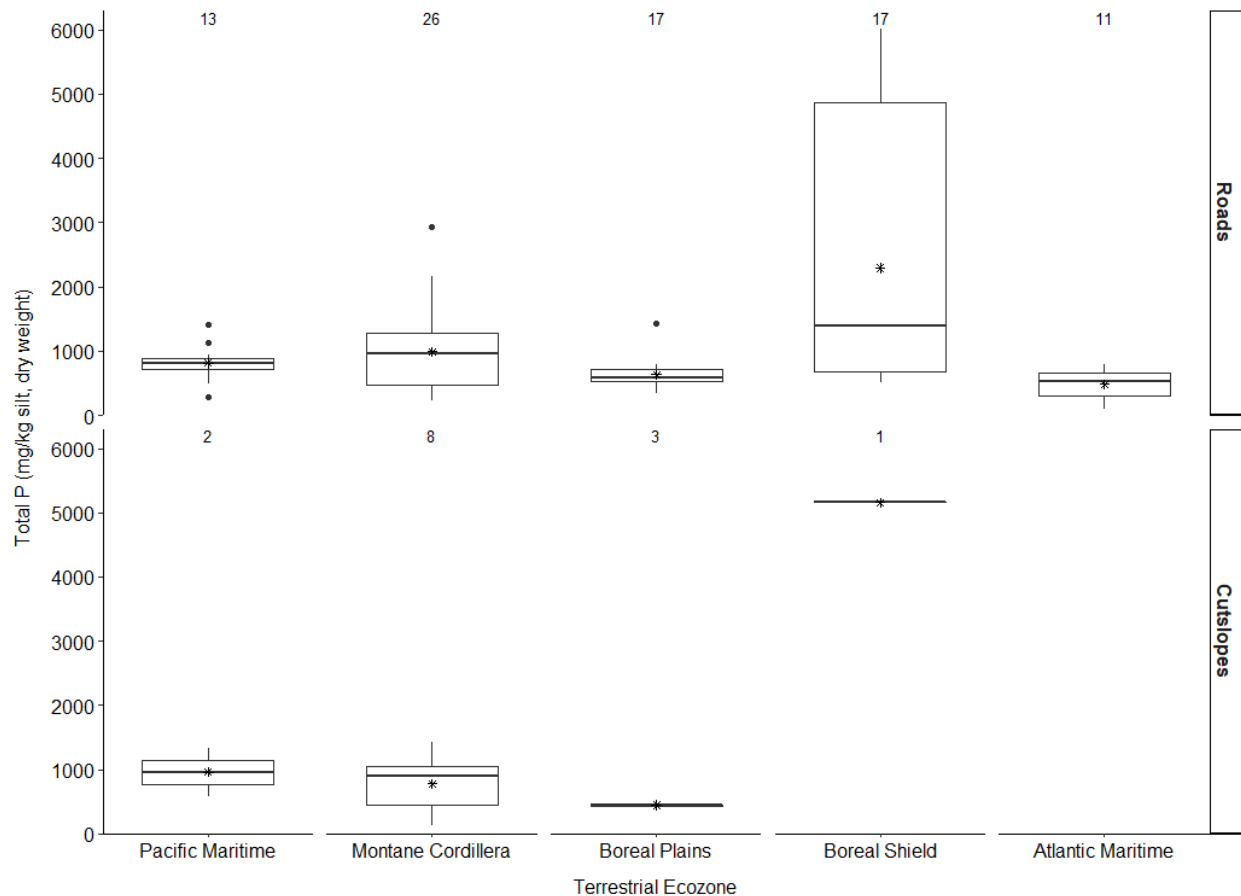


Figure 3.2. Total phosphorus (P) in silt (particles <63 μm) from road surface materials and cutslopes across ecozones, where total P is the sum of non-apatite inorganic P (NAIP) + apatite P (AP) + organic P (OP). Values within plots denote (n); bold bars indicate medians and asterisks (*) indicate means.

Table 3.1. Multiple comparisons of total phosphorus (P) in silt (particles <63 μm) from road surface materials, where total P is the sum of non-apatite inorganic P (NAIP) + apatite P (AP) + organic P (OP). Bold face denotes significant pairwise tests ($\alpha_{family} = 0.05$).

	<i>p</i> -value			
	Pacific Maritime	Montane Cordillera	Boreal Plains	Boreal Shield
Montane Cordillera	0.5268	—	—	—
Boreal Plains	0.0271	0.1517	—	—
Boreal Shield	0.0687	0.0340	0.0024	—
Atlantic Maritime	0.0104	0.0271	0.3161	0.0018

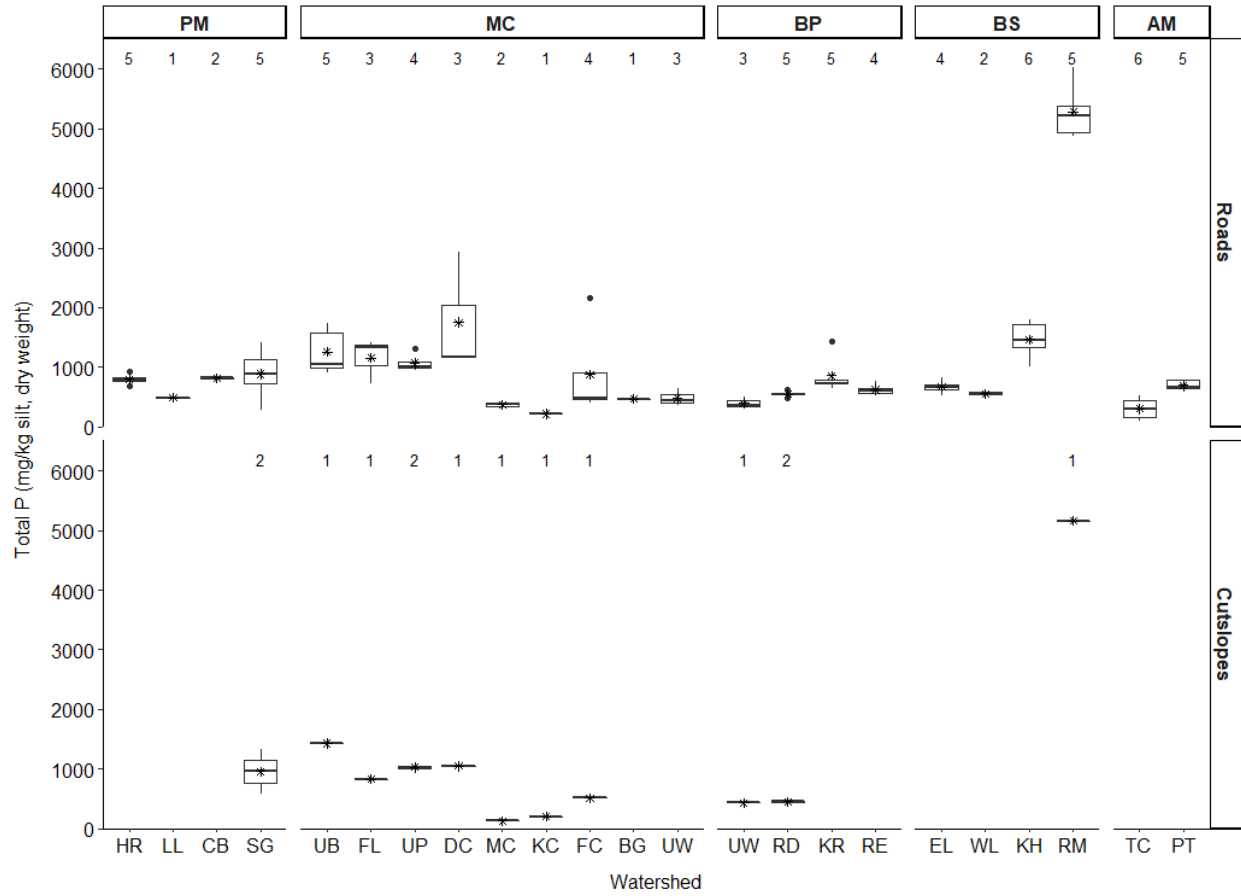


Figure 3.3. Total phosphorus (P) in silt (particles <63 μm) from road surface materials and cutslopes by watershed, where total P is the sum of non-apatite inorganic P (NAIP) + apatite P (AP) + organic P (OP). Note that the UW watershed spans two ecozones; median watershed total P concentration in road surface materials is 406 mg/kg. Values within plots denote (n). Bold bars indicate medians and asterisks (*) indicate means. Data for separate watersheds serving the same water utility were summarized as single groups (i.e., CB, SG, DC, UW, and PT).

Table 3.2. Median non-apatite inorganic phosphorus (NAIP) concentration (mg/kg of silt) in road surface materials and cutslopes across ecozones. Letters represent similar NAIP distributions ($\alpha = 0.05$); ranges (minimum, maximum) are in brackets. NA = not available (no samples collected).

Terrestrial Ecozone					
	Pacific Maritime	Montane Cordillera	Boreal Plains	Boreal Shield	Atlantic Maritime
Roads	112a (34, 293)	72a (6, 358)	120a (<12, 286)*	94a (23, 262)	92a (21, 291)
n	13	26	17	17	11
Cutslopes	779 (468, 1089)	95 (22, 190)	128 (17, 152)	766 –	NA –
n	2	8	3	1	NA

*All NAIP fractions below LOQ, minimum value is <12 mg/kg.

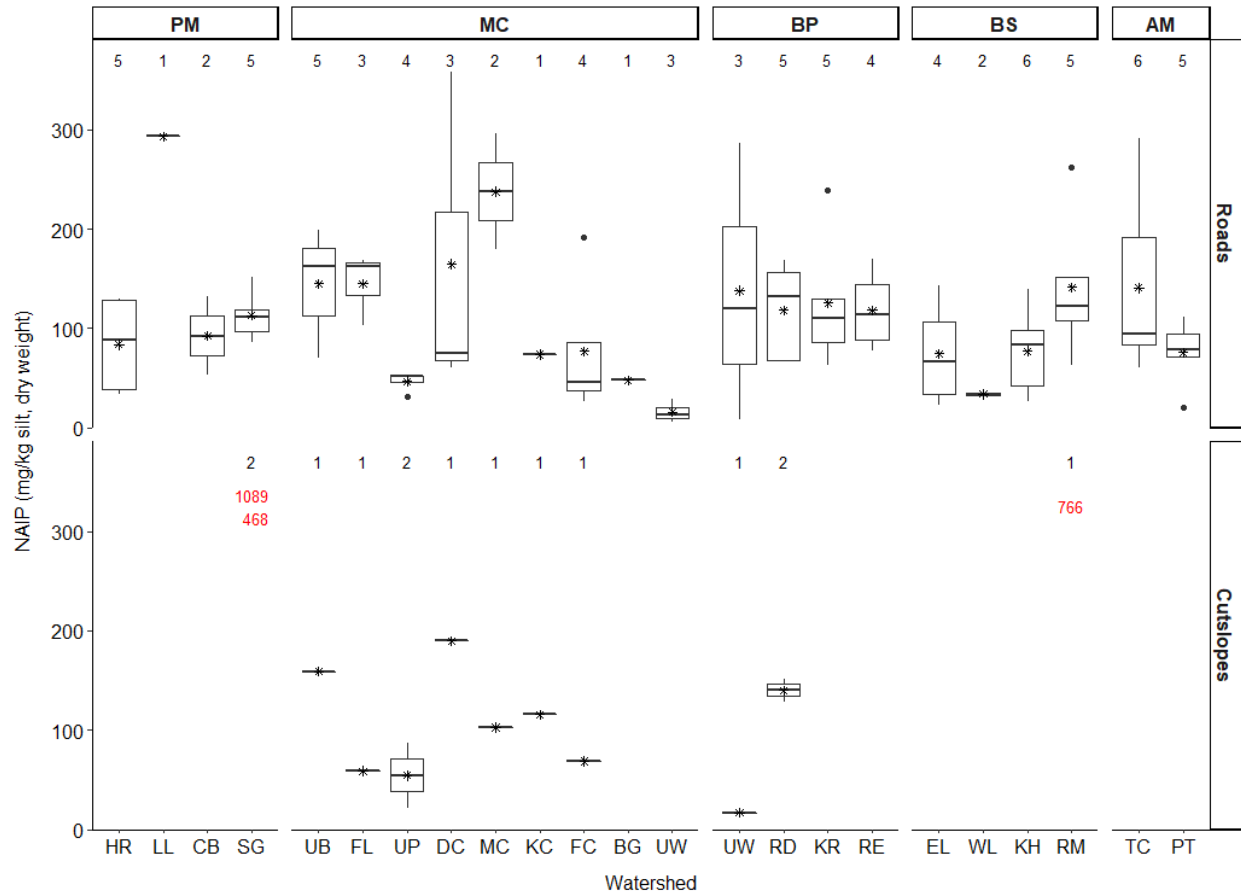


Figure 3.4. Non-apatite inorganic phosphorus (NAIP) concentrations in silt (particles $<63 \mu\text{m}$) from road surface materials and cutslopes by watershed. Note that the UW watershed spans two ecozones; median watershed NAIP concentration in road surface materials is 21 mg/kg. Values within plots denote (n). Bold bars indicate medians and asterisks (*) denote means. Cutslope concentrations $>370 \text{ mg/kg}$ are given in red text. Data for separate watersheds serving the same water utility were summarized as single groups (i.e., CB, SG, DC, UW, and PT).

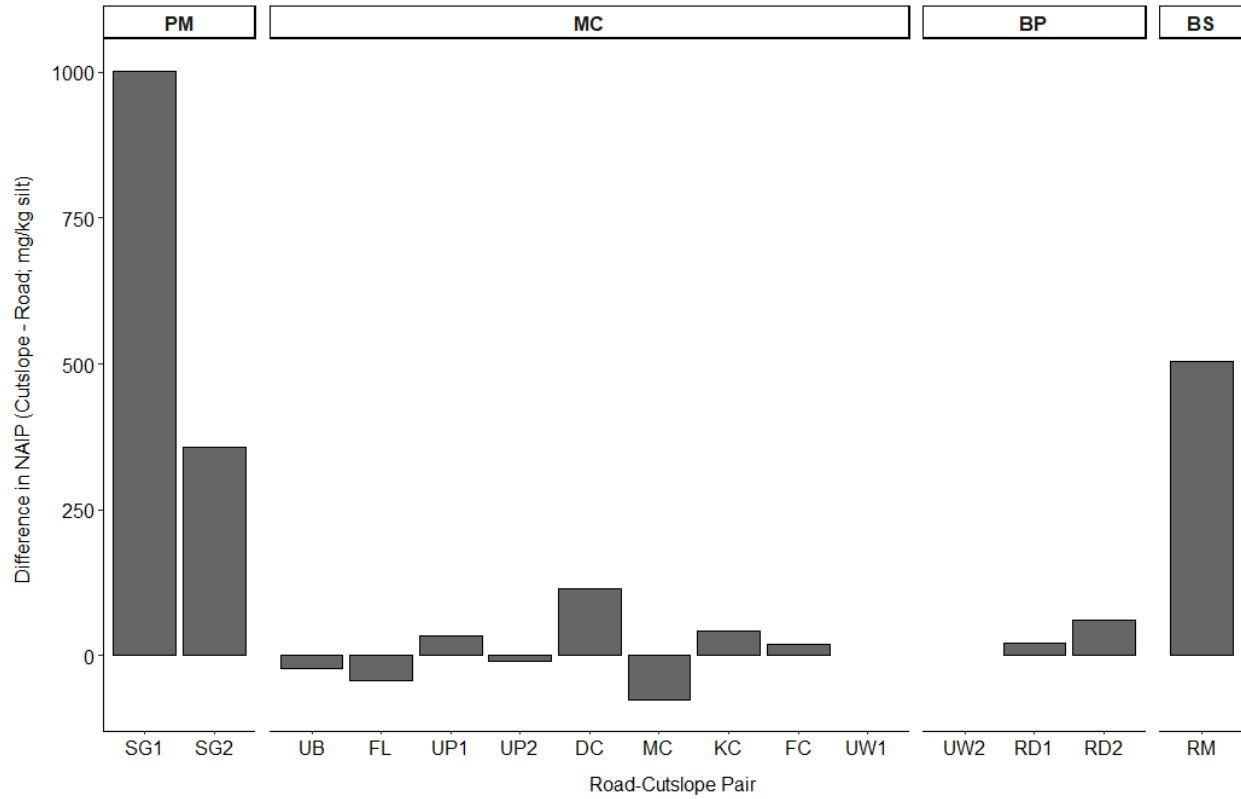


Figure 3.5. Difference in non-apatite inorganic phosphorus (NAIP) concentration (mg/kg of silt) between road surface materials and cutslopes on the same road segment. Blanks indicate difference could not be calculated (insufficient sample or censored value). Positive values denote cutslope NAIP > road surface NAIP.

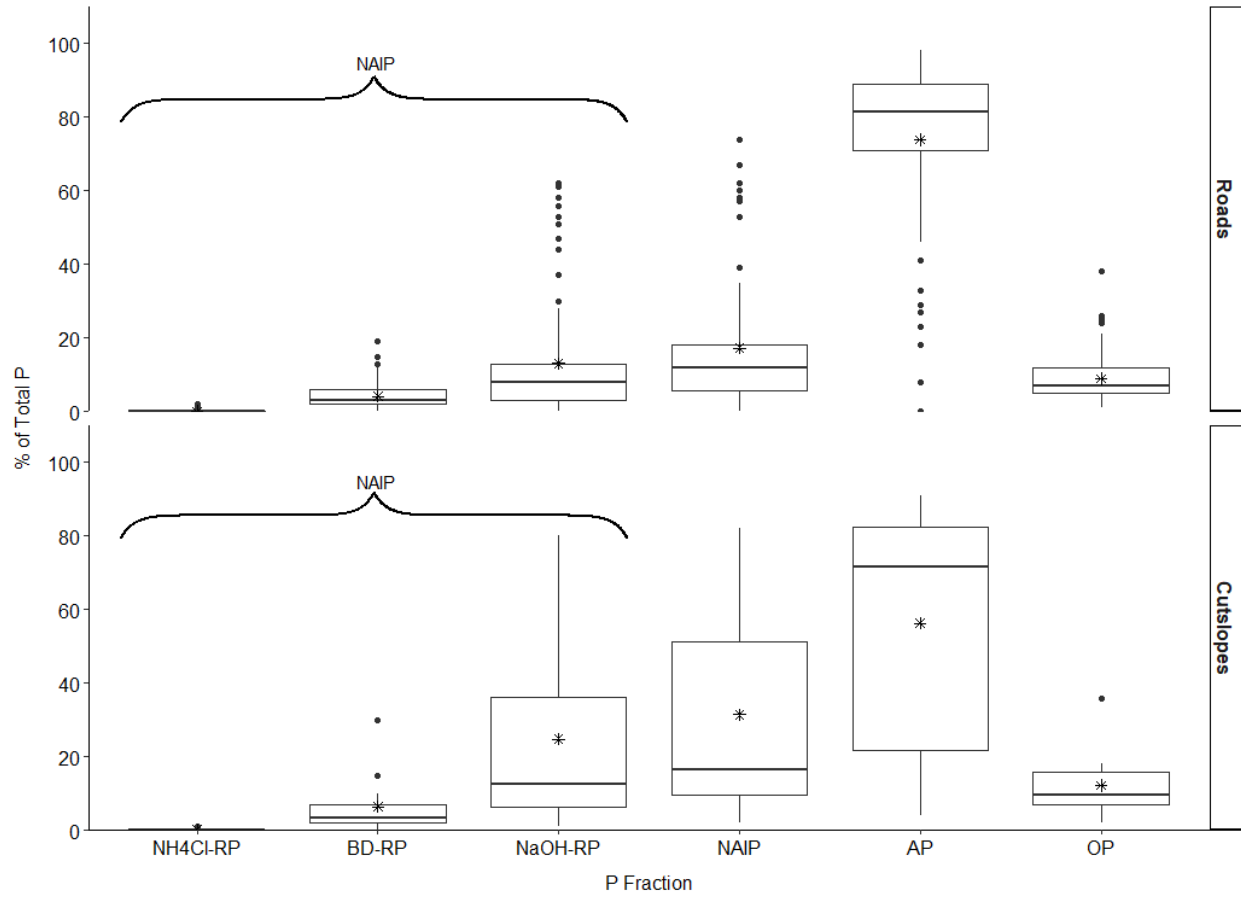


Figure 3.6. Composition of phosphorus (P) in silt (particles <math><63 \mu\text{m}</math>) from road surface materials and cut slopes as a percentage of total P, where total P is the sum of non-apatite inorganic P (NAIP) + apatite P (AP) + organic P (OP). Solid bars indicate medians and asterisks (*) denote means.

Table 3.3. Controls on non-apatite inorganic phosphorus (NAIP) content in road-related materials.

Process/Factor	Explanation	References
Weathering	Weathering of primary mineral phosphorus (e.g., apatite phosphorus, or AP) yields secondary forms (NAIP) and organic phosphorus (OP). Materials that are minimally weathered have a high percentage of AP.	Walker and Syers, 1976 and others (e.g., Blecker et al., 2006; Cross and Schlesinger, 1995; Frossard et al., 1989; Mehmood et al., 2018; Schoenau et al., 1989; Smeck, 1985; Yang and Post, 2011; Yang et al., 2013)
Age/Exposure	The degree of weathering will be higher the longer materials have been exposed at the surface.	Boyle et al., 2013; Press et al., 2003; Walker and Syers, 1976
Geology	Rock composition determines how and at what rate materials weather. For example, AP may weather faster on road surfaces or outslope faces consisting of rocks (or materials derived from rocks) that contain acid-generating minerals (e.g., pyrite).	Cross and Schlesinger, 1995; Guidry and MacKenzie, 2003; Maloney and Carson, 2018; Mehmood et al., 2018; Press et al., 2003
Climate and Environment	Weathering is higher in regions with high mean annual temperatures and precipitation.	Blecker et al., 2006; Cross and Schlesinger, 1995; Guidry and MacKenzie, 2003; Press et al., 2003; Schoenau et al., 1989; Walker and Syers, 1976; Yang and Post, 2011; Yang et al., 2013
	Higher soil temperatures due to forest harvesting may increase weathering rates. It follows that higher temperatures could occur on daylighted roads and accelerate weathering and NAIP production.	Feller, 2005
	Common apatite minerals, such as igneous fluorapatite and carbonate fluorapatite, are sensitive to low pH. Therefore, weathering may be greater in regions with higher levels of acid deposition.	Guidry and MacKenzie, 2003; Smeck, 1985
	Weathering rates could be higher on non-daylighted roads due to acidic leaching from forest litter.	Mehmood et al., 2018

Process/Factor	Explanation	References
Texture	Dissolution of common apatite minerals, such as igneous fluorapatite and carbonate fluorapatite, is a surface-driven process. Physical weathering (e.g., as vehicle traffic breaks down road surface materials) could enhance P release by increasing particle surface area.	Guidry and MacKenzie, 2003; Newman, 1995
Slope	Chemical weathering is higher on lower gradient slopes. However, steep slopes are prone to physical weathering, which increases the surface area for chemical weathering (see above).	Press et al., 2003
Biological Activity	Surface biofilms (which could include bacteria, algae, fungi, and lichens), microorganisms, and plant roots enhance weathering (chemical and physical).	Newman, 1985; Press et al., 2003; Viles, 2013
Other Factors		
Geology	Parent material (initial) P controls how much P is available to be released over time.	Yang et al., 2013
	Some materials may naturally contain higher proportions of OP (note: this would affect the relative amount of NAIP (%) but not the concentration).	Alexander and Robertson, 1968; Walker and Syers, 1976; Yang and Post, 2011
Texture	Surface-bound forms of NAIP can be retained on particle surfaces, so materials with a higher clay content may have higher percentages of NAIP.	Blecker et al., 2006
Deposition	Atmospheric dust (could include fine soil particles, pollen, burned plant material and fossil fuels) can be deposited on roads, potentially contributing additional bioaccessible P. Atmospheric inputs can become especially important in older materials in which P is depleted.	Boyle et al., 2013; Newman, 1985; Porder and Ramachandran, 2013; Yang and Post, 2011
Biological Activity	P is exchanged between the NAIP and OP pools through biological processes (e.g., uptake, mineralization). NAIP should exceed OP (or they may be roughly similar) since the latter is limited by the availability of inorganic P.	Blecker et al., 2006; Frossard et al., 1989; Smeck, 1985; Walker and Syers, 1976; Yang and Post, 2011; Yang et al., 2013; Yuan et al., 2018

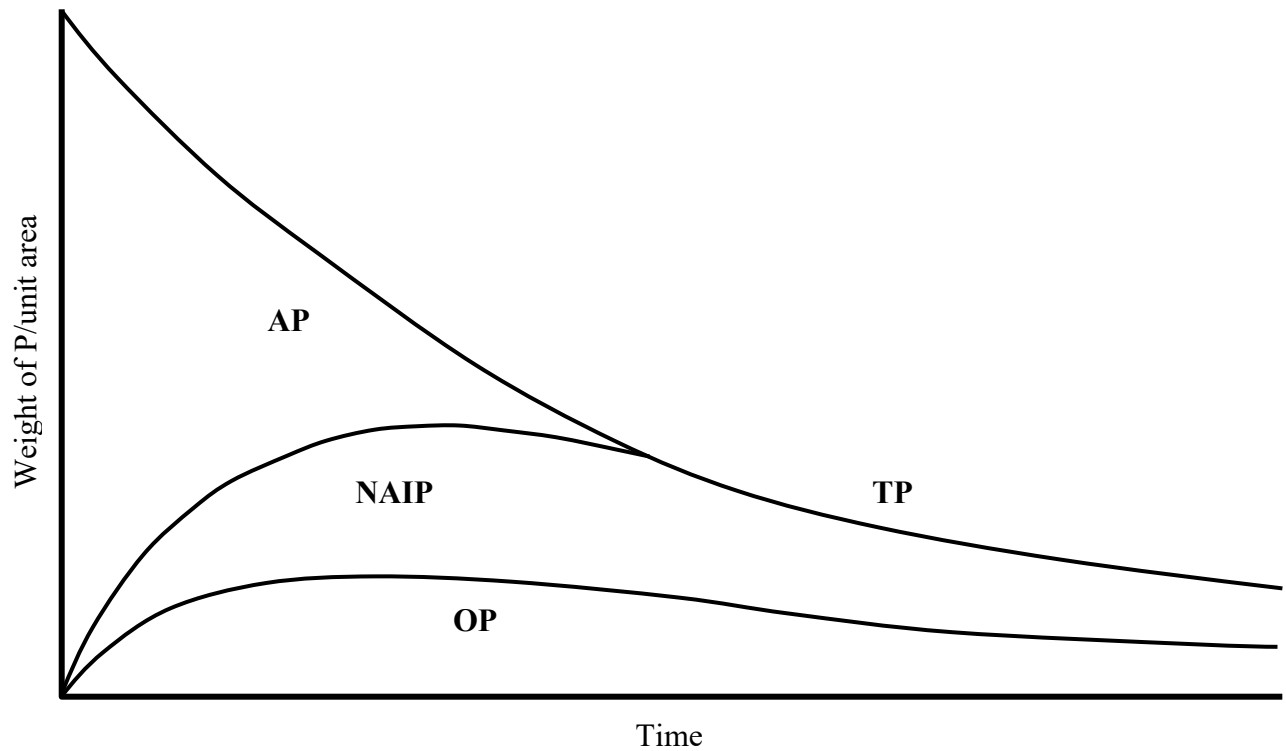


Figure 3.7. Changes in total phosphorus (TP) and functional pools of P over time. The conceptual model is adapted from Walker and Syers (1976) in terms of the operational definitions for the P fractions extracted using the modified Psenner method (described in section 3.2.3). These include apatite P (AP), non-apatite inorganic P (NAIP), and organic P (OP).

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Chapter 4. Synthesis

The silt and P mobilized in runoff from unpaved access roads have implications for water quality, yet these hazards have rarely been assessed and discussed in a drinking water context. Thus, the goal of this thesis was to improve understanding of the potential drinking water source quality and treatment challenges associated with unpaved access roads in forested watersheds. Silt and P availability in unpaved access road-related materials, along with the mobilization of silt from road-related sediment sources, reflects what could be transported in runoff either directly to surface water supplies or to their contributing areas upstream. This study is unique in its broad geographical scope, which represents a range of ecophysiological settings across Canada. It may also be the first to quantify P and—more importantly—bioaccessible P in unpaved access road-related materials in Canada.

The first data chapter of this thesis (Chapter 2) focused on 1) silt supply in road surface materials and cutslopes, and 2) silt mobilization from road surfaces and cutslopes. Silt content (particles $<63 \mu\text{m}$, in this thesis) was assessed as a relative measure of silt availability in unpaved access road-related materials from forested source watersheds across Canada. The results highlight the range and variability of silt contents across unpaved access road surface materials and, to a lesser extent, cutslopes at regional (ecozone) and local (watershed) scales. Higher road surface silt contents were more common in western Canadian ecozones. Within ecozones, the hazards associated with silt availability (e.g., turbidity, potential for sediment-bound contaminant transport to surface waters) appear to be higher in certain watersheds. Still, the range of silt contents within watersheds—up to ca. 50–74%, in some cases—demonstrates that “hotspots” of comparatively higher silt supply are common across watersheds. The differences in road surface material silt content could reflect the availability (or lack thereof) of more suitable, coarser-textured road

surface materials due to local geology and management factors, such as road use intensity. Cutslope silt content can likely be attributed to geology, though natural weathering processes (e.g., freeze-thaw cycles and wetting-drying cycles) could also play a role.

Although texture is indicative of the range of particle sizes available to be eroded (Grismer et al., 2008), the silt content of road surface materials did not predict the silt content of sediments generated under simulated rainfall. More specifically, source and sediment silt contents did not exhibit a positive linear relationship. Rather, silt mobilization was typically elevated (median = 95%) across the rainfall simulation plots. Further, sediments were enriched in silt—they contained, on average, 3.6 times more silt than the corresponding road surface material. Thus, overall, the rainfall simulation results were consistent with selective erosion. Selective erosion from unpaved access roads has been well-documented (e.g., Bilby 1985; Bilby et al., 1989; Commandeur, 1992; Costantini et al., 1999; Croke et al., 2005; Fahey and Coker, 1992; Foltz, 1996; Zemke, 2016). It suggests that the hazards to water quality—i.e., turbidity and sediment-bound contaminant transport—are higher when silt availability is comparatively higher, since the silt that is present in the source material tends to be mobilized from the road surface. Silt mobilization from cutslopes was assessed only through literature review. Findings across studies that examined erosion from steep slopes were inconsistent, so it is unclear whether silt is likewise selectively eroded from cutslopes.

Modelling and experimentation by others (e.g., Al-Chokhachy et al., 2016, Luce and Black, 1999) suggests that “hotspots” of high sediment production are responsible for most—as much as 90%—of the sediment delivered to streams. It follows that identifying silt-supply hotspots, particularly where road drainage is highly connected to surface waters, could present an opportunity to significantly reduce silt inputs to source watersheds by implementing BMPs to

minimize erosion and sedimentation. However, silt supply alone is not likely a reliable indicator of water quality hazard at specific sites (i.e., road segments). Other factors, including infiltration, slope, contributing area, vegetation or rock cover, rainfall intensity, surface roughness, and connectivity to surface waters (as reviewed by MacDonald and Coe, 2008), can all influence erodibility and sediment delivery. Instead, silt content in road-related materials may be more useful for understanding the hazard at larger (regional, or watershed) scales, as the results suggest that silt transport can be supply-limited at very low road surface silt contents ($\leq 10\%$). To that end, silt content in absolute rather than relative units (e.g., g/m^2 of road surface vs. percent) may provide more information on silt supply, and subsequently, the potential for silt limitation.

The second data chapter (Chapter 3) assessed 1) total P concentrations, 2) bioaccessible P concentrations, and 3) the relative composition of P in silt from road surface materials and cutslopes. This chapter is closely related to Chapter 2, since the $<63 \mu\text{m}$ particle size fraction typically contains the highest P concentrations (e.g., Dudley et al., 1997; Kerr et al., 2011; Stone and Mudroch, 1989) and it tends to be preferentially mobilized from unpaved access roads (e.g., Bilby, 1985; Bilby et al., 1989), especially from road surfaces (e.g., Bilby et al., 1989; Costantini et al. 1999; Fahey and Coker, 1992; Foltz, 1996; Lane and Sheridan, 2002; Zemke, 2016; Chapter 2). The results reported in Chapter 3 emphasize the range and variability of total P and bioaccessible P across unpaved access road surface materials and, again to a lesser extent, cutslopes at different spatial scales. The variability in total P and bioaccessible P concentrations may reflect a number of controlling factors. According to Walker and Syers' (1976) conceptual model of P transformations over time, geology (which determines how much P is present) and weathering (which determines how much P remains and in what forms) are the primary influences on the concentrations and the forms of P present. Generally, only a portion of the total P in road

surface materials and cutslopes (ca. 12–17%, respectively) was bioaccessible. This finding has important implications for how the hazard associated with sediment-bound P transport from unpaved access roads is assessed. As the type of P that could be mobilized in the aquatic environment—thus becoming available for algal uptake and potentially leading to detrimental water quality impacts—future studies should focus on bioaccessible P rather than total P.

Similar to silt content, higher total P and bioaccessible P concentrations occurred in some watersheds, and “hotspots” also occurred within watersheds. Bioaccessible P “hotspots” may be of particular interest to watershed managers, especially where they coincide with high silt availability (e.g., the Mark Creek watershed, denoted as MC, in the east Kootenay region of British Columbia). Areas where high bioaccessible P concentrations overlap with high silt supply represent an elevated water quality hazard due to the increased potential for bioaccessible P transport associated with the selective erosion of silt. However, the hazard associated with low bioaccessible P concentrations should not be underestimated. Unpaved access roads are chronic sources of sediment (Al-Chokhachy et al., 2016; Brown et al., 2013; Carson and Younie, 2003; Elliot, 2013; Gucinski et al., 2001; Karr et al., 2004; MacDonald and Coe, 2008), so they could contribute significant amounts of bioaccessible P over time. Furthermore, the impact of unpaved access road-related P inputs on source water quality is currently not well understood, and it is expected to vary among watersheds due to differences in the characteristics and processes related to road erosion, sediment delivery, and the receiving environments.

Together, Chapters 2 and 3 reveal key characteristics of unpaved access road-related materials from forested source watersheds across Canada. The results provide insight on what may be transported in runoff from unpaved access roads and subsequently impact source water quality and treatment. Variability was a common theme between Chapters 2 and 3. Both spatial and

temporal factors likely contribute to variability in silt content and bioaccessible P concentrations. First, sample sizes were small compared to the length and potential diversity of roads in most watersheds. An ideal sampling design would be representative of different types of roads, road materials, road age, and road use intensity across each watershed; however, detailed road information and mapping is required for this approach. In larger watersheds with many different road owners, detailed information may be difficult to obtain. Instead, a sufficient number (e.g., proportional to road density or length) of random samples or stratified random samples (e.g., based on mapped surficial geology or other available information) could effectively capture the range of silt and P availability across the diversity of roads. Next, silt content, P, and bioaccessible P are expected to change over time due to processes such as erosion (e.g., Bilby et al., 1989; Ziegler et al., 2000; Ziegler et al., 2001) and weathering (Megahan et al., 2001; Walker and Syers, 1976). Silt content on road surfaces, in particular, likely varies over short time scales (days or weeks) due to the effects of road use (i.e., vehicle traffic), maintenance and rainfall (i.e., erosion, runoff). As such, long-term silt supply cannot be determined with confidence from one-time measurements. Significant changes in bioaccessible P concentrations are expected to occur over much longer periods of time (hundreds to thousands of years); however, it is not known whether (or by how much) the rate of bioaccessible P production could be enhanced by increased physical weathering on road surfaces.

4.1 Future work and recommendations

This study exposed the variability of silt and P—both total and bioaccessible—on unpaved access road surfaces in forested source watersheds in Canada. The findings raise additional research questions, such as:

- What are the predominant controls on silt availability and mobilization from road surfaces?
- Is silt preferentially eroded from cutslopes?
- What are the predominant controls on bioaccessible P production in road-related sediment sources?

There may be practical applications gained from examining these topics—for example, understanding whether daylighting unpaved access roads limits or increases bioaccessible P production, or understanding the relative importance of slope and material texture for silt mobilization. However, the opportunity to manage the water quality hazards posed by silt and P seems clear: the use of well-established BMPs to minimize unpaved access road erosion and sediment delivery to surface waters (e.g., Carson and Younie, 2003) will help reduce silt and bioaccessible P inputs to water supplies and their contributing areas.

There are challenges to remediating erosion and sedimentation issues, including having the resources (human and capital) required to inventory sites or run models and field-truth model outputs across extensive road networks. Resources are also required to implement BMPs and to monitor their effectiveness. Furthermore, problematic road construction practices, such as routing ditch drainage to surface waters, are still accepted as industry standard. As a result, the hazards associated with unpaved access road erosion and sedimentation will likely persist into the future. Several tools have already been developed to assess watershed- and site-scale erosion and sediment delivery. Road erosion and sediment delivery models, such as the Water Quality Effectiveness Evaluation (WQEE; Maloney et al., 2018), the Road Erosion and Sediment Delivery Index (READI; Benda et al., 2019), and the Geomorphic Roads Analysis and Inventory Package (GRAIP; Black et al., 2012; Cissel et al., 2012), can help watershed managers estimate sediment

loading from unpaved access roads and target sediment delivery “hotspots” at which to implement BMPs. Thus, further study of the amount and the impact of road-related bioaccessible P contributions, particularly in relation to other P sources, will further improve watershed managers’ ability to prioritize and implement source control measures at a watershed scale to protect drinking water resources. To better understand the potential impacts to drinking water resources caused by road-related bioaccessible P, the following areas of study are recommended:

- 1) **Quantify the spatial and temporal variability of bioaccessible P at a watershed scale.** Because of the broad geographical scope of this study, the results reflect a limited number of samples collected at a single point in time. To better understand bioaccessible P contributions from unpaved access roads in a watershed, quantifying the uncertainty and distribution of bioaccessible P concentrations in road-related materials is a logical first step. This could be accomplished using the methods presented here (e.g., sweep sampling, P fractionation), an increased number of samples, and repeat sampling. The results could be used in conjunction with road erosion and sediment delivery models to inform where implementing BMPs may be most effective at reducing bioaccessible P inputs to surface waters.
- 2) **Are unpaved access roads a significant source of bioaccessible P in source watersheds?** The next step to assess the importance of road-related bioaccessible P on drinking water resources is to use sediment tracing or apportionment techniques (e.g., Motha et al., 2003; Stone et al., 2014; Tiecher, 2019) to estimate the amount of bioaccessible P contributed by the various sediment sources within a forested source watershed. This type of study will clarify the relative influence of unpaved access road-related bioaccessible P contributions in a watershed and assist managers in identifying

which sediment sources pose the largest threat to drinking water sustainability. The results can be used to implement targeted mitigation and monitoring programs to improve or protect source water quality.

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Appendix A. Phosphorus fractionation procedure

Sample Preparation

1. Dry samples at low temperature (<35°C) or air dry for 72 hours.
2. Sieve to <63 µm.

Prepare Reagents

1. Prepare the desired volume of 1 M NH₄Cl

- a. Due to the large volume required, this extractant was prepared, according to density, in a 10 L HDPE carboy:

$$\text{Volume}_{\text{solution}} * \text{Density}_{\text{solution}} = \text{Mass}_{\text{solution}}$$

$$\text{Mass}_{\text{solution}} - \text{Mass}_{\text{salt}} = \text{Mass}_{\text{H}_2\text{O}}; \text{ recall } 1 \text{ g H}_2\text{O} = 1 \text{ mL}$$

- i. Density of 1 M NH₄Cl is 1.01 g/mL
- b. Mix until fully dissolved (use magnetic stirrer and stir bar, or cap and invert).
 - c. Check the pH (will be slightly acidic, approximately pH 5). Raise to pH 7, dropwise, using concentrated NH₄OH.
 - d. Top up to final volume with DI water.
 - e. Decant 500 mL of extraction solution into a HDPE bottle. Submit to lab to prepare standards and for calibrations.

2. Prepare the desired volume of 0.11 M NaHCO₃·Na₂S₂O₄

- a. This solution must be made fresh (same day).
- b. Use the formula: $\frac{X \text{ g}}{\text{mol}} * \frac{0.11 \text{ mol}}{\text{L}} * \text{required solution volume (in L)}$ to determine the masses of each solute required (in g) for the desired volume.
 - i. MW NaHCO₃ = 84.006 g/mol
 - ii. MW Na₂S₂O₄ = 174.11 g/mol

- c. Decant 500 mL of extraction solution into a HDPE bottle, add 25 drops of 1 mM EDTA, and sparge with air for 2 hours. Submit to lab to prepare standards and for calibrations.
3. Prepare the required volume of 1 M NaOH
 - a. Use the formula in 1.a. to prepare a large volume in a 10 L HDPE carboy.
 - i. Density of 1 M NaOH is 1.04 g/mL
 - ii. Add NaOH pellets gradually while mixing. Caution: the carboy will get hot.
 - b. Decant 500 mL of extraction solution into a HDPE bottle and add 25 drops of 1 mM EDTA. Submit to lab to prepare standards and for calibrations.
4. Order prepared, certified 0.5 N HCl.
 - a. Decant 500 mL of extraction solution into a HDPE bottle. Submit to lab to prepare standards and for calibrations.
5. Prepare 1 mM EDTA (note that 60 drops from a transfer pipet equals approximately 2.5 mL)
 - a. Use the formula in 2.b.
 - i. $MW\ EDTA = 372.24\ g/mol$
 - b. Add EDTA to a volumetric flask, fill to line with DI water, and mix until dissolved.

Wash Tubes and Caps[§]

1. Wash with phosphate-free dish detergent and triple-rinse with DI water.
2. Soak in 5% HCl bath for 30 minutes.
3. Soak in DI water bath.
4. Triple-rinse with DI water.
5. Air dry in clean fume hood or on countertop.
6. Cap clean, dry tubes and place in clean plastic bags. Store in a box or cupboard until use.

[§]Also wash used syringes, if they will be re-used.

Subsampling Procedure

1. Mix sample well and empty bag contents onto a clean piece of parchment or weigh paper.
2. Using a clean, stainless steel spatula, cone and quarter the sample until a ~0.50xx g subsample remains.
3. Weigh 0.50xx g of sample into a clean, labeled 50 mL polypropylene centrifuge tube (must be flat-cap type). Record exact mass of subsample.

Extraction Procedure

1. NH₄Cl-reactive P

- i. Tare balance with 25 mL graduated cylinder. Measure 25 mL of 1 M NH₄Cl into cylinder. Verify weight (25.3 g) and add to centrifuge tube with subsample.
- ii. Shake (on table shaker, at low setting) for 2 hours.
- iii. Centrifuge at 8950 g for 20 minutes.
- iv. Using a 0.45 µm syringe filter (use filters with polypropylene housing and membrane for this step and all subsequent steps) and syringe, filter supernatant into a labeled 20 mL scintillation vial.
- v. Carefully, without disturbing the residue, remove the remaining supernatant with a transfer pipet and discard into a waste container.
- vi. Repeat steps ii-vi.
- vii. Analyze filtrate for molybdate-reactive P (see Notes).

2. NaHCO₃.Na₂S₂O₄-reactive P

- i. Tare balance with 25 mL graduated cylinder. Measure 25 mL of 0.11M NaHCO₃.Na₂S₂O₄ into cylinder. Verify weight (25.5 g) and add to tube with residue from Extraction 1.
- ii. Shake manually to break up and re-suspend sediment pellet.

- iii. Heat in a 40°C water bath for 30 minutes, shaking occasionally (e.g., every 10 minutes).
- iv. Centrifuge at 8950 g for 20 minutes.
- v. Using a 0.45 µm syringe filter and syringe, filter supernatant into a labeled 20 mL vial with a septum cap. Add 1 drop of 1 mM EDTA to stabilize dissolved Fe.
- vi. Carefully, without disturbing the residue, remove the remaining supernatant with a transfer pipet and discard into a waste container.
- vii. Air sparge the filtrate for at least 1 hour.
 - a. A colour change from clear to yellow–dark orange will occur in most samples.
 - b. Some samples may require sparging beyond 1 hour. A colour test (addition of reagent R1) can be performed on an aliquot to determine whether the dithionite has been sufficiently removed so as not to interfere with the colourimetric reagents, and subsequently, with the analysis.
- viii. Analyze filtrate for molybdate-reactive P (see Notes).

3. NaOH-reactive P

- i. Tare balance with 25 mL graduated cylinder. Measure 25 mL of 1 M NaOH into cylinder. Verify weight (26.0 g) and add to tube with residue from Extraction 2.
- ii. Shake manually to break up and re-suspend sediment pellet.
- iii. Shake (on table shaker, at low setting) for 16 hours.
- iv. Loosen cap briefly to allow gas to escape prior to centrifuging. Centrifuge at 8950 g for 20 minutes.
- v. Using a 0.45 µm syringe filter and syringe, filter supernatant into a labeled 50 mL centrifuge tube. Add 1 drop of 1 mM EDTA.

- vi. Carefully, without disturbing the residue, remove the remaining supernatant with a transfer pipet and discard into a waste container.
- vii. Analyze filtrate for molybdate-reactive P (see Notes).

4. HCl-reactive P

- i. Tare balance with 25 mL graduated cylinder. Measure 25 mL of 0.5M HCl into cylinder. Verify weight (25.3 g) and add to tubes with residues from Extraction 3.
- ii. Shake manually to break up and re-suspend sediment pellet.
- iii. Shake (on table shaker, at low setting) for 24 hours.
- iv. Loosen cap briefly to allow gas to escape prior to centrifuging. Centrifuge at 8950 g for 20 minutes.
- v. Using a 0.45 μm syringe filter and syringe, filter supernatant into a labeled 50 mL centrifuge tube.
- vi. Carefully, without disturbing the residue, remove the remaining supernatant with a transfer pipet and discard into a waste container.
- vii. Analyze filtrate for molybdate-reactive P (see Notes).

5. NaOH_(85°C)-reactive P

- i. Tare balance with 25 mL graduated cylinder. Measure 25 mL of 1 M NaOH into cylinder. Verify weight (26.0 g) and add to tube with residue from Extraction 4.
- ii. Shake manually to break up and re-suspend sediment pellet.
- iii. Heat in a 85°C water bath for 24 hours, shaking occasionally (e.g., every 2 hours, except overnight).
 - a. Secure lids finger-tight, only tighten for shaking.
- iv. Allow to cool for 10 minutes.

- v. Loosen cap briefly to allow gas to escape prior to centrifuging. Centrifuge at 8950 g for 20 minutes.
- vi. Using a 0.45 μm syringe filter and syringe, filter supernatant into a labeled 50 mL centrifuge tube. Add 1 drop 1 mM EDTA.
- vii. Analyze filtrate for molybdate-reactive P (see Notes).

Notes:

- If possible, analyze samples immediately after filtering. Analyze filtrates from Extraction 2 within 12 hours; remaining filtrates should be analyzed within 24 hours.
- If not analyzed immediately, filtrates should be refrigerated.
- Residues can be refrigerated overnight if subsequent extraction steps cannot proceed immediately.
- Filtrates were analyzed as per Thermo Fisher Scientific (2015). All calibration standards and dilutions (except NH_4Cl) were matrix-matched with same-batch extraction solutions. The NaOH and HCl extracts were neutralized automatically with HCl and NaOH, respectively.

Appendix B. Phosphorus fractionation quality control

B.1 Moisture content

Fifteen random samples from 2018 were selected for gravimetric moisture content (Topp et al., 2006). Using 2 g of dried silt (particles <63 µm, in this thesis), the analysis served to verify whether P concentrations needed to be corrected for moisture content, as the samples were not oven-dried (i.e., at 105°C). Only three samples had any detectable moisture, and the maximum moisture content was 0.4%. Therefore, all analyte concentrations were converted directly to a dry weight basis without any correction for moisture content.

B.2 Method blanks

Each sample batch included one method blank (n = 5 batches) to check for contamination due to reagents, supplies, and procedures. All method blank results were at least one order of magnitude below the LOQ, so any P introduced during the procedure (including any background P concentrations in the extractants) was negligible.

B.3 Repeatability and subsampling error

Twelve percent (12%) of road surface material and cutslope samples were extracted in triplicate to assess within-batch consistency and subsampling error. A certified reference material (TILL-1, Canadian Certified Reference Materials Project, Ottawa, ON, CA) was also used to evaluate within- and among-batch consistency: the reference material was analyzed in triplicate in one batch, and one subsample of the reference material was extracted and analyzed in each batch. The results were assessed on a dry weight basis (mg/kg).

For the road surface material and cutslope samples run in triplicate (within batches), the mean coefficient of variation (CV) for all five P fractions was below 4%. For subsamples of reference materials run in triplicate, the CV for any fraction did not exceed 5% (note that a CV for the NH₄Cl-RP could not be calculated, as the results were below the LOQ). The results indicated that there was minimal variation due to subsampling and showed consistency within batches. The among-batch CVs, assessed using the reference material, ranged from 4% (for the NaOH-RP fraction) to 12% (for the HCl-RP fraction). Again, a CV for the NH₄Cl-RP fraction could not be calculated as the results were below the LOQ. The CVs indicate the level of uncertainty in the reported concentrations.

B.4 Performance of the modified Psenner method

The performance of the P fractionation procedure was assessed by comparing the sum of the fractions to total P using a standard method. Subsamples of the reference material (1 subsample) and road materials (23 subsamples = 24% of samples analyzed for P) were submitted to the NRAL for total P analysis. Subsamples of 50–80 mg were digested with HNO₃ in a microwave (MARS Xpress Digestion Microwave System, CEM Corporation, Matthews, NC, USA) according to U.S. EPA Method 3051A (2007). Digests were analyzed using a Thermo iCAP 6000 series inductively-coupled plasma-optical emission spectrometer (ICP-OES; Thermo Fisher Scientific, Cambridge, UK). Total P values were averaged for samples run in triplicate, laboratory duplicates, or analytical duplicates.

The relative percent difference (RPD) between the sum of the P fractions and total P measured using the EPA method ranged from 0.4–29%, but on average, the values were within 10% (s.d. = 8%) of each other. The two values were strongly correlated ($\rho = 0.97, p < 0.001$;

Figure B.1). The fractionation procedure did not show any tendency toward under- or over-estimating total P among the road materials. However, the P fractionation procedure consistently underestimated total P in the reference material by an average of 24% (s.d. = 1%). This indicates that the P fractionation procedure performs better on some types of materials (e.g., different mineral composition) than others.

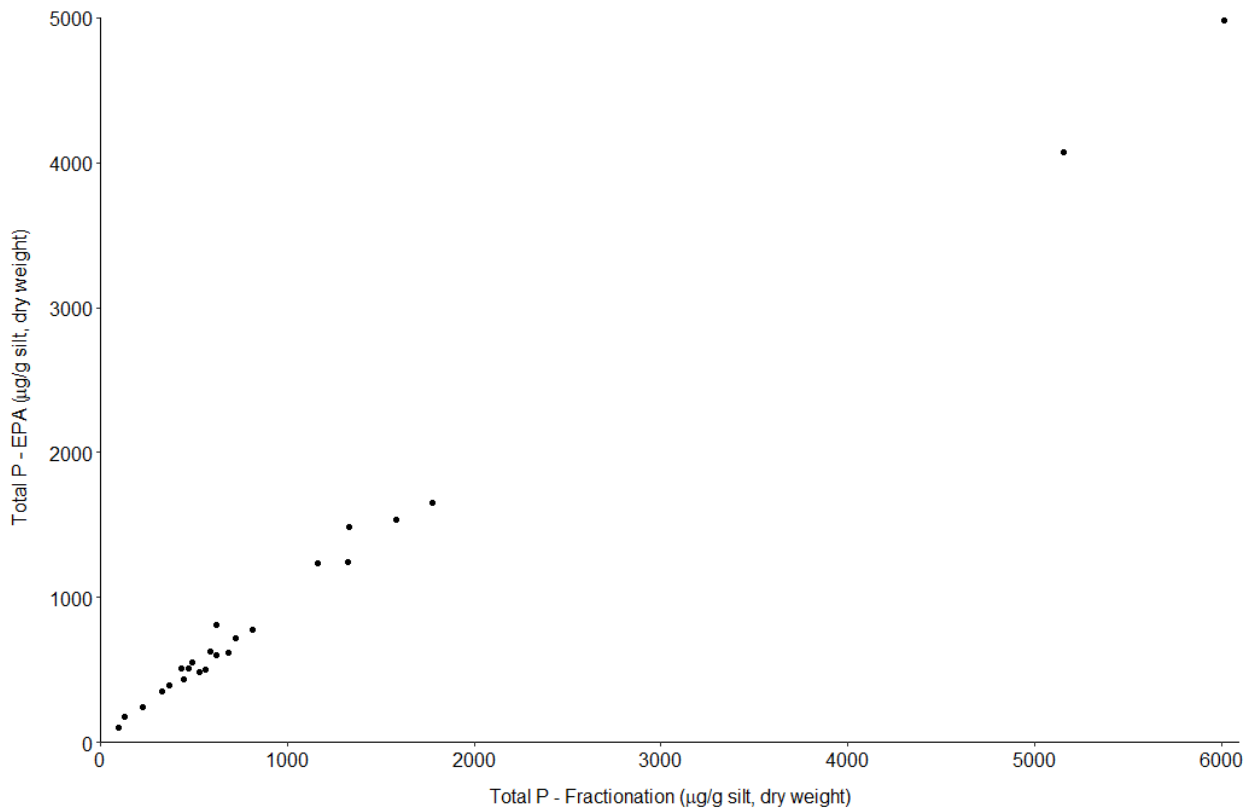


Figure B.1. Performance of the modified Psenner method (Total P–Fractionation) compared to a standard method (Total P–EPA).

Appendix C. Data

Sample No.	Terrestrial Ecozone	Watershed	Material Type	Silt Content - Sediment (%)	Silt Content - Material (%)	Total P (mg/kg) ⁵	NH4Cl-P (%)	BD-P (%)	NaOH-P (%)	NAIP (%)	AP (%)	OP (%)	NAIP (mg/kg)
120	Pacific Maritime	HR	Road	NA	25	685	0	2	3	5	89	6	34
121	Pacific Maritime	HR	Road	NA	32	770	0	4	13	17	76	7	128
122	Pacific Maritime	HR	Road	NA	15	788	0	3	8	11	82	7	88
123 ^b	Pacific Maritime	HR	Road	NA	27	931	0	1	3	4	91	5	39
124	Pacific Maritime	HR	Road	NA	22	818	0	3	13	16	77	7	130
43	Pacific Maritime	LL	Road	NA	15	492	0	7	53	60	23	17	293
44 ^a	Pacific Maritime	CB	Road	NA	42	826	0	4	12	16	78	6	132
49 ^a	Pacific Maritime	CB	Road	NA	46	812	0	2	5	7	89	5	53
50	Pacific Maritime	SG	Road	NA	9	NA	NA	NA	NA	NA	NA	NA	NA
54	Pacific Maritime	SG	Road	NA	9	NA	NA	NA	NA	NA	NA	NA	NA
65	Pacific Maritime	SG	Road	NA	61	1136	0	3	5	8	89	3	86
52 ^b	Pacific Maritime	SG	Cutslope	NA	14	1329	0	2	80	82	13	5	1089
46 ^b	Pacific Maritime	SG	Road	NA	15	718	0	6	7	13	83	4	97
53 ^a	Pacific Maritime	SG	Road	NA	11	1419	0	3	5	8	89	4	112
51	Pacific Maritime	SG	Cutslope	NA	29	587	0	3	77	80	7	14	468
66	Pacific Maritime	SG	Road	NA	61	285	0	6	47	53	33	14	152
47	Pacific Maritime	SG	Road	NA	15	878	0	3	11	14	82	5	119
81	Montane Cordillera	UB	Road	NA	NA	1058	0	4	11	15	77	7	163
3	Montane Cordillera	UB	Road	97	39	1582	0	4	9	13	82	6	199
34	Montane Cordillera	UB	Road	NA	27	NA	NA	NA	NA	NA	NA	NA	NA
38	Montane Cordillera	UB	Road	NA	27	NA	NA	NA	NA	NA	NA	NA	NA
39	Montane Cordillera	UB	Road	NA	14	NA	NA	NA	NA	NA	NA	NA	NA
23	Montane Cordillera	UB	Road	NA	17	NA	NA	NA	NA	NA	NA	NA	NA
59	Montane Cordillera	UB	Road	NA	45	917	0	3	5	8	83	10	70
10	Montane Cordillera	UB	Road	NA	31	NA	NA	NA	NA	NA	NA	NA	NA
32	Montane Cordillera	UB	Road	NA	44	1745	0	4	7	10	83	6	181
58	Montane Cordillera	UB	Cutslope	NA	87	1432	0	4	7	11	80	9	159
17 ^a	Montane Cordillera	UB	Road	NA	24	983	0	4	8	12	80	8	113
7	Montane Cordillera	FL	Road	92	51	1325	0	4	8	12	82	5	163
35	Montane Cordillera	FL	Road	NA	19	NA	NA	NA	NA	NA	NA	NA	NA
25 ^a	Montane Cordillera	FL	Road	NA	11	1410	0	3	9	12	84	4	169
40 ^a	Montane Cordillera	FL	Road	NA	12	734	0	5	9	14	80	6	103
67	Montane Cordillera	FL	Cutslope	NA	70	829	0	3	4	7	87	5	59
37	Montane Cordillera	FL	Road	NA	15	NA	NA	NA	NA	NA	NA	NA	NA
11 ^b	Montane Cordillera	UP	Road	NA	49	992	0	2	3	5	86	8	53
75	Montane Cordillera	UP	Cutslope	NA	88	985	0	2	6	9	81	10	87
13	Montane Cordillera	UP	Road	NA	44	1305	0	1	1	2	90	8	32
77	Montane Cordillera	UP	Cutslope	NA	66	1075	0	1	1	2	91	7	22
69	Montane Cordillera	UP	Road	NA	68	954	0	3	2	6	89	6	52
2 ^b	Montane Cordillera	UP	Road	99	19	1023	0	3	2	5	87	8	51
42	Montane Cordillera	DC	Road	NA	10	2934	0	1	1	2	96	2	61
61 ^b	Montane Cordillera	DC	Road	NA	40	1161	2	8	21	31	61	8	358
68	Montane Cordillera	DC	Road	NA	61	1166	1	3	3	6	89	5	75
48	Montane Cordillera	DC	Cutslope	NA	45	1050	1	7	10	18	75	7	190
9	Montane Cordillera	MC	Road	75	41	440	0	6	62	67	18	15	296
24	Montane Cordillera	MC	Road	NA	68	308	0	15	44	58	29	13	179
72 ^{a,c}	Montane Cordillera	MC	Cutslope	NA	96	128	0	30	50	80	4	15	103
29 ^{a,b}	Montane Cordillera	KC	Road	NA	25	222	0	9	24	33	46	21	74
71	Montane Cordillera	KC	Cutslope	NA	91	205	1	15	40	57	7	36	116
70	Montane Cordillera	FC	Road	NA	68	482	0	4	6	11	78	11	51
56	Montane Cordillera	FC	Cutslope	NA	33	515	0	4	10	13	68	18	69

Sample No.	Terrestrial Ecozone	Watershed	Material Type	Silt Content - Sediment (%)	Silt Content - Material (%)	Total P (mg/kg) ⁵	NH4Cl-P (%)	BD-P (%)	NaOH-P (%)	NAIP (%)	AP (%)	OP (%)	NAIP (mg/kg)
80	Montane Cordillera	FC	Road	NA	70	NA	NA	NA	NA	NA	NA	NA	NA
22	Montane Cordillera	FC	Road	NA	23	2157	0	1	1	2	96	2	41
5 ^a	Montane Cordillera	FC	Road	99	21	396	0	3	3	7	83	10	26
26	Montane Cordillera	FC	Road	NA	19	NA	NA	NA	NA	NA	NA	NA	NA
60	Montane Cordillera	FC	Road	NA	8	NA	NA	NA	NA	NA	NA	NA	NA
31 ^b	Montane Cordillera	FC	Road	NA	19	491	0	9	30	39	47	14	191
57	Montane Cordillera	BG	Road	NA	41	NA	NA	NA	NA	NA	NA	NA	NA
4 ^b	Montane Cordillera	BG	Road	100	28	472	1	5	5	10	83	7	48
18 ^c	Montane Cordillera	UW	Road	NA	16	445	0	1	1	3	91	6	13
19 ^c	Montane Cordillera	UW	Road	NA	14	366	0	0	1	2	92	6	6
16	Montane Cordillera	UW	Road	NA	11	NA	NA	NA	NA	NA	NA	NA	NA
62	Montane Cordillera	UW	Cutslope	NA	79	NA	NA	NA	NA	NA	NA	NA	NA
28	Montane Cordillera	UW	Road	NA	7	640	0	2	3	5	88	8	29
1 ^b	Boreal Plains	UW	Road	98	10	330	0	0	0	0	95	5	<12
63	Boreal Plains	UW	Cutslope	NA	48	433	1	2	2	4	87	9	17
73	Boreal Plains	UW	Road	NA	81	363	0	8	25	33	52	15	120
14 ^a	Boreal Plains	UW	Road	NA	81	NA	NA	NA	NA	NA	NA	NA	NA
76 ^b	Boreal Plains	UW	Road	NA	81	503	0	19	37	57	27	16	286
27	Boreal Plains	RD	Road	NA	21	548	0	8	16	24	63	13	132
74	Boreal Plains	RD	Cutslope	NA	76	442	0	10	25	34	48	18	152
41	Boreal Plains	RD	Road	NA	44	562	0	3	9	12	77	11	68
78	Boreal Plains	RD	Cutslope	NA	86	462	0	7	21	28	57	16	128
6	Boreal Plains	RD	Road	100	19	NA	NA	NA	NA	NA	NA	NA	NA
20	Boreal Plains	RD	Road	NA	43	543	0	9	19	29	60	11	157
21 ^b	Boreal Plains	RD	Road	NA	25	622	0	4	6	11	81	8	66
36	Boreal Plains	RD	Road	NA	19	NA	NA	NA	NA	NA	NA	NA	NA
30	Boreal Plains	RD	Road	NA	14	482	0	7	28	35	49	16	169
12	Boreal Plains	KR	Road	NA	51	654	0	2	8	10	74	16	63
79	Boreal Plains	KR	Road	NA	80	722	0	4	11	15	72	13	110
8	Boreal Plains	KR	Road	100	24	792	0	4	7	11	80	9	86
33 ^b	Boreal Plains	KR	Road	NA	28	720	0	6	12	18	67	15	130
15 ^b	Boreal Plains	KR	Road	NA	28	1428	1	7	9	17	71	12	239
83 ^b	Boreal Plains	RE	Road	NA	9	526	0	7	7	15	76	10	77
82	Boreal Plains	RE	Road	73	39	617	0	9	13	22	67	11	135
84	Boreal Plains	RE	Road	NA	16	585	0	7	9	16	74	11	92
85	Boreal Plains	RE	Road	NA	14	764	0	9	13	22	63	15	170
118	Boreal Shield	EL	Road	NA	8	670	0	2	2	3	93	4	23
94	Boreal Shield	EL	Road	22	10	826	0	2	3	5	91	4	38
92	Boreal Shield	EL	Road	NA	10	664	0	3	18	22	73	6	143
91	Boreal Shield	EL	Road	NA	31	530	0	3	15	18	73	9	94
117	Boreal Shield	WL	Road	NA	9	595	0	2	5	6	81	13	37
96	Boreal Shield	WL	Road	NA	7	513	0	3	3	6	86	8	31
104	Boreal Shield	KH	Road	NA	8	1775	0	0	5	6	91	3	100
112	Boreal Shield	KH	Road	NA	5	1395	0	1	1	2	96	2	26
97	Boreal Shield	KH	Road	NA	6	1309	0	0	6	6	91	3	72
93 ^b	Boreal Shield	KH	Road	8	7	1527	0	0	9	9	86	5	140
98 ^b	Boreal Shield	KH	Road	NA	7	1017	0	0	9	9	84	7	94
113	Boreal Shield	KH	Road	NA	6	1796	0	0	2	2	96	2	32
100	Boreal Shield	RM	Road	NA	6	5373	0	0	2	2	96	2	108
99	Boreal Shield	RM	Road	NA	16	5215	0	0	5	5	94	1	262
101	Boreal Shield	RM	Cutslope	NA	24	5158	0	0	15	15	83	2	766

Sample No.	Terrestrial Ecozone	Watershed	Material Type	Silt Content - Sediment (%)	Silt Content - Material (%)	Total P (mg/kg) [§]	NH4Cl-P (%)	BD-P (%)	NaOH-P (%)	NAIP (%)	AP (%)	OP (%)	NAIP (mg/kg)
119	Boreal Shield	RM	Road	NA	6	4871	0	0	3	3	95	2	151
116 ^b	Boreal Shield	RM	Road	NA	8	6016	0	0	2	2	96	2	123
103	Boreal Shield	RM	Road	NA	7	4927	0	0	1	1	98	1	63
89	Atlantic Maritime	TC	Road	NA	7	276	0	7	28	35	41	24	98
109 ^b	Atlantic Maritime	TC	Road	NA	24	97	0	12	51	62	0	38	61
114	Atlantic Maritime	TC	Road	NA	23	125	0	13	61	74	0	26	92
106 ^b	Atlantic Maritime	TC	Road	NA	10	526	0	3	13	15	78	6	81
105	Atlantic Maritime	TC	Road	NA	15	472	0	6	56	62	23	16	291
107	Atlantic Maritime	TC	Road	55	11	330	0	10	58	67	8	25	222
111	Atlantic Maritime	PT	Road	NA	10	586	0	4	9	12	81	7	72
108	Atlantic Maritime	PT	Road	9	6	794	0	1	11	12	84	5	95
90	Atlantic Maritime	PT	Road	NA	30	649	0	2	15	17	71	12	112
110	Atlantic Maritime	PT	Road	NA	17	663	0	3	9	12	81	7	79
95	Atlantic Maritime	PT	Road	NA	9	790	0	1	2	3	91	7	21

[§] Total P calculated as the sum of P in the apatite P (AP), non-apatite inorganic P (NAIP), and organic P (OP) fractions

^a Reported result is an average of duplicate or triplicate subsamples with the lowest relative standard deviation (RSD)

^b Reported result is an average of replicate subsamples and/or re-runs

^c Sample was re-run but reported result is for a single run only

<LOQ Result below limit of quantitation

NA Not analyzed, except for rainfall simulations where NA = no rainfall simulation