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Presence of organic compounds in river surface water in a neotropical environment of south Brazil

Presença de compostos orgânicos em água superficial de rio em ambiente neotropical no sul do Brasil

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ABSTRACT

Many industrially synthesized human - therapeutic agents, agrochemicals, and additives used by industries are heterocyclic compounds. Many of these contribute to increased environmental contamination in localized and diffuse sources of water bodies, reflecting soil quality, communities, and human health. This concern led us to develop this research to evaluate the presence of organic compounds in the surface waters of the Pirapó river basin, Paraná, Brazil, influenced by the soybean and safflower corn crops that are cultivated in adjacent areas around the river. The water samples were collected from October 2017 to January 2019, at three collection points. Organic compounds were analyzed by GC-MS, after the solid phase extraction (SPE). Two organic compounds were analyzed: azetidine and sarcosine that are used in the pharmaceutical industry. Organic compounds existing in drugs and/or contaminants that were observed in this study reveal the importance of further investigation into their origins and the consequences for the health of the biota and the population.

Keywords: Pharmaceutical compounds; GC-MS method; Environmental monitoring.

RESUMO

Muitos agentes terapêuticos humanos sintetizados industrialmente, agroquímicos e aditivos, são compostos heterocíclicos. Muitos destes contribuem para o aumento da contaminação ambiental em fontes pontuais e difusas dos corpos de água, o que reflete na qualidade do solo, das comunidades e na saúde humana. Com isso, este estudo teve objetivo de avaliar a presença de compostos orgânicos nas águas superficiais da bacia do rio Pirapó, influenciados pelas culturas de soja e milho safrinha que são cultivadas nas áreas adjacentes em torno do rio. As amostras de água foram coletadas no período de outubro de 2017 a janeiro de 2019, em três pontos de coleta. Os compostos orgânicos foram analisados por CG-EM, após a extração em fase sólida (SPE). Dois compostos orgânicos foram detectados: a azetidina e a sarcosina, que são utilizados na indústria farmacêutica. Os compostos orgânicos existentes em medicamentos e/ou contaminantes observados neste estudo revelam a importância de investigações mais aprofundadas sobre suas origens e as consequências para a saúde da biota e da população.

Palavras-chave: Compostos farmacêuticos; Método CG/EM; Monitoramento ambiental.

INTRODUCTION

Water, both a source of life and a limited resource, has been degraded because of the increase in urban population and environmental pollution. Thousands of people around the world, including in Brazil, typically have some kind of disease because of the poor quality of water from the impacts that water bodies have been suffering (Mendes et al., 2017).

Water contamination can be due to numerous causes, among them: deforestation, lack of soil conservation, silting, crops and roads, great urban expansion with the use and disorderly occupation of the soil, absence of adequate environmental planning, discharge of domestic and industrial effluents, solid waste from hospitals, and from domestic and agricultural origin, as well as the introduction of discharges of agrochemical residues and their packaging, and compounds such as those used by the pharmaceutical or chemical industry (Geissen et al., 2015; Carvalho et al., 2015, 2022; Fernandes et al., 2017).

The presence of organic contaminants in an aquatic environment is a growing concern due to their potential effects on human health and the ecosystem (Hansen et al., 2016; Tomaz et al., 2023). They can be divided into several categories, including hydrocarbons, volatile organic compounds (VOCs), persistent organic compounds (POCs), organic waste, pharmaceuticals and/or personal chemicals, and miscellaneous synthetic organic compounds (Liang et al., 2021; McBeath et al., 2021; Nas et al., 2021; Tomaz et al., 2023).

To address these challenges, governments and environmental organizations around the world develop regulations and guidelines to monitor, control, and reduce the emission and presence of organic contaminants in the environment (Tomaz et al., 2023). In addition, sustainable management practices, advanced treatment technologies, public awareness, and analytical methodologies for identifying and quantifying these contaminants play a key role in minimizing the negative impacts by these contaminants (Liang et al., 2021; McBeath et al., 2021; Nas et al., 2021).

The proper management, regulation of production, and elimination combined with the development of analytical methodologies capable of identifying and quantifying the presence of these contaminants in water resources are essential to mitigate their negative impacts. In this way, the objective of the present study was to determine the presence of organic contaminants in the surface water of the Pirapó River, a river in the Neotropical region of Southern Brazil.

MATERIALS AND METHODS

Study area

The Pirapó river basin is located in the northern Paraná state mesoregion, within the polygon delimited by the latitudes 22°30' and 23°30' South and longitudes 51°15' and 52°15' West (Schneider et al., 2011). Its sources are located in the municipality of Apucarana at an altitude of 1,000 m, extending for 168 km in a north-northwest direction, and its mouth in the Paranapanema River. This basin is the main source of supply for 35 municipalities in the state of Paraná, including Apucarana and the city of Maringá. This is an important regional hub, from economic, industrial, agricultural, services, leisure, and social points of view (Instituto Água e Terra, 2017).

Three sampling points (P1, P2 and P3) were selected. P1 is close to the source (23°27'8.57"S - 51°33'25.10"W), the second (P2) upstream of the water intake for public supply of Maringá (23°18'43.92"S-51°50'52.55"W), and the third (P3) near the meeting with the Bandeirantes River (23°11'31.37"S-51°57'57.25"W), all on the Pirapó River (Figure 1).

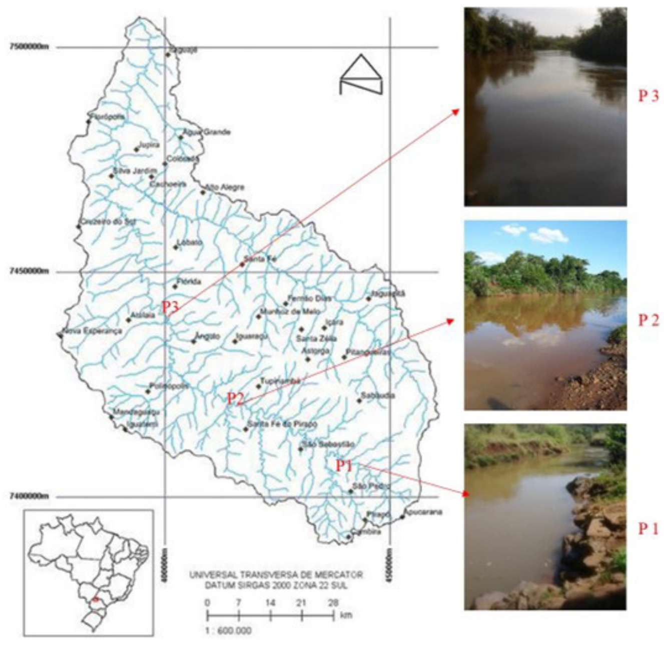

Figure 1. Drainage basin in the Pirapó river, Paraná State, Brazil. P = Sampling point.
Source: Oliveira et al. (2012) – adapted by author.

Sampling

To verify the water quality, on-site analyses were carried out using a portable multiparameter probe (HORIBA U50). Electrical conductivity (mS/cm), dissolved oxygen (mg/L), pH, water temperature (ºC), turbidity (NTU) and total dissolved solids (mg/L) were measured.

For the analysis of the presence of organic compounds, 3 liters of water were collected per collection point, at a depth of 10 cm, against the current, in sterile 1-liter vials, between October 2017 and January 2019, totaling four collections per sampling site, corresponding to October 2017, April 2018, October 2018 and January 2019. After collection, the water samples were frozen and stored for further extraction and chromatographic analysis.

Processing of samples

The analysis of organic compounds in the collected water samples (1L) was performed by pre-concentration and purification by Solid Phase Extraction (SPE), with an ElutNexus Bond cartridge as adsorbent and a SupelcoVisiprep SPE vacuum system.

The extraction of the water sample in the SPE cartridge was performed with a flow of 10 mL/min, after adsorption the cartridge was washed with 10 mL of deionized water, drying the cartridge under vacuum for 20 min to eliminate the traces of water and its elution was performed with 3 mL of ethyl acetate, followed by elution with 3 mL of dichloromethane. The final aliquots were combined, concentrated by flow of N2 to dryness, and resuspended to a 2 mL vial with dichloromethane to then be submitted to analysis by GC-MS.

The concentration factor for the water samples was 500, as established by the method applied at the Paraná Institute of Technology – TECPAR (American Public Health Association, 2005). The analyses in the GC-MS were performed in a gas chromatograph (model Agilent 7890B) coupled to a mass spectrometer (model Agilent 5977A MSD), equipped with HP-5MS UI Agilent column with 5% phenyl methyl siloxane phase (30.0 m x 250 µmd. i. x 0.25 µm film thickness) and one with automatic injector (CTC PAL Control).

For the proper separation of the analytes in the GC-MS system, the following optimized furnace temperature programming was used: initial temperature of 92°C maintained for 2.5 min, then ramp from 15°C min-1 to 175°C maintained for 13 min, and ramp from 20°C min-1 to 280 °C and maintained for 15 min. The other conditions of the analysis were: injection volume of 1.0 µL, the flow of the carrier gas (He, purity 99.99999 1.0 mL min-1, ionization by the electronic impact of 70 eV and temperatures of the ionization source of 230°C, the quadrupole of 150°C, the transfer line of 280°C and the injector of 250°C. Data acquisition was performed by the MassHunter software and qualitative analysis of the mass spectra by the NIST 11 library. The limits of quantification established in the analysis methodology were: 0.0016 µg L^{-1} (azetidine) and 0.0028 µg L^{-1} (sarcosine).

RESULTS AND DISCUSSION

The results of the analysis of the parameters involving water quality, based on CONAMA Resolution No. 357 of 2005 (Brasil, 2005), were compared with the limits established for class II water bodies, since P2 is located upstream of the water bodies for the municipality's supply. The water analysis showed that the temperature was higher along the course of the river and in the last two collection periods. According to INMET (Instituto Nacional de Meteorologia, 2019), for the year 2019, the month of January presented a temperature variation between 22.4 ºC and 32.5 ºC (Table 1).

Comparing the results with CONAMA resolution 357/2005, which controls several parameters in its resolution, the turbidity was above what is allowed (maximum value up to 100 UNT) for the classification of freshwater class II. The river has sandy soil, collaborating with the transport of particles, and it thus promotes the increase in the number of suspended solids in the water, as observed in this study, since the dissolved solids did not undergo high alteration. In general, the water quality parameters are within the normal range.

In their study, Harfuch et al. (2019) analyzed 14 points of the same stretch of coverage, including P1, P2, and P3. The authors observed that the environmental quality indexes, in general, were considered reasonable.

Table 1. Results of the physical and chemical analysis of the water at the sampling points in the Pirapó River, during the collection period (October 2017 to January 2019).

Sample point	Date	T (ºC)	CE (mS/cm)	OD (mg/L)	pH	Turbidity (NTU)	STD (g/L)	PIM (m^3)
P1	Oct/17	21	10.00	9.01	7.39	21.43	60	311.9
P2	Oct/17	20.86	12.00	7.60	7.61	12.60	80	
P3	Oct/17	22.97	13.40	7.31	7.18	18.00	87	
P1	Apr/18	21.32	10.70	7.80	7.23	47.30	69	26.7
P2	Apr/18	21.89	9.70	7.68	6.18	78.70	63	
P3	Apr/18	24.87	12.60	7.94	7.11	39.70	82	
P1	Oct/18	24.03	9.80	7.50	7.70	99.50	64	319.3
P2	Oct/18	23.62	12.40	8.03	7.25	40.30	89	
P3	Oct/18	26.21	13.90	8.92	7.38	89.70	90	
P1	Jan/19	24.23	12.00	35.83	7.79	24.30	78	201.4
P2	Jan/19	28.61	10.90	29.60	7.82	252.00	71	
P3	Jan/19	29.54	13.70	9.45	7.66	221.00	89	

Source: The authors. Legend: T = Temperature; OD = Dissolved Oxygen; STD = Total Dissolved Solids; CE = Electrical Conductivity; PIM = The mean monthly rainfall for each collection period - INMET (Instituto Nacional de Meteorologia, 2019).

Monitoring of organic contaminants in bodies of water has developed due to the population increase of urban centers over decades, coupled with increasing concern for these water bodies (Lima et al., 2017). The presence of these compounds may be associated with remediation and release in these bodies of water (Borrely et al., 2012; Fekadu et al., 2019). Despite their importance, there are still few studies addressing these compounds.

In this study, the presence of two organic compounds in the surface water of the Pirapó River was analysed: Azetidine and Sarcosine (Table 2). These compounds are not described in the Legislation by Resolution 430 of CONAMA (Brasil, 2011), which provides for the classification of water bodies and establishes the conditions and standards for the release of effluents (Brasil, 2011).

Azetidine is a heterocyclic organic compound, derived from 2-azetidine, and arouses much interest due to its usefulness and biological action; it can be used in medicines, especially those with anticoagulant properties and inhibitory action of cholesterol absorption (Boto et al., 2012). In addition, it is a compound of degradation of drugs, and it is used as an intermediate in the pharmaceutical industry or the chemical industry for various applications, including in the production of agrochemicals (Ganelin, 2013).

Its composition can be used as a synthetic basis for the creation of other compounds. The study conducted by Carrillo et al. (2021) demonstrated that this compound can also be used as a pharmacological alternative for the treatment of heart failure (Yoda et al., 2011). It can still be used as an antibiotic, since azetidine derivatives are a class of antibiotics widely used in the fight against bacterial infections (Avilés Zepeda, 2008), in addition to its use as an analgesic (Pereira et al., 2022).

Because of its prominence as a pharmacological alternative, azetidine is one of the compounds that are found in surface waters. In this study, azetidine was observed only at P2, which is situated upstream of the water abstraction for public supply. The presence of this substance may be related to the intense urbanization process of the 37 municipalities that are supplied totally or partially by the waters of this basin.

Azetidine, along with other drugs and derived substances in the environment, can go through different processes, making them biologically active. Chemical synthesis may then be one of the strategies used in the development of new molecules with biological activity (Luna Herrera et al., 2017).

The presence of drugs and organic compounds can increase considerably in sewage treatment plants, due to the disposal of numerous effluents such as fluoxetine, diclofenac, paracetamol, and others (Wilkinson et al., 2017; Sehonova et al., 2019; Dick et al., 2020), and degraded compounds from the use of antibiotics and anticancer can also be found.

In turn, sarcosine was found at all collection points and in all periods of this study. Sarcosine is an amino acid produced by the human body that is essential to the synthesis and composition of muscle, and is the main metabolic source of glutathione, creatine and serine. This amino acid is present in urine, muscles and other structures (Sreekumar et al., 2009).

Sarcosine can also be used as a biological marker for prostate cancer (PCa) because it is considered an important intermediate metabolite of invasion and aggressiveness of cancer cells, being useful as a potential marker of the disease (Sreekumar et al., 2009; Khan et al., 2013; Araújo, 2015). The use of sarcosine as a marker of early stages of prostate cancer has been little discussed, but in its application as tumor marker new analytical methods are being developed with low cost in its determination in urine, tissue and blood plasma samples (Cernei et al., 2013).

Wang et al. (2016) demonstrated the relationship between glyphosate and sarcosine. This author observed two routes of glyphosate degradation, and in one of them, sarcosine is formed by the action of the bacterium *Enterobacter aeroneges*, while in the other route, the sediment itself plays a fundamental role in the microbial degradation of glyphosate by the sarcosine pathway in water (Figure 2). Thus, sarcosine can be found both by degradation in the environment and by biological processes in humans.

However, in this study, it was not possible to observe the presence of glyphosate, since the methodology used does not detect this compound. Even so, the presence of sarcosine may be an indicator of the presence of glyphosate in the environment.

The effect and concentration of these compounds found in surface water may be related to the increase in urbanization, as well as the presence/absence of basic sanitation. In addition, environmental and climatic factors can also influence the accumulation of these compounds by organisms, promoting the process of bioaccumulation and/or biomagnification of these contaminants (Flaherty & Dodson, 2005; Ginebrada et al., 2010; Canela et al., 2014; Wilkinson et al., 2017).

Table 2. Compounds identified in chromatographic analyses, in the period between October 2017 and January 2019, along the Pirapó River.

Sampling points	Date Month/Year	Organic compounds	
		Azetidine (μg L^{-1})	Sarcosine (μg L^{-1})
P1	Oct/17	-	0.0091
P2	Oct/17	0.0073	0.0099
P3	Oct/17	-	0.0095
P1	Apr/18	-	0.0197
P2	Apr/18	0.0175	0.0205
P3	Apr/18	-	0.0189
P1	Oct/18	-	0.0121
P2	Oct/18	0.0098	0.0116
P3	Oct/18	-	0.0126
P1	Jan/19	-	0.0172
P2	Jan/19	0.0150	0.0163
P3	Jan/19	-	0.0181

Source: The authors.

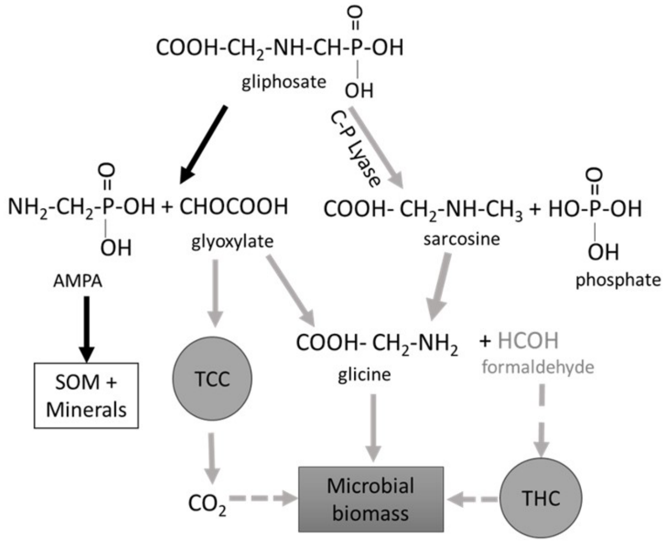

Figure 2. Part of the microbial degradation pathways of glyphosate via production of the metabolites aminomethylphosphonic acid (AMPA) and sarcosine. Gray arrows = biogenic residual formation; black arrows = xenobiotic formation; THC (tetracarboxylic cycle); TCC (tricarboxylic acid cycle); SOM (sedimentary organic matter).
Source: adapted from Wang et al. (2016).

Organic contaminants, or those resulting from use by urban or agricultural occupation in surface or deep-water bodies, represent a danger to both human and environmental health (Ritter et al., 2002). The lack of legislation for pollutants of pharmacological use, as well as its limitations, is an aggravating factor for the monitoring of surface waters. Therefore, to obtain a single health or one health, it is necessary to know and monitor water resources, organisms, and the population.

CONCLUSION

Organic compounds existing in drugs and/or contaminants that were observed in this study reveal the importance of further investigation into their origins and the consequences for the health of the biota and the population supplied by this resource. In addition, future studies regarding agrochemicals and their effect on biota will also serve to confirm the presence of glyphosate and its influence on the presence of sarcosine.

This study, therefore, constitutes an important tool for the management of water resources and public policies regarding the quality of water and quality public supply, in addition to being an important tool for further studies on public health.

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