

Chapter 8

Constraints of Radiocarbon Dating in Southeastern Baltic Lagoons: Assessing the Vital Effects

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Abstract During the past decades, a suite of radiocarbon (^{14}C), infrared optically stimulated luminescence (IR-OSL), and electron spin resonance (ESR) dates were compiled on a variety of materials from the Curonian and Vistula lagoons and spits of the southeastern Baltic Sea. These dated materials generally included lagoon sediments and mollusc shells, along with samples of fossil fish remains, peat, wood, and water bicarbonates. A growing number of ^{14}C dates (conventional and AMS) demonstrates disparities and contradictions with the associated IR-OSL dates and palaeobotanical investigations of contemporary materials. Detailed analyses of ^{14}C , IR-OSL and ESR chronologies and experimental ^{14}C dating of modern live molluscs and water bicarbonates from the Curonian Lagoon and its main tributary – Nemunas-Neris River system – reveals a substantial influx of “old” carbonates into the lagoon. As a result, the uncertainty of reservoir effect added a considerable error to the ^{14}C ages. Moreover, fossil molluscs of the same species extracted from boreholes and outcrops yield significantly younger ages (up to several millennia) than the enclosing sediments. Both of these trends – aging and rejuvenation – highlight an urgent need for constraining the local reservoir correction (ΔR). Several scenarios are presented to explain the impact of vital effects on radiocarbon

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chronology of carbonates and to offer strategies to account for them in future studies.

Keywords Curonian lagoon • Vistula lagoon • Molluscs • Radiocarbon • IR-OSL • ESR • Reservoir effect

8.1 Introduction

Our understanding of the geological structure of the southeastern Baltic Sea spits and lagoons, as well as reconstruction of their evolutionary stages during the Holocene, are based on a relatively wide spectrum of field investigations: palynological, diatom and mollusc analysis, integrated geophysical and lithological studies, and a compilation of dates based on different methods of absolute geochronology. During the past decades, a suite of conventional and accelerator-mass spectrometry (AMS) radiocarbon (^{14}C) dates were performed on a variety of materials from the Curonian and Vistula lagoons and spits (Fig. 8.1). Dating included bulk sediment samples (organic carbon and/or inorganic carbonates) and mollusc shells, as well as several samples of associated fish remains, wood fragments, and peat. In addition, water bicarbonates were dated from the Baltic Sea, Curonian Lagoon, and its main tributaries – Nemunas and Neris (the last one is tributary of the Nemunas River in the eastern part of Lithuania; not indicated in the figures) Rivers. A growing number of radiocarbon dates demonstrates inconsistencies and disagreements with the results of IR-OSL dating and palaeobotanical investigations of contemporary materials taken from the same sampling locations. A special attention was given to the ages of numerous shells of the zebra mussel – *Dreissena polymorpha* (Pallas 1771), which occur in middle-late Holocene sediments beneath the Curonian Spit and Curonian Lagoon (Damušytė 2009). Recent dating of zebra mussel valves from the middle Holocene deposits contradict the currently held view that this species migrated into the Baltic Sea during the early 1800s through the canals joining the watersheds of rivers that drain into the Black and Baltic Sea basins (Karatayev et al. 1997, Olenin et al. 1999, etc.). A few scenarios to explain the old radiocarbon dates of *D. polymorpha* were suggested (Buynevich et al. 2011), but they do not address all the nuances and contradictions associated with the growing database of mollusc-based chronologies. The goal of this study is to present the potential mechanisms responsible for the observed age discrepancies and to explore the need for alternative methods of absolute chronology for accurate chronostratigraphic correlation and palaeogeographic reconstruction in this region.

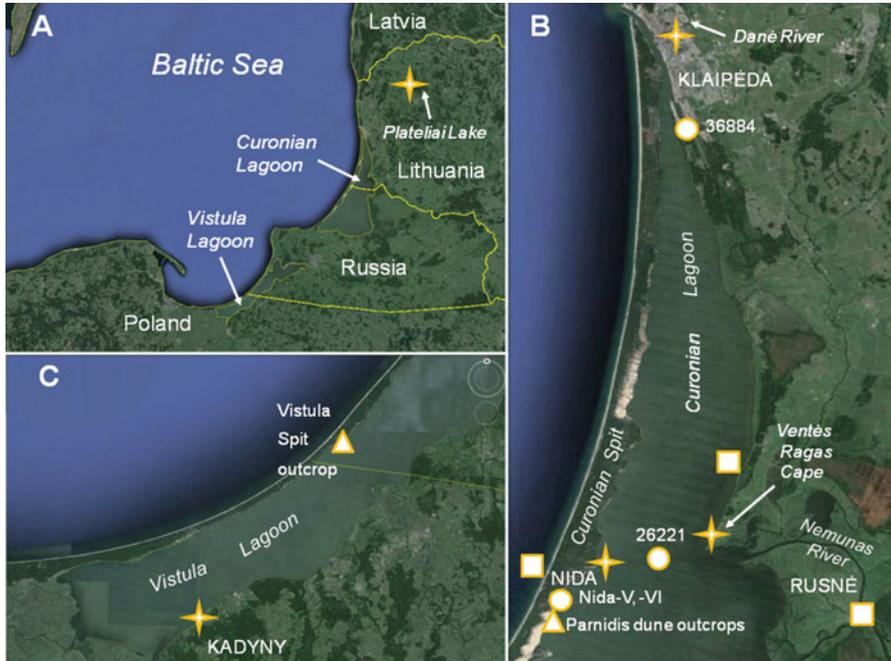


Fig. 8.1 Locations of study sites (Google Earth Images): map of the Southeastern Baltic Sea region (a) and detailed areas of the Northern part of the Curonian lagoon (b) and Southwestern part of the Vistula lagoon (c). Outcrops are marked by *triangles*, boreholes – *circles*, sampling sites of live molluscs – *stars*, sampling points for water bicarbonate dating – *squares*

8.2 Geological Setting

The area of investigations generally covers the Lithuanian part of the Curonian Lagoon and lagoon coast of the Curonian Spit, so the more detailed geological characteristic of this region are presented. The Curonian Spit is a 98-km-long, 0.4–4.0 km-wide sandy barrier that separates the Curonian Lagoon from the Baltic Sea (Fig. 8.1). This landform represents dynamic sedimentation processes at the land-sea interface during the Holocene: barrier spit formed as a result of the Semba Peninsula erosion (i.e. peninsula located between Curonian and Vistula lagoons; Fig. 8.1a) together with alongshore sediment flow and is linked with Litorina and Post-Litorina Sea stages of the Baltic Sea development (Gudelis 1997; Gelumauskaitė 2002; Kabailienė 2006; Damušytė 2011). Currently the Curonian Spit represents sand barrier with developed sandy dunes reached 50–60 m. The Baltic Ice Lake sediments prevails below the Litorina and Post-Litorina Sea lagoon and marine barrier sediments in the northern part of the Curonian Spit and Lagoon; whereas in the southern part of this region the till of the Last Glacial somewhere is detectable beneath the mentioned Litorina and Post-Litorina sediments (Sergeev et al. 2015). According to Trimonis et al. (2003) surfacial bottom sediments of the

Curonian Lagoon are heterogeneous, but recent clastogenic (medium- and fine-grained sand, silt) sediments obviously prevail and cover almost the whole lagoon. Biogenic sediments generally are represented by mollusc shells. In some limited areas – generally around the Ventės Ragas Cape – relicts of till (gravel and boulders) are outcropping due to erosional activity of currents (Trimonis et al. 2003). River runoff (23.1 km^3 per year) reaches the Curonian Lagoon from the area $100,458 \text{ km}^2$, 98% of which is from the Nemunas River basin (Pustelnikovas 1998). The tributaries of the Curonian Lagoon, as well as tributaries of the Vistula Lagoon, are draining the territories totally covered by Pleistocene deposits what have been formed due to erosion of Palaeozoic and Mesozoic rocks and are enriched by carbonates (Lithuania's Geology 1994).

The organic-rich freshwater lagoon sediments with abundant and diverse fossil mollusc fauna – the full list and characteristic of fossil molluscs is published by A. Damušytė (2009) – and fragments of fossil fish and wood were the focus of chronological investigation in the southeastern Baltic lagoonal sequences. The greatest number of dated materials was sampled from the so-called “lagoon marl” – a specific type of back-barrier deposit widespread in the western part of the Curonian Lagoon and extending beneath the Curonian Spit. “Lagoon marl” actually encompasses carbonaceous silty clay or clayey silt with admixture of finely dispersed organic matter. According to laboratory investigations, the main components have variable contribution: clay – 30–60%, organic matter – 10–50%, and carbonates – 10–25%. These sediments were accumulated in the ancient lagoon (the similar sedimentation in the recent lagoons of the southeastern Baltic is not appointed) due to relatively fast and intense accumulation when the inflow of mineral and organic matter probably was significantly bigger than at the recent. The term “lagoon marl” comes from the monographs and maps of Prussian investigators published in late XIX – early XX centuries and are used by a number of recent researchers (Kabailienė 1995, 1997; Gudelis 1998; Linčius 1993, and others). Here, we continue to use this broad lithological designation. The undisturbed (*in situ*) deposits of “lagoon marl” occur in the middle part of post-glacial sedimentary sequence: approximately 7–9 m below mean sea level (b.m.s.l.), their base extending down to 14–16 m b.m.s.l. Late Holocene sand typically caps the “lagoon marl”. At several sites along the landward edge of the spit, large fragments of lagoon strata are extruded onto the surface from beneath massive sand dunes. Formation of the “lagoon marl” extrusions in different parts of the Curonian Spit is a multistage and complicated process, partially addressed in the special paper (Sergeev et al. *in press*). As a result, the layers of the outcropping “lagoon marl” could be folded, and even overturned, but this non-*in situ* context can be verified in the field. Examples of such exposures of “lagoon marl” include two sections at the base of the Parnidis dune, south of Nida settlement: (1) a historical outcrop (or so-called “old” outcrop, >60 years old), ~25–30 m in length and ~2.5 m high (Fig. 8.2), and (2) a recent segment (or “new” outcrop emerged during the winter of 2007/2008) ~50–60 m long and 1.5 m high, located 80 m north of the “old” one (Buynevich et al. 2010; Fig. 8.3) and actively eroding at present.



Fig. 8.2 Field view of the “old” Parnidis dune outcrop and results of radiocarbon dating (calibrated age in years). Despite the fact that the sequence is not *in situ* (sediments were upturned during extrusion), the small-scale context is preserved and ^{14}C dating results of “lagoon marl” show quasi-normal chronological sequence of sedimentation and strongly support the Early Atlantic age of sediments. In contrast, the age of *Dreissena polymorpha* fossil shells is much younger

In the Vistula Spit, sediments similar to “lagoon marl” (described here as gyttja) are confined to a single site in the central part of the lagoon side of the spit (Figs. 8.1c and 8.4). In one section of the outcrop (Section A) gyttja occurs *in situ* 1.5–2.5 m above m.s.l., with another exposure (Section B) located close to mean sea level (Bitinas et al. 2008).

8.3 Methods

8.3.1 Field Observations and Sampling

All chronological material was collected during the past two decades as part of various geological surveys and stratigraphic investigations: geological mapping of the Lithuanian Maritime Region (1:50,000 scale) carried out by the Lithuanian Geological Survey, detailed engineering-geological mapping of the Klaipėda harbor, thematic scientific projects, as well as independent and collaborative initiatives by several researchers.

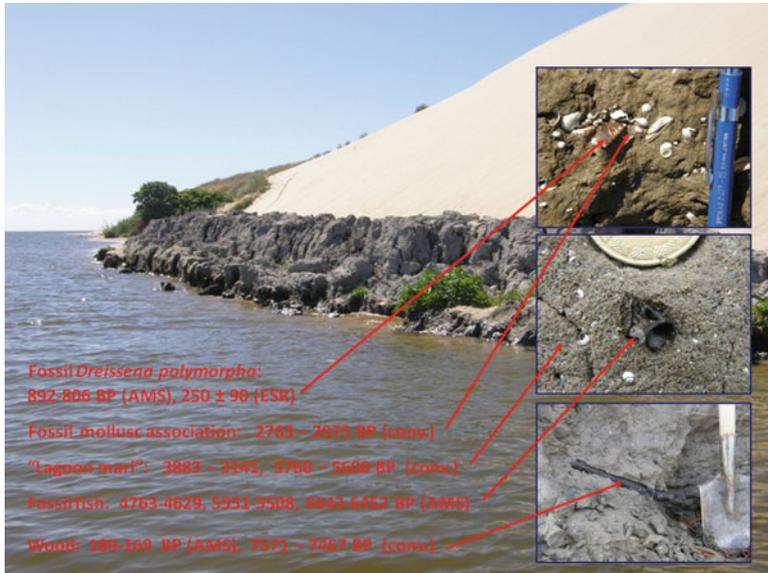


Fig. 8.3 Recent ("new") Parnidis dune outcrop and results of radiocarbon dating of different materials (calibrated age in years). Note the differences in raw age between adjacent *Dreissena polymorpha* fossil shells dated by different methods and the stark contrast with the age of the enclosing "lagoon marl"



Fig. 8.4 Gytja layer in the Vistula spit outcrop (sediments similar to the "lagoon marl" in the Curonian spit) and result of its radiocarbon dating (calibrated age in years)

Along the Curonian Spit lagoon shore, samples for radiocarbon dating of sediments, fossil mollusc shells (*Dreissena polymorpha*, *Valvata* sp., *Unio pictorum*, etc.), fish remains, and wood fragments were generally collected from both historical and recent outcrops of “lagoon marl” beneath the Parnidis dune. A number of sedimentological and chronological samples were taken from two closely spaced land-based boreholes Nida-V and Nida-VI located between the Parnidis dune and Nida settlement (Fig. 8.1b), as well as boreholes in the Curonian Lagoon (No. 26221) and Klaipėda harbor (No. 36884). Several samples of gyttja and fossil mollusc shells were obtained from the outcrop along the Russian part of the Vistula Spit (Fig. 8.1c).

Borehole Nida-VI was established as a type-section locality to address the chronostratigraphic problems within the framework of special thematic research supported by the Lithuanian Science Foundation (Fig. 8.1b). Boreholes were drilled mechanically, using auger technology for extracting partially disturbed sediments. The descriptions of borehole section were done in the field during the drilling process (see Table 8.1 and Fig. 8.5 for the borehole section description), along with sampling for subsequent laboratory investigations. Core samples were examined by a suite of methods, including radiocarbon (^{14}C) dating of organic sediments (conventional and AMS), sand dating by infrared optically stimulated luminescence (IR-OSL), and pollen analysis of the entire sediment column. Sand samples (~1 kg) for IR-OSL dating, sensitive to light exposure, were collected in special dark bags upon core extraction. Samples for pollen analysis were collected every 10 cm and sediments for ^{14}C dating were taken from the all organic-rich horizons (1–4 samples depending on the thickness of the target layer). According to visual lithological inspection of the sediment column, some sampled organic matter was noted to not occur *in situ*, i.e. older sediments were re-deposited in the younger layers (for example, fragments of peat between sand horizons at 13.90–14.20 m, Table 8.1; Fig. 8.5).

The IR-OSL analysis of Nida-VI borehole section focused on sand layers to a depth of 20.7 m. The deeper part of borehole Nida-VI section is composed of mixed sand and gravel with variable clay fraction (glaciofluvial sediments). Luminescence dating of this type of material is considered problematic due to the possibility that sand grains were not thoroughly bleached (“zeroed”) (Bitinas et al. 2001). Lithological, geochemical, and low-field magnetic susceptibility analyses were also performed on Nida-VI samples, but their treatment is beyond the scope of this paper and partially addressed in recent publications (Kaminskas and Bitinas 2013).

A large number of living molluscs were collected for comparison of dating results to the fossil counterparts of the same species (Fig. 8.1). For this purpose, living *Dreissena polymorpha*, *Valvata* sp., and *Unio pictorum* were sampled along the eastern shores of the Curonian Lagoon (Ventės Ragas Cape) and Vistula Lagoon (near Kadyny settlement, Poland). At both places, glacial sediments (till) are widespread along the nearshore and coastal sections of the lagoon. Sampling of *D. polymorpha* was also performed from the nearshore of the Curonian Lagoon near Nida (from a thriving population on the sandy lagoon bottom) and the Plateliai Lake (glacial substrate), as well as from the lower reaches of the Danė River (sandy

Table 8.1 Lithological description of Nida-VI borehole section (altitude: 0.5 m above m.s.l.)

Depth, m	Lithology	Age of sediments		
		IR-OSL, ka	¹⁴ C, cal. years BP	
			Organic carbon	Inorganic carbonates
0.0–0.9	Sand, medium-grained, well sorted, brownish-yellow and grayish-yellow, composed by feldspar-quartz, ³ HCl ⁻			
0.9–1.7	Sand, fine- and medium-grained, greenish-grey and black-grey, composed by feldspar-quartz, HCl ⁻			
1.7–3.3	Sand, fine- and very-fine grained, greenish-grey and yellowish-grey, composed by feldspar-quartz, HCl ⁻	0.9 ± 0.1		
		0.9 ± 0.1		
3.3–7.1	Sand, fine-grained, well sorted, greenish-grey and yellowish-grey, composed by feldspar-quartz, in the lowermost part (6.9–7.1 m) – silty sand with admixture of organic matter, HCl ⁻	0.9 ± 0.1		
		0.9 ± 0.1		
		0.9 ± 0.1		
7.1–12.5	“Lagoon marl”, black-grey, in the lowermost part from 11.0 m – black, in some intervals – with greenish tint, massive, with remnants of fossil mollusc shells, especially in the lowermost part of layer, HCl ⁺		2745–2458	
			5768–5609	
			7029–6664	
			7424–7243	10,160–9704
			7524–7430	11,750–11,311
		7786–7560	11,268–11,061	
12.5–12.7	Sand, fine- and very-fine grained, with admixture of gyttja, grey with greenish tint, composed by carbonates-feldspar-quartz, with remnants of fossil mollusc shells, HCl ⁺	5.7 ± 0.4		
12.7–12.9	“Lagoon marl”, black-grey (analogous to int. 7.1–12.5 m), massive, with thin interlayers of fine-grained sand, HCl ⁺		8392–8276	
12.9–13.9	Sand, very-fine grained, grayish-yellow and yellowish-grey, composed by carbonates-feldspar-quartz, in lowermost part with admixture of thin (1–2 cm) interlayers of silt enriched by organic matter and macro-remnants of plants, HCl ⁺⁺	5.7 ± 0.4		
13.9–14.2	Peat, black and dark brown, well decomposed, with very thin interlayers of fine-grained sand, with rotten pieces (Ø until 5 cm) of wood and seeds, in the uppermost part – with remnants of freshwater molluscs (<i>Valvata sp.</i>), HCl ⁻		9901–9629	
			8645–8417	
			(AMS)	
			8811–8588	
14.2–15.6	Sand, very fine-grained, grey with greenish tint, composed by	5.8 ± 0.4		
		5.9 ± 0.4		

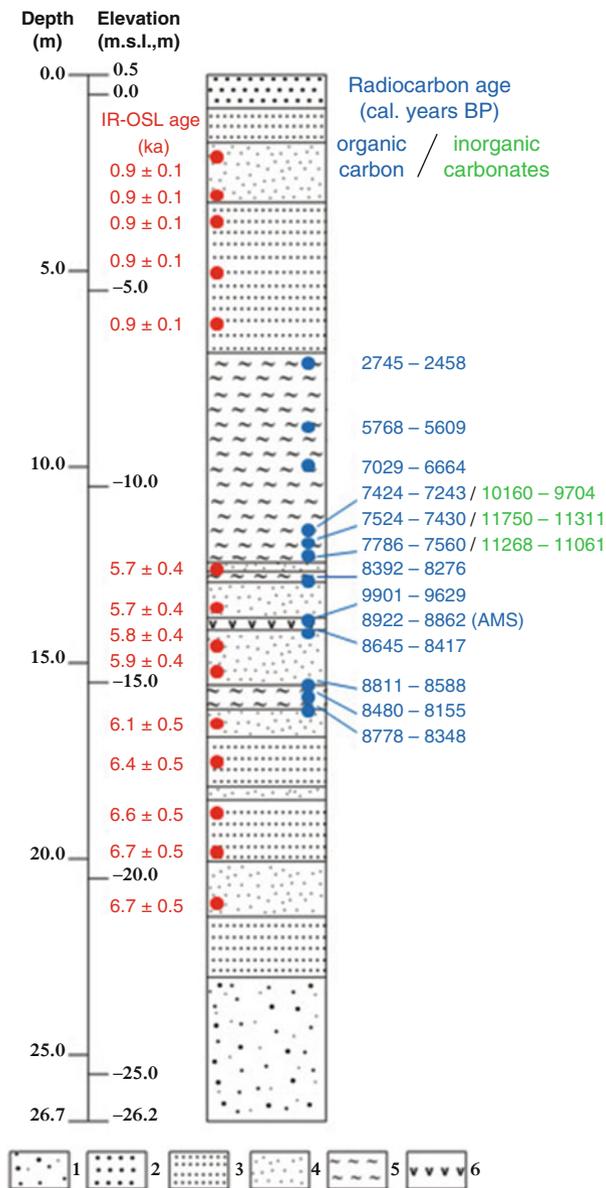
(continued)

Table 8.1 (continued)

Depth, m	Lithology	Age of sediments		
		IR-OSL, ka	¹⁴ C, cal. years BP	
			Organic carbon	Inorganic carbonates
	carbonates-feldspar-quartz, with rare admixture of dispersive organic matter and single macro-remnants of plants, HCl ⁺⁺			
15.6–16.2	“Lagoon marl”, grey, massif, in the lowermost part – black-grey and patched, carbonaceous, with abundant of very small remnants of fossil mollusc shells, HCl ⁺⁺		8811–8588	
			8480–8155	
			8778–8348	
16.2–16.6	Sand, fine-grained, well sorted, grey with greenish tint, composed by carbonates-feldspar-quartz, HCl ⁺	6.1 ± 0.5		
		6.4 ± 0.5		
16.6–16.7	Sand, very fine-grained, well sorted, greenish-grey and grey with greenish tint, composed by carbonates-feldspar-quartz, HCl ⁺⁺			
16.7–20.2	Sand, fine-grained, greenish-grey and grey with greenish tint, composed by carbonates-feldspar-quartz, with interlayers of sand with gyttja (2–3 cm thick), silty sand (2–3 cm thick), with admixture of single pieces of wood (Ø 2–3 cm), HCl ⁺	6.6 ± 0.5		
		6.7 ± 0.5		
20.2–21.6	Sand, very fine-grained, yellowish-grey, composed by carbonates-feldspar-quartz, in the lowermost part of layer – with small admixture of dispersive organic matter and single pieces of mollusc shells, HCl ⁺	6.7 ± 0.5		
21.6–23.0	Sand, fine-grained (somewhere – until medium grained), yellowish-grey, composed by carbonates-feldspar-quartz, in the lowermost part – with interlayers of silty sandy (5–6 cm thick), HCl ⁺			
23.0–26.7	Moderately sorted sand (prevailed medium-grained), more coarse in the lower half of the layer – with single gravel (Ø until 4 cm), yellowish-grey, composed by carbonates-feldspar-quartz, in the lowermost part – with interlayers of fine grained silty sand, HCl ⁺			

^aReaction with hydrochloric acid (10%): HCl⁻ – non-react; HCl⁺ – temperate/good; HCl⁺⁺ – intense

Fig. 8.5 Geological section of the Nida-VI borehole and results of ¹⁴C and IR-OSL dating. Lithological legend: 1 – moderately sorted sand, 2 – medium-grained sand, 3 – fine-grained sand, 4 – very fine-grained sand, 5 – “lagoon marl”, 6 – peat (Note the discrepancy between the two dating techniques, as well as between different materials dated by ¹⁴C method)



deposits). For the analysis of recent water bicarbonates, a suite of samples was collected from the Baltic Sea near Nida, eastern (Ventės Ragas Cape) and western (Nida) nearshore sites within the Curonian Lagoon, the lower reaches of the Nemunas River (Rusnė settlement), and from the Neris River – the main tributary of the Nemunas River in eastern Lithuania.

8.3.2 Radiocarbon (^{14}C) Dating Analyses

Radiocarbon analyses were performed using both conventional (^{14}C) dating of bulk samples and Accelerator Mass Spectrometry (AMS) dating of individual mollusc valves and other organic materials. The bulk samples were analyzed at the Laboratory of Nuclear Geophysics and Radioecology, State Research Institute Nature Research Centre, Vilnius, Lithuania (Tables 8.1, 8.2 and 8.3; laboratory index Vs). Samples included associated assemblages of fossil and recent molluscs, organic sediments (“lagoon marl”, gyttja), wood, peat, and water bicarbonates. The technological lines for benzene synthesis and purification used for analysis were produced at the Kiev Radiocarbon Laboratory, Ukraine, where several additional dates were obtained. The specific activity of ^{14}C in benzene was measured by liquid scintillation counting (LSC) method (Gupta and Polach 1985; Arslanov 1985; Kovaliukh and Skripkin 1994) using the liquid scintillation analyzer Tri-Carb 3170TR/SL. The radiocarbon calibration program OxCal v. 4.1 (Bronk Ramsey et al. 2010) and the calibration curve IntCal13 (Reimer et al. 2013) were used for the calibration of radiocarbon dates (calendar years BP). The data of recent mollusc shells are presented uncalibrated.

Mollusc valves (mostly from articulated specimens), organic sediment fraction, as well as several fragments of fossil fish (without collagen extraction) and wood were dated at the National Ocean Sciences Accelerator Mass Spectrometry (NOSAMS) facility, Woods Hole Oceanographic Institution, USA, using the new continuous-flow (SFAMS) system with a gas ion source (Table 8.5; laboratory index OS). Samples were calibrated using Calib 7.0 software.

8.3.3 Palaeodosimetric Dating (IR-OSL, ESR)

Palaeodosimetric dating techniques used in the present study – potassium feldspar-based infrared optically stimulated luminescence (IR-OSL) and mollusc-based electron spin resonance (ESR) – have played a significant role in establishing and refining the chronologies for archaeology (Molodkov 2001) and Quaternary geology of Northern Eurasia (Molodkov and Bolikhovskaya 2009), including the Lithuanian coastal region (Molodkov and Bitinas 2006; Molodkov et al. 2010; Bitinas et al. 2011).

The “luminescence clock” used by IR-OSL dating is the trapped charges (electrons) in the crystal structure of feldspar minerals. The number of these charges is related to the time elapsed since mineral grains were buried after their last episode of exposure to the natural light.

The ESR dating method of the mollusc shells consists in a direct measurement of the number of radiation-induced paramagnetic centers, created in the shell matter due to exposure to natural radiation. The number of these centers is also related to the age of the shell (see Molodkov 1993 for ESR methodology).

Table 8.2 Results of conventional radiocarbon ^{14}C dating of sediments and fossil molluscs from the Southeastern Baltic sea spits and lagoons

No.	Lab. index	Sampling site; coordinates	Altitude (mean sea level, meters)	Analyzed material	^{14}C age, years BP ($\pm 1\sigma$)	Calibrated age, years BP (1σ ranges)	Calibrated age, years BP (2σ ranges)
1.	Vs-1158	Curonian spit, Parnidis dune "old" outcrop (sediments not <i>in situ</i>);	1.5	"Lagoon marl", organic carbon (OC)	6500 \pm 90	7565–7530 BP (17.5%)	7620–7317 BP (95.4%)
		55° 17' 34" N, 20° 56' 16" E				7524–7430 BP (50.7%)	
2.	Vs-1159	-"	1.0	-"	6130 \pm 100	7161–6901 BP (68.2%)	7257–6779 BP (95.0%) 6763–6756 BP (0.4%)
3.	Vs-1160	-	0.5	-	7380 \pm 105	8329–8153 BP (54.1%)	8386–8005 BP (95.4%)
						8140–8133 BP (1.8%)	
						8119–8109 BP (2.6%)	
						8092–8055 BP (9.7%)	
4.	Vs-1565	-"	1.5 \div 1.7	<i>Dreissena polymorpha</i> , carbonates	1570 \pm 120	1004–1247 BP (1)	883–1358 BP (1)
5.	Vs-1497	Vistula spit outcrop, section B;	~2.1	<i>Unio pictorum</i> , carbonates	2690 \pm 90	2288–2545 BP (1)	2170–2675 BP (1)
		54° 28' 40" N, 19° 42' 06" E					
6.	Vs-1545	Vistula spit outcrop, section A;	~1.5	Gytija, OC	2310 \pm 190	2699–2633 BP (7.9%)	2770–1898 BP (95.4%)
		54° 28' 32" N, 19° 41' 43" E				2617–2587 BP (3.4%) 2539–2124 BP (56.9%)	

7.	Vs-1591	Curonian Lagoon coast, borehole No 36884; 55° 38' 51" N, 21° 09' 02" E	-1.3 ÷ -1.6	"Lagoon marl", OC	6600 ± 90	7566–7434 BP (68.2%)	7650–7645 BP (0.3%) 7623–7323 BP (95.1%)
8.	Vs-1596	-"-	-5.9 ÷ -6.1	-"-	9180 ± 80	10,476–10,471 BP (1.4%) <u>10,421–10,245 BP</u> (66.8%)	10,557–10,222 BP (95.4%)
9.	Vs-1665	Curonian spit, borehole Nida-V; 55° 17' 55" N, 20° 59' 58" E	-7.7 ÷ -8.0	-"-	4930 ± 60	<u>5715–5602 BP</u> (68.2%)	5887–5819 BP (7.8%) 5761–5583 BP (87.1%) 5499–5492 BP (0.4%)
10.	Vs-1831	Curonian spit, Parnidis dune "new" outcrop (sediments not <i>in situ</i>); 55° 17' 35" N, 20° 59' 56" E	~0.0 ÷ 1.5	-"-	3260 ± 300	<u>3883–3141 BP</u> (66.5%) 3125–3112 BP (0.8%) 3093–3080 BP (0.8%)	4293–2775 BP (95.4%)
11.	Vs-1869	-"-	-"-	Wood, OC	6630 ± 70	7571–7467 BP (68.2%)	7616–7424 BP (95.4%)
12.	Vs-1902	-"-	-"-	Association of molluscs, carbonates	3100 ± 90	2763–2975 BP (1)	2714–3123 BP (1)
13.	Vs-1948	-"-	-"-	"Lagoon marl", OC	4970 ± 110	5889–5821 BP (17.4%) <u>5760–5600 BP</u> (50.8%)	5939–5573 BP (90.4%) 5552–5473 BP (5.0%)

Primary treatment of mollusc shells was done by washing with H₂O₂ in ultrasound bath, for all the rest samples the acid-alkali-acid pre-treatment was applied OxCal v.4.2.3 software with IntCal 13 atmospheric curve are used for OC Calib Rev 7.0.2 software with Marine 13 curve are used for carbonates Age range of highest probability from 1 σ ranges is underlined

Table 8.3 Results of conventional radiocarbon ^{14}C dating of borehole Nida-VI sediments Coordinates: $55^{\circ} 17' 49'' \text{ N}$, $20^{\circ} 59' 52'' \text{ E}$

No	Lab index	Depth, meters below m. s.l.	Analyzed material	^{14}C age, years BP ($\pm 1\sigma$)	Calibrated age, years BP (1σ ranges)	Calibrated age, years BP (2σ ranges)
1.	Vs-2083	-6.6	“Lagoon marl”, organic carbon (OC)	2510 ± 120	<u>2745–2458 BP</u> (67.4%) 2447–2443 BP (0.8%)	2846–2335 BP (95.4%)
2.	Vs-2080	-8.0	-, -	4990 ± 120	5890–5806 BP (22.7%) <u>5768–5609 BP</u> (45.5%)	5993–5572 BP (91.0%) 5553–5473 BP (4.4%)
3.	Vs-2081	-9.8	-, -	6010 ± 170	7155–7111 BP (5.7%) 7084–7079 BP (0.6%) 7068–7056 BP (1.4%) <u>7029–6664 BP</u> (60.5%)	7267–6475 BP (95.4%)
4	Vs-2237	-10.8 ÷ -10.9	“Lagoon marl”, OC	6380 ± 105	<u>7424–7243 BP</u> (64.1%) 7197–7180 BP (4.1%)	7489–7150 BP (88.9%) 7125–7018 BP (6.5%)
5.	Vs-2234		“Lagoon marl”, inorganic carbonates (IC)	8840 ± 165	10,160–9704 BP (68.2%)	10,265–9529 BP (95.4%)
6.	Vs-2238	-11.0 ÷ -11.1	“Lagoon marl”, OC	6590 ± 90	7565–7530 BP (17.5%) <u>7524–7430 BP</u> (50.7%)	7620–7317 BP (95.4%)
7.	Vs-2236		“Lagoon marl”, IC	$10,020 \pm 120$	11,750–11,311 BP (68.2%)	11,996–11,231 BP (95.4%)
8.	Vs-2248	-11.2	“Lagoon marl”, OC	6790 ± 140	<u>7786–7560 BP</u> (62.7%) 7540–7514 BP (5.5%)	7930–7892 BP (3.1%) 7876–7432 BP (92.3%)
9.	Vs-2246		“Lagoon marl”, IC	9730 ± 145	<u>11,268–11,061 BP</u> (37.2%) 11,033–10,991 BP (4.8%) 10,976–10,787 BP (26.2%)	11,612–11,571 BP (2.9%) 11,510–10,664 BP (92.5%)

(continued)

Table 8.3 (continued)

No	Lab index	Depth, meters below m. s.l.	Analyzed material	^{14}C age, years BP ($\pm 1\sigma$)	Calibrated age, years BP (1σ ranges)	Calibrated age, years BP (2σ ranges)
10.	Vs-2082	-12.3	"Lagoon marl", OC	7500 \pm 100	8392–8276 BP (44.2%)	8519–8494 BP (1.5%)
					8269–8201 BP (24.0%)	8482–8153 BP (90.5%)
						8141–8132 BP (0.4%)
						8120–8107 BP (0.6%)
						8094–8052 BP (2.4%)
11.	Vs-2079	-13.5	Peat, OC	8760 \pm 70	9901–9629 BP (68.2%)	10,135–10,061 BP (6.8%)
						10,039–10,024 BP (0.9%)
						10,013–9990 BP (1.5%)
						9951–9549 BP (86.1%)
12.	Vs-2078	-13.6	-, -	7770 \pm 100	8645–8417 BP (68.2%)	8974–8913 BP (3.5%)
						8898–8883 BP (0.8%)
						8866–8828 BP (2.4%)
						8792–8385 BP (88.6%)
13.	Vs-2084	-15.4	"Lagoon marl", OC	7890 \pm 140	8978–8878 BP (17.1%)	9089–9045 BP (1.6%)
					8872–8825 BP (8.2%)	9039–8410 BP (93.8%)
					8811–8588 BP (42.9%)	
14.	Vs-2087	-15.5	-, -	7500 \pm 190	8516–8493 BP (2.8%)	8760–7937 BP (95.4%)
					8480–8155 BP (60.6%)	
					8117–8112 BP (0.7%)	
					8090–8057 BP (4.1%)	

(continued)

Table 8.3 (continued)

No	Lab index	Depth, meters below m. s.l.	Analyzed material	¹⁴ C age, years BP ($\pm 1\sigma$)	Calibrated age, years BP (1σ ranges)	Calibrated age, years BP (2σ ranges)	
15.	Vs-2086	-15.6	-,,-	7730 \pm 190	8929–8925 BP (0.4%)	9076–9060 BP (0.3%)	
					8858–8835 BP (2.2%)		9032–8169 BP (95.1%)
					<u>8778–8348 BP (65.6%)</u>		

$\delta^{13}\text{C}$ value of organic carbon from the depth –11.7 metres m.s.l. is –20.5‰ PDB, whereas $\delta^{13}\text{C}$ value of organic carbon in the recent bottom sediments of the Curonian Lagoon is changing between –30 and –28‰ PDB (Mažeika et al. 2009)

OxCal v.4.2.3 software with IntCal 13 atmospheric curve used for calibration

Age ranges of highest probabilities at 1σ are underlined

IR-OSL and ESR age determinations were carried out at the Research Laboratory for Quaternary Geochronology (RLQG), Institute of Geology, Tallinn University of Technology, Estonia. An overview of the IR-OSL dating procedure used in RLQG is presented in Molodkov and Bitinas (2006). Good consistency of comparative results obtained using both dating methods are demonstrated in Molodkov (2012).

IR-OSL dating of 14 samples was used for age determination of sand layers in the key section – borehole Nida-VI (Table 8.6). ESR dating aided in age determination of a single fossil *D. polymorpha* shell from “lagoon marl” in the Parnidis dune “new” outcrop. All IR-OSL and ESR dates are reported as calendar ages.

8.3.4 Pollen Analysis

Pollen analysis of the Nida-VI type section was used for a high-resolution stratigraphic subdivision and correlation. Fifty samples were prepared according to standard procedures (see Grichuk and Zaklinskaya 1948, Erdtman 1960), with identification of pollen taxa based on Faegri and Iversen (1989) and Moore et al. (1991). The pollen percentage diagram was plotted using “TILIA” (v. 2) and “TILIAGRAPH” software (v. 2.0 b.5) (Grimm 1990, 1992).

8.4 Results

8.4.1 Radiocarbon Analysis

Parnidis Dune Outcrop The radiocarbon dating results of “lagoon marl” in the “old” Parnidis dune outcrop yield 8.3–7.2 cal ka BP (conventional ^{14}C), whereas the dating of the fossil zebra mussel (*D. polymorpha*) shells from the top of this outcrop fall consistently within a much younger age range of 1.0–0.8 cal ka BP (AMS) (Tables 8.2 and 8.5; Fig. 8.2). The “lagoon marl” in the recent (“new”) Parnidis dune outcrop dates to 5.6–3.1 cal ka BP (conv. ^{14}C) (Fig. 8.3). The ages of other materials from the same section yielded very different ages: the fossil fish remains are as old as 4.6 to 6.7 cal ka BP (AMS) (Table 8.5); the various mollusc associations (bulk sample) are 2.9–2.7 cal ka BP old (Table 8.2); *D. polymorpha* shells yield more recent ages of 0.85 cal ka BP (all conv. ^{14}C) (Table 8.4); and two wood fragments date to 7.6 and 0.2 cal ka BP, conv. ^{14}C and AMS, respectively (Tables 8.2 and 8.5).

Borehole Nida-VI Conventional ^{14}C dates of “lagoon marl” (organic carbon) from Nida-VI borehole vary in age from 8.8–8.3 to 2.7–2.5 cal ka BP (Fig. 8.5; Tables 8.3 and 8.5). The sequence of ages is in chronological order, i.e. the age of sediments is progressively increasing with depth, except for an inversion at 13.6–13.5 m b.m.s.l. where two samples of peat were dated. A single wood fragment from 13.5 m yielded an AMS age of 9.0–8.7 ka BP. Three samples of “lagoon marl” from the 11.2–10.8 m interval were duplicated – i.e. were analyzed organic and inorganic components of the same samples. Inorganic carbon was dated to 11.3–9.7 cal ka BP, compared to the organic sample age of 7.8–7.2 cal ka BP, i.e. the latter yielded a 4.2–2.5 ka younger age compared to the sediment date based on inorganic fraction (Table 8.2).

Borehole Nida-V In this borehole, samples from the depth 7.8–8.0 m b.m.s.l. show a range of results (Tables 8.2 and 8.5): the age of “lagoon marl” varied between 5.6 (conv. ^{14}C) and 2.0 (AMS) cal ka BP, a *Valvata sp.* shell from the same layer dated to ca. 4.0 cal ka BP (AMS; Table 8.5).

Curonian Lagoon AMS dating of *D. polymorpha* shells from 8.75 m b.m.s.l. in the central part of the Curonian Lagoon (borehole No 26221; Fig. 8.1; Table 8.5) produced an age that varied from 1.1–1.2 cal ka BP to modern. According to geological mapping (unpublished materials at the Archive of the Lithuanian Geological Survey), the enclosing sand layer is attributed to the Litorina Sea stage, i.e. about 8.3–3.7 cal ka BP.

Conventional ^{14}C dates of “lagoon marl” along the eastern coast of the Curonian Lagoon, in the vicinity of Klaipėda harbour (borehole No. 36884; Fig. 8.1; Table 8.2) cluster within 10.5–10.3 cal ka BP and 7.5 cal ka BP for sediment samples taken at 5.9–6.1 m and 1.3–1.6 m b.m.s.l., respectively. Meanwhile,

Table 8.4 Results of conventional radiocarbon ^{14}C dating (uncalibrated) of the recent mollusc shells and water's bicarbonate of the Baltic Sea, Curonian lagoon, and related Nemunas-Neris tributary system

No	Lab. index	Sampling site; coordinates	Analyzed object, material	^{14}C activity, pMC ($\pm 1\sigma$)	^{14}C age, years BP ($\pm 1\sigma$)	$\delta^{13}\text{C}$, ‰ PDB
1.	Vs-1642	Curonian lagoon,	<i>Dreissena polymorpha</i>	83.0 ± 0.7	1495 ± 55	n/m
2.	Vs-1681	Ventės ragas cape;	“-“	96.4 ± 0.6	295 ± 55	n/m
3.	Vs-1682	$55^\circ 22' 14''$ N, 21°	<i>Unio pictorum</i>	91.2 ± 0.6	740 ± 55	n/m
4.	Vs-1683	$12' 51''$ E	<i>Viviparus sp.</i>	94.9 ± 0.6	420 ± 55	n/m
5.	Vs-1703	Vistula lagoon, Kadyny	<i>Dreissena polymorpha</i>	90.5 ± 0.6	1440 ± 70	-9.5
6.	Vs-1705	$54^\circ 18' 17''$ N, 19°	<i>Viviparus sp.</i>	89.4 ± 0.6	900 ± 55	-8.2
7.	Vs-1706	$28' 26''$ E	<i>Unio pictorum</i>	92.4 ± 0.7	635 ± 60	n/m
8.	Vs-2331	Curonian lagoon, Nida	<i>Dreissena polymorpha</i> , recent mollusc shells from bottom surface, mixture from six locations	99.3 ± 1.5	modern	-9.1 ÷ -8.1 (range from 6 measurements)
9.	Ki-X	Curonian lagoon, Ventės ragas cape;	<i>Dreissena polymorpha</i> , \emptyset 0–5 mm	85.6 ± 0.9	1280 ± 90	n/m
10.	Vs-1797	$55^\circ 22' 14''$ N, 21°	<i>D. polymorpha</i> , \emptyset 5–10 mm	94.0 ± 0.7	510 ± 60	-8.9
11.	Vs-1794	$12' 51''$ E	<i>D. polymorpha</i> , \emptyset 10–15 mm	97.4 ± 0.4	220 ± 50	-9.3
12.	Vs-1796		<i>D. polymorpha</i> , \emptyset 15–20 mm	92.6 ± 0.5	630 ± 60	-10.0
13.	Vs-2261		<i>D. polymorpha</i> , \emptyset 20–25 mm	91.5 ± 0.5	710 ± 40	-10.2
14.	Vs-2330		<i>D. polymorpha</i> , \emptyset 25–30 mm	93.2 ± 0.9	570 ± 70	-10.1
15.	Ki-Y		<i>D. polymorpha</i> , \emptyset 30–35 mm	93.9 ± 0.8	520 ± 70	n/m
16.	Vs-1810	Danė River, Klaipėda;	<i>Dreissena polymorpha</i>	86.0 ± 0.5	1210 ± 40	-9.5
		$55^\circ 42' 34''$ N, $21^\circ 08' 18''$ E				

(continued)

Table 8.4 (continued)

No	Lab. index	Sampling site; coordinates	Analyzed object, material	^{14}C activity, pMC ($\pm 1\sigma$)	^{14}C age, years BP ($\pm 1\sigma$)	$\delta^{13}\text{C}$, ‰ PDB
17.	Vs-1824	Plateliai Lake; 56° 02' 53" N, 21° 51' 31" E	<i>Dreissena polymorpha</i>	104.2 ± 0.6	modern	-5.5
18.	Vs-1890	Nemunas River, Rusnė, 2009; 55° 17' 33" N, 21° 23' 19" E	Water's biocarbonate	78.9 ± 0.5	1960 ± 70	-6.7
19.	Vs-457	Curonian lagoon, Nida, 1982; 55° 18' 41" N, 21° 00' 43" E	-"-	81.1 ± 0.9	1760 ± 90	n/m
20.	Vs-483	Curonian lagoon, Nida, 1983; 55° 18' 41" N, 21° 00' 43" E	-"-	95.8 ± 0.6	320 ± 70	n/m
21.	Vs-1891	Curonian lagoon, Šturmai, 2009; 55° 21' 55" N, 21° 12' 38" E	-"-	78.8 ± 0.5	1970 ± 70	-8.9
22.	Vs-2293	Neris River, Paneriai, 2012; 54° 47' 13" N, 24° 54' 47" E	-"-	83.3 ± 0.8	1510 ± 80	-9.4
23.	Vs-2292	Neris River, Buivydžiai, 2012; 54° 50' 22" N, 25° 44' 30" E	-"-	80.8 ± 0.7	1765 ± 65	-9.2

(continued)

Table 8.4 (continued)

No	Lab. index	Sampling site; coordinates	Analyzed object, material	^{14}C activity, pMC ($\pm 1\sigma$)	^{14}C age, years BP ($\pm 1\sigma$)	$\delta^{13}\text{C}$, ‰ PDB
24.	Vs-484	Baltic Sea, Nida, 1983; 55° 18' 57" N, 20° 59' 05" E	--	114.4 \pm 1.7	modern	n/m

Primary treatment of mollusc shells was done by washing with H_2O_2 in ultrasound bath

n/m – not measured

Ki-X,-Y samples were dated in Kiev Radiocarbon Laboratory but lab index numbers were not applied

$\delta^{13}\text{C}$ values of samples were measured with the reproducibility of 0.1‰ at the Tallinn University of Technology, Estonia, and at the Institute of Geochemistry and Geophysics of National Academy of Sciences of Belarus

AMS-dated *D. polymorpha* shell from a sandy section located between the aforementioned layers produced an age of 100-years, i.e. practically modern (Table 8.5).

The ages of the living zebra mussel valves from the eastern Curonian Lagoon coast (Ventès Ragas Cape; Fig. 8.1) varied from 710 ± 40 to 220 ± 50 uncalibrated ^{14}C years, with a single sample as old as 1280 ± 90 uncalibrated years BP (AMS and conv. ^{14}C ; Tables 8.4 and 8.5). Due to possible correlation between mollusc size and age, a series of *D. polymorpha* shells were grouped according to size fractions before dating (Table 8.4). Analysis of co-occurring *Unio pictorum* and *Viviparus sp.* mussels fall into the same age range of 740 ± 55 and 420 ± 55 uncalibrated years BP (conv. ^{14}C), respectively. An assemblage of modern *D. polymorpha* shells from the bottom sediments of the western Curonian Lagoon near Nida has apparent age very close to modern with ^{14}C specific activity of 99.3 ± 1.5 pMC.

Continental Waters Several zebra mussel shells from other water basins were dated for comparison to the results from coastal lagoons. In the lower reaches of the Danė River, which flows through Kalaipėda city into the northern Curonian Lagoon, these species are up to 1210 ± 40 uncalibrated ^{14}C years old. In contrast, the dates obtained on the shells from nearshore of the Plateliai Lake, located in the upland part of Lithuania (Fig. 8.1a) are consistent with their negative post-bomb age (^{14}C activity is 104.2 ± 0.6 pMC; Table 8.4).

Vistula Spit and Lagoon According to bulk ^{14}C dating results of samples from the Vistula Lagoon outcrop (Fig. 8.1c, 4), the gyttja in Section A was formed ~ 2.5 – 2.1 cal ka BP (Table 8.2). *Unio pictorum* from the adjacent Section B has a similar conventional date of 2.5–2.3 cal ka BP (Table 8.2), whereas *D. polymorpha* from the same layer yielded an AMS age of 0.9–0.8 ka cal BP (Table 8.5).

Table 8.5 Results of AMS ^{14}C dating

No	Lab index	Field number	Sampling site ^a	Analyzed material	^{14}C age, years BP (1σ range)	Calibrated age, years BP 1σ ranges (%)	Calibrated age, years BP 2σ ranges (%)	$\delta^{13}\text{C}$, ‰
1.	OS-57403	BD-P1	Curonian spit, Parmidis dune "old" outcrop (sediments not <i>in situ</i>);	<i>Dreissena polymorpha</i> , fossil, carbonates	1260 ± 30	856–758	893–724	–5.4
2.	OS-57404	BD-L-26221	Curonian lagoon, borehole No 26221, depth: –8.75 m ^b	-“-	285 ± 30	modern	modern	–8.6
3.	OS-59536	BD-L-26221A	-“-	-“-	1600 ± 25	1218–1134	1243–1078	–7.6
4.	OS-57405	BD-V-1	Vistula spit outcrop, section A	-“-	1340 ± 30	926–845	951–792	–8.9
5.	OS-57522	ND-5-830	Borehole Nida-V, depth: –7.8 m ^b	"Lagoon marl", organic carbon (OC)	2050 ± 30	2053–1969 (89.9) 1962–1951 (10.1)	2114–2077 (12.4) 2073–1932 (87.6)	–30.2
6.	OS-59535	ND-5-850	Curonian spit, borehole Nida-V, depth: –8.0 m ^b	Gastropod (<i>Valvata</i> sp), carbonates	3970 ± 30	4021–3905	4075–3864	–12.9
7.	OS-69633	BD-VR-0	Curonian lagoon, Ventés ragas cape	<i>Dreissena polymorpha</i> , recent, carbonates	885 ± 30	522–480	546–452	–9.9
8.	OS-69635	BD-L-36884	Curonian lagoon coast, borehole No 36884, depth: –1.6 – -2.1 m ^b	<i>Dreissena polymorpha</i> , fossil, carbonates	110 ± 25	modern	modern	–6.7
9.	OS-69634	BD-PD-1	Curonian spit, Parmidis dune "new" outcrop (sediments not <i>in situ</i>)	-“-	1300 ± 30	892–806	917–763	–5.2
10.	OS-71606	P-W-01	-“-	Wood, OC	175 ± 20	281–268 (19.8) 213–193 (26.3) 189–169 (30.9) 153–146 (8.5) 14–4 (14.5)	285–261 (19.1) 221–163 (50.8) 158–140 (11.3) 24–0 (19.0)	–26.2

(continued)

Table 8.5 (continued)

No	Lab index	Field number	Sampling site ^a	Analyzed material	¹⁴ C age, years BP (1 σ range)	Calibrated age, years BP 1 σ ranges (%)	Calibrated age, years BP 2 σ ranges (%)	$\delta^{13}\text{C}$, ‰
11.	OS-71411	P-FISH-2008	-“-	Fossil fish, OC	4760 \pm 20	5582–5574 (12.2) 5551–5508 (66.8) 5488–5475 (21.0)	5585–5470	–27.9
12.	OS-97116	PD-F-2010	-“-	-“-	4160 \pm 40	4822–4798 (15.4) 4792–4791 (0.6) 4763–4629 (84.0)	4832–4573	–24.4
13.	OS-97292	PD-F-2011	-“-	-“-	5750 \pm 85	6643–6452	6742–6394 (97.9) 6370–6347 (1.6) 6334–6324 (0.5)	–21.1
14.	OS-90184	NIDA-6-1400	Curonian spit, borehole Nida-VI, depth: –13.5 m ^b	Wood, OC	7990 \pm 35	8991–8949 (24.6) 8943–8934 (4.2) 8922–8862 (37.4) 8834–8720 (33.8)	9003–8720	–30.1

^aCoordinates of sampling sites are presented in Tables 1–3

^bDepth below mean sea level, meters

Calibrated ages are 2 σ years BP (CALIB 7.0 software; MARINE curve used for carbonates)

Age ranges of highest probabilities at 1 σ and 2 σ are marked in bold

Dating of living molluscs from Vistula Lagoon (Fig. 8.1c) was undertaken on the same assemblage as in the Curonian Lagoon: *D. polymorpha*, *Unio pictorum*, and *Viviparus sp.* show age variations from 1470 ± 70 to 635 ± 60 uncalibrated ^{14}C years (conv. ^{14}C ; Table 8.4).

Water Bicarbonates Conventional ^{14}C dating of water bicarbonates in the eastern part of the Curonian Lagoon, to the north of the Ventès Ragas Cape (Fig. 8.1b), shows that the recent water is 1960 ± 70 uncalibrated ^{14}C years old. An identical result was obtained from the lower reaches of the Nemunas River in front of Rusnė settlement – 1970 ± 70 uncalibrated ^{14}C years BP (Table 8.4). The other two water samples from the Neris River, the main tributary of the Nemunas in the eastern part of Lithuania, are 1765 ± 65 and 1510 ± 80 uncalibrated ^{14}C years BP. In contrast to these freshwater settings, a radiocarbon date of nearshore water bicarbonates from the Baltic Sea side of Nida settlement is negative as derived from post-bomb ^{14}C excess (114.4 ± 1.7 pMC)

Isotopes The $\delta^{13}\text{C}$ values of dated samples of different materials vary in a large range: carbonates of *D. polymorpha* – from -10.2 to -5.2‰ , water carbonates – from -9.4 to -6.7‰ , organic carbon of sediments – from -30.2 to -20.5‰ , wood – from -30.1 to -26.2‰ , and fish bones – from -27.9 to -21.1‰ . The $\delta^{13}\text{C}$ values indicate the main carbon sources, however their variation does not reveal clear tendencies that could be used to predict the reservoir effect.

8.4.2 Palaeodosimetric Dating

Results of infrared optically stimulated luminescence (IR-OSL) dating of 14 samples from the borehole Nida-VI core show that the age of lacustrine and lagoon sand is gradually increasing with depth, from 0.9 ± 0.1 ka in the upper part to 6.7 ± 0.5 ka at 20.7–20.5 m b.m.s.l. (Fig. 8.5; Table 8.6).

The results of ESR dating on a single *D. polymorpha* shell extracted from the middle part of the “new” Parnidis dune outcrop provides an independent age of 250 ± 90 years (Fig. 8.3; Table 8.7).

8.4.3 Pollen Analysis

In the Nida-VI borehole, the highest concentrations of pollen and spores were detected between 5.25–26.40 m. Based on palynological analysis, five chronozones were established, ranging from Boreal to Sub-Atlantic (Fig. 8.6). The pollen and spore diagram indicates the sedimentary and, possibly, stratigraphic hiatus during a particular time span between Boreal and Atlantic, and nearly stable and relatively continuous sedimentation during the Atlantic. These findings fit well with the

Table 8.6 Results of IR-OSL dating and radioactivity data from the Nida-VI borehole sand samples

No	Elevation (m.s.l., m)	Lab. no	Field no	U (ppm)	Th (ppm)	K (%)	IR-OSL age (ka)
1	-1.2 to -1.8	RLQG 2008-101	OSL 2	0.32	0.01	0.26	0.9 ± 0.1
2	-2.2 to -2.3	RLQG 2009-101	OSL 3	0.01	0.00	0.28	0.9 ± 0.1
3	-3.0 to -3.1	RLQG 2010-101	OSL 4	0.01	0.00	0.25	0.9 ± 0.1
4	-4.8 to -4.9	RLQG 2011-101	OSL 6	0.00	0.00	0.27	0.9 ± 0.1
5	-6.4 to -6.5	RLQG 2012-101	OSL 8	0.00	0.00	0.36	0.9 ± 0.1
6	-12.1 to -12.2	RLQG 2013-101	OSL 9	0.21	0.76	0.93	5.7 ± 0.4
7	-13.2 to -13.4	RLQG 2014-101	OSL 10	0.25	0.91	1.07	5.7 ± 0.4
8	-13.8 to -13.9	RLQG 2015-101	OSL 11	0.50	0.61	0.97	5.8 ± 0.4
9	-14.8 to -15.0	RLQG 2016-101	OSL 13	0.20	0.30	0.98	5.9 ± 0.5
10	-16.0 to -16.1	RLQG 2017-101	OSL 14	0.18	0.23	0.92	6.1 ± 0.5
11	-16.5 to -16.7	RLQG 2018-101	OSL 15	0.05	0.00	0.83	6.4 ± 0.5
12	-18.1 to -18.2	RLQG 2019-101	OSL 17	0.26	0.19	0.88	6.6 ± 0.5
13	-19.1 to -19.2	RLQG 2020-101	OSL 19	0.12	0.62	0.81	6.7 ± 0.5
14	-20.5 to -20.7	RLQG 2021-101	OSL 20	0.08	0.76	0.91	6.7 ± 0.5

Notes:

U, Th, K are the uranium, thorium and potassium content in sediments.

Uncertainties: U determination: ± 2–3%; Th: ± 3–4%; K: ± 1–2%; gamma irradiation: ± 3–5%.

Table 8.7 Result of ESR dating of *Dreissena polymorpha* shell sample and radioactivity data on enclosing “lagoon marl” from the Parnidis dune “new” outcrop

No	Elevation (m.s.l., m)	Lab. no	Field no	U (ppm)	Th (ppm)	K (%)	ESR age (a)
1	0.2	RLQG 430-090	NIDA 2010	1.06	2.31	1.09	250 ± 90

Notes:

U, Th, K are the uranium, thorium and potassium content in sediments

Uncertainties: U determination: ± 2–3%; Th: ± 3–4%; K: ± 1–2%; gamma irradiation: ± 3–5%

results of IR-OSL dating: both methods indicate that the bulk of the borehole section was deposited during the Atlantic chronozone.

8.5 Discussion

Analysis of radiocarbon dating results of a diverse suite of geological materials and comparison to other chronometric techniques reveals several clear trends and highlights important disparities. These are believed to go beyond the issues that characterize chronological datasets with large numbers of dates and the inherent possibility of statistically overlapping ranges. The first issue involves age inversions and discrepancies in borehole sections. The second relates to the substantially “aged” live molluscs from the Curonian and Vistula Lagoons and their tributaries. The third, the most problematic issue in our view, is the inconsistency between dating results of different materials within the same layer or sample, especially between mollusc shells and enclosing “lagoon marl”. The last two concerns constitute the main reason for why the majority of controversial radiocarbon dating results collected from a number of Curonian and Vistula sites have not yet been published.

The contradictions associated with borehole sections may be largely explained based on sedimentological criteria. For example, age reversal in Nida-VI borehole observed at ~13.5 m b.m.s.l. (Fig. 8.4; Table 8.3, samples Vs-2079 and Vs-2078) could be explained by the fact that the sampled organic horizon with the stratigraphically inverted age of 9.9–9.6 cal ka BP is not in its primary context. Rather, it represents re-deposited peat and thus an older organic matter as suspected during original visual description of the borehole (see *Field observations and sampling*). Thus, the dated fragments of peat, including AMS-dated wood fragment (Table 8.5; sample OS-90184, 8.9–8.7 cal ka BP) extracted from the same layer, were likely eroded and re-deposited from older sediments, compared to the rest of the enclosing layers of “lagoon marl”.

The problem of overestimating the ages of recent mollusc shells could be explained by a significant influence of the reservoir effect (or so-called “hard-water” effect) in the semi-enclosed Curonian and Vistula Lagoons. Based on analyses of pre-bomb mollusc shells across a strategic salinity transect in the Baltic Sea the reservoir age for Eastern Baltic is evaluated as 200–250 years (Lougheed et al. 2013). Dating of water bicarbonates Curonian Lagoon shows that the local reservoir effect depends strongly on water composition of the river tributaries (Table 8.4; samples Vs-1890, -1891, -2292, -2293) and is more variable (Mažeika et al. 2010). These findings have major ramifications for establishing the local reservoir correction (ΔR) for southeast Baltic region. Dating of water bicarbonates indicates that the main tributary of the Nemunas River, the Neris, is transporting a substantial amount of dissolved older bicarbonates (Table 8.4; Mažeika et al. 2010). But it is clear that the “hard water” effect is greater at the mouth of the Nemunas River compared to Neris-Nemunas junction in the eastern part of Lithuania, with

the amount of dissolved old carbonates gradually increasing along the fluvial course. Thus, the greatest “hard water” effect in the lagoon is close to the mouths of tributaries and decrease with distance. For example, according to the dating results of samples collected in 1983 adjacent to Nida, this effect was slightly more than 300 years (Table 8.4: sample Vs-483) most likely due to the marine water inflow occasionally observed in this part of the lagoon (Galkus 2007). The radiocarbon dating of mollusc shells suggest that the total reservoir effect in the Curonian Lagoon and its fluvial tributaries (for example, in the Danė River) may reach up to 1200–1500 years (Table 8.4; samples Vs-1642, Vs-1810) and thus caution must be taken when only the standard marine correction (maximal of 400 years) is applied (Fischer and Heinemeier 2003).

The “hard water” in the Curonian Lagoon also confirms the recent findings based on dating modern fish bones. AMS dates of pike-perch and bream caught in the lagoon in October 2014 yielded conventional ^{14}C ages of 782 ± 30 BP (90.72 pMC) and 539 ± 30 BP (93.5 pMC) (Piličiauskas and Heron 2015), respectively.

A similar scenario may exist in the Vistula Lagoon, with mollusc-based chronology suggesting a local reservoir effect of up to 1400 years (Table 8.4: sample Vs-1703). In contrast, preliminary dating of live *D. polymorpha* shells from the Plateliai Lake indicates that this vital effect is not an issue in mainland lakes (Table 8.4: sample Vs-1824). This is likely due to different hydrological conditions and less important metabolic role of dissolved old carbonates, compared to coastal lagoons. Modern age of *D. polymorpha* shells was also obtained from the Curonian Lagoon nearshore close to Nida (Table 8.4: sample Vs-2331). This fact can be explained by the aforementioned periodic influence of marine water, as well as by fresh groundwater discharge along the nearshore region of the Curonian Spit. The main source of the groundwater is atmospheric precipitation filtered through aeolian sand of the Curonian Spit dunes that do not contain carbonate minerals (Gudelis 1989–1990). It is important to note that freshwater reservoir effect in radiocarbon dating of molluscs within landlocked or semi-enclosed basins is a very characteristic and widely analyzed issue in a large number of recent scientific publications (Petersen et al. 1992; Fischer and Heinemeier 2003; Philippsen 2013).

The most pressing issue to date is the contradiction between radiocarbon dating results of “lagoon marl” and mollusc shells. This question remains after a decade of data compilation, beginning with dating of fossil *D. polymorpha* shells (1.2–1.0 cal ka BP) and “lagoon marl” (8.3–7.4 cal ka BP; Table 8.2) from the “old” Parnidis dune outcrop. The same problem persisted when several years ago the “new” outcrop was investigated. New dates of fossil *D. polymorpha* shells, obtained by both radiocarbon (~3.0–2.8–~0.9–0.8 cal ka BP; Fig. 8.3; Tables 8.2 and 5) and ESR (~250 years, Fig. 8.3; Table 8.7) dating, shows significantly younger age than the enclosing “lagoon marl” (~5.6–3.1 cal ka BP; Fig. 8.3; Table 8.2). The same incongruity was observed in the Vistula Lagoon outcrop (Fig. 8.1b) where the age of *D. polymorpha* fossil shell of ~0.9–0.8 cal ka BP (Table 8.5) contrasts with that of the surrounding gyttja layer (~2.5–2.1 cal ka BP; Table 8.2). It is possible to conclude that in all cases, whether from Parnidis dune outcrops, boreholes in the Curonian Spit and lagoon, or the Vistula Spit outcrop, there arises a pattern in the

disparity between ages of mollusc shells and enclosing sediments: most molluscs yield younger ages. This discrepancy varies widely, from a few centuries to several millennia. Thus, the key question is: which ages are the most accurate? Only in recent years, when the special type-section borehole Nida-VI was drilled and a series of radiocarbon ages of “lagoon marl”, IR-OSL dates of sand, and pollen analyses were completed, it became possible to clarify some of the chronometric disagreements. At the same time, dating of water bicarbonates provided a substantial independent contribution to the addressing this issue.

Comparison of radiocarbon and IR-OSL dating results in the sedimentary sequence of the Nida-VI borehole shows clear discrepancies: the optical age of sand horizons is up to several millennia younger than the adjacent “lagoon marl” layers (Fig. 8.5). IR-OSL dating results fit well with the pollen analysis of the entire section (Fig. 8.6). On the other hand, there is a large intra-sample radiocarbon dating discrepancy. The “lagoon marl” age based on inorganic carbonates is ~4.2–2.5 ka older than that obtained by dating the organic carbon fraction (Table 8.3, sample Nos. 4–10). This raises serious concerns regarding the reliability of dating the lagoon sediments by radiocarbon method. Thus, the IR-OSL dates may represent a “foothold” from which to assess the accuracy of radiocarbon ages. According to IR-OSL and palynological databases, the entire sequence of “lagoon marl” penetrated by the Nida-VI borehole was formed during the Atlantic chronozone, i.e. starting ~6.5–6.0 ka BP and ending ~4.0 ka BP. Thus, we argue that the “lagoon marl” outcropping in both Parnidis dune exposures should date to the same time interval.

In contrast to mollusc ages, AMS radiocarbon ages of three fossil fish remains (4.5 to ~6.5 cal ka BP; Table 8.5) collected during different field seasons from three separate sections of the “new” Parnidis outcrop fit well with the results of IR-OSL dating and pollen analysis. Remains of fossil fish (bones) are mainly composed of calcium phosphate, keratin, hydroxyapatite (ca. 60–70%), and collagen (ca. 30%). The latter, as the main component of the bone targeted for dating, has only minor carbonate content and is less influenced by “hard-water” effect. Thus, although their findings *in situ* or in boreholes are extremely rare, fish remains have the potential to serve as more reliable objects for radiocarbon dating in the lagoon sediments. On the other hand, AMS dating of fish (food residue) remains on the pottery obtained during archaeological investigations demonstrates a substantial reservoir effect in the lagoon conditions and suggest that these materials be avoided in chronological reconstructions (Fischer and Heinemeier 2003; Philippsen 2013). Further analysis of the taphonomy and intra-sample chronological variability in fish skeletal assemblages will undoubtedly contribute to resolving this issue.

In addition to samples of “lagoon marl”, fish bones, and molluscs in the “new” Parnidis outcrop, two samples of wood yielded controversial dates (~8.0 and ~0.17 cal ka BP; Table 8.5). Whereas the older date can be theoretically explained by the piece of old wood being reworked from elsewhere and re-deposited in the younger section of the “lagoon marl”, the younger age may be the result of a more recent incorporation of woody debris during the extrusion of the marl. In general, because wood is not part of autochthonous lagoon sedimentation, it is not a good

material for chronological control, except for providing a maximum limiting for the overlying strata.

Interestingly, ESR dating of a *D. polymorpha* shell sample from the same layer of the “lagoon marl” yielded a very similar age (250 ± 90 years; Table 8.7) to the younger wood fragment, but is much younger than the associated ^{14}C -dated molluscs ($\sim 3.0\text{--}2.8$ to $\sim 0.9\text{--}0.8$ cal ka BP). It is noteworthy that in spite of geologically extremely young shells, which are most likely the youngest ever dated by ESR, the shells turned out quite suitable for ESR analysis and dating: ESR spectra from these shells revealed sufficiently distinguishable radiation-induced signals that allowed to reliably record the analytical line at $g=2.0012$ (for methodology details, see Molodkov 1993, Molodkov et al. 1998). Sensitivity to irradiation of the shell matter was also quite high. Therefore, we conclude that the ESR age of these *D. polymorpha* shells is very close to accurate.

8.5.1 Vital Effects

It becomes increasingly evident that very similar trends in disagreement between chronometric results of molluscs and sediments are characteristic for other parts of northern Europe. For example, AMS dating of sediments from the cores on the Barents Sea shelf indicates that mollusc shells are much younger than enclosing sediments dated by thermoluminescence (Krapivner 2006; Gusev et al. 2012). Recent studies in the southern North Sea region of the Netherlands show that multi-dating analyses performed on the same sediments may produce different results: AMS radiocarbon dating of mollusc shells yields MIS 3 (36–50 ka BP) ages, whereas optically stimulated luminescence dating of quartz and feldspar grains point to sedimentation that occurred much earlier, >117 ka (Busschers et al. 2012). Similarly, Polish investigators report a discrepancy of 3.9–2.4 ka between younger molluscs and surrounding mud from the Gulf of Gdansk coast (Jeglinski et al. 2012).

According to the sedimentation regime in the Curonian Lagoon, fluvial input is the main source of terrigenous material entering the lagoon, whereas the finely dispersed organic component of lagoon sediments is forming primarily through the contribution of local phytoplankton (Žilius 2011). As suggested by our recent findings, the rivers should be considered as the key contributors of dissolved bicarbonates. The amount of dissolved carbonates in river water depends on the geological structure of the drainage basin. As it has been mentioned, the tributaries of the Curonian and Vistula Lagoons drain the Pleistocene deposits enriched in Palaeozoic and Mesozoic carbonates (Lithuania’s Geology 1994). This contributes to a large reservoir (“hard-water”) effect reflected in the results of radiocarbon dating of inorganic carbonates. Dissolved atmospheric carbon has the reverse effect on metabolic processes, skeletal construction, and potential elemental fractionation in the lagoonal phytoplankton by mitigating the impact of dissolved old carbonates (Philippesen 2013). As a result, dating of organic carbon fraction will likely yield a

younger age compared to that of inorganic fraction (i.e., reduced contribution of reservoir effect). Unlike the formation of organic sediments, sand sedimentation in the lagoon subenvironments is proceeding without any influence of aforementioned processes, so IR-OSL dating results are emerging as the most reliable chronometric technique.

Based on the age of living mollusc shells from the Curonian and Vistula Lagoons, the reservoir effect is estimated to be 1400–1500 years. However, such correction of radiocarbon dating results cannot be applied to fossil molluscs due to taphonomic effects. Our results suggest that following their burial, the molluscs were likely affected by complex post-depositional processes. The timing of inception of these taphonomic effects, either upon burial or some time following the formation of the death assemblage, is not clear. What is becoming increasingly evident is a substantial “younging” trend of the molluscs relative to the enclosing sediments. Thus, the taphonomic overprinting remains the key issue pending resolution.

According to Busschers et al. (2012) the “rejuvenation” of fossil mollusc shells is a result of re-crystallization inside the shell structure accompanied by contamination by younger carbon from CO₂-rich groundwater. However, active groundwater circulation beneath the Curonian Lagoon floor is unlikely due to a thick low-permeability layer of the “lagoon marl” producing inadequate geological-hydrogeological conditions for this process. Besides, the long experience of one of the authors (A.M.) in the field of ESR dating of fossil mollusc shells (over 30 years and more than 500 shell samples from 860,000 to 250 years in age) indicates that re-crystallization of the initial aragonite structure even in the Pleistocene mollusc shells is highly unlikely. The “lagoon marl” itself is rich in organic matter, serving as a methane source in southeastern Baltic lagoons (Pimenov et al. 2013). Perhaps this gas could be an important factor that influences the epigenetic alterations in the mollusc shells. However, at present, we can only conclude that this issue remains a challenge that requires in-depth investigation.

8.5.2 Conceptual Model of Chronometric Pathways

Based on our findings, we propose a model of syn- and post-depositional geological processes to address the chronological issues pertinent to the southeastern Baltic Sea lagoons (Fig. 8.7). An idealized “sedimentary box” contains different types of sediments, such as “lagoon marl”/gyttja and sand, which were formed contemporaneously in a hypothetical lagoon. The highest influence of the “hard water” effect during the sedimentation involves the influx of inorganic carbonates (in our case, minerogenic constituents of “lagoon marl”), so that their radiocarbon dating shows the maximum age (green pathway). An organic constituent of “lagoon marl” is influenced by reservoir effect to a lesser degree and its ¹⁴C age is also older than the true age (blue pathway), but younger than inorganic carbonates. Similarly, mollusc shells that have been exposed to a substantial reservoir effect yield older ages (dark

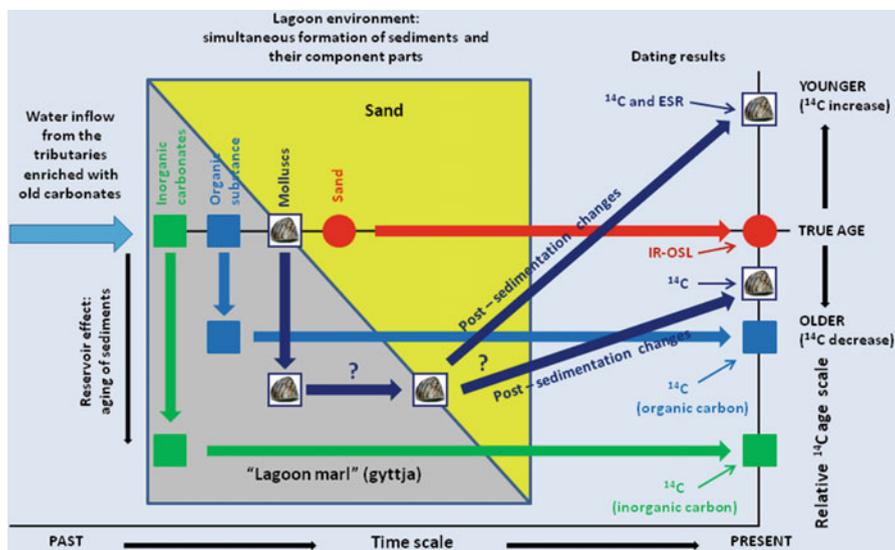


Fig. 8.7 Conceptual model of sedimentary processes and pathways responsible for deviations in radiocarbon and other chronological results for a suite of materials from the lagoons of the Southeastern Baltic Sea region. Note the diverse post-depositional and taphonomic pathways that likely explain the discrepancies between the analytical ages of dated materials and their true age

blue pathway), but the taphonomic effects (see squares with the shell label). As a result, their ^{14}C age is always younger than that of the “lagoon marl”, but the degree of this “younging” can be quite variable. Thus, IR-OSL dating of sandy deposits (red pathway) serves as the “fulcrum” for the most reliable chronological reconstruction.

The results of this study have wide-ranging applications to geological, archaeological, and biogeographical research, which relies heavily on accurate dating of sediments and enclosing organic remains. Future work will aim to refine the chronology of lagoonal sequences in the southeast Baltic region, with the ultimate goal of establishing the types of samples that are unsuitable for radiocarbon dating, as well as those “elite” materials that will provide the most reliable reconstruction of regional chronology, especially where luminescence ages cannot be obtained.

8.6 Conclusions

Radiocarbon dating of lagoon sediments enriched by organic carbon and carbonates (“lagoon marl”, gyttja), as well as ubiquitous fossil mollusc shells in recent sediments beneath the lagoons and spits of the southeastern Baltic Sea, has

demonstrated a substantial influence of fractionation and post-depositional processes (local reservoir effect) in the mollusc shells. The vital processes affecting the fossil zebra mussel (*Dreissena polymorph*) and other species cause substantially younger ages than the enclosing sediments. The age discrepancy can exceed 1000 years and thus cannot be used for accurate millennium-to-century scale reconstructions of the sedimentation history based on biostratigraphy alone. Optically-stimulated luminescence (potassium feldspar-based IR-OSL) supplemented by the results of palynologic investigations, is emerging as the most reliable chronological technique for lagoon deposits in the southeastern Baltic Sea. Dating results of organic remains (molluscs, wood fragments, or re-deposited peat horizons) or inorganic carbonates are untrustworthy, with further investigation required for unconventional types (e.g., fish remains). Thus, the earlier chronology of lagoon and spit deposits in the southeastern Baltic Sea region that relied heavily on radiocarbon dating of organic sediments, especially mollusc shells, should be critically revised. Understanding of the taphonomic effects on the traditional organic dating materials remains an important theoretical and practical issue that requires an integrated multidisciplinary approach. A conceptual model of chronological pathways is presented as a working hypothesis and a basis for future research.

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