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ASSESSING THE LITHOLOGICAL COMPOSITION OF CORE SEDIMENT SAMPLES TO INVESTIGATE THE INFLUENCE OF NATURAL AND ANTHROPOGENIC STRESSORS

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Abstract. Naturally occurring processes, as well as anthropogenic activities, leave their marks on terrestrial and aquatic ecosystems which are undergoing extensive material and energy exchanges. Both natural and anthropogenic factors have played a significant role in creating the past and present hydro-morpho-dynamic conditions at the Danube Delta Biosphere Reserve edifice. This study was aimed to assess the anthropogenic and natural factors influencing spatial changes in the lithological composition of core sediment gathered from specific transitional environments. In this sense, six sediment cores were retrieved from Tătaru Lake (located in the fluvial delta plain), Puiu Lake (located in the marine delta plain), and Musura Bay (located in the coastal environment). To understand how lacustrine ecosystems *versus* coastal ecosystems respond to the natural and anthropogenic factors we assess between-core variation of the main lithological components (total organic matter - TOM%, total carbonates - CAR%, siliciclastic fraction - SIL%) and their spatial accumulation and layer distribution. Core sediments were analyzed for their lithological composition by means of LOI Method, attempting to figure the degree of natural and or/anthropogenic influence to the investigated transitional ecosystems. Results indicated a considerable vertical (in-depth) variation of the investigated parameters in function of the natural location of the sampling sites, and particularly, showed notable differences between the layers. The spatial variability, as well as in-depth fluctuations of the investigated parameters in core-sediments retrieved from different environs, indicated that the sediments have different susceptibilities to local environmental changes, which is important to improve understanding of deltaic/coastal processes and dynamics using sediment cores. The results obtained in this paper are based on a preliminary approach to better understand the hydro-sedimentary processes that occurred within these depositional settings. Further detailed new information and complex investigations are necessary to decipher the active deltaic sedimentation mechanisms in a fluvial-delta-sea environment.

Keywords: Danube Delta, natural factors, sediment cores, total organic matter, transitional environment

1 INTRODUCTION

An aquatic ecosystem acquires its identity from the interaction between its watery environment, as well as sediments, and the living fauna and flora communities that inhabit it. Both freshwater and saltwater ecosystems are extremely important to the environment and biosphere, and subsequently for human populations concerning the ecosystem services they yield (Wetzel, 2001). Following this, aquatic sediments comprise variable and complex mixtures of organic and inorganic materials (Bortone, 2006), as clays, silts, quartz grains, terrestrial organic detritus, planktonic material, and calcareous shell fragments. They are important archives for the study of the past environmental changes as they act as transporters, storage containers (deposition or redeposition), and contamination sources, recording the history of human-environment interactions (Dearing et al., 2006). As well, the sediments may be used for reconstructing climatic and environmental changes at different time scales, assess the impacts of human activities on ecosystems (Mills et al., 2017) and their watersheds (Boyle et al., 2015). Sediment quality assessment tools and approaches

include a wide spectrum of specific studies (e.g., paleo-ecological, physical, chemical, biological, geochemical, ecotoxicological etc.) (Loizeau et al., 2001; Minyuk et al., 2007; Wang et al., 2009) linked to hydro-environmental research. Among that, the assessment of the lithological composition of sediments is another useful environmental proxy for the preliminary characterization of lake ecosystems. The main lithological components i.e., total organic matter - TOM%, total carbonates - CAR%, siliciclastic fraction - SIL% can be influenced by both natural and anthropogenic factors and these lithological assortments can be used to estimate the contributions of different external or internal forcing mechanisms to changes within a depositional system. Locally, the naturally occurring processes are related to the water discharge and sediment load of the Danube River, hydro-meteorological circumstances, the basin morphology and clogging/siltation of lakes (Gâstescu & Știucă, 2008). On the other hand, over the past several decades, human related activities have influenced the deltaic ecosystems in responding to the changes brought on, especially, by the hydro-technical works carried out along the Danube River and its tributaries, as well as within the Danube Delta. These have considerably influenced the river water and sediment discharge and consequently the particle flux in the coastal area (Panin et al., 2016). Ecosystems belonging to the Danube Delta Biosphere Reserve are known to be the transition areas between the Black Sea and the drylands, being recognized for their socio-economic and ecological importance, as well as for their complexity and vulnerability to natural and anthropogenic pressures (Giosan, 2012). These territories have been officially nominated, under different conventions, in terms of cultural heritage protection, as UNESCO World Heritage Sites, Biosphere Nature Reserve and Wetland of International Importance (Ramsar Convention, 1987). The natural and/or anthropogenic stressors produce changes in the dynamics of the entire region, i.e., Danube River-Danube Delta-Black Sea system, resulting in changes related to flora and fauna species (Rose, 1992; Rose et al., 1993) sediment budget (transport and dynamics) (Panin et al., 1999; Panin & Jipa, 2002; Habersack et al., 2013), environmental flows and droughts between the tributaries river mouths and the adjacent coastal zone (Stănică, 2012), erosion (Stănică, 2011) and so on. The aim of the present study was to investigate between-core variation within depth slices, of the main lithological components (total organic matter - TOM%, total carbonates - CAR%, siliciclastic fraction - SIL%) and their spatial accumulation and distribution. Sediment cores were collected from three aquatic ecosystems (i.e., Tătaru Lake, Puiu Lake and Musura Bay) located in different transitional environments and supposing differing sediment composition. Core sediments were analyzed for their lithological composition by means of LOI Method, attempting to figure the degree of natural and or/anthropogenic influence to the investigated transitional ecosystems. The exploratory results obtained from this study may contribute to better insights into the complex relationships occurring within such depositional environments.

2 MATERIALS AND METHODS

2.1 Sampling sites

The three aquatic systems were chosen to cover a supposing wide range of sediment types (Fig. 1). **Tătaru Lake** is a medium-sized, shallow lake located within Sireasa-Șontea-Fortuna intertributary depression that belongs to the fluvial delta plain. Specifically, Tătaru L. is situated southward of Lungu Lake and Draghilea Canal, which ensures double protection against the direct Danube River alluvial input (Rădan&Rădan, 2007). This intermediate location within the Danube Delta gives it some water body stagnation environmental conditions that allow the accumulation of nutrients, sediments, and particles. These circumstances increase the chances of abundant development of aquatic vegetation, implicitly algal blooms (Wurtsbaugh et al., 2019). Generally, the catchment lithology is characterized by sediments accumulating on the bottom lake which are highly organic muds (silts) composed predominantly from autochthonous sedimentation. **Puiu Lake** is a relatively large lake (with an area of 8.8 km²), located in the Lumina-Roșu intertributary depression in the framework of the marine delta plain. More precisely, is located between Sulina and Sf. Gheorghe Branches, east of the Caraorman Spit (Panin et al., 2016). In terms of the environmental circumstances, it can be appreciated that Puiu L. is characterized by the same similar water body stagnation environmental conditions. Its catchment is relatively geomorphologically quiescent with plenty of underwater vegetation. The lithology of the lake catchment is predominantly related to intrabasinal organic muds (silts) deposits originated from autochthonous sources. **Musura Bay** is a shallow semi-enclosed water body (1-2 m depth) with low salinity (1-5‰). The bay is located in the delta front (Stănică et al., 2007), in the area where marine and freshwater mix, namely there where Chilia and Sulina branches empty into the Black Sea. Musura Bay is characterized by a high dynamic environment, permanently changing due to the amount of Danube's water flowing out, and, as a result of the action of the waves of the Black Sea. Consequently, its catchment lithology

is made from sediment material linked to extrabasinal sources, meaning a mixture of allochthonous silts, sands as well as autochthonous organic muds/silts.

2.2 Coring and Laboratory Analysis

Two sediment cores were extracted from Tătaru Lake, during a field campaign carried on 2019. The other ones were gathered from Puiu Lake and, respectively from the Musura Bay during 2020. The field activities occurred onboard the RV-”Istros” (NIRD - GeoEcoMar, Romania). The sampling site locations were georeferenced by a handheld GPS receiver (Garmin Montana 680, Garmin International Inc.). All the cores were retrieved using a Hydro-Bios corer, with a transparent plastic tube. The length of the cores depended on the bottom lake substrate consistency (not longer than 56 cm). The cores preserved their original depositional sequences with undisturbed sediment material. Onboard, the cores were sliced along the transversal plane (a horizontal cut with a sterile palette knife), withdrawn from the plastic tube, and divided into 2 cm-thick slices. Then, the sediment samples were described in details in terms of their structure, texture, grain size, sorting, stratification, the main lithoclast components (clay, mud, silt, sand, gravel), and bioclasts (shells and detritus, faunal and vegetal remains), including color, odor and general appearance etc. After that, the samples of core sediment were transferred into plastic holders (100 g) and stored at 4°C in darkness until further laboratory analysis. In the laboratory, core sediment sub-samples (~ 0.10 g) were tested for the present study. Primarily, the sediment sub-samples were oven-dried at 105°C (Mettler Etuve) to determine the moisture content (%) by Loss On Drying (LOD) method (Smith and Mullins, 2000; ASTM-D221). After oven drying, the samples were weighed and exposed to sequential heating (Snol 8.2/1100 Calcination Furnace) and measuring weight loss between heating stages (%) by Loss On Ignition (LOI) method. The loss in mass by combustion at 550°C (Dean, 1974; Bengtsson & Enell, 1986; Beaudoin, 2003; Boyle, 2001; Boyle, 2004), confers an assessment of the total organic matter (TOM%). The loss in mass by calcination at 950°C-1000°C (Digerfeldt et al., 2000; www.geog.cam.ac.uk), imparts an estimation associated to the total carbonate content (CAR%). The residual material is ascribed to the siliciclastic fraction (SIL%). The results are expressed as percentages of the total sample mass.



Figure 1. Map showing the research area and the location of the investigated sites

3 RESULTS AND DISCUSSIONS

The main physical-chemical and biological characteristics of sediments relate to a multitude of aspects, including variations in sediment flow, sorting during sediment transport, physical and chemical weathering, the geochemical background, catchment processes etc. The lithological determination is an exploratory tool for investigating aquatic sediments in order to decipher past and recent sedimentary depositional environments. This study analyzed the lithological characteristics (i.e., TOM-%, CAR-%, SIL-%) of six sediment cores retrieved from aquatic ecosystems as follows: Tătaru L. (DD19-55 & DD19-66), Puiu L. (DD20-47 & DD20-66) and Musura Bay (DD20-34 & DD20-35), to decipher their spatial as well as vertical distribution. The assessment of the main lithological

components was performed by LOI method aimed to estimate the relative total organic matter, total carbonates and siliciclastic contents from every core sample. Predominantly, the sediments encompass various organic and inorganic constituents, such as mineral grains and organic matter. Both freshwater and marine sediments (porous, soft, or lithified) incorporate three main components i.e., organic matter, carbonate, and siliciclasts that integrates their solid fraction (Ricken, 1993). In this research, the percentage concentration (%) of the main lithological components (TOM-%, CAR-%, SIL-%) varied considerably between the investigated sampling sites as regards their geographical position and lithological content. In view of this, were noticed discernible vertical distribution aspects and trends. The synthetic results are summarized in the following table (Table 1), including minimum, maximum, and the average values obtained from the analysis. The obtained results from this study were converted to binary and ternary diagrams (Fig. 2, Fig. 3).

Table 1. The percentage concentration (%) of the main lithological components

Locati on	Core name and length (cm)	Lithological components of core sediments								
		TOM (%)			CAR (%)			SIL (%)		
		Min	Max	Mean	Min	Max	Mean	Min	Max	Mean
<i>Tătaru Lake</i> (n = 23)	DD19-55 (46.5 cm)	53,00	79,15	69,07	2,55	10,02	4,72	16,77	36,98	26,21
<i>Tătaru Lake</i> (n = 25)	DD19-66 (50 cm)	55,28	75,83	63,10	8,61	14,85	11,56	12,47	36,10	25,34
<i>Puiu Lake</i> (n = 26)	DD20-47 (52 cm)	2,47	75,37	43,92	14,9 3	35,66	25,89	9,32	68,59	30,19
<i>Puiu Lake</i> (n = 28)	DD20-48 (56 cm)	4,40	65,13	36,13	24,5 3	60,05	44,13	10,35	44,00	19,74
<i>Musur a Bay</i> (n = 28)	DD20-34 (56 cm)	10,70	36,14	25,65	6,86	12,54	8,80	56,98	79,31	65,54
<i>Musur a Bay</i> (n = 28)	DD20-35 (56 cm)	37,00	55,22	37,00	5,95	11,11	7,62	34,77	56,17	45,23

Total organic matter (TOM %). Organic matter can be roughly recognized as an important environmental indicator of aquatic sediments, hinting at the potential contributions of autochthonous and allochthonous sources with respect to their origin within a depositional setting. In this study, our results were related to a standard classification (Perrin, 1974; Tate, 1987; Van der Veer, 2006), which synthesizes two following main types, as mineral sediments (≤ 15 -30% organic matter), and organic sediments (≥ 15 -30% organic matter). An overall assessment between-core variation showed that the total organic matter (TOM %) was the predominant sediment fraction in the samples gathered from Tătaru L., with values more than 50% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3). These values remained higher from the top of the cores and up to the bottom layers. That was somehow to be expected if we consider the environs of the Tătaru L., such as a confined area characterized by low hydrodynamics slightly perturbed by dynamic bottom currents and plenty of vegetation. Concurrently, we may appreciate that Tătaru L. is a sink for sediments mainly produced in the catchment (autochthone) by means of in-lake production (Fig. 4). On the other side, the level of TOM % concentration in the sediment-cores retrieved from Puiu L., showed higher values on the top of the cores which decrease as we descend in-depth layers. The average of the obtained values is less than 50% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3). Even Puiu L. is characterized by

the same similar water body stagnation environmental conditions, we should consider further detailed analyses related to the natural geochemical background of this depositional setting. However, overall, it can be appreciated that Puiu L. is a sink for sediments predominantly generated in the catchment (autochthone) as a result of the magnitude of the in-lake processes (Fig. 4). In terms of the TOM % percentage distribution in the sediment-cores collected from the Musura Bay, the average of the obtained values less than 50% were also noticed (Table 1, Fig. 2, Fig. 3). The obtained results within this study reflect a different environmental setting, thereby Musura Bay is a sink for sediments both originated in the catchment (autochthone) and in the bay (allochthone) (Fig. 4).

Total carbonates (CAR%). Carbonates in sediments can be derivable from organic origin (humus, plant remains, biogenic debris) and inorganic forms (calcite, aragonite) (Kennedy and Woods, 2013). The carbonate content of the sediment can be roughly regarded as indicators of paleoenvironmental and depositional biogeochemical conditions (Clayton and Degens, 1959; Zhao et al., 2016). In this study, our results were compared to informal classification (Emelyanov & Shimkus, 1986), presenting the following categories: non-carbonated sediments ($\text{CaCO}_3 \leq 10\%$), low calcareous sediments ($10\% < \text{CaCO}_3 \leq 30\%$) and calcareous sediments ($30\% < \text{CaCO}_3 \leq 50\%$). The inter-comparison amongst the investigated sampling sites showed an interesting variation along the vertical profile of core-sediments. For example, the range of variation for CAR % content is quite narrow in core-sediment samples retrieved from Tătaru L., with values less than 10% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3, Fig. 4). The relatively carbonate-rich sediments may be associated with the presence of the friable shell debris firmly attached by the sediment material that cannot be detached during sample preparation analytical stage.

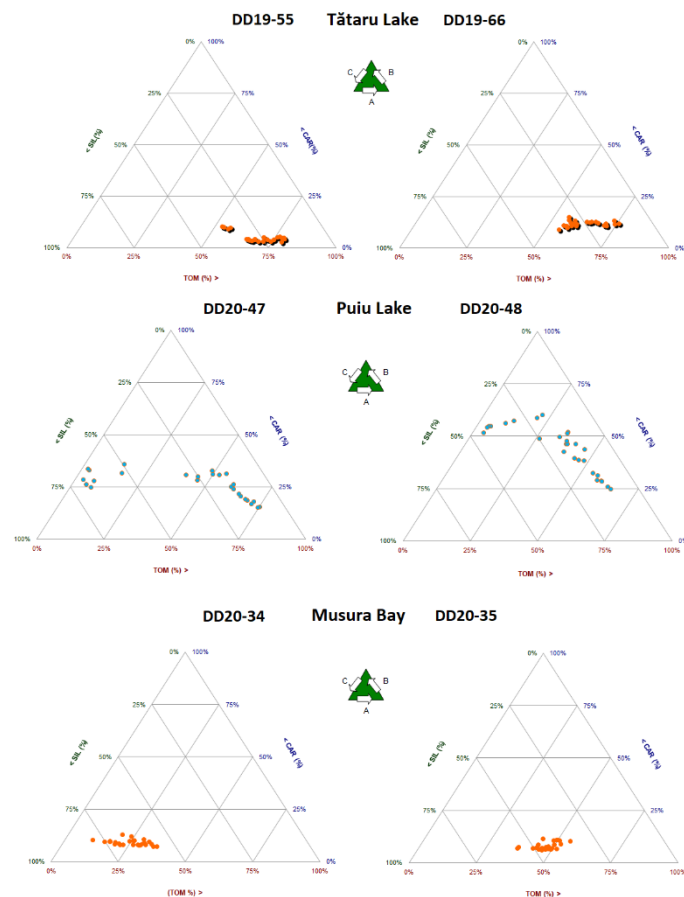


Figure 2. Ternary diagram indicating the percentage distribution (%) of investigated parameters

Instead, the level of CAR % concentration in the sediment-cores extracted from Puiu L. exhibited higher amounts, with values around $30\% < \text{CAR} \leq 50\%$, of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3, Fig. 4). In this case, besides the organic carbonate content, originated from shell debris and other carbonate residues, it can be taken into account the number of carbonates occurred as a result of the chemical precipitation or recrystallization within this depositional setting. The vertical distribution of the CAR % amounts within Musura Bay sediment-cores revealed a uniform distribution pattern across the depth profiles with values less than 15% of the total weight of the dry

residue (Table 1, Fig. 2, Fig. 3, Fig. 4). And in this case, similar to Tătaru L., the relatively higher carbonate values may be due to organic carbonate residues. Probably, the mixed environmental conditions with multiple environmental changes interact to modify the dynamics and development rate of aquatic organism species.

Siliciclastic fraction (SIL%). The siliciclastic components (mineral residues) represent an indicator that may be used to establish the origin of extrabasinally (allochthone) sedimentary particles (fluvial/aeolian input stream, volcanic and hydrothermal events etc.), or, to intrabasinal (autochthone) genesis (geological setting, erosion, wind, waves, and bottom currents). Tătaru L. is characterized by a relatively low siliciclastic content, with values less than 25% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3), that may be attributed to the long-term environmental changes in pathways and depositional circumstances of intermittent terrigenous material brought by the Danube River via canals and streams (Fig. 4). Rather similar, the siliciclastic content did not range significantly throughout the sediment-cores from Puiu L., having average values lower than 30% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3). In this situation, the lower values could be related to the same long-term environmental changes in pathways and depositional circumstances of allochthonous material. The sediment material accumulations were deposited within comparable depositional environments (confined area with low hydrodynamics) (Fig. 4). By contrast, the siliciclastic content determined throughout the sediment-cores from Musura Bay, displayed average values higher than 45% of the total weight of the dry residue (Table 1, Fig. 2, Fig. 3). In this context, where the siliciclastic content is highest, it seems quite clear that the sedimentary sequences belonging to the sediment-cores from Musura Bay were deposited within very different depositional environments. This fact may indicate that terrigenous sediment input is allocated to both allochthonous (Danube River alluvial input) and autochthonous sources (local sedimentary processes) (Fig. 4).

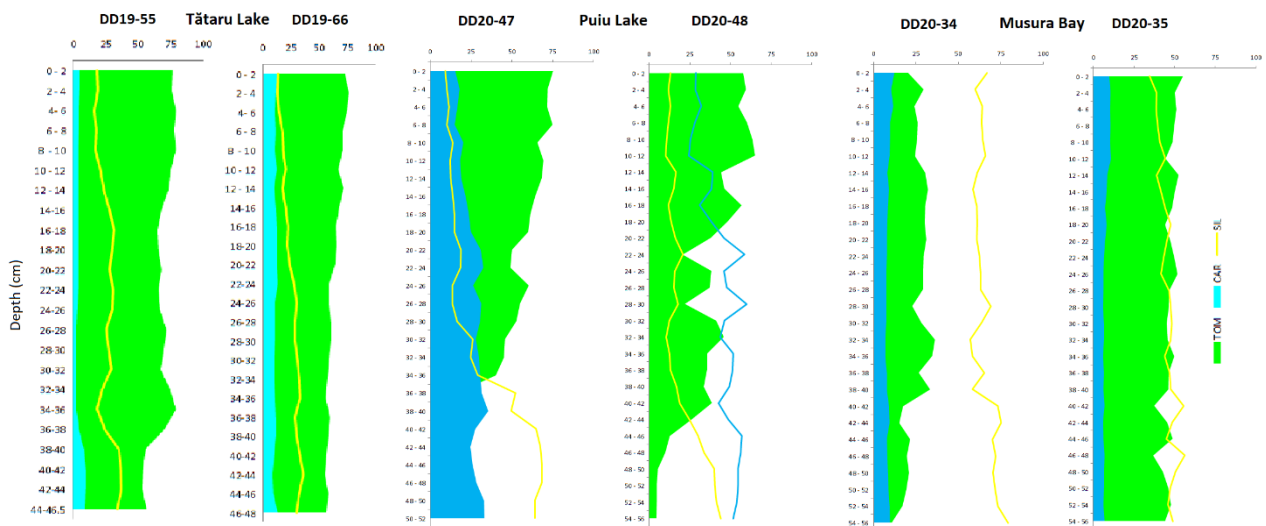


Figure 3. Vertical distribution of the investigated parameters in core-sediment samples

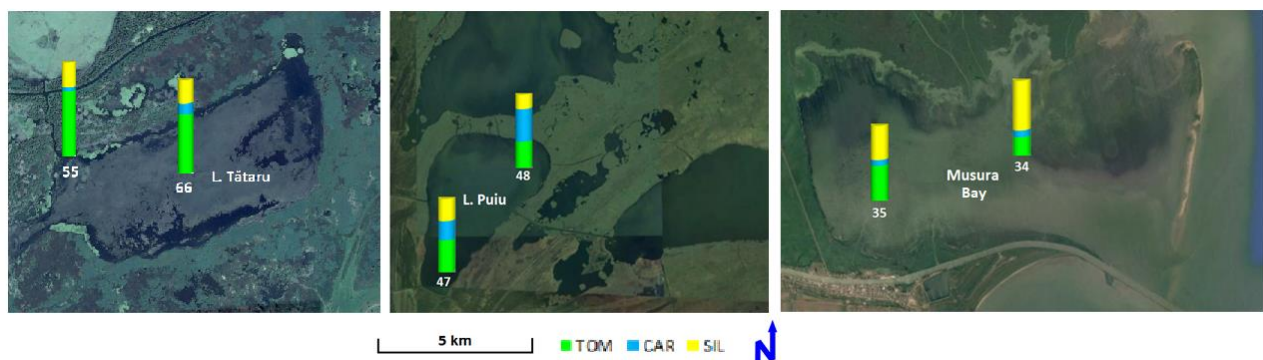


Figure 4. Vertical distribution of the environmental indicators (TOM%, CAR%, SIL%) in core sediments from Tătaru L., Puiu L. and Musura Bay

Conclusively, the overall results for core-sediment investigations demonstrated a variable mixed lithology, depending on the participating proportion of the main lithological components (TOM%, CAR%, SIL%). Thereby, the sedimentary deposits in Tătaru L. are characterized by a mixed lithology between high organic-rich, low carbonate-rich, and low siliciclastic-rich contents. Then, Puiu L. accumulations are ascertained by a mixed lithology between high organic-rich, high carbonate-rich, and low siliciclastic-rich contents. Finally, the depositions within Musura Bay are distinguished by a mixed lithology between low-moderate organic-rich, low carbonate-rich, and high siliciclastic-rich contents (Fig. 4).

4 CONCLUSIONS

This study aimed to assess the vertical variability of the main lithological components of core sediment samples gathered from different environs belonging to the DDBR area. The Danube Delta is a transition zone between upstream Danube River water and sediment input and its downstream receiver, namely the Black Sea. In this context, delta provides an outlet buffer zone, that protects several inland freshwater deltaic and lacustrine sedimentary systems from sediments delivered from the Danube River catchment.

This is the situation of Tătaru and Puiu lakes studied in this paper, whose morpho-sedimentary evolution is conditioned by the long-term environmental changes in pathways and depositional circumstances of allochthonous material. Tătaru and Puiu lakes are characterized by a typical deltaic lacustrine sedimentation which contains accumulations deposited in a shallow lake setting.

Reversing it, Musura Bay which is naturally located in a more dynamic area of intersection, between the mouth of the Danube River via Chilia and Sulina branches and the vicinity of the Black Sea, is characterized by different morpho-sedimentary evolutionary trends. Musura Bay is characterized by specific transitional depositional environment (erosion and deposition) which contains accumulations deposited in a shallow coastal lagoon setting. Here, the long-term contribution of both allochthonous (Danube River alluvial input) and autochthonous suppliers (local sedimentary processes) will lead to the path of natural transformation into a coastal lagoon area.

The results obtained in this paper are based on a preliminary approach to better understand the hydro-sedimentary processes that occurred within these depositional settings. Further detailed new information and complex investigations are necessary to decipher the active deltaic sedimentation mechanisms in fluvial-delta-sea environment.

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