# DETERMINATION OF TOTAL ORGANIC CARBON AND TOTAL NITROGEN FROM SOIL SEDIMENTS FROM HORIA LAKE, TULCEA

## Adina Popescu, Mirela Cretu, Angelica Docan

*Key words:* Horia lake, Tulcea county, Romania, sediments samples, total organic carbon (TOC), total nitrogen (TN), C/N ratio, statistical analysis

## **INTRODUCTION**

An important parameter for the environmental status estimation of aquatic ecosystem is the total organic carbon (TOC) and total nitrogen (TN) content in sediments (Pavlos Avramidisa et al., 2014), which represents an important reservoir for the global carbon cycle (Weston and Joye, 2005). Sediment can be viewed as a sink for nutrients, pollutants and metals in aquatic ecosystems due to the association of these compounds with particulate material (IAEA, 2003). Sediment is also considered a source for these compounds due to processes that release nutrients back into the water (Kelsey L. Berrier, 2015). The presence of TOC in aquatic ecosystems proves the occurrence of living organisms and decomposable materials (US-EPA 2002). According to the Seiter et al., 2004, the stored organic matter as organic carbon and nutrients offers a model of phytoplankton growth in an area. Total nitrogen (TN) plays an important role as a source of nutrients (Moss 2001; Maberly et al., 2002; Fütterer, 2000), decreasing with depth due to the remineralization of organic matter and non-biological oxidation (Stein, 1990; Wakeham, 2002).

At 2 km northwest of Horia village, a 150meter-long dike stops the waters of Taita in a reservoir lake (Lake Horia). The lake is limited to north-west and south by agricultural field, and the eastern limit is represented by the intercounty road 222 A. The lake has a surface around 230 ha.

The aim of this paper is to study was to determine the content of total organic carbon and total nitrogen content from the sediments of the Horia lake, from Tulcea county, Romania.

# MATERIALS AND METHODS

*Station location.* For this study the sediments samples were collected from six stations (established in the symmetry axle of the lake, Figure 1) using Marinescu grab (Photo 1). From each station were collected three samples. All the samples were collected in the autumn season (November 2016).



Figure 1. Study area location and sampling station distribution



Photo 1. Collection of sediment samples from the sampling stations

Methodology. A total number of 18 sediments samples were analysed for their TOC and TN content and all the samples were performed in duplicate. All the collected samples were pre-treated according to the ISO 11464 and 11465. Its principle is drying soil samples to constant mass at  $105^{\circ}$ C and using the difference in mass of an amount of soil before and after the drying procedure to calculate the dry matter and water contents on a mass basis. After that, a representative sub-sample has to be milled until it passes a 250 µm aperture sieve.

TOC content from sediments were measured using Primacs<sup>SLC</sup> equipment and TN content using the Primacs<sup>SNC</sup> Analyzer from Skalar Company. All the samples were made in the Nutrition Laboratory of Romanian Centre for Modelling Recirculating Aquaculture Systems (MoRAS), University Dunărea de Jos, Galați, Romania.

The principle for the TOC method. Total Carbon (TC) determination: a small quantity of sample (0.1 to 1 mg) is placed into a quartz crucible is cleared with oxygen to create a CO<sub>2</sub> free environment. Then the sample is vertically introduced into the combustion zone at temperature of 1050°C. In the presence of the catalyst cobalt oxide, all organically and inorganically bound carbon is oxidized or decomposed in the flow of pure oxygen into the gaseous carbon dioxide. The flow of oxygen transports the carbon dioxide to the IR detector and the carbon dioxide is measured at 4.2 µm by IR detector and recalculated to the total carbon content according to the calibration by the standards; Inorganic Carbon (IC) determination: the analysis is performed at low temperature (150°C). The sample is added in a test tube in which oxygen will be purged before analysis begin, in order to remove CO<sub>2</sub>. Then is added orthophosphoric acid to the sample to decompose the inorganically bound carbon to the gaseous carbon dioxide. The flow of oxygen purges the carbon dioxide from the liquid

into the IR detector to be measured again. The concentration of TOC = TC-IC.

The principle for the TN: The samples are weighed in a quartz crucible and placed in the autosampler. By a unique vertical "bottom-to-top" sample introduction system the samples are introduced in the high temperature combustion furnace. At 1100°C the Nitrogen is converted into NxOy in the presence of Oxygen. After the catalyst furnace, where the oxidation reaction takes place, the sample passes through a Peltier cooler to remove moisture formed by condensation. Then the sample gas is splitted and transferred, by the carrier gas Helium, to the reduction oven. In the reduction oven of 600°C NxOy is reduced, in the presence of a copper reductor, to N2. Then the N2 gas is measured by Thermal Conductivity Detection (TCD) and to remove interferences a background correction is performed by measuring the He gas. The signal from the detector is transferred to the equipment software for the calculation according to the calibration by the standards.

Data analysis. All the statistical analysis was made using SPSS 21 for Windows. Data were analysed using Anova test and if significant differences were found, a Duncan test was applied. Difference were found significant at  $\alpha \leq 0.05$ .

#### **RESULTS AND DISCUSSION**

In the Table 1 are presented the results of Total TOC, TN and Carbon/Nitrogen (C/N) ratio.

The TOC content averaging varied from 1.81% in station 2 to 2.51% in station 3, with significant differences (Anova,  $p \le 0.05$ ) between the six stations. In fact, Duncan analysis divided the values of TOC in three sets of data: the lowest values were registered in the Station 1 and Station 2, followed by the Station 4, while the highest values were recorded in the Stations 3, 5 and 6.

Parameter	Station 1	Station 2	Station 3	Station 4	Station 5	Station 6
*TOC (%)	1.91±0.03	1.82±0.04	2.51±0.18	2.06±0.02	2.36±0.09	2.41±0.05
*TN (%)	$0.44{\pm}0.04$	0.36±0.01	0.67±0.02	0.51±0.02	0.30±0.04	0.28±0.06
C/N	4.36±0.35	4.96±0.24	3.75±0.39	4.02±0.15	7.90±1.19	8.53±0.20

Table 1. The TOC and TN content in sediments from the six stations of Horia lake, Tulcea county

\*The values are expressed as mean and standard deviation for the three samples/station (each sample was made in duplicate)

Regarding the TN content significant differences (Anova,  $p \le 0.05$ ) were found between the six stations. The highest TN value was recorded, also, in the Station 3, followed by the Station 1 and 4, Station 2 and 5. The lowest TN content was recorded in the Station 6.

The C/N ratio represent an important factor for determining how easily bacteria are able to decompose an organic material. Usually, a C/N ratio of 5-8 indicate unaltered algal organic matter, while C/N ratios of 25-35 indicate fresh land-derived organic matter (Meyers, 1994).

In our study, the ratios of C/N for the sediment samples ranged between 3.75% in Station 3 and 8.53 % in Station 6, without significant differences ( $p \ge 0.05$ ) between the Stations 1,2,3 and 4, but with significant differences in comparison with the Station 5 and 6 ( $p \le 0.05$ ). The obtained values of C/N, indicate a source of organic matter sourced from phytoplankton (lake sourced algae). In fact, our previous study (Popescu and Docan 2017) has demonstrated the presence in relatively large abundances of the dinoflagellates, due to the fact that the slope of the ground allows for the accumulation of water from the adjacent lakes, with suspension contributions, fertilizers and herbicides used for the adjacent corn crop.

#### CONCLUSIONS

In conclusion, the result of our study shows significant differences in the content of TOC, TN and C/N ratio between the six stations. The higher TOC and TN values from the Station 3 may be due to the increase of corn culture from the adjacent lake area. Also, the content of TOC and TN from sediment may be influenced by many factors such as the physical, biological and chemical processes.

#### ABSTRACT

Carbon and Nitrogen have an important role in maintaining trophic levels in lake ecosystems and can be used us pollution indices, soil quality and productivity indicators. In this context, the aim of this research was to determine the total organic carbon (TOC), total nitrogen (TN) and the carbon nitrogen ratio (C/N) from the sediment samples collected from lake Horia, Tulcea county, Romania. The lake is located in the area of Horia, being limited to the north, west and south by the agricultural field, and the eastern limit is represented by the intercounty road 222 A. The surface of the lake is 230 ha, and the depth varies between 0.5 - 1 m in summer time in the foot rope zone: the maximum depth is 3.962 m in the dam area. The incline of the ground permits the accumulation of the waters from the versants adjacent to the lake, with suspension contributions, fertilizers and herbicides used for the adjacent corn crop. The samples were collected in the autumn season, from six stations of the lake and the sediment TOC and TN contents were measured using  $Primacs^{SLC}$  and  $Primacs^{SNC}$  Analyzer, from the laboratory of Romanian Center for Modelling Recirculating Aquaculture Systems, University Dunărea de Jos, Galați. The higher values of TOC and TN were registered at station 3 (TOC =  $2.53 \pm$ 0.18%; TN=  $0.67 \pm 0.02\%$ ). The C/N ratio registered values between (3 - 9) indicating a source of protein rich, lignin poor organic matter sourced from phytoplankton (lake sourced algae).

#### ACKNOWLEDGEMENTS

This work was supported by a grant of the Romanian National Authority for Scientific Research and Innovation, CNCS/CCCDI – UEFISCDI, project number PN-III-P2-2.1-BG-2016-0417, within PNCDI III".

#### REFERENCES

- FÜTTERER, D.K., 2000 The solic phase of marine sediments, in Schulz, H.D., Zabel, M., (eds.), Marine Geochemistry: Berlin, Springer, 1-26;
- BERRIER, KELSEY L., 2015 -"Analysis of Nitrogen and Phosphorus Nutrients in Lake Sediment". Senior Honors Projects, 2010current. 70. http://commons.lib.jmu.edu/honors201019/70Ma berly, S.C., King, L., Gibson, C.E., May, L., Jones, R.I., Dent, M.M. & Jordan, C. (2003): Linking nutrient limitation and water chemistry in upland lakes to catchment characteristics. Hydrobiologia 506: 83-91;
- MEYERS, P.A., 1994 Preservation of elemental and isotopic source identification of sedimentary organic matter. Chemical Geology, 114, 289-302;

- MOSS, B., 2001 The Broads. The people's wetland. The New Naturalist. Harper Collins Publishers, London;
- PAVLOS AVRAMIDISA, KONSTANTINOS NIKOLAOU AND VLASOUL BEKIARI, 2014
  Total Organic Carbon and Total Nitrogen in Sediments and Soils: A Comparison of the Wet Oxidation – Titration Method with the Combustion-Infrared Method, Agriculture and Agricultural Science Procedia 4, 425 – 430;
- POPESCU ADINA, DOCAN ANGELICA, 2017 - Preliminary aspects concerning structure plankton in the lake horia – Tulcea county, Current Trends in Natural Sciences, Vol. 6, Issue 12, pp. 170-173;
- SEITER, K., HENSEN, C., SCHRÖTER J., ZABEL, M., 2004 - Organic carbon content in surface sediments defining regional provinces: Deep-Sea Research part I. 51. 2001–2026;
- STEIN, R., 1990 Organic carbon content/sedimentation rate relationship and its paleoenviromental significance for marine sediments: GeoMarine Letters, 10, 37–44;
- WAKEHAM, S., 2002 Diagenesis of organic matter at the water-sediment interface, in Gianguzza, A., Pelizzetti, E., Sammartano, S.

(eds.), Chemistry of marine water and sediments: Berlin, Springer 147-164;

- WESTON, N.B., JOYE, S.B., 2005 -Temperature-driven decoupling of key phases of organic matter degradation in marine sediments: PNAS, 102, 17036–17040;
- \*\*\* U.S. Environmental Protection Agency (EPA), (2002), Mid-Atlantic Integrated Assessment (MAIA) Estuaries 1997-98: Summary Report, EPA/620/R- 02/003,115 pp.
- \*\*\* International Atomic Energy Agency (IAEA). (2003), Collection and preparation of bottom sediment samples for analysis of radionuclides and trace elements. p. 1-130.

#### **AUTHORS' ADDRESS**

POPESCU ADINA, CRETU MIRELA, DOCAN ANGELICA - Department of Food Science, Food Engineering, Biotechnology and Aquaculture, Faculty of Food Science and Engineering, University of "Dunarea de Jos" Galati,

e-mail: adina.popescu@ugal.ro; mirela.cretu@ugal.ro; adocan@ugal.ro.