



Occurrence of cocaine and benzoylecgonine in drinking and source water in the São Paulo State region, Brazil



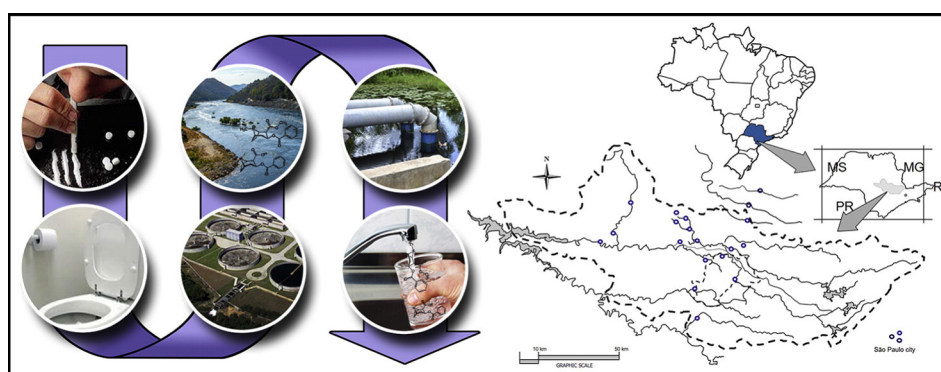
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HIGHLIGHTS

- Cocaine was investigated in natural waters from important watersheds in Brazil.
- High use of cocaine and poor sanitation affect the water quality in Brazil.
- COC and BE levels found are one of the highest found in urban surface waters.
- Cocaine levels detected in rivers in the São Paulo State may pose some risk to aquatic species.
- The presence in tap water indicates human indirect exposure to multiple compounds.

GRAPHICAL ABSTRACT



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ABSTRACT

The occurrence of illicit drugs in natural waters (surface, source and drinking water) is of interest due to the poor sanitation coverage and the high consumption of drugs of abuse in Brazil. In addition, little is known about the effects of these compounds on aquatic organisms and human health. This work investigates the occurrence of cocaine (COC) and its major metabolite, benzoylecgonine (BE), in surface and drinking water collected in rivers from a populated geographic area in Brazil. Surface water samples were collected in 22 locations from 16 different rivers and one dam from São Paulo State, whereas drinking water samples were collected in 5 locations. Samples were collected during the dry and wet season. Among the 34 surface water samples analyzed, BE was detected above the LOD in 94%, while COC in 85%. BE concentrations ranged from 10 ng L⁻¹ to 1019 ng L⁻¹ and COC concentrations from 6 ng L⁻¹ to 62 ng L⁻¹. In the drinking water samples analyzed, BE and COC were found in 100% of the samples analyzed. For BE, concentrations were found in the range from 10 ng L⁻¹ to 652 ng L⁻¹, and COC was quantified in concentrations between 6 and 22 ng L⁻¹. These concentrations are one of the highest found in urban surface waters and may pose some risk to aquatic species. However, no human health risk was identified using the Hazard Quotient. BE is proposed as a reliable indicator of sewage contamination in both source and drinking water.

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1. Introduction

Illicit drug abuse causes effects on health, social life and world economy. In the last years, the increase in consumption and consequently the detection in many matrices has motivated the investigation of

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these “emerging contaminant” in the environment (Zuccato et al., 2008).

Many illicit drugs are continuously released into the aquatic environment due to their incomplete removal in the wastewater treatment plants (WWTP). Once consumed, these substances are partially metabolized in the human organism and reach the wastewater network as a complex mixture of parental compounds and metabolites, where they can be detected from ng L^{-1} to $\mu\text{g L}^{-1}$ levels (Berset et al., 2010; Maldaner et al., 2012; Thomas et al., 2012). However, as the majority of WWTP have been designed to remove organic matter, many specific compounds like hormones, pesticides and illicit drugs, which are not included in the current local legislation, could persist and reach the receiving surface waters (Zuccato et al., 2008; Postigo et al., 2010). In countries with poor sanitation coverage, such as Brazil, high loads of raw and poorly treated sewage are discarded directly into water bodies, resulting in a severe contamination scenario in these watersheds (Sodré et al., 2010; Montagner and Jardim, 2011; Martins et al., 2014; Carreira et al., 2015; Frena et al., 2016).

The frequency and the levels that illicit drugs are detected in the environment depend on the local prevalence, seasonality, efficiency and coverage of sanitation infrastructure. The literature shows illicit drugs concentrations at ng L^{-1} in surface waters, but even at such low levels, these substances can affect the ecosystem and reach the drinking water supply (Berset et al., 2010; Binelli et al., 2013; van Der Aa et al., 2013; Mendoza et al., 2014). The removal efficiency of illicit drugs depends on the water treatment process used, which means that it is necessary to monitor the presence of these substances also in drinking waters to protect human health. However, considering the impossibility to monitor all emerging contaminants or every type of illicit drugs and metabolites that may be found in surface and drinking waters, selected target substances that might work as an indicator of contamination from sewage is necessary and desired for a rapid and less costly screening of the water quality.

According to the World Drug Report of the United Nations Office on Drugs and Crime (UNODC, 2016), about 246 million people used some illicit drug in 2013, representing an increase of 3 million compared to the previous year (UNODC, 2015). Cocaine (COC) is one of the most used illicit drugs in the world. While the use of opioids is a serious problem in Europe, the prevalence of COC use in South America is the highest in the world, particularly in Brazil, which has been facing serious problems with COC and crack, representing the second highest market in the world.

In Brazil, around 59% of the sewage produced is not treated and dumped “in nature”. Consequently, the contamination of source waters occurs, compromising the quality of drinking water concerning the presence of illegal substances and its major metabolites, which are only partially removed in drinking water treatment plants (DWTP) (SNSA, 2016; Sodré et al., 2010; Montagner and Jardim, 2011). This contamination may pose a risk to humans, especially considering that 17% of Brazilian cities are not fully served by the water distribution network (SNSA, 2016).

Few studies investigated the presence of these substances in the drinking water distribution network around the world. In Madrid region, COC was quantified (1.6 ng L^{-1}) in only one out of the six sampling points (Mendoza et al., 2014). COC and other illicit drugs were investigated in cities around Europe (Austria, France, Germany, Switzerland, United Kingdom), Latin America (Panama, Brazil, Colombia, Argentina, Uruguay) and in Japan by Boleda et al. (2011). COC was detected in 66% (0.2 to 2.3 ng L^{-1}) and its major metabolite, benzoylecgonine (BE), in 34% (0.2 to 3.1 ng L^{-1}) of the samples analyzed in Spain. In drinking water samples from the other countries, these compounds were detected at sub ng L^{-1} levels. The highest mean values of COC and BE were detected in tap water samples coming from South America. For BE, the mean values found were: 0.2 ng L^{-1} in Europe, $<\text{LOQ}$ in Japan, 0.4 ng L^{-1} in Spain and 4.5 ng L^{-1} in Latin America.

There are few studies evaluating the presence of drugs of abuse in both Brazilian surface and drinking water, considering their probable adverse effects for aquatic life and human health (Thomas et al., 2014; Boleda et al., 2011; Locatelli, 2011). The present study investigated COC and BE occurrence in surface and drinking water in different areas of the São Paulo State. Also, COC and BE levels were evaluated according to regional and seasonal variability, exploring the potential use of BE as an indicator of wastewater contamination in natural waters. To the best of our knowledge, this is the first monitoring study of both substances in surface and drinking waters, covering the most populated geographic area in some major rivers in the São Paulo State, Brazil.

2. Material and methods

2.1. Chemicals

COC and BE analytical reference standards were purchased from Cerilliant Corporation (Round Rock, TX, USA), in liquid form with purity higher than 98%, at concentrations of 0.1 mg mL^{-1} or 1.0 mg mL^{-1} in methanol (MeOH) or acetonitrile (ACN) (stock solution). All stock solutions were stored in the dark at $-4 \text{ }^\circ\text{C}$. Work standard solutions containing both target compounds at 10 and $1 \mu\text{g L}^{-1}$ were prepared in ACN/water ($50:50 \text{ v v}^{-1}$). Cerilliant Corporation BE-d3 was used as internal standard (IS), and it was prepared separately at $10 \mu\text{g L}^{-1}$ following the same procedure. Both solvents (99% purity MeOH and ACN) were purchased from Mallinckrodt Baker, Inc. (Phillipsburg, NJ - USA). Formic acid (98% purity) was acquired from Sigma-Aldrich Co., LLC (St Louis, MO - USA). Ultrapure water (Resistivity of $18 \text{ M}\Omega \text{ cm}$) was prepared using a Millipore Milli-Q Plus water purification system (Millipore Corporation - Burlington, MA, USA).

2.2. Sampling

The investigated area included some of the main rivers in the state of São Paulo, and an important dam, located in São Paulo city. These waterbodies together supply over 10 million people. Surface water samples were collected in 16 rivers from São Paulo State from important watersheds, which supply approximately 6.3 million people (Fig. 1). Besides, three sampling points (GD1, GD2, and GD3) were selected in the Guarapiranga dam (São Paulo city), an important source of drinking water for nearly 5.6 million people in the neighborhood (SABESP, 2014). In some cases, surface water samples were collected in more than one point along the river flow, reaching 22 sampling points in total. Some sampling was performed during dry season (between February and March 2014) and at the beginning of the rainy season (between September and October 2014), and 34 surface water samples were analyzed in total. Drinking water samples were collected during the same period in five cities supplied by some of the selected rivers, reaching eight drinking water samples.

2.3. Sample treatment and analyte extraction

Surface and drinking water samples were collected as grab sample in amber flasks (1 L) previously rinsed with ultrapure water, followed by the sample. Once collected, and during shipment, the samples were kept refrigerated. In the laboratory, the samples were vacuum filtered using $1.2 \mu\text{m}$ pore-size glass microfiber filters (Schleicher&Schuell - Dassel, LS, Germany) followed by $0.45 \mu\text{m}$ pore size cellulose acetate membranes (Sartorius - Göttingen, LS, Germany). Solid phase extraction (SPE) was performed with Oasis HLB Cartridges.

The SPE method was modified from the previous study to investigate COC and BE in wastewater (Maldaner et al., 2012). Commercial HLB Oasis® cartridges (6 mL, 500 mg) were obtained from Waters Corporation (Milford, MA, USA).

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