Use of pre-industrial floodplain lake sediments to establish baseline river metal concentrations downstream of Alberta oil sands: a new approach for detecting pollution of rivers

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Abstract
In the Alberta oil sands region, insufficient knowledge of pre-disturbance reference conditions has undermined the ability of the Regional Aquatics Monitoring Program (RAMP) to detect pollution of the Athabasca River, because sampling began three decades after the industry started and the river naturally erodes oil-bearing strata. Here, we apply a novel approach to characterize pre-industrial reference metal concentrations in river sediment downstream of Alberta oil sands development by analyzing metal concentrations in sediments deposited in floodplain lakes of the Athabasca Delta during 1700–1916, when they were strongly influenced by Athabasca River floodwaters. We compared results to metal concentrations in surficial bottom sediments sampled by RAMP (2010–2013) at downstream sites of the Athabasca River and distributaries. When normalized to lithium content, concentrations of vanadium (a metal of concern in the oil sands region) and other priority pollutants (Be, Cd, Cr, Cu, Pb, Ni, Zn) in nearly all of the RAMP river sediment samples lie below the upper 95% prediction interval linearly extrapolated from the river-derived lake sediments. Assuming the RAMP protocols obtained recently deposited sediment, this indicates that the metal concentrations in downstream Athabasca River sediment have not increased above pre-disturbance levels. Reference conditions derived from the lake sediment data were used to develop profiles of metal residual concentrations versus time for the RAMP river sediment data, which provides an excellent tool for decision-makers to identify and quantify levels of metal pollution for any given sample, and to monitor for future trends. We recommend that the approach be applied to resurrect the utility of RAMP data at other river sampling locations closer to the development, and for ongoing risk assessment. The approach is also readily transferable to other rivers where insufficient pre-disturbance reference data impairs...
it is important to establish as rigorously as possible the background or baseline level of pollution, against which any future trends can be assessed’ (Dowdeswell et al 2010, p 31).

Here, we respond to this recommendation of Dowdeswell et al (2010) by developing the use of downstream floodplain lake sediments, deposited prior to development of the Alberta oil sands (and before evidence of regional atmospheric metal pollution; Wiklund et al 2012), as an archive of pre-industrial baseline river sediment metal concentrations. Our objective was to employ these pre-industrial baseline data to evaluate RAMP river bottom-sediment metal concentrations and test whether oil sands development is polluting the downstream Athabasca River. Specifically, we report results for several metals on the US Environmental Protection Agency (EPA) list of priority pollutants (Be, Cd, Cr, Cu, Pb, Ni, Zn) and V. We analyzed for these metals because Kelly et al (2010) reported elevated deposition to the snowpack near the oil sands development and higher concentrations in river water downstream of development. Thus, we aimed to determine if this evidence of enhanced metal concentrations, which has been associated with mining activity, was detectable in downstream surface sediments of the Athabasca River. Also, bitumen in the McMurray Formation contains higher V content than conventional crude oils and other bitumen formations (Jacobs and Filby 1982, Reynolds et al 1989, Gosselin et al 2010), and studies have demonstrated V can be toxic to plants, invertebrates, fish, wildlife and humans (Irwin et al 1997).

2. Methods

We performed metal analyses on previously obtained $^{210}\text{Pb}$-dated sediment cores from lakes ‘PAD 23’ and ‘PAD 31’ in the Athabasca Delta (Hall et al 2012; figure 1). These lakes were flood-prone from the 1700s to the early 1900s and, therefore, the Athabasca River was the main source of sediment during this time (Hall et al 2004, Wolfe et al 2008, Johnston et al 2010). Samples of freeze-dried sediment were analyzed for metals (method: EPA 200.2/6020A) at ALS Canada (Edmonton). Results are reported for pre-1920 sediments from PAD 23 (1823–1916 CE; sediment core depths: 31–43 cm) and PAD 31 (1699–1912 CE; sediment core depths: 26–39 cm). Concentration data for metals from Athabasca River surficial bottom sediment were obtained from the RAMP database from six sampling stations located in the Athabasca Delta near our lake sediment core sites (RAMP 2014a; figure 1). The samples collected by RAMP are assumed to represent recently deposited sediment.

We first developed linear relations for the pre-1920 lake sediment metal concentrations relative to Li concentration, as is commonly done to normalize for grain-size effects, because metals adsorb preferentially to clay-sized sediment (Loring 1991, Kersten and Smedes 2002). Geochemical normalization of metal concentration data is important because spatial and temporal fluctuations in the energy of Athabasca River flow generate grain-size variations, and floodplain lakes preferentially receive the low energy, fine-grained fraction carried by floodwaters. We assumed that the metals of interest and Li in the pre-1920s lake sediment have a common source for both lakes (i.e., the Athabasca River) and thus their concentrations should cluster along a line that passes through the origin when graphed as crossplots (Loring 1991, Kersten and Smedes 2002). We used the 95% prediction interval (PI)
about this relation (appendix B, C; equation (C.1)) to define the natural range of variability of metal concentrations, consistent with Loring (1991). We then tested whether the RAMP river sediment metals data plotted below the linearly-extrapolated upper 95% PI of the pre-1920 lake sediment linear relation, which would indicate a common natural source for both datasets. On the other hand, if >2.5% of the RAMP river sediment metals data fall above the upper 95% PI, this would indicate that the metal concentrations are greater than predicted by the pre-1920 lake-sediment metal-Li relation; a result that identifies an additional source of the metal possibly due to pollution (Loring 1991, Kersten and Smedes 2002). Of common normalizing agents, we selected Li because it showed the strongest linear relations with metals of interest. Although Li data are only available for recent years of RAMP sampling (2010–2013), this captures a period of rapid growth of the oil sands industry and peak production (CAPP 2014). Also, the RAMP samples were analyzed by ALS Canada (Edmonton) during these years using the same methodology (EPA 200.2/6020A; RAMP, 2014b) as we used for the pre-1920 lake sediment samples.

3. Results

We illustrate the application of this approach using V, a metal commonly associated with oil and oil-related pollution (Khalaf et al 1982, Juichang et al 1995), including the Alberta oil sands (Baker et al 2012). The pre-1920s lake sediments at PAD 23 and PAD 31 possess concentrations of V and Li that overlap and cluster along a regression line that is fixed at the origin (figure 2(a)), supporting the assumption that the V and Li are derived from a common source (i.e., Athabasca River). Thus, the 95% PIs about the regression line define the expected natural range of V concentration, relative to Li concentration, for individual samples of Athabasca River sediment deposited in these lakes by floodwaters. Samples with relatively high metal concentrations are associated with lower energy conditions and finer-grained sediment, whereas lower concentrations are associated with higher energy conditions and coarser-grained sediment.

The RAMP river sediment samples plot along the same V-Li linear relation as the pre-1920 lake sediment data and fall within the 95% PIs (figure 2(b)), indicating that the river and lake sediments have a common, natural source of these elements whose composition has not changed. The RAMP values partially overlap with the pre-1920 lake sediment data and also span lower concentrations, as expected given the higher energy conditions and coarser grain size of bottom sediments in the Athabasca River. We obtained similar results for the other metals analyzed (figure 3). Of the 135 metal-Li measurements obtained by RAMP on river bottom-sediment samples collected during 2010–2013 from the Athabasca Delta sampling locations, only four (2.96%) fall above the linearly-extrapolated 95% PIs (Cr: GIC-1 2013, FLC-1 2013; Cu: EMR-2 2010, EMR-1 2011; figures 3(C)–(D)). Of these four measurements, only one (Cu: EMR-2 2010) lies distinctly above the linearly-extrapolated upper 95% PI (figure 3(d)). Thus, use of our approach indicates that the RAMP river sediment concentration data for Be, Cd, Cr, Cu, Pb, Ni, V and Zn provide little to no evidence that they have been influenced by pollution, and therefore have not been enriched by oil sands development. Instead, the RAMP river sediment metal concentration data we examined (2010–2013) align within the ranges expected under natural conditions, based on metal concentration data from river-sourced sediment deposited in nearby lakes before 1920.

To facilitate identifying and quantifying pollution in river sediment samples, as well as the detection of trends in future monitoring data, we converted the results presented in crossplots of figures 2 and 3 to time-series of metal concentration ‘residuals’ in figure 4. With this graphical display,
temporal trends of past and future monitoring data can be assessed relative to the baseline (i.e., residual metal concentration = 0) established from the pre-industrial lake-sediment data, and individual sample results can be evaluated for pollution immediately upon data acquisition using values provided in appendix B and equations provided in appendix C. For the available RAMP data, we were able to determine that three of the 135 RAMP river sediment analyses (=2.20%) plot above the linearly-extrapolated upper 95% PI, after accounting for analytical uncertainty of metal concentrations and statistical uncertainty of the reference metal-Li relation (see appendix C).

4. Discussion and conclusions

Based on our analysis utilizing newly acquired pre-industrial baseline data of metal concentrations in river-derived floodplain lake sediment, we detect little to no evidence of pollution by the oil sands development in downstream surficial bottom sediments of the Athabasca River. Our study used RAMP data from river sample locations ~200 km downstream of the oil sands development, because the lower Athabasca River, Lake Athabasca, Athabasca Delta and downstream community of Fort Chipewyan are locations where concerns have been expressed regarding effects of increasing oil sands development on human and ecosystem health (Timoney 2007, Timoney and Lee 2009, Schindler 2010). Also, the delta is where sediments and their contaminant load carried by the Athabasca River accumulate (Dillon et al 2011), and this is where we had pre-disturbance lake sediment core data to serve as reference material for applying this approach. Notably, our data show that the Athabasca River has been a source of metals to the Athabasca Delta for centuries before oil sands development, equivalent to that measured in recent years. This conclusion is similar to findings based on sediment core analyses of river-transported bitumen-associated indicator polycyclic aromatic compounds from the same lakes (Hall et al 2012). Although the Athabasca Delta has long been a natural repository of contaminants, metal concentrations have remained below the threshold effects level (TEL) of the interim sediment quality guidelines for the protection of aquatic life for those metals where this has been established (including Cd (TEL = 0.6 mg kg⁻¹), Cr (37.3 mg kg⁻¹), Cu (35.7 mg kg⁻¹), Pb (35.0 mg kg⁻¹) and Zn (123.0 mg kg⁻¹); CCME 2014).

We advocate that this approach be used for ongoing risk assessment of oil sands development on metal pollution in the Athabasca River. We show that metal concentrations in surficial river bottom sediments have not yet become elevated above pre-industrial reference conditions at the downstream terminus of the Athabasca River, but our study does not address concerns of river sediment metal pollution closer to the oil sands development. Consequently, we recommend that pre-disturbance sediments be obtained from flood-prone lakes located adjacent to the Athabasca River and its tributaries closer to oil sands development. Such lakes may provide more representative reference conditions for assessing pollution due to oil sands development from data archived from RAMP’s upstream river-bottom-sediment sampling stations. We consider the collection of these data a high priority to better inform the interpretation of river sediment quality data collected from ongoing monitoring.

Importantly, the approach we developed here, based on paleolimnological determination of pre-disturbance contaminant concentrations in sediments deposited into floodplain lakes from the river system of interest, allows for use of powerful before-after study designs in situations where such designs otherwise would not be possible (Forstner and Müller 1981, Bowman and Somers 2005). For example, paleolimnological inference of baseline conditions offers an alternative for situations where representative reference sites are difficult or impossible to establish using space-for-time approaches (e.g., control-impact and reference condition approaches), and where absence of real-time pre-disturbance measurements from the river of interest permit only use of weaker upstream-downstream comparison and temporal-change assessment approaches (Bowman and Somers 2005).

Ability of the latter two approaches to detect pollution can be compromised when natural processes also supply the contaminants of concern and when trends in confounding factors...
Figure 3. Relations of priority pollutants versus lithium in lake and river sediment. Concentrations are shown for pre-1920 sediment from delta lakes (PAD 23, PAD 31) and RAMP river bottom sediment (collected in 2010–2013) for (a) Be, (b) Cd, (c) Cr, (d) Cu, (e) Pb, (f) Ni and (g) Zn. Relation is linear regression, fixed at the origin (solid line), and includes 95% prediction intervals (dashed lines) for the pre-1920 sediment from the delta lakes. Dashed-dotted lines represent the upper 95% prediction interval based on linear extrapolation of the widest 95% prediction interval from the pre-1920 lake sediment data (appendix B). RAMP Samples positioned above the 95% prediction intervals are labeled with their site code and year, as reported by RAMP (2014a).
(e.g., declining river flow) are correlated with the pace of industrial development, respectively (Gosselin et al. 2010, Timoney and Lee 2011). In situations where our approach can be applied to a comparable unaffected reference river as well as to the impacted river, then investigators can apply the most rigorous ‘before-after, control-impact’ (or, BACI) study design to detect pollution from an anthropogenic source (Bowman and Somers 2005). Thus, we anticipate our approach will find broad applicability to other river ecosystems where monitoring efforts are hampered by absence of

**Figure 4.** Temporal trends in metal concentrations as residuals relative to the pre-1920s lake sediment baseline. Panels (a) Be, (b) Cd, (c) Cr, (d) Cu, (e) Pb, (f) Ni, (g) V and (h) Zn include pre-1920 sediment core data from lakes PAD 23 and PAD 31 versus date of deposition, and RAMP river-bottom sediment data from Athabasca Delta sampling locations versus date of sediment collection. See appendix C for equations used to generate the 95% prediction intervals for the linear regression and error bars for the residual metals concentrations. Horizontal solid line at 0 represents pre-industrial (pre-1920) reference. Dashed-dotted lines represent linear extrapolation of the widest 95% prediction interval from the pre-1920 lake sediment data. Sediment samples with lower error bar above the upper 95% prediction interval can be interpreted as enriched in the metal of concern, possibly due to pollution (including Cr: FLC-1 2013; Cu: EMR-2 2010, EMR-1 2011).
pre-disturbance data, or where duration of monitoring records is too short to detect a pollution trend.

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Appendix A

Table A1. Athabasca Delta lake coring locations and RAMP river bottom sediment quality monitoring locations (WGS84).

| Sites               | Latitude | Longitude |
|---------------------|----------|-----------|
| PAD 23              | 58.391617| -111.443333|
| PAD 31              | 58.493767| -111.517150|
| ATR-ER (Athabasca River upstream of Embarras River) | 58.353316 | -111.541848|
| BPC-1 (upper Big Point Channel) | 58.590791 | -110.795243|
| EMR-1 (upper Embarras River) | 58.358268 | -111.550145|
| EMR-2 (lower Embarras River) | 58.567500 | -111.092222|
| FLC-1 (Fletcher Channel) | 58.564539 | -111.062198|
| GIC-1 (Goose Island Channel) | 58.588101 | -110.835251|

Appendix B

Table B1. Values to generate the regression lines and 95% PIs for the metal-Li relations. Linear regression coefficients and statistical error terms required to calculate 95% PIs and uncertainty of residual metals concentrations. Also included are the precision values for the replicate metal concentration measurements. Formulas used to calculate the 95% PIs and error bars on RAMP surficial bottom sediments from stations in the Athabasca Delta are presented in appendix C.

| Metal | Slope (m) coefficient | Std. error (μm) | MSresidual (S_{xy}^2) | % Precision of replicatesa |
|-------|-----------------------|-----------------|------------------------|-----------------------------|
| Be    | 4.047×10^{-2}         | 6.114×10^{-4}   | 0.0039                 | 3.30                         |
| Cd    | 1.727×10^{-2}         | 3.389×10^{-4}   | 0.0012                 | 9.23                         |
| Cr    | 1.0586                | 1.355×10^{-2}   | 1.9172                 | 4.72                         |
| Cu    | 1.2565                | 1.529×10^{-2}   | 2.4416                 | 4.56                         |
| Pb    | 0.6386                | 1.345×10^{-2}   | 1.8883                 | 8.74                         |
| Ni    | 1.4160                | 1.971×10^{-2}   | 4.0533                 | 2.46                         |
| V     | 1.8748                | 3.217×10^{-2}   | 10.8002                | 5.32                         |
| Zn    | 4.2703                | 1.053×10^{-1}   | 115.7424               | 2.33                         |

| Predictor (X) | Mean value ( ) |
|---------------|----------------|
| Li            | 19.5426        |

a Mean % precision calculated from all duplicate PAD 23 and PAD 31 lake sediment samples sent to ALS Canada (Edmonton) for metals determinations. As the RAMP data generally show lower absolute values for metal concentrations, the mean % precision values of PAD lake sediment samples may underestimate the variability in RAMP metal determinations.

Appendix C

Below, we present equations used to determine statistical uncertainties for the metal-Li regressions (figures 2, 3) and metal residuals concentrations (figure 4).

C1. Prediction intervals for metal-Li concentration regressions

The 95% prediction intervals (PIs) shown in figures 2 and 3 for the metal-Li relations are calculated as:

\[ E \pm \tilde{t}_{0.025,n-2} \sqrt{S_{\text{res}}^2 + \left( \frac{1}{n} + \frac{(X_i - \bar{X})^2}{\sum (X_i - \bar{X})^2} \right)} \tag{C.1} \]

where \( E \) is the expected mean concentration of a metal \((E = mX_i)\) in mg kg\(^{-1}\), where \( m \) is the estimated slope coefficient from linear regression of the lithium-metal relation (see appendix B for values), \( \tilde{t}_{0.025,n-2} = 2.0595 \); the two-tailed \( t \)-value for the sample size \((n = 27)\).

\( S_{\text{res}}^2 \) is the mean square residual \((S_{\text{res}}^2 \text{ in appendix B) from linear regression, } X_i \text{ is the lithium concentration (mg kg}^{-1}\); \( \bar{X} \) is the average concentration of \( X \) (lithium; 19.54 mg kg\(^{-1}\)) and \( \sum (X_i - \bar{X})^2 \) is the sum of squared residuals of lithium concentration; in this case =125.4035.

In figures 2–4, dashed-dotted lines represent linear extrapolation of the widest 95% prediction interval from the pre-1920 lake sediment data, which corresponds to the highest measured lithium concentration \((X_i = 23.4 \text{ mg Li kg}^{-1})\), which provides a conservative estimate of the expected range of values for the RAMP data.
C2. Uncertainty of residuals

Error bars in figure 4 are the uncertainty (u) of the residual (observed—expected) metal concentrations for individual RAMP river bottom sediment samples:

\[
u_{\text{residual}} = \sqrt{\left(\frac{Y_i \times \left(\frac{\%\text{PrecMetal}}{100}\right)}{m}\right)^2 + \left(u_E\right)^2 }, \quad (C.2)
\]

where \(Y_i\) is the measured metal concentration for sample \(i\), \(\%\text{PrecMetal}\) is the mean % precision for a metal concentration from replicate sample analyses (see appendix B for values), \(u_E\) is the uncertainty of the mean expected value \(E (E=mXi)\), based on regressions presented in figures 2 and 3, where

\[
u_E = E\sqrt{\left(\frac{\mu_m}{m}\right)^2 + \left(\frac{\%\text{PrecLithium}}{100}\right)^2 }, \quad (C.3)
\]

where \(m\) is the estimated slope coefficient from linear regression and \(\mu_m\) is the uncertainty of the slope estimate for a given lithium-metal relation (see appendix B for values). \(\%\text{PrecLithium}\) is the % precision for lithium concentration from replicate sample analyses (see appendix B for values) and \(E\) is the expected mean concentration of a metal \((E=mXi; \text{ in mg kg}^{-1})\).

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