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USING RADIOCARBON AGES ON ORGANICS AFFECTED BY FRESHWATER—A GEOLOGIC AND ARCHAEOLOGIC UPDATE ON THE FRESHWATER RESERVOIR AGES AND FRESHWATER DIET EFFECT IN MANITOBA, CANADA

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ABSTRACT. Freshwater reservoir ages and a potential freshwater diet effect are examined for Manitoba (Canada), using paired samples of contemporaneous terrestrial, freshwater, and omnivore-diet species (*Homo sapiens*, *Gavia* sp.). The freshwater diet effect (n=4) varies from 220 to 370 ^{14}C years. The FRE (n=12) is temporally variable, with a median of 230 ^{14}C years and values up to 880 ^{14}C years (or more). Understanding the FRE is important for geologists who reconstruct the postglacial history of an area. It is also important for archaeologists who rely on ^{14}C dating to develop cultural histories, as inhabitants of the Canadian boreal forest have relied upon freshwater resources as a part of their diet. An analysis of the $\delta^{13}C$ on 93 freshwater mollusk samples from 11 species demonstrates significant variability, which highlights the difficulty in assigning an "assumed" fractionation correction.

KEYWORDS: Canada, fractionation, freshwater reservoir age, freshwater shells, mollusk, radiocarbon dating.

INTRODUCTION

Radiocarbon (^{14}C) ages are the foundation of cultural and geologic histories. ^{14}C ages become even more important in areas that have slow soil accumulation, which limits the application of other dating techniques. Wherever possible, conventional wisdom suggests that terrestrial plant macrofossils, wood and/or charcoal should be sampled for radiocarbon instead of aquatic plant macrofossils, shell-bearing organisms (ostracods, gastropods, pelecypods), and organisms that eat the above. There are many times, however, where the "less-ideal" freshwater and/or freshwater-diet organic material is the only material available for radiocarbon dating. The user must then correct for the freshwater reservoir effect (Broecker and Walton 1959; Keaveney and Reimer 2012). This is because organic materials are also affected by inorganic carbon present within freshwater environments (the reservoir) that overlie both carbonate rocks (Deevey et al. 1954; Andree et al. 1986; MacDonald et al. 1987), and/or lignite, coal, and carbonaceous shales (Nambudiri et al. 1980). Uptake of dissolved old bicarbonate in freshwater leads to disequilibrium and "hardwater," as the waters no longer contain the same ^{14}C as the atmosphere (Godwin 1951). Hence, a freshwater reservoir age is needed to correct radiocarbon ages obtained on freshwater-derived organics. In Canada, freshwater reservoir age studies indicate that corrections can range from 0 to ~6400 years (MacDonald et al. 1987; McMartin 2000; Lewis et al. 2001; Daubois et al. 2015; Patterson et al. 2017) and vary due to the following:

- availability of dissolved old bicarbonate; linked to geology of the substrate (Arnlond 1957; Broecker and Walton 1959; Moore et al. 1998);
- changes in groundwater storage time, runoff intensity, direction of flow (Geyh et al. 1998; Hutchinson et al. 2004; Osterkamp et al. 2014) or the influence of glacial meltwater (Hall and Henderson 2001);
- taxa dated, as old carbon settles differently in the environment, stratification of water can lead to a lack of exchange with the atmosphere, and organisms uptake carbon differently (Shotton 1972; Marty and Myrbo 2014).

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Problems interpreting a freshwater reservoir effect increase with the trophic level of the taxa dated. Keaveney and Reimer (2012) show that there are variations due to the diet of individual fish of the same species, exclusive of variations between different species. Similar freshwater diet variations are expected for carnivorous animals and human bones, depending on the proportion of freshwater in an individual's diet (Lanting and van der Plicht 1998; Philippsen 2013). The aim of this study is to examine the order of magnitude and degree of variability of the freshwater reservoir age in Manitoba. This incudes observation on omnivore-diet organisms, which may have eaten food derived from freshwater.

METHODS

The recently updated Manitoba radiocarbon database includes 1371 radiocarbon ages on material from 25 different laboratories (Gauthier 2021; Table 1). We examined all paired terrestrial–freshwater shell and live-collected pre-bomb mollusk samples, one paired deer–loon sample, and terrestrial–omnivore diet samples. The freshwater reservoir age at each site is calculated by Equation (1) below. Data on individual radiocarbon ages, including location and depositional setting, is located within the Manitoba radiocarbon database (Gauthier 2021).

$$\text{Freshwater reservoir age} = \text{terrestrial } ^{14}\text{C age-freshwater } ^{14}\text{C age} \tag{1}$$

An additional correction is needed for the live-collected pre-bomb mollusk samples, to account for the radioactively inert carbon dioxide produced during the industrial revolution (Suess 1955). This can done by using estimated atmospheric $\Delta^{14}C$ values from Stuiver and Quay (1981) in Equation (2) below, to calculate the estimated age offset for each sample (Hua 2009; Moore et al. 1998). The freshwater reservoir age for the live-collected samples is calculated using Equation (3).

$$\text{Age offset} = -8033\ln\left(1 + \Delta^{14}\text{C}/1000\right) \tag{2}$$

$$\text{Freshwater reservoir age} = \text{fractionation-corrected } ^{14}\text{C age-pre-1950 true age} \tag{3}$$

RESULTS

Freshwater Reservoir Age

Radiocarbon ages obtained from paired terrestrial–freshwater (n=14) and live-collected pre-bomb (n=3) organic samples in Manitoba (Figure 1) are presented in Table 2. Equation (2) is factored into column "Conventional ^{14}C age (yr BP)" of Table 2. This additional correction was not originally applied to sample TO-_4289 in Last and Teller (2002), leading to an incorrect freshwater reservoir age of 120 instead of 88 ^{14}C years. The uncorrected age of the freshwater mollusk GCS-3281 is reported as 570 ± 100 (Blake 1982). It was incorrectly normalized as 440 ± 100 in Nielsen et al. (1982), leading to an incorrect freshwater reservoir age in Lewis et al. (2001). The terrestrial–omnivore diet pairs were previously published in Syms (2018), and we include them here to provide comparison with the lower trophic level organisms.

Data Exclusion

Eleven additional terrestrial–freshwater paired-sites in Manitoba were not included in Table 2. These pairs have been used to calculate freshwater reservoir age in the past, and but are based

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Table 1 Measured $\delta^{13}C$ values for freshwater shell samples in Manitoba

	Amnicola limosa	*Fossaria parva*	*Gyraulus parvus*	*Lampsilis radiata*	*Lymnaea stagnalis*	*Marstonia gelida*	*P. lacustris*
	n=1	n=2	n=1	n=17	n=2	n=17	n=1
min	–	−8.3	–	−12.6	−9.6	−1.2	–
max	–	−7.2	–	−2.9	−8.6	1.0	–
mean	−1.2	−7.7	−8.0	−5.7	−9.1	0.0	−4.4
median	–	−7.7	–	−5.0	−9.1	0.1	–
st. dev.	–	–	–	2.0	–	0.6	–

	Sphaerium sp.	*Stagnicola* sp.	*Strophitus undulatus*	*Valvata tricarinata*	Gastropoda	Bivalvia	**All shells**
	n=31	n=11	n=1	n=1	n=41	n=51	**n=93**
min	−9.0	−8.8	–	–	−9.6	−12.6	**-12.6**
max	−1.9	−0.6	–	–	1.0	−1.9	**1.0**
mean	−5.2	−3.8	−7.9	−4.6	−2.7	−5.4	**-4.3**
median	−4.7	−2.8	–	–	−1.3	−4.7	**-4.3**
st. dev.	1.9	3.0	–	–	3.3	2.1	**3.0**

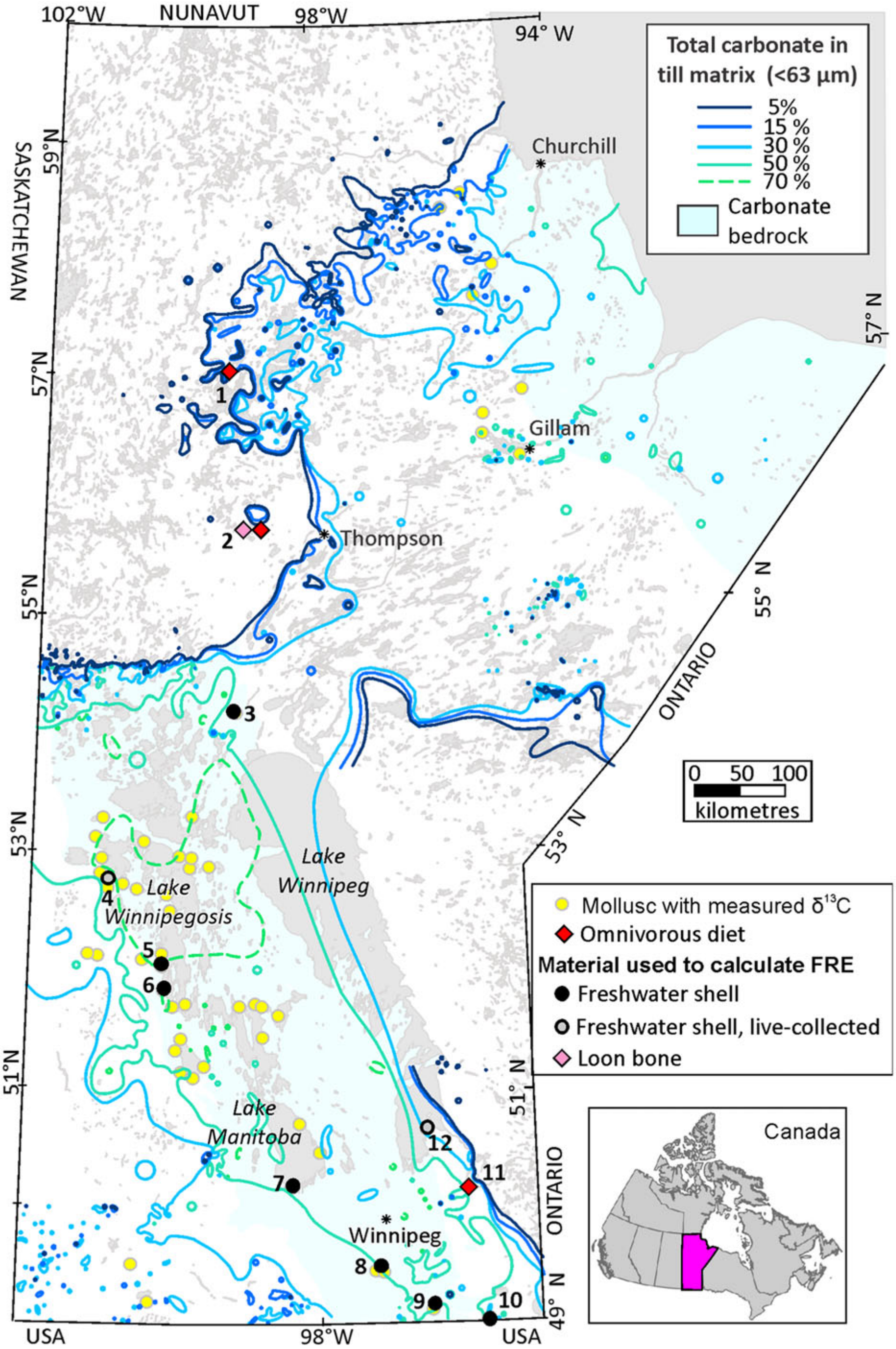

Figure 1 Map of Manitoba, with locations of mollusks with measured $\delta^{13}C$ values, paired terrestrial-freshwater, terrestrial-freshwater diet, and terrestrial-omnivorous diet samples (Table 3). Mapped carbonate bedrock is modified after Manitoba Geological Survey (2006) and hand-drawn contouring of the total carbonate in the till (glacial sediment) matrix is from Manitoba Agriculture and Resource Development (2020a). The index map shows Canada, with the province of Manitoba in pink.

Table 2 ^{14}C data for paired terrestrial-freshwater, terrestrial-freshwater diet, and terrestrial-omnivorous diet samples in Manitoba.

Site number	Estimated CO_3[1]	Laboratory number	Material dated	Dated taxa	Latitude[2]	Longitude[2]	^{14}C age[3] (yr BP)	$\delta^{13}C$	Omnivoire diet effect[4]	Freshwater reservoir age[4]	References
1	<5 %	Beta-106475	plant (seeds)	*Prunus pensylvanica*	57.08	−99.25	220 ± 50	−25.3 ‰	220, 370	–	Brownlee and Syms 1999; Syms 2018
		Beta-107745	bone (mixed diet)	*Homo sapians*	57.08	−99.25	440 ± 30	−23.3 ‰			Brownlee and Syms 1999; Syms 2018
		TO-_5228	bone (mixed diet)	*Homo sapians*	57.08	−99.25	590 ± 40	−25.0 ‰			Brownlee and Syms 1999; Syms 2018
2	<5 %	CAMS-13187	bone (mixed diet)	*Homo sapians*	55.75	−98.92	4370 ± 60	−21.6 ‰	320	–	Morlan et al. 2000; Syms 2018
		CAMS-13188	bone (terrestrial diet)	*Alces alces*	55.75	−98.92	4050 ± 50	−22.1 ‰			Morlan et al. 2000; Syms 2018
	<5 %	TO-_6031	bone (antler; terrestrial diet)	*Cervidae* sp.	55.75	−98.92	3700 ± 60	AMS	–	220	Brownlee 2005; Morlan et al. 2000; Syms 2018
		TO-_6032	bone (freshwater diet)	*Gavia* sp.	55.75	−98.92	3920 ± 60	AMS			Brownlee 2005; Morlan et al. 2000; Syms 2018
3	~50 %	GSC-6087	limnic peat	unidentified	54.208	−99.169	4500 ± 130	−27.1 ‰	–	380	McMartin 2000
		TO-_5699	freshwater gastropod shells	*Gyraulus* sp.	54.208	−99.169	4880 ± 60	AMS			McMartin 2000
4	~70 %	BGS-1099	freshwater shells	*Lampsilis radiata siliquoidea*	52.833	−101.000	310 ± 100	−10.3 ‰	–	241 ± 100*	Nielsen et al. 1987
5	~50–70 %	Beta-68102	charred wood	unidentified	52.062	−100.158	300 ± 90	−25.7 ‰	–	290	Tackman et al. 1998
		Beta-68105	freshwater shells	*Stagnicola*	52.062	−100.158	590 ± 60	−5.0 ‰			Tackman et al. 1998
6	~50–70 %	Beta-77523	freshwater shells (0.05 m above)	*Lampsilis radiata*	51.857	−100.112	460 ± 100	−5.3 ‰	–	40	Tackman et al. 1998
		Beta-77524	marsh peat	unidentified	51.857	−100.112	420 ± 40	−28.7 ‰			Tackman et al. 1998
7	~50 %	Beta-68106	freshwater shells (<0.2 m depth)	*Stagnicola*	50.184	−98.368	480 ± 70	−8.8 ‰	–	190, 210	Tackman 1997
		Beta-68083	freshwater shells	*Sphaerium*	50.184	−98.368	460 ± 90	−1.9 ‰			Tackman 1997
		Beta-68088	peat (below shells)	unidentified	50.184	−98.368	270 ± 50	−27.3 ‰			Tackman 1997
8	~50 %	GSC-6253	freshwater shells; whole valves	*Sphaerium* sp.	49.4944	−97.229	4320 ± 70	−8.16 ‰	–	270	Morlan et al. 2000
		GSC-6266	charcoal fragments	*Fraxinus*	49.4944	−97.229	4050 ± 80	−26.4 ‰			Morlan et al. 2000
9	~50 %	GSC-5363	bulk freshwater shells	unidentified	49.1633	−96.5461	10916 ± 100	−5.61 ‰	–	880 minimum	Morlan et al. 2000
		GSC-5296	wood (1.3 m lower)	*Picea sp.*	49.1633	−96.5461	10039 ± 90	−28.7 ‰			Morlan et al. 2000

(Continued)

Table 2 (*Continued*)

Site number	Estimated CO_3[1]	Laboratory number	Material dated	Dated taxa	Latitude[2]	Longitude[2]	^{14}C age[3] (yr BP)	$\delta^{13}C$	Omnivoire diet effect[4]	Freshwater reservoir age[4]	References
10	30–50 %	TO-_4865	plant macrofossil - 2	unidentified	49.0182	−95.844	4080 ± 70	AMS	–	-190	Teller et al. 2000
		TO-_4866	freshwater shells	unidentified	49.0182	−95.844	3890 ± 60	AMS			Teller et al. 2000
	30–50 %	TO-_4862	wood; 2 fragments	unidentified	49.0182	−95.844	4430 ± 60	AMS	–		Teller et al. 2000
		TO-_4863	bulk lake sediment	unidentified	49.0182	−95.844	5060 ± 60	AMS		630	Teller et al. 2000
		TO-_4864	freshwater shell fragments	unidentified	49.0182	−95.844	4630 ± 70	AMS		200	Teller et al. 2000
11	15–30%	NSRL-_3127	bone (mixed diet)	*Homo sapians*	50.12	−96.04	1570 ± 60	AMS	340	–	Ens 1998
		GX-_3603	charcoal	unidentified	50.12	−96.04	1230 ± 155	−25.0 ‰			Buchner 1982; Meiklejohn et al. 1994
12	15–30%	GSC-3281	freshwater shells	*Strophitus undulatus*	50.667	−96.583	840 ± 100	−7.9 ‰	–	670 ± 100*	Blake 1982; Nielsen et al. 1982; Todd et al. 2000
unknown	~50 %	TO-_4289	freshwater shells	*Andonta grandis simpsonia*	'Lake Manitoba'		170 ± 60	AMS	–	88 ± 60*	Last and Teller 2002

[1]Based on regional total carbonate (weight percent) in the till (Figure 1). These should be considered as a relative way to compare the availability of old carbon in the respective depositional environments.

[2]Geographic coordinate system North American 1983.

[3] ^{14}C - conv = conventional method; ^{14}C - AMS = accelerator mass spectrometry method.

[4]Equation (1), in ^{14}C years.

*Equation (3).

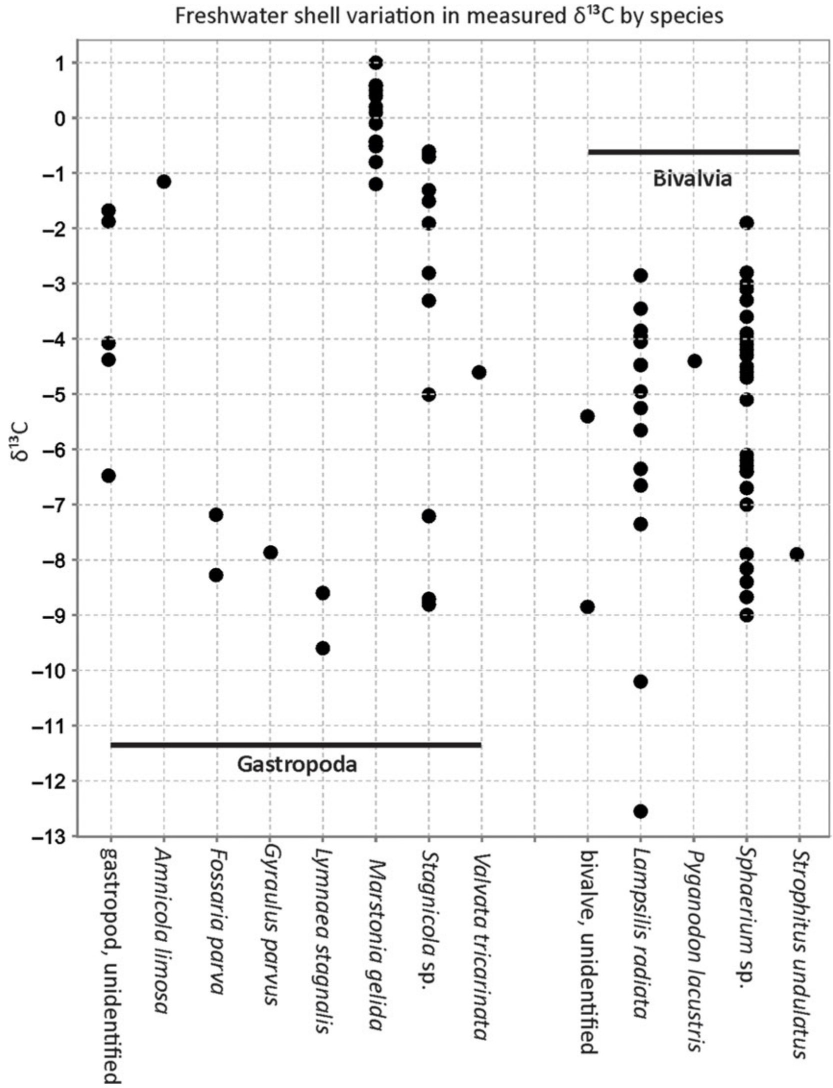

Figure 2 The range of $\delta^{13}C$ measured on various taxa of freshwater shells from Manitoba.

on assumptions that are incorrect. Two sites were analyzed before fractionation correction relative to $\delta^{13}C = -25.0‰$ PDB was conventional (GSC-215 and GSC-216, Dyck et al. 1965; GSC-689 and GSC-797/-870,Lowdon and Blake 1968). Three additional sites used assumed $\delta^{13}C$ values on plant seeds (CAMS-34555 and CAMS-71709; CAMS-_38680 and CAMS-71710 and CAMS-34550 and CAMS-71708; Lewis et al. 2001; Todd et al. 2000; Todd et al. 1996), moss (Y-165, GSC-1319, TO-11762 and Beta-193587; Boyd 2007; Lowdon et al. 1971; Preston et al. 1955; Ritchie and Lichti-Federovich 1968; Teller 1989) or wood (BGS-1851 and TO-_330; Morlan et al. 2000; Nielsen and Thorleifson 1996; Rannie et al. 1989). Data on 51 wood samples in from Manitoba shows that an assumed value is inacceptable, given that measured $\delta^{13}C$ values on wood can range from −30 to −23.5‰ with a median of −26.9‰ and a standard deviation of 1.5 (Gauthier 2021).

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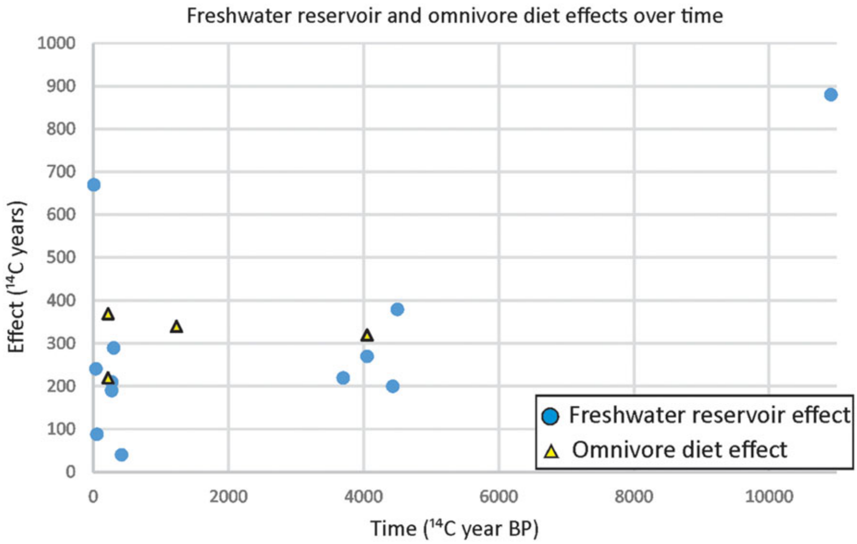

Figure 3 Freshwater reservoir age and possible freshwater-diet effects in Manitoba over time, calculated from paired terrestrial-mollusk and terrestrial-omnivore diet radiocarbon ages.

Another freshwater reservoir age was calculated using a terrestrial sample with significant error, which makes accurate quantification of the FRE impossible (7450 ± 680 BP, TO-5816, Last and Teller 2002; Risberg et al. 1999). An additional freshwater reservoir age was calculated by comparing with lake gyttja (TO-4858 and TO-4859, Teller et al. 2000). Gyttja is by definition a bulk sample that may contain older detritus, in addition to have its own FRE (Grimm et al. 2009).

δ^{13}C Variation on Freshwater Mollusks

Another three excluded terrestrial–freshwater paired-sites used assumed δ^{13}C values on mollusks (assumed a δ13C = −7.0‰ on unidentified shells, CAMS-32193 and CAMS-35495; Vance and Telka 1998) or ostracods (assumed a δ13C = −5.0‰, CAMS-38676 and CAMS-38678; Vance and Telka 1998). To see if the assumed fractionation correction is valid, the measured (IRMS) δ^{13}C values were collated from 93 freshwater mollusks sampled within Manitoba (Figure 1). This data spans the entire range from live-collected (pre-bomb) to 8460 BP (not corrected for freshwater reservoir age). Measured δ^{13}C values range from −12.6 to 1‰, and there is variability both within a single species and cross-species (Figure 1, Figure 2, Table 1). The gastropod *Marstonia gelida* (synonym of *Marstonia lustrica*) has similar median and mean, with a standard deviation of just 0.6‰. This data suggests it may be possible to estimate an assumed δ^{13}C ratio of 0 ± 1‰ for *Marstonia gelida*. For all other species, however, the spread of measured δ^{13}C is simply too wide to assign an assumed ratio (c.f. Morlan 1999; Stuiver and Polach 1977). This could be because environmental dissolved inorganic carbon is also a factor in controlling shell δ^{13}C (McConnaughey and Gillikin 2008), meaning that shells cannot be compared from different substrates/water bodies.

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Freshwater-Diet Effect

Radiocarbon ages obtained from terrestrial–omnivore diet organic samples in Manitoba (Figure 1) are presented at the bottom of Table 2. The four pairs show a discrepancy between the terrestrial age and the human age, indicating the presence of a freshwater-diet effect between 220 to 370 ^{14}C years.

DISCUSSION

Assumed Fractionation Correction on Freshwater Mollusks?

The mean and standard deviation of all Manitoban freshwater shell values, $\delta^{13}C = -4.3 \pm 3‰$, could be used as a tentative assumed fractionation correction. This would normalize an unidentified shell with an uncorrected age of 1050 BP to 1388 BP (correction of 338 ^{14}C y). If, however, the accurate $\delta^{13}C$ value is more like −9‰ or −2‰, the same fractionation corrected ages would be 1321 BP (correction of 262 ^{14}C y) or 1425 BP (correction of 375 ^{14}C y), respectively. This 104 ^{14}C year difference (potential error) may have a significant impact on the chronology of recent histories, or for identifying cultural changes. This potential error is not acceptable for calculating a freshwater reservoir age. As more data becomes available, use of an assumed fractionation correction should stop.

Freshwater Reservoir Age Variability

The freshwater reservoir age in Manitoba has not remained constant through time (Figure 3). The freshwater reservoir age varies between 40 and 880 ^{14}C years for the 12 freshwater mollusk samples. The largest freshwater reservoir age was measured from the oldest sample pair and likely reflects the contribution of depleted glacial meltwater into Lake Agassiz. There are no other Lake Agassiz-related freshwater reservoir age studies to compare spatial variability to. Southwest of James Bay, an even larger 4020 ^{14}C year correction is tentatively suggest for Lake Ojibway, though the two samples uses to calculate this are from different strata with an erosional contact in between (Daubois et al. 2015). The dataset has a mean of 310 ^{14}C years, a median of 230 ^{14}C years and a standard deviation of 230 ^{14}C years—or roughly an average freshwater reservoir age of 310 ± 230.

In Lake Winnipeg, cessation of Agassiz sediments and initiation of Lake Winnipeg sediments is dated at 7.75 ka ^{14}C BP (twigs, CAMS-32189; Vance and Telka 1998). After separation from the larger meltwater-fed postglacial lake, the data suggests it may possible to use freshwater and freshwater-diet radiocarbon ages in large-scale regional data compilations as long as the error bars are respected. Similar-magnitude modern freshwater reservoir ages (250 to 500 ^{14}C years) were calculated for lakes Michigan and Huron, which also overlie carbonate bedrock (Moore et al. 1998).

Freshwater Diet Effect

The freshwater diet effect in Manitoba shows less variability through time (Figure 3), though there are only 4 samples. The values range between 220 and 370 ^{14}C years. Unfortunately, neither $\delta^{15}N$ nor $\delta^{34}S$ values were measured on these samples. As such, it is impossible to fully understand the dietary make up, nor quantify the proportion of diet due to the freshwater reservoir effect.

Geologic Link?

Direct measurements of water alkalinity are not available to investigate a potential link between relative availability of old carbon and calculated freshwater reservoir age in Manitoba. This is partially because five of the freshwater reservoir age samples are >3500 BP. Instead, we used the surficial geology as a proxy. Till (sediment derived directly from glaciation), meltwater-related glacial sediments (glaciofluvial, glaciolacustrine, glaciomarine), and/or postglacial sediments (fluvial, lacustrine, marine), cover most of Manitoba (Manitoba Agriculture and Resource Development a). The total-carbonate content of surface till in Manitoba varies between 0 and 80 weight percent, due to changes in underlying bedrock lithologies as well as variable transport and comminution of calcareous detritus by glaciers (Manitoba Agriculture and Resource Development b). This is measured on the <63 μm size-fraction of the till matrix. Figure 1 shows that the majority of the paired-radiocarbon sites are overlying calcareous till (~ 50 weight % CO_3), and/or within waterbodies that largely overlay calcareous till. This coincidence makes it difficult to determine if there is a predictable link between the calcareous substrate and freshwater reservoir age. It does, however, suggest that the variability in calculated freshwater reservoir age (Table 2) is not a factor of substrate alone.

CONCLUSIONS

The freshwater reservoir age in Manitoba is variable over time and space; one specific ^{14}C year correction cannot be applied without significant error bars. Previous Manitoban studies that have either negated the freshwater reservoir age (Tackman et al. 1998; Risberg et al. 1999; Last and Teller 2002) or calculated it incorrectly (Nielsen et al. 1982, 1987), should be re-addressed in this light. Radiocarbon ages obtained on other omnivorous or carnivorous taxa in Manitoba, and other similar boreal environments, may also need partial freshwater reservoir age corrections due to a freshwater diet effect. This study provides the first compilation of regional data and should be used to guide future projects.

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