

A chemical analysis of sediment sources in the
Le Sueur River, southern Minnesota

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Abstract

Recently, it has been found that the Minnesota River is contributing a disproportionate amount of sediment to the Mississippi River. Much of this unnatural influx comes from the Le Sueur River watershed in Blue Earth County near Mankato, MN. Using geochemical techniques we attempted to determine the source of Le Sueur River sediment. By using rare earth and trace elements as “fingerprints”, we analyzed topsoil, bluff, and ravine sediments of the river valley. We found that the geochemical composition of heavy minerals can be used to discriminate between bluff and ravine sediments. However, due to a shortage of suspended sediment sample we could not measure the heavy mineral fraction of suspended sediments and thus could not use our findings to determine the sediment source proportions.

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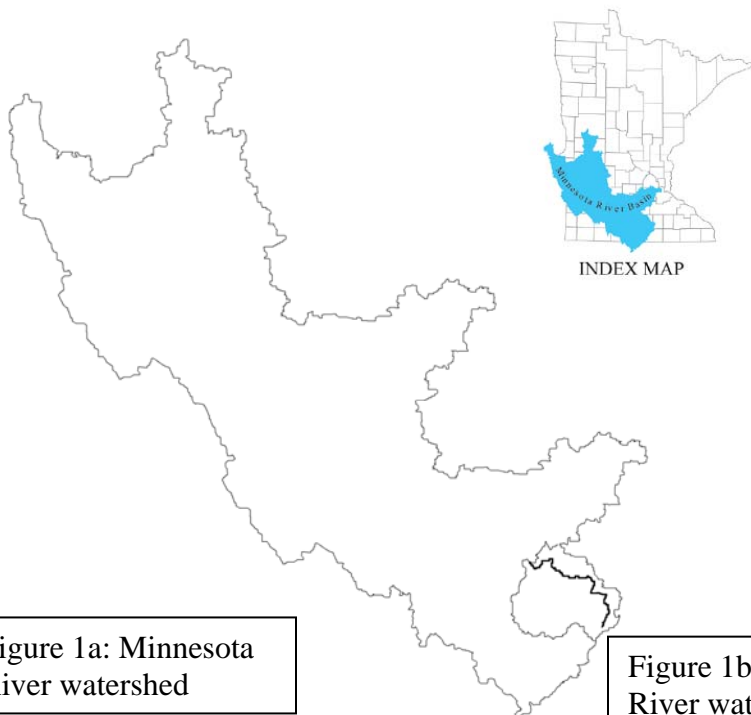
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INTRODUCTION:

In recent years, the Minnesota River and its watershed (fig. 1) have been implicated in the pollution of Lake Pepin, a natural widening in the river downstream of the confluence of the Minnesota River with the Mississippi River. According to Environmental Protection Agency regulations, Lake Pepin does not meet the standard for turbidity, a measure of suspended sediment in water, largely due to unnatural fluxes of sediment and nutrients entering the lake from tributaries upstream (Engstrom et al, 2009).



However, the precise source of this sediment on the landscape is unknown.

To identify the most important source of sediments entering the lake, Kelly (et al., 2000) attempted to geo-chemically fingerprint

the major tributaries of

Lake Pepin. By analyzing 42

elements in suspended sediment samples from the Upper Mississippi, St. Croix and Minnesota rivers, Kelly was able to statistically distinguish sediment between the three. Using a sediment core from Lake Pepin, the group then used a chemical mass balance

model to determine the proportions each river contributed to Lake Pepin sediment over time. The results they found were groundbreaking.

Of the total sediment entering Lake Pepin, 90% (+/- 1.4) of it is contributed by the Minnesota River (Kelly 2000). More importantly, this proportion has risen since the early 1800's and the onset of European settlement, indicating anthropogenic causes to the increased sediment load. Because the Minnesota River is the primary source for sediment entering the lake, recent efforts have been focused on the river and its tributaries.

According to the National Center for Earth Surface Dynamics at the University of Minnesota, 33% of sediment in the Minnesota River comes from the Le Sueur River even though it is only 7% of the total watershed (fig. 1). With such a large input for its size, there is high probability that human activity is cause to the increased sediment load.

Located in southern Minnesota, the Le Sueur River drains parts of four counties before flowing into the Blue Earth River near Mankato, Minnesota in Blue Earth County.

Draining largely agricultural land, these rivers contribute to the greater Minnesota River watershed that empties much of southern and southwestern Minnesota (Lorenz D., 1990).

Within the river, bluffs, ravines, and topsoil have been cited as the three possible sources of sediment contributing to the river turbidity. Defined as a steep riverbank or cliff, bluffs are steep slopes where active erosion or steep slopes undermine vegetative growth. Bluffs along the Le Sueur river are composed primarily of till deposited by the glaciers during the last glaciation. Ravines are erosional features that feed into the river system. The fluvial deposits within ravines are in contrast to the sides of ravines which presumably have similar composition to the unchanged till in bluffs. Originally, the land

was covered uniformly with till, but the processes of erosion and creation of ravines may accumulate specific parts of the soil, giving ravines their own distinct chemical signature.

Similar to the river valley, ravines are cut by running water but are narrower with steeper slopes. In the Le Sueur River watershed, these features contain water seasonally, but still could contribute to the large influx of sediment into the river. Both bluffs and ravines are often a river's response to a lowering of base level, the lowest point to which a river can flow. Because rivers have an ideal profile of efficiency, a drop in base level causes the river to erode away sediment until it reaches achieves a more efficient slope. Bluffs and ravines can indicate that a river is actively eroding sediment to reach equilibrium.

Finally, topsoil is simply the sediment horizon closest to the land surface. Farms in southern Minnesota thrive off of the region's thick, nutrient rich top soil. The disturbance of the land by farming could mobilize this resource and increase sediment load into the Le Sueur River. However, other initial studies indicate that suspended sediment in the river is not contributed by top soils (Schottler, personal communication, 2008) making this sediment source a non-factor in this study.

With down cutting and erosion in the river valley, it is possible that while the overlying till was once uniform in composition, weathering processes due to ravine formation and topsoil development could have affected it differently as sediment makes its way to rivers and eventually downstream. These processes could create measurable differences in the geo-chemical signatures of ravines and bluffs.

Using a similar method to Kelly et al (2000), we attempted to geochemically distinguish bluff and ravines and compare them to suspended sediment in the river.

Successfully differentiating the sediment sources can help in deciding what actions need to be taken in cleaning up the watershed to protect Lake Pepin. Bluffs, for example would take different stabilization techniques than ravines would.

To best study these sources, we tested two different components of each sediment sample, the “whole” sediment, and the “heavy mineral” sediment. Testing the heavy mineral fraction of the sediment is helpful in that it eliminates some of the “background noise” potentially added to the sediment by other components. It is possible that organics, carbonates, iron oxides, and light minerals can enter the various sediment sources via random processes such as organismal activity or groundwater percolation. These processes could mask the true signature of the sediment sources we are trying to study because they are not necessarily the actual “fingerprint” of the source. Testing heavy minerals for their chemical content could serve as a control to each sediment source. On the other hand, rivers are not selective with what they carry, so it is of equal importance that each source is tested for chemical composition as a whole component as well.

The section of the Le Sueur River watershed that contributes most of the sediment to the river is below the town of St. Clair, MN to just upstream of Blue Earth county road 90 (Minnesota Pollution Control Agency, personal communication, 2008). St. Clair is approximately the location of a knick point in the Le Sueur River (Patrick Belmont, NCED, personal communication, 2008), which is defined as an abrupt change in slope. Knick points are a river’s in response to a lowering of base level or other hydrological changes. By actively down cutting and eroding along the knick point, a river is attempting to reach equilibrium with the new conditions.

Because the amounts of sediment in the Le Sueur and Minnesota Rivers are unnaturally high, anthropogenic activity is undoubtedly a large contributor regardless of the sediment source. In ravines, added drain tile focusing the flow to one outlet could lead to increased erosion and subsequent sediment load. If bluffs are a significant source, it may be that the tremendous amount of drain tiles installed in the watershed (and throughout southern Minnesota) has significantly changed the Le Sueur River's hydrograph. More rapid and high discharge floods due to more rapid drainage of water from the landscape via drain tile could destabilize bluffs and lead to more and rapid bluff retreat (P. Belmont, personal communication, 2008).

GEOLOGIC SETTING:

From its origin in Freeborn County to its confluence with the Blue Earth River in Blue Earth County, the river drains primarily farmland overlying glacial till left behind

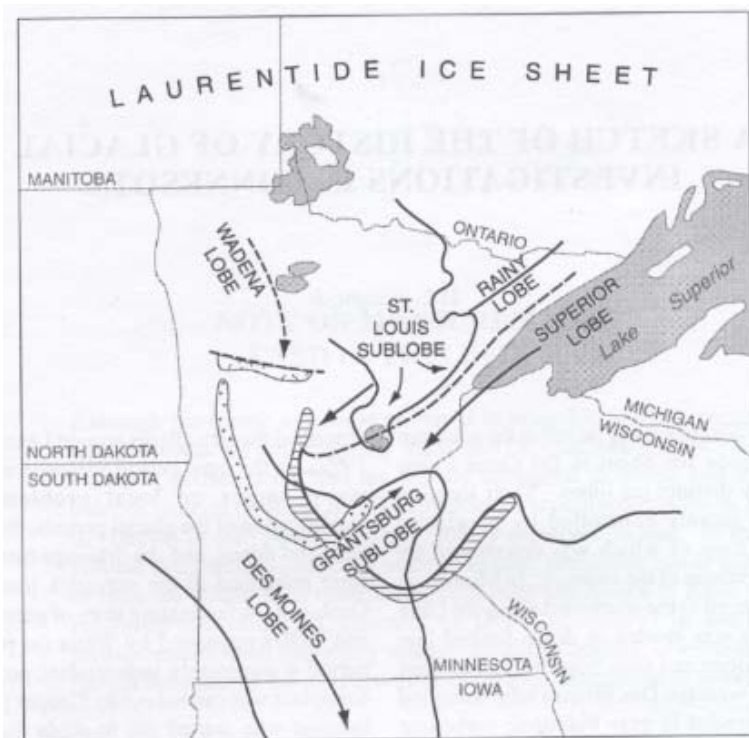


Figure 2: Glacial lobes of the Upper Midwest (Wright, 1998)

from the Wisconsin glacial episode at the last glacial maximum (Waseca county soil survey, 2001). In particular, this watershed lies within the area covered by the Des Moines Lobe, a section of glacier part of the Laurentide ice sheet.

Two advances of this lobe occurred during the late Wisconsin glaciation. The first advance, around 20-30 ka BP, flowed

through western Minnesota to just south of the Iowa border of Minnesota. The second advance of the Des Moines Lobe generally followed the path of the first but continued further south into Iowa, reaching its maximum extent at 14 ka BP (fig. 2). At this maximum, the Des Moines lobe was 200 km wide and reached 900 km beyond the body of the Laurentide ice sheet (Patterson 1996).

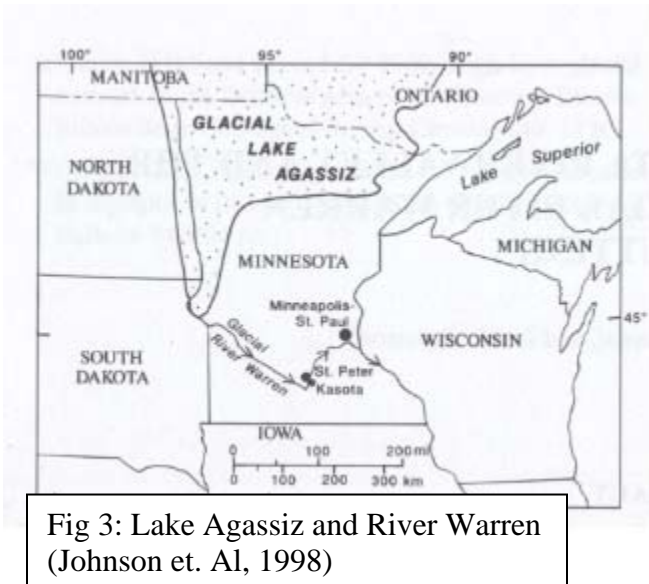
The Des Moines Lobe advanced further south than other parts of the ice sheet and geologists have studied possible mechanisms that allowed it to extend over such a large area. Patterson (1996) suggested that the lobe was an outlet glacier of an ice stream draining portions of the Laurentide Ice Sheet. A high proportion clayey material may also have contributed to the lobe extending further south than other lobes in the region.

The Des Moines Lobe till is dominated by Paleozoic carbonate, Cretaceous shales, and clay minerals. This results in a lower content of feldspars relative to other lobes (Grimley 2000). Geologists believe the source for carbonate is near Winnipeg, Canada, but some uncertainty remains (Wright 1998). According to Gowan (1998) till in western Minnesota appears blue or grayish color, while the oxidized layers near the surface are transformed to yellowish. Kaolinite is the most abundant clay mineral in Des Moines Lobe tills. This is partially due to the effects of weathering on the shale. (Grimley, 2000). These clay minerals, along with the dominant presence of limestones, dolomites, and Cretaceous shale make up the bulk of Des Moines Lobe till composition.

Because of similar geologic history, tills within the depositional region of the lobe are often difficult to differentiate. For example, New Ulm and Granite Falls tills that Gowan (1998) sampled are geochemically similar. Both have comparable proportions of granite, carbonate, and sandstone. A higher proportion of Cretaceous shale is observed in

the New Ulm till, but shale is not distinguished from sandstone in the grain counts that Gowan performed. Overall, it appears that while till units in Minnesota can be differentiated from each other, sites within each unit are too similar to be differentiated by conventional till descriptors such as grain size analysis, carbonate content, sand grain composition, and rock magnetic properties.

With the retreat of the glaciers 12,000 years ago, a large glacial lake formed in



what is now northwest Minnesota and Canada (fig. 3). Known as Lake Agassiz, this enormous body of water experienced one or more sudden drainage events around 11,700 years ago (Wright et al). The massive amounts of flowing water, known as Glacier River Warren, carved the present day Minnesota River Valley,

effectively creating a new base level for the region nearly 200 feet lower than the surrounding uplands. Tributaries in the Minnesota River Valley such as the Le Sueur River are down cutting through the till and rock to reach equilibrium with this base level.

11,000 years have passed since the glaciers receded and Glacier River Warren emptied Lake Agassiz. Because of down cutting and erosion, it was possible that while the overlying till was once uniform, weathering processes could have affected it differently as sediment makes its way to rivers and eventually downstream. This could potentially create altered chemical compositions in top soils, ravines, or bluffs that we can statistically measure.

SITE DESCRIPTION:

Located in southern Minnesota, the Le Sueur River drains parts of Freeborn, Steele, Faribault, Waseca, and Blue Earth counties. Originating in Freeborn County, it traverses 112 miles before flowing into the Blue Earth River near Mankato, Minnesota in Blue Earth County. Draining 1,100 miles², the watershed is primarily used for agricultural purposes (Lorenz 1991). Upstream from St. Clair, the river flows in a shallow river bed between small banks. Downstream from St. Clair, the river cuts through a much deeper valley (fig. 4). Adjacent bluffs can reach heights up to approximately 60 ft, while active ravines cut perpendicular from the river into the farmland (fig. 5-8).

To obtain relevant samples for our experiment, we limited our sampling to the second knick point zone of the Le Sueur River. Stretching from the city of St. Clair to just upstream of Blue Earth county road 90 (fig. 4), the Le Sueur River's banks are filled with actively eroding bluffs and dotted with ravines entering the system. Though relatively short in length, this stretch is the most actively eroded section of the river and therefore the largest sediment contributor.

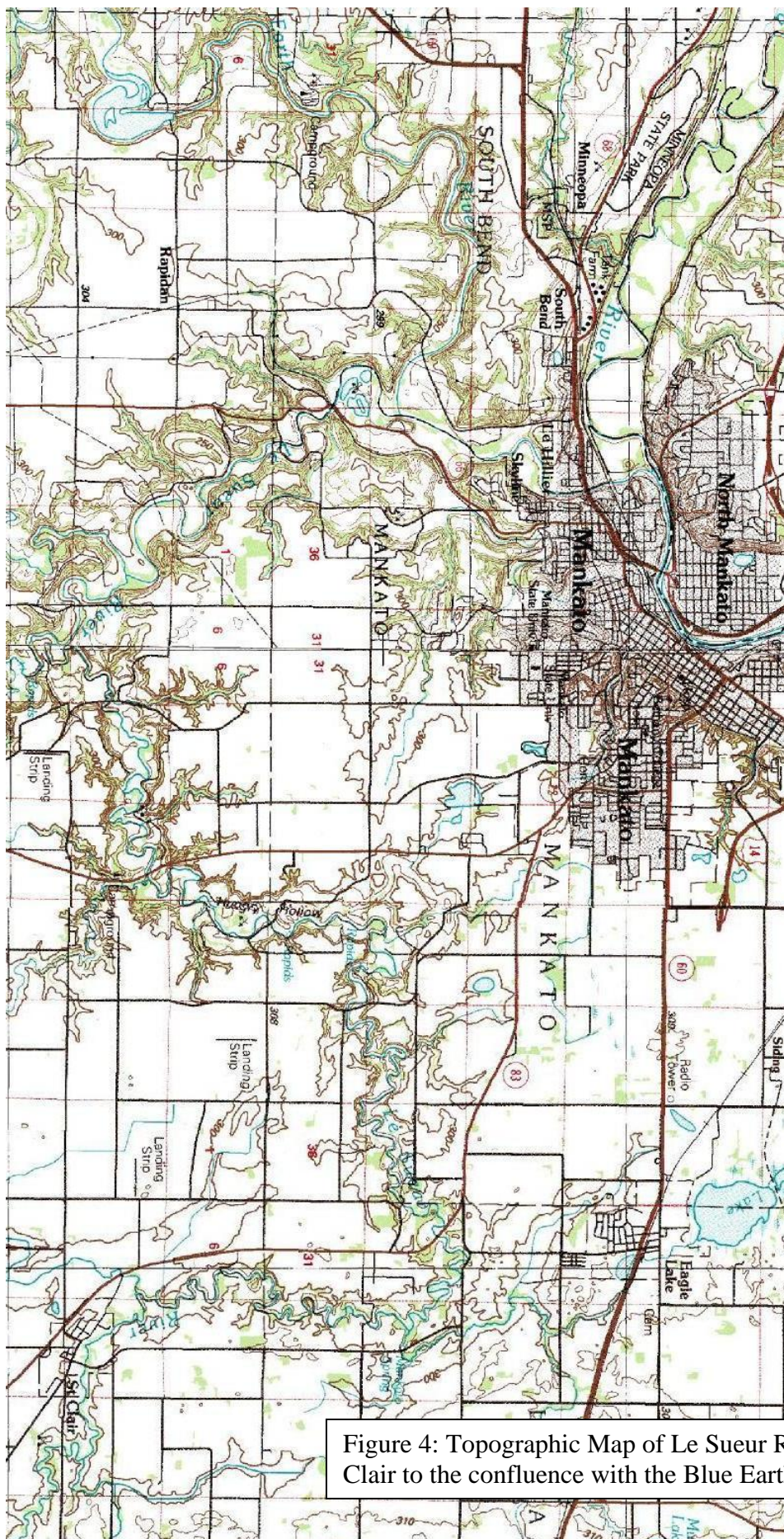


Figure 4: Topographic Map of Le Sueur River from St. Clair to the confluence with the Blue Earth River. (USGS)



Figures 5 and 6: Large bluff near Monks road (left) and bluff near Wildwood park (right).



Figures 7 and 8: Ravine pictures courtesy of Stephanie Day

METHODS:

Twenty-five samples were collected in total; eleven from bluffs, twelve from ravines, and two from top soils. However, one of the bluff samples was too low in silt content to test, while another was from an older variety of till originating at low enough elevations to be only seen near the mouth of the Le Sueur. This limited our tested number to twenty three sediment samples to compare with six suspended sediment samples collected from the Le Sueur River by the Minnesota Pollution Control Agency in 2007 and 2008. Three of the suspended sediment samples were collected at St. Clair above the knick point (May 2006, May and June 2008), two were collected at County Road 8 (May and June 2008) and one at Red Jacket (May 2008).

Bluff samples were taken as far upstream as Wildwood Park (fig. 6) and downstream to Blue Earth County Road 66. Taken at roughly even intervals along the river, bluff height ranged from <10 feet high to approximately 60 feet in some places (fig. 5 and 6).

Ravine samples were gathered from two ravines directly south of Mankato (fig. 7 and 8). The first ravine is located just off of Blue Earth County Road 8, 3.5 miles south of Stadium Road. The second ravine is also near Monks Road. It is intersected by 192nd street near the Terrace View Golf Course, though we collected all of our samples downstream from the road. Both ravines are seasonal in water flow but gradually gain water the closer you get to the river. Lastly, top soil samples were taken from fields adjacent to Monks Road 2 miles south of Mankato.

In sampling the bluffs, we collected sediment unexposed to the surface, and not part of erosive features like slumps. This was done in attempt to collect the till in a

natural, unaltered state. Ravine samples were gathered from apparent depositional areas in the fluvial part of each ravine; for example, shallow terraces or point bars, where sediment may be temporarily stored during transit from its source (ravine walls or uplands) to the LeSueur.

After gathering our samples, we separated silt-sized particles with a 63 μm sieve. Rivers typically carry only silt-sized particles or smaller as suspended load, so this was the portion of the sample that we kept to test. From the 63 μm or smaller sediment, we extracted at least 0.25 grams of dry material for each of our 23 “whole sediment” samples.

The remaining sediment was treated to isolate the heavy minerals; organic matter, carbonate and iron oxides were removed, all of which may act as cements or glues binding heavy minerals with other minerals. First, we removed the organic matter. Sediment samples were placed into 600 ml beakers with about 40-mL of 30% hydrogen peroxide solution and heated in an 85°C water bath. The effervescent reaction can be quite violent and took anywhere from 35-55 minutes before all the organic matter had reacted. Ten mL of hydrogen peroxide were added at a time to keep the reaction going, with 100 mL as the overall average that was added. Upon completing the reaction, each sample was then centrifuged for 35 minutes at 3500 rpm, decanted, and rinsed with de-ionized water three times.

Next, carbonates were removed. Using a similar method to the Soil Survey Investigations Report (1996), each sample was soaked overnight in 35 mL of NaOAc acid solution. The next day, samples were heated in a 90 degree C water bath to further react all the carbonate material. Reactions took one to two hours to complete. After

bubbles had stopped rising to the surface, samples were centrifuged, decanted, and rinsed with de-ionized water.

Next, we extracted the iron oxides. Employing a similar method to the Soil Survey Investigations Report (1996), 20 ml of sodium citrate and, 2.5 ml of the sodium bicarbonate were added to each sample tube before adding 0.5 g of sodium dithionite. Samples were reacted for ~15 minutes in a 80°C water bath before the reaction was stopped by adding 5 mL of NaCl. Samples were then centrifuged, decanted, and rinsed.

Finally, we used sodium polytungstate to remove the light minerals. Utilizing an open file report from the U.S. Geological Survey (Skipp et al), we set our liquid to a density of 2.91 g/cm³ by mixing the polytungstate powder and small amounts of water. Samples were then centrifuged to separate the minerals lighter than the liquid from minerals heavier than 2.91 g/cm³. After decanting the light minerals and polytungstate liquid, the remaining sediment presumably contained only the heavy minerals of the sediment.

“Whole sediment” and “heavy mineral” fractions were then sent to the University of Minnesota for further processing. Samples were fused with lithium metaborate in graphite crucibles at 1000°C and poured into 1N HCl solution for analysis by inductively coupled plasma optical emission spectroscopy (ICP-OES). U.S. Geological Survey rock standards were used for calibration. Major elements (Al, Ba, Ca, Fe, K, Mg, Mn, Na, P, Si, Sr, Ti) were measured as oxides directly from the fused solution. Before measuring trace elements (Ba, Be, Ce, Co, Cr, Cu, Dy, Er, Eu, Ga, Gd, Ho, La, Mn, Nd, Ni, Pb, Pr, Rb, Sc, Sm, Sr, V, Y, Yb, Zn) major cations were removed by passing the solutions through a Dionex strong-acid exchange column.

Upon receiving the elemental data for bluff and ravine samples of both heavy minerals and whole sediments, each element was then statistically tested to determine if there were significant differences between bluffs and ravines. Using the Mann-Whitney U test, each element's "p" value was determined for both the heavy mineral and whole sediment data samples. Following Walling's (2005) procedure, compared sample sets were considered significantly variant for a given element if the "p" value was less than 0.05. When comparing two elements from a sediment set, a value of less than 0.05 infers that there is less than a five percent chance that the sample populations are from the same source based on that element. In comparing the entire sediment population, a combination of five elements from majors and traces must vary significantly to consider the populations significantly different.

To help determine the accuracy of our results, multiple duplicate samples, split samples, and rerun samples were tested. Duplicate samples were samples from the same raw sediment, but brought through each of the preparation steps as a separate entity. Split samples were samples that were brought through all the steps together, but then split right before the fusion and optical emission spectroscopy steps. Lastly, reruns were samples that were simply tested twice during the optical emission spectroscopy.

RESULTS:

Of the 36 elements tested for in heavy minerals and whole sediment samples, a total of fourteen in the heavy minerals and three in the whole sediment samples were found to significantly differ in bluff and ravine samples ($p < 0.05$). In the heavy mineral

comparison; Al, Fe, P, Ce, Cr, Eu, Ga, Ad, La, Sr, V and Zn had p values of less than 0.05 (Table 1). P, Ho, and Zn were found to significantly differ in whole sediments.

Element	Heavy Mineral Comparison	Whole Sediment Comparison	Element	Heavy Mineral Comparison	Whole Sediment Comparison
Al	0.021	0.456	Ga	0.023	0.546
Ba	0.213	0.67	Gd	0.001	0.859
Ca	0.972	0.095	Ho	0.263	0.043
Fe	0.025	0.374	La	0.023	0.337
Mg	0.749	0.095	Mn	0.616	0.166
Mn	0.456	0.145	Nd	0.097	0.145
Na	0.145	0.214	Ni	0.787	0.241
P	0.014	0.009	Pb	0.375	0.06
SiO	0.456	0.145	Pr	0.07	0.456
Sr	0.075	0.188	Rb	0.049	0.804
TiO	0.06	0.546	Sc	0.203	0.456
Ba	0.263	0.499	Sm	0.083	0.456
Ce	0.028	0.749	Sr	0.049	0.189
Cr	0.015	0.972	V	0.019	0.271
Dy	0.132	0.271	V	0.012	0.414
Dy	0.153	0.594	Y	0.114	0.374
Er	0.097	0.095	Yb	0.059	0.749
Eu	0.012	0.337	Zn	0.001	0.017

Table 1: “p” values of the 36 tested elements for both Heavy and Whole sediments. Be, Cu, K, and Co were also tested, but did not have a comparative “p” value of less than .3

In both the heavy and whole sediment populations, ravine sediments had higher concentrations in all of the significantly variant elements (Tables 2 and 3).

Bluff (ug/g)	Ravine (ug/g)	Element
426	533.74	Ba
47.181	89.337	Ce
52.169	97.241	Cr
0.5296	1.7915	Er
7.572	10.422	Ga
0.5913	3.273	Gd
23.203	45.305	La
165.063	192.763	Sr
51.86	80.531	V
25.939	44.613	Zn (whole)
0.695	0.928	Ho (whole)
94.208	132.601	Zn (whole)

Bluff (% wt.)	Ravine (% wt.)	Element
6.9859	8.2201	Al ₂ O ₃
1.6102	1.9479	Fe ₂ O ₃
0.1338	0.2091	P ₂ O ₅
0.1393	0.1711	P ₂ O ₅ (whole)

Tables 2 and 3: Average concentrations of elements from trace (left) and major (right) elements. Elements are from heavy minerals unless otherwise stated Major elements were measured in percent weight. Trace elements were measured in micrograms per gram.

Elements Er and Ho, Gd from the heavy mineral comparisons and Ho, Gd from the whole sediment comparison had high relative standard deviations in the determined value of many of the original samples, making them poor indicators of variability.

The major elements had average percent standard deviation of less than 10% between the “original” test and the various repeatability checks. For the most part, trace elements followed suit. Reruns all had an average of less than 15% error for each sample, with the majority less than 10%. Split samples as well were of reasonable percent error with B7 having 19% error between the original and the split and R1B having 10% error. Duplicates were less satisfactory. One of the duplicates, B8, had reasonable percent error from the original at 18%. The other, R3C, was much higher than desired at 70%.

DISCUSSION:

Answering our original research question in determining if bluffs and ravines are geo-chemically different, we found that heavy minerals can be good indicators in distinguishing between bluff and ravine sediment. When considering the heavy mineral component, fourteen elements in bluff and ravine sediments met Walling’s criteria for significant difference (2005). With fourteen in total, the sediment sources can be considered significantly different as according to Walling’s criteria of five variant elements. Two of these elements, Sr and Rb had p values of 0.049, barely below the 0.05 line set by Walling and one, Gd had higher than desirable relative standard deviation values. Nonetheless, the other eleven had values well below the threshold, indicating separate sediment populations.

Our suspended sediment solids were only tested with the “whole” sediment fraction. Even though our Heavy Mineral samples from bluffs and ravines were significantly different, we could not compare them to suspended sediment river samples without the heavy mineral fraction from the suspended sediment. When comparing our “whole” sediment samples from bluffs and ravines, we found only three discriminatory elements, one of which, Ho was low enough in concentration that analytical uncertainty was high. At only two significantly variant elements, we could not compare our whole sediment samples to our suspended sediment samples either as they did not meet Walling’s five element minimum (Walling 2005).

Heavy minerals apparently can be used to discriminate between bluffs and ravines while whole sediment cannot. The combination of organics, carbonates, iron oxides, and light minerals must not be preferentially staying in bluffs or ravines before they enter the river. These parts of the sediment must be weathering in bluffs and ravines similarly enough to indicate no chemical difference between the two sediment sources. With similar weathering, “whole” sediments cannot be statistically distinguished between bluff and ravine populations. Conversely, the heavy minerals must have some sort of difference in what is weathering out in ravines and bluffs.

Interestingly, of the seventeen elements found to be significantly different in heavy mineral or whole sediment samples, all of them had greater average concentrations in the ravine samples. This implies that the minerals carrying these elements enter the ravine and have a longer residence time than other components of the sediment. Specifically, heavy minerals containing the discriminatory elements must be more

resistant to weathering conditions in the ravines than other heavy minerals originally found in the till or topsoil.

The split sample and rerun sample results were encouraging. Though the values seemed high at first, we determined that a handful of elements significantly raised the average percent error due to concentrations near the detection limit for the ICP-OES. Removing Er, Gd and Ho, we found the accuracy to be within acceptance levels. Without more duplicates as gauges, it is difficult to determine how accurate a repeated study would be if completely different samples were collected.

CONCLUSIONS

Overall, we were not able to find the actual proportions of bluff and ravine sediments eroding into the Le Sueur River because we did not have enough sample to analyze heavy mineral composition in suspended sediment samples. However, our research indicates that heavy minerals can be used to geochemically distinguish between bluff and ravine sediments in the river. We successfully found fourteen heavy mineral elements that varied significantly between bluffs and ravines. If heavy mineral fractions of suspended river sediment could be collected and tested, bluff and ravine heavy mineral compositions could be used in a mixing model to determine proportions of each delivered to the river. While our duplicate samples from our raw material had higher than ideal percent error, our heavy mineral samples had far more discriminative elements than Walling's minimum of five. Perhaps having duplicates from a wider range of samples might lower the uncertainty.

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