

## Article

# Monitoring and Assessment of the Quality of Chlorinated Water in Schools

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**Abstract:** All water destined for human consumption must be subjected to disinfection processes via chlorination with the aim of eliminating the risk of the transmission of waterborne diseases. However, the treatment of water using this method leads to the formation of trihalomethanes (THMs), which are toxic compounds that may be ingested, inhaled, or absorbed by the skin, increasing the risk of carcinogenic and mutagenic processes. High trihalomethane concentrations in water may be directly related to physicochemical properties, such as temperature, pH, hardness, and potentially toxic metal concentrations. In this work, physicochemical characterization was performed and water quality was assessed with regard to the presence of trihalomethanes in 17 samples collected from points before and after storage in reservoirs. Atomic absorption spectroscopy to determine potentially toxic metals and the measurement of physicochemical properties demonstrated that all results were in accordance with the standards established by Brazilian legislation. Gas chromatography–mass spectrometry was used to determine levels of trihalomethanes in the water samples (trichloromethane, bromodichloromethane, chlorodibromomethane, and bromoform), which were also in accordance with the limits established by Brazilian legislation. Hierarchical clustering on principal components confirmed changes in the water quality depending on location. This work constitutes a paradigm for future studies on the monitoring of toxic organic compounds in water to avoid health problems in humans and animals.

**Keywords:** water; trihalomethane; potentially toxic metal; schools



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## 1. Introduction

The supply of water of adequate quality is a constant concern of environmental agencies and public health officials [1]. Besides microbiological control, the assessment of water quality includes the identification of potentially toxic chemicals, such as inorganic compounds, metals from industrial waste, and disinfection byproducts (DBPs) [2]. For microbiological control, waters destined for human consumption must be subjected to treatment processes, with emphasis on disinfection for the inactivation of pathogenic microorganisms [1]. The purpose of such procedures is to prevent illnesses, as conditions such as cholera, typhoid fever, giardiasis, amoebiasis, hepatitis A, acute diarrhea, and leptospirosis are water-borne diseases and are mainly associated with critical sanitary problems [3].

Chlorine-releasing and chlorine-based compounds are commonly used for disinfection due to the ease of use, low cost, readily available technology, high antimicrobial activity, and total dissolution in water [4–6]. The addition of chlorine-based disinfectants (e.g., chlorine

gas and sodium/calcium hypochlorite) to water leads to the formation of hypochlorous acid (HOCl) which, depending on the pH of the water, can dissociate, generating the hypochlorite ion ( $\text{OCl}^-$ ) [6]. Both compounds (HOCl, and  $\text{OCl}^-$ ) are capable of performing disinfection as well as reacting with natural organic matter (NOM), leading to the formation of DBPs [7,8]. Chlorine in the form of hypochlorous acid and hypochlorite in water is denominated free residual chlorine [7]. Chlorine is also used in pre-chlorination prior to the addition of the coagulant to facilitate the removal of suspended or dissolved impurities as well as reduce the amount of chlorine used in the treatment process [9]. Even in lower quantities, chlorine reacts with NOM during pre-chlorination, generating DBPs [10]. Chlorine has also been widely used as a strong oxidizing agent for the removal of inorganic compounds (Fe and Mn), reducing issues related to taste, smell, and color [11].

The use of chlorine as a disinfectant compound may lead to the formation of trihalomethanes (THMs), which are toxic byproducts that can have cytotoxic, genotoxic, and mutagenic effects [12]. These compounds have a carbon atom and  $\text{CHX}_3$  as their general formula, with X representing one or more halogen substituents, such as fluorine, chlorine, bromide, or iodine, and their combinations [13]. As a result of the presence in waters destined for human consumption, trihalomethanes may be found in meat and dairy products, vegetables, roasted products, beverages, and other foods; although, information on the concentrations and origins is considerably limited [14,15]. THMs are considered one of the most important groups of DBPs generated by chemical reactions between chlorine and NOM present in water, including humic and fulvic substances [16,17]. The formation of DBPs depends on the nature and concentration of NOM, levels of bromide ions ( $\text{Br}^-$ ) and chlorine (mainly residual chlorine), pH, and temperature [18]. Total THMs are formed by the sum of four species: trichloromethane ( $\text{CHCl}_3$ ), bromodichloromethane ( $\text{CHCl}_2\text{Br}$ ), chlorodibromomethane ( $\text{CHBr}_2\text{Cl}$ ), and bromoform ( $\text{CHBr}_3$ ) [16,19]. According to Annex XX of Consolidation Ordinance No. 5/2017, the maximum acceptable level of THMs in chlorinated water in Brazil is  $0.10 \text{ mg L}^{-1}$ , with supply companies required to perform quarterly analyses [20].

Epidemiological studies have investigated the action of these compounds during chronic exposure even at low concentrations [12]. Inhalation, ingestion, and skin contact are the main routes linked to exposure to THMs [21,22]. Every system that uses disinfection by chlorination has, as a result, the generation of either THMs or other byproducts with a carcinogenic potential of higher or lower intensity [2,5]. Besides THMs, elements such as potentially toxic metals, which generally have high levels of bioaccumulation, may be present in water [23]. These species pose major risks to human health and the ecosystem in the long term and may indirectly alter the amount of THMs generated [24].

Thus, it is necessary to monitor and carefully assess the physicochemical and microbiological aspects of treated water, particularly that supplied to schools, which mainly host children and adolescents [19]. In the present study, the monitoring and assessment of different water samples from schools were conducted to determine levels of THMs and potentially toxic metals as well as the relationship to the chlorination process. One of the aims of this study was to create a paradigm for future studies on the monitoring of toxic organic compounds in water to avoid health problems in humans and animals.

## 2. Material and Methods

### 2.1. Reference Study Area

A reference study was performed in an urban area of the municipality of Pinhalzinho, Brazil. The water supply in the city is the responsibility of the *Companhia Catarinense de Águas e Saneamento* (CASAN) and is the result of capturing water from the Lageado Ramos and Limeira Rivers. The treatment process to ensure water potability consists of two water treatment plants (WTPs). After treatment, potable water is stored in two reservoirs with a total capacity of  $750 \text{ m}^3$  and is provided to the consumption points through the distribution network. Water samples were collected from schools in different periods, comprising eight different regions of the supply system, as well as from the water treatment plant.

The collection points are shown in Figure 1. These points were chosen based on their distribution after the water exits the WTP. Point 1 is the closest to the WTP and Point 7 is the farthest.



**Figure 1.** Distribution map of sampling points in study reference area.

## 2.2. Sampling

All samples were collected at points located before and after the water storage reservoirs, comprising nine locations and 17 collection points. At each point, water flow was measured for 3 min under low flux to expel the water from the pipes and avoid divergences in the results. Amber vials with a screw cap were used to collect the samples. One vial was used for each type of characterization (potentially toxic metals, THMs, physicochemical properties, and microbiological properties). Thus, four vials were used at each sampling point. These vials were stored under refrigeration in a cooler with recycled ice, followed by transference to the laboratory and storage in a refrigerator (Electrolux BCD-281EA, Brazil). Field tests were also conducted for the measurement of pH, temperature, and free chlorine. Special care was taken in the collection of samples for the THM analyses to avoid the presence of air. To achieve this, the receptacles were closed under running water, avoiding the formation of air bubbles in the vials. To preserve the samples during analysis, 0.10 mL of 10% sodium thiosulfate was added to the vials. In this case, these samples were not affected by air during the experimental measurements in the chromatography equipment as described in Section 2.5.

## 2.3. Physicochemical and Microbiological Analyses

Physicochemical and microbiological analyses were performed following standard methods for the examination of water and wastewater [25]. The following analyses were performed: pH (method 4500-H+ with the aid of an Akso<sup>®</sup> pocket pH meter, São Leopoldo, RS, Brazil); free chlorine (COD) (Method 5220 D); total solids (TS) (Method 2540 B); fluorine (Instrutherm FD-570 Digital Portable Fluorometer, São Paulo, SP, Brazil—SPADNS method); and total and thermotolerant coliforms (Method 9223 B).

## 2.4. Determination of Potentially Toxic Metals

Samples were prepared according to Method 210/IV of the Adolfo Lutz Institute [26]. Potentially toxic metal concentrations were determined using an Analytik Jena contrAA 700 high-resolution continuum source atomic absorption spectrometer equipped with background correction and a flame atomizer (acetylene/air at flow of 40 to 120 L h<sup>−1</sup>), São Paulo, SP, Brazil. Standards were prepared using stock solutions (1000 mg L<sup>−1</sup>) of

nickel (Ni), iron (Fe), zinc (Zn), and copper (Cu) purchased from Sigma-Aldrich, Barueri, SP, Brazil. These solutions were prepared in 0.2% nitric acid (Sigma-Aldrich) and stored in polyethylene flasks. Calibration curves were plotted for each species using linear regression and all analyses were performed in triplicate.

### 2.5. Determination of Trihalomethanes

Concentrations of trichloromethane ( $\text{CHCl}_3$ ), bromodichloromethane ( $\text{CHCl}_2\text{Br}$ ), chlorodibromomethane ( $\text{CHBr}_2\text{Cl}$ ), and bromoform ( $\text{CHBr}_3$ ) in the water samples were determined using a Shimadzu-GCMS-QP2010 ultra gas chromatograph with an AOC-20i injector (São Paulo, SP, Brazil), as described elsewhere [27]. The stationary phase was a Restek® Rtx-5MS column containing 95% dimethyl/5% diphenyl, with a length of 30 m, an inner diameter of 0.25 mm, and a film thickness of 0.25  $\mu\text{m}$ . The chromatographic conditions for the measurements of THMs were 30 °C (3 min), 5 °C  $\text{min}^{-1}$  up to 65 °C (0 min), 40 °C  $\text{min}^{-1}$  up to 200 °C for 1 min, and a He flow of 1.40  $\text{mL min}^{-1}$ . The temperatures of the interface and ion source were 220 and 200 °C, respectively. All data were recorded with ion scanning in a range of 35–265  $m/z$ . CAR/DVB/PDMS 50/30  $\mu\text{m}$  (Supelco, Bellefonte, PA, USA) fibers were employed for the extraction processes. To accomplish this, 10 mL of sample were transferred to 15 mL amber vials with a screw cap equipped with a silicon septum internally coated with PTFE. A magnetic bar coated with PTFE was used for agitation. The sample was incubated for 5 min under agitation at a temperature of 30 °C. The fiber was then inserted into the headspace of the sample and extraction was conducted for 15 min at 30 °C under magnetic agitation. Fiber desorption was conducted in the injector port at 240 °C for 3 min (1 min in splitless mode), with a ratio of 1:5 for the remainder of the time. To create the standard calibration curve for each analyte, 2000  $\text{mg L}^{-1}$  of a THM standard mixture purchased from Sigma-Aldrich was used. The results were expressed in  $\mu\text{g L}^{-1}$  and total THM concentration was calculated by summing the four species of THMs and converting the values into  $\text{mg L}^{-1}$ . The same procedure was applied for both samples and calibration standard solutions.

### 2.6. Statistical and Principal Component Analysis

The data were submitted to a normality distribution test, followed by analysis of variance (ANOVA). In cases of significant differences, means were submitted to Tukey's test with a significance level of 5% ( $p < 0.05$ ). All statistical analyses were performed using R 3.6.1 software as described elsewhere [28]. Hierarchical clustering is a multivariate analysis technique employed to classify different treatments according to their similarity. From a general perspective, the aim of hierarchical clustering is to find a ranking of clusters that are easily interpretable in a dendrogram [29]. To reduce the dimension of data and provide more stable clustering, principal component analysis has been applied to hierarchical clustering in different studies [30–32]. Hierarchical clustering on principal components was conducted with the R packages FactoMineR and factoextra [28,33].

## 3. Results and Discussion

### 3.1. Physicochemical and Microbiological Characterization

The physicochemical characteristics before and after water storage in reservoirs are shown in Tables 1 and 2, respectively. These data are the results of a master thesis [34]. No significant differences were found for chloride ( $\text{Cl}^-$ ) or total solids, among the sampling points, before water storage in reservoirs. However, differences in temperature and free chlorine were found at most sampling points. Temperatures remained high due to the season in which the samples were collected and were significantly lower at the WTP compared to the other points. This may be explained by the gradual increase in temperature in the piping system during water distribution. Hardness values ranged from soft water (lower than 50  $\text{mg L}^{-1}$ ) to slightly hard water (50 to 100  $\text{mg L}^{-1}$ ) [7], with results below the maximum allowable level (MAL) expressed in Brazilian legislation (Annex 10 of Annex XX of Consolidation Ordinance No. 5/2017, in which the MAL is 500  $\text{mg L}^{-1}$ ). In the comparison



between samples collected before and after water storage, a decrease in hardness occurred at most of the study points after storage.

**Table 1.** Physicochemical characteristics at different sampling points before water storage in reservoirs.

|     | T<br>(°C)                 | pH                        | Cl<br>(mg L <sup>-1</sup> ) | F<br>(mg L <sup>-1</sup> ) | Cl <sup>-</sup><br>(mg L <sup>-1</sup> ) | Hardness<br>(mg L <sup>-1</sup> ) | TS<br>(mg L <sup>-1</sup> ) | COD<br>(mg L <sup>-1</sup> ) |
|-----|---------------------------|---------------------------|-----------------------------|----------------------------|------------------------------------------|-----------------------------------|-----------------------------|------------------------------|
| P1  | 28.20 ± 0.00 <sup>c</sup> | 7.60 ± 0.00 <sup>a</sup>  | 1.50 ± 0.00 <sup>g</sup>    | 1.03 ± 0.06 <sup>ab</sup>  | 13.49 ± 0.00 <sup>a</sup>                | 56.04 ± 0.00 <sup>b</sup>         | 466 ± 230 <sup>a</sup>      | 413 ± 187 <sup>ab</sup>      |
| P2  | 28.90 ± 0.00 <sup>a</sup> | 7.50 ± 0.00 <sup>ab</sup> | 1.08 ± 0.01 <sup>a</sup>    | 1.00 ± 0.00 <sup>a</sup>   | 13.49 ± 0.00 <sup>a</sup>                | 53.38 ± 2.31 <sup>ab</sup>        | 466 ± 230 <sup>a</sup>      | 624 ± 6 <sup>a</sup>         |
| P3  | 31.27 ± 0.06 <sup>b</sup> | 7.60 ± 0.00 <sup>a</sup>  | 1.30 ± 0.01 <sup>b</sup>    | 1.10 ± 0.00 <sup>b</sup>   | 13.85 ± 0.00 <sup>a</sup>                | 50.71 ± 4.62 <sup>abc</sup>       | 333 ± 115 <sup>a</sup>      | 599 ± 77 <sup>a</sup>        |
| P4  | 28.13 ± 0.12 <sup>c</sup> | 7.60 ± 0.00 <sup>a</sup>  | 1.46 ± 0.01 <sup>c</sup>    | 1.10 ± 0.00 <sup>b</sup>   | 13.61 ± 0.20 <sup>a</sup>                | 45.37 ± 4.62 <sup>acd</sup>       | 466 ± 230 <sup>a</sup>      | 457 ± 149 <sup>ab</sup>      |
| P5  | 29.37 ± 0.12 <sup>d</sup> | 7.43 ± 0.12 <sup>b</sup>  | 1.40 ± 0.01 <sup>d</sup>    | 1.00 ± 0.00 <sup>a</sup>   | 13.49 ± 0.36 <sup>a</sup>                | 54.71 ± 2.31 <sup>b</sup>         | 400 ± 0 <sup>a</sup>        | 605 ± 55 <sup>a</sup>        |
| P6  | 28.93 ± 0.06 <sup>a</sup> | 7.40 ± 0.00 <sup>b</sup>  | 1.12 ± 0.00 <sup>e</sup>    | 1.00 ± 0.00 <sup>a</sup>   | 13.49 ± 0.00 <sup>a</sup>                | 42.70 ± 2.31 <sup>cd</sup>        | 533 ± 115 <sup>a</sup>      | 565 ± 50 <sup>ab</sup>       |
| P7  | 29.40 ± 0.00 <sup>d</sup> | 7.60 ± 0.00 <sup>a</sup>  | 1.30 ± 0.00 <sup>b</sup>    | 1.06 ± 0.06 <sup>ab</sup>  | 13.49 ± 0.00 <sup>a</sup>                | 54.71 ± 2.31 <sup>b</sup>         | 400 ± 0 <sup>a</sup>        | 480 ± 77 <sup>ab</sup>       |
| P8  | 29.00 ± 0.00 <sup>a</sup> | 7.23 ± 0.12 <sup>c</sup>  | 1.10 ± 0.00 <sup>f</sup>    | 1.10 ± 0.00 <sup>b</sup>   | 13.61 ± 0.20 <sup>a</sup>                | 53.38 ± 2.31 <sup>ab</sup>        | 400 ± 0 <sup>a</sup>        | 531 ± 87 <sup>ab</sup>       |
| WTP | 24.97 ± 0.06 <sup>e</sup> | 7.50 ± 0.00 <sup>ab</sup> | 1.50 ± 0.00 <sup>g</sup>    | 1.00 ± 0.00 <sup>a</sup>   | 13.96 ± 0.20 <sup>a</sup>                | 41.37 ± 2.31 <sup>d</sup>         | 466 ± 230 <sup>a</sup>      | 312 ± 55 <sup>b</sup>        |

Note(s): Data are expressed as mean ± standard error, which are the results of a master thesis [34]. Means followed by the same letter in columns do not differ significantly according to Tukey's test at the significance level of 0.05.

**Table 2.** Physicochemical characteristics at different sampling points after water storage in reservoirs.

|    | T<br>(°C)                 | pH                        | Cl<br>(mg L <sup>-1</sup> ) | F<br>(mg L <sup>-1</sup> ) | Cl <sup>-</sup><br>(mg L <sup>-1</sup> ) | Hardness (mg<br>L <sup>-1</sup> ) | TS<br>(mg L <sup>-1</sup> ) | COD (mg<br>L <sup>-1</sup> ) |
|----|---------------------------|---------------------------|-----------------------------|----------------------------|------------------------------------------|-----------------------------------|-----------------------------|------------------------------|
| P1 | 28.23 ± 0.06 <sup>f</sup> | 7.60 ± 0.00 <sup>ab</sup> | 1.40 ± 0.00 <sup>d</sup>    | 0.93 ± 0.06 <sup>ab</sup>  | 13.49 ± 0.36 <sup>a</sup>                | 56.04 ± 0.00 <sup>b</sup>         | 333 ± 115 <sup>a</sup>      | 340 ± 1 <sup>a</sup>         |
| P2 | 28.50 ± 0.00 <sup>a</sup> | 7.77 ± 0.06 <sup>a</sup>  | 0.80 ± 0.00 <sup>ab</sup>   | 0.90 ± 0.00 <sup>a</sup>   | 13.49 ± 0.00 <sup>a</sup>                | 54.71 ± 2.31 <sup>ab</sup>        | 466 ± 115 <sup>a</sup>      | 269 ± 35 <sup>a</sup>        |
| P3 | 29.33 ± 0.06 <sup>b</sup> | 7.60 ± 0.00 <sup>ab</sup> | 0.77 ± 0.02 <sup>a</sup>    | 1.00 ± 0.00 <sup>ab</sup>  | 13.85 ± 0.36 <sup>a</sup>                | 52.04 ± 0.00 <sup>a</sup>         | 333 ± 115 <sup>a</sup>      | 252 ± 6 <sup>a</sup>         |
| P4 | 28.53 ± 0.06 <sup>a</sup> | 7.67 ± 0.06 <sup>ab</sup> | 1.13 ± 0.06 <sup>c</sup>    | 1.00 ± 0.00 <sup>ab</sup>  | 13.49 ± 0.00 <sup>a</sup>                | 48.03 ± 0.00 <sup>c</sup>         | 400 ± 0 <sup>a</sup>        | 285 ± 16 <sup>a</sup>        |
| P5 | 28.70 ± 0.00 <sup>c</sup> | 7.40 ± 0.17 <sup>cd</sup> | 1.17 ± 0.01 <sup>c</sup>    | 0.97 ± 0.06 <sup>ab</sup>  | 13.49 ± 0.36 <sup>a</sup>                | 41.36 ± 2.31 <sup>d</sup>         | 400 ± 0 <sup>a</sup>        | 257 ± 40 <sup>a</sup>        |
| P6 | 27.07 ± 0.06 <sup>d</sup> | 7.50 ± 0.00 <sup>bc</sup> | 0.80 ± 0.00 <sup>ab</sup>   | 0.93 ± 0.06 <sup>ab</sup>  | 13.37 ± 0.20 <sup>a</sup>                | 40.03 ± 0.00 <sup>d</sup>         | 400 ± 346 <sup>a</sup>      | 267 ± 6 <sup>a</sup>         |
| P7 | 27.80 ± 0.00 <sup>e</sup> | 7.63 ± 0.06 <sup>ab</sup> | 0.87 ± 0.06 <sup>b</sup>    | 1.00 ± 0.00 <sup>ab</sup>  | 13.37 ± 0.20 <sup>a</sup>                | 52.04 ± 0.00 <sup>a</sup>         | 333 ± 115 <sup>a</sup>      | 309 ± 84 <sup>a</sup>        |
| P8 | 28.30 ± 0.00 <sup>f</sup> | 7.30 ± 0.00 <sup>d</sup>  | 0.79 ± 0.01 <sup>ab</sup>   | 1.03 ± 0.06 <sup>b</sup>   | 13.61 ± 0.20 <sup>a</sup>                | 52.04 ± 0.00 <sup>a</sup>         | 333 ± 115 <sup>a</sup>      | 349 ± 134 <sup>a</sup>       |

Note(s): Data are expressed as mean ± standard error, which are the results of a master thesis [34]. Means followed by the same letter in columns do not differ significantly according to Tukey's test at a significance level of 0.05.

Chloride concentrations were similar in the distribution system and reservoirs, with a minimum of 13.34 mg L<sup>-1</sup> for samples collected before water storage and a maximum of 13.95 mg L<sup>-1</sup> at the WTP. All the samples had chloride concentrations below the MAL (250 mg L<sup>-1</sup>). Low levels of chloride in potable water are necessary to ensure the health of the population, as high concentrations can restrict water intake due to the taste and the laxative effect for humans and animals [1,3]. Free chlorine concentrations in the distribution system were in accordance with the values described in legislation (minimum of 0.2 mg L<sup>-1</sup>), with higher levels at the exit of the WTP and Point 1 (1.5 mg L<sup>-1</sup>). A similar situation was found when comparing samples collected at the same points before and after water storage. This occurs because chloride is consumed by organic matter that remains in water even after treatment [26]. A decrease was also found in fluoride concentrations after storage at all sampling points when compared to the points before this stage, with values less than 1.5 mg L<sup>-1</sup>.

Total solid content in most samples remained unchanged at Points 1, 2, and 5, with values less than 1000 mg L<sup>-1</sup> (MAL). There is no legally established MAL for COD. However, lower COD levels are associated with a lower diluted organic load in the reference sample [35]. COD at the study points was lower when the water samples were collected after entering in the reservoir due to the presence of retention filters at the school systems, which help sorb organic matter and solids. The results of the microbiological characterization indicate no contamination by total coliforms or *Escherichia coli* in the 17 samples, making this water suitable for consumption.

### 3.2. Distribution of Potentially Toxic Metals

Considering all the potentially toxic metals studied in this work, only iron was detected at the water sampling points. As iron is removed by chemical oxidation during the control of organoleptic properties (taste, color, and odor), it may be found at low pH and in the absence of oxygen as a soluble, imperceptible, chemically reduced species ( $\text{Fe}^{+2}$ ). However, iron minerals precipitate during aeration or with the addition of chlorine, generating water that is red to black in color [36] and requires treatment. The absence of potentially toxic metals in the water samples indicates low organic matter (OM) content in the water course, as OM increases the levels of species complexed and sorbed in the system, serving as substrate for the growth of microorganisms in the distribution system [10]. Low concentrations of iron suggest that the oxidizing process was effective, and the system has good structural conditions. The physical conditions of water distribution systems, their conception, and layout contribute to varying levels of Fe and Mn in water, altering the metallic taste during consumption by humans [11,23,37].

### 3.3. Distribution of Trihalomethanes

The chromatographic analysis of the 17 sampling points enabled completely splitting all analytes. Concentrations of  $\text{HCl}_3$ ,  $\text{CHCl}_2\text{Br}$ ,  $\text{CHClBr}_2$ , and  $\text{CHBr}_3$  at the sampling points are displayed in Tables 3 and 4.

**Table 3.** Mean concentration (MC) and standard deviation (SD) of  $\text{CHCl}_3$ ,  $\text{CHCl}_2\text{Br}$ ,  $\text{CHBr}_2\text{Cl}$ , and  $\text{CHBr}_3$  ( $\mu\text{g L}^{-1}$ ) before (B) and after (A) water storage in reservoirs.

|      | $\text{CHCl}_3$ |     | $\text{CHCl}_2\text{Br}$ |     | $\text{CHBr}_2\text{Cl}$ |      | $\text{CHBr}_3$ |      |
|------|-----------------|-----|--------------------------|-----|--------------------------|------|-----------------|------|
|      | MC              | SD  | MC                       | SD  | MC                       | SD   | MC              | SD   |
| P1 B | 13.6            | 1.3 | 3.5                      | 0.3 | 0.37                     | 0.07 | ND              | -    |
| P1 A | 25.1            | 4.2 | 5.3                      | 0.6 | 0.69                     | 0.02 | 0.01            | 0.01 |
| P2 B | 18.1            | 1.3 | 4.3                      | 0.1 | 0.60                     | 0.02 | 0.01            | 0.01 |
| P2 A | 27.8            | 1.9 | 7.2                      | 0.4 | 1.51                     | 0.05 | 0.06            | 0.01 |
| P3 B | 22.9            | 0.4 | 6.0                      | 0.7 | 6.22                     | 0.42 | 0.03            | 0.01 |
| P3 A | 21.0            | 3.1 | 5.4                      | 1.1 | 1.05                     | 0.27 | 0.04            | 0.02 |
| P4 B | 17.4            | 2.4 | 4.5                      | 0.4 | 0.63                     | 0.05 | 0.01            | 0.01 |
| P4 A | 14.0            | 4.4 | 4.6                      | 0.8 | 0.90                     | 0.08 | 0.03            | 0.01 |
| P5 B | 33.0            | 4.1 | 7.7                      | 0.2 | 0.94                     | 0.03 | 0.02            | 0.01 |
| P5 A | 16.9            | 0.4 | 5.8                      | 0.1 | 0.70                     | 0.03 | 0.02            | 0.01 |
| P6 B | 14.8            | 1.0 | 4.2                      | 0.3 | 0.44                     | 0.03 | ND              | -    |
| P6 A | 19.8            | 2.0 | 7.5                      | 0.3 | 1.31                     | 0.02 | 0.04            | 0.01 |
| P7 B | 14.4            | 2.1 | 6.9                      | 0.5 | 1.55                     | 0.11 | 0.10            | 0.01 |
| P7 A | 12.7            | 2.2 | 4.4                      | 0.5 | 0.57                     | 0.07 | ND              | -    |
| P8 B | 27.5            | 1.0 | 6.0                      | 0.1 | 0.80                     | 0.02 | ND              | -    |
| P8 A | 14.4            | 1.1 | 4.5                      | 0.5 | 0.86                     | 0.11 | 0.05            | 0.01 |
| WTP  | 12.9            | 2.8 | 4.9                      | 0.4 | 0.68                     | 0.04 | 0.01            | 0.01 |

Note(s): Concentrations between DL and QL are “detected” but not necessarily quantified (D). Concentrations below DL are considered “not detected” (ND). Quantification limits are  $0.01 \mu\text{g/L}$  for all THMs.

Higher mean  $\text{CHCl}_3$  concentrations were found at all sampling points, including the exit of the WTP and within the distribution system of the schools, followed, in descending order, by  $\text{CHCl}_2\text{Br}$ ,  $\text{CHBr}_2\text{Cl}$ , and  $\text{CHBr}_3$ . This is in agreement with results described elsewhere for treated waters from other WTPs [22,38,39]. The lowest and highest concentrations of total THMs were  $0.017$  and  $0.041 \text{ mg L}^{-1}$ , respectively, at the distribution system points as well as in the pipes of the school buildings. All these values were lower than those proposed as MALs in different countries, including Brazil ( $0.1 \text{ mg L}^{-1}$ ), Canada ( $0.35 \text{ mg L}^{-1}$ ), Mexico ( $0.2 \text{ mg L}^{-1}$ ), the United States ( $0.08 \text{ mg L}^{-1}$ ), and the Netherlands ( $0.075 \text{ mg L}^{-1}$ ). Points 3B, 5B, 8B, 1A, 2A, 3A, and 6A had values higher than the MAL established in Germany, which is  $0.025 \text{ mg L}^{-1}$  [10]. All sampling points had values higher than that established by French law ( $0.010 \text{ mg L}^{-1}$ ). The considerable variation in the

maximum allowed or recommended values of THMs in different countries is likely due to the omission of information of the toxic potential of these compounds, implying not only a need for more scientific studies (as well as disclosure) on their toxicity, but also a need to develop methods to minimize and/or remove THMs after water treatment [10].

**Table 4.** Concentrations of total trihalomethanes (THMs) at sampling points before (B) and after (A) water storage in reservoirs.

| Total THMs (mg L <sup>-1</sup> ) |       |      |       |
|----------------------------------|-------|------|-------|
| P1 B                             | 0.017 | P1 A | 0.031 |
| P2 B                             | 0.023 | P2 A | 0.036 |
| P3 B                             | 0.035 | P3 A | 0.027 |
| P4 B                             | 0.022 | P4 A | 0.019 |
| P5 B                             | 0.041 | P5 A | 0.023 |
| P6 B                             | 0.019 | P6 A | 0.028 |
| P7 B                             | 0.022 | P7 A | 0.017 |
| P8 B                             | 0.034 | P8 A | 0.019 |
| WTP                              | 0.018 |      |       |

The production of THMs at some sampling points may be attributed to longer contact times between oxidant species and organic matter, influenced by a possible lack of periodic cleaning of the water reservoirs. Concentrations of total THMs in the distribution system were higher than those values found at the exit of the WTP (0.018 mg L<sup>-1</sup>), except for Point 1B (0.017 mg L<sup>-1</sup>), which is in agreement with information described elsewhere [40,41]. This occurs due to the combination of continuous reactions between NOM and chlorine, generating THMs, in addition to hydrolysis reactions of decomposing intermediate DBPs to form THMs. External levels of DBPs may also be influenced by the potential difference in characteristics of the piping networks and the way water is distributed in different regions, such as piping network extension, leakage rate, mean nighttime water flow, mean daily demand, spatial and temporal variability, existing microbiota, as well as the age and condition of the distribution system. Moreover, it is rarely considered that residual chlorine reacts with organic matter to generate more DBPs in water transmission and distribution processes [41–45].

Equation (1) expresses the relationship between the amount of THMs and the temperature (T), pH, and concentration of free chlorine (Cl) at points before water storage in reservoirs. Such values were expressed as an estimation (standard error) and all coefficients obtained were statistically significant ( $p < 0.05$ ). Applying the Shapiro–Wilk test, the residuals were normally distributed, indicating that the adjustment is suitable for describing the experimental results. The coefficient of determination ( $R^2$ ) was 0.6081.

$$\text{THM} = 0.190(0.071) + 0.004(0.001)T - 0.044(0.011)\text{pH} + 0.028(0.009)\text{Cl} \quad (1)$$

According to Equation (1), an increase of one degree in temperature corresponds to an increase of 0.004 mg L<sup>-1</sup> in the concentration of THMs, an increase of one unit in pH implies a decrease of 0.044 mg L<sup>-1</sup>, and an increase of 1 mg L<sup>-1</sup> in free chlorine results in an increase of 0.028 mg L<sup>-1</sup>. These results show that pH is the variable that most affects the concentration of THMs in the water distribution system before storage in reservoirs, which is in agreement with results published elsewhere [8,46]. An increase in temperature also results in a higher production of THMs due to the increase in the reaction rate between organic precursors and chlorine and the need to reapply chlorine throughout the system.

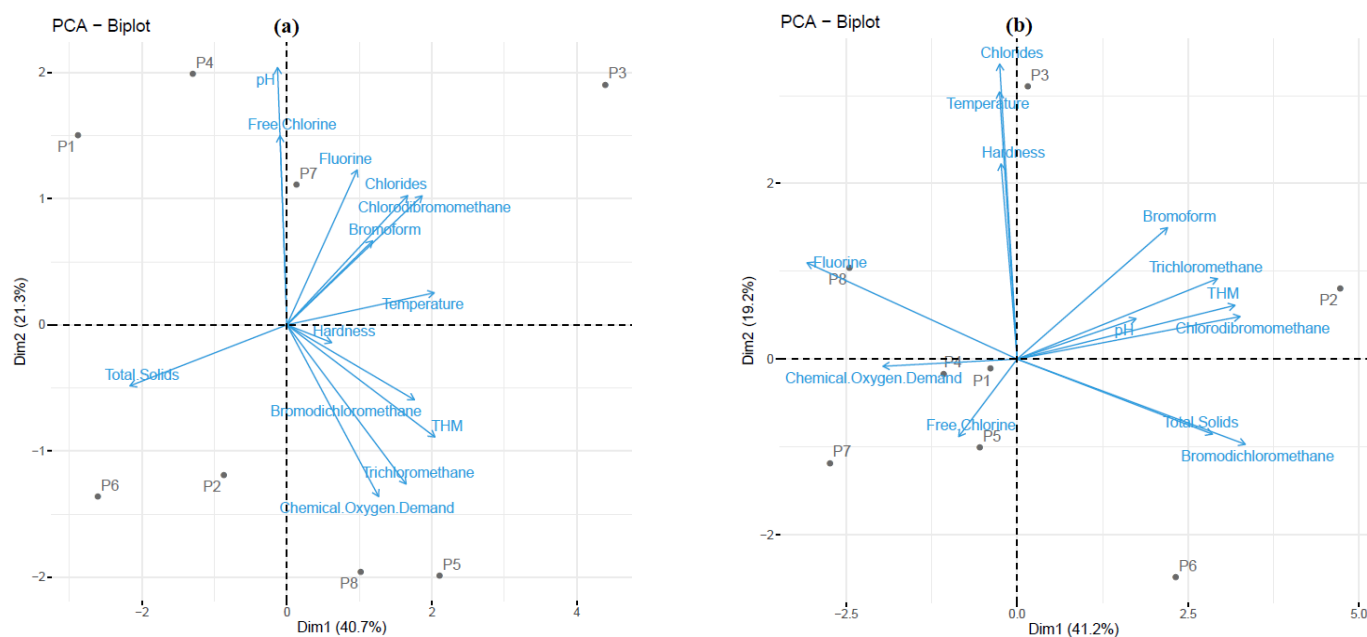
Equation (2) indicates the dependence of the concentration of THMs on the physico-chemical variables of the water after storage in reservoirs.

$$\text{THM} = 0.003(0.000)\text{pH} \quad (2)$$

This time, pH is the only significant term in the linear regression. The results show that an increase of one unit in pH increases the level of THMs by  $0.003 \text{ mg L}^{-1}$  (coefficient of determination = 0.9441, with residuals normally distributed according to the Shapiro–Wilk test). The production of THMs is higher when pH increases due to its catalytic action on haloforms. The haloform reaction comprises a number of basic catalytic substitution reactions of an  $\alpha$ -carbonyl group, followed by hydrolysis to generate trihalomethane. For simple compounds that contain acetyl, the low velocity determination threshold is proton breakdown, which does not depend on the concentration of halogens but is highly dependent on pH [47,48]. Lastly, it is possible to verify the influence of physicochemical variables in the production of DBPs, especially THMs. Such parameters can also be useful tools for monitoring the potential production of THMs through physicochemical and toxicological studies of water destined for human consumption.

### 3.4. Hierarchical Clustering on Principal Components

Figure 2 presents the biplots obtained at the points studied before and after water storage in reservoirs. As Figure 2a,b are very different from each other, it is apparent that water storage significantly affects the physicochemical properties of the treated water, especially in collection sites located close to the WTP. Before storage, P3 can be considered an outlier, as it is relatively isolated from the other points, whereas P2, P5, and P8 are characterized by a higher COD content and higher trichloromethane ( $\text{CHCl}_3$ ) concentration before water storage compared to the other sites. After storage, P3 is characterized by high values of chlorides and temperature, whereas P8 is characterized by a large concentration of fluorine.

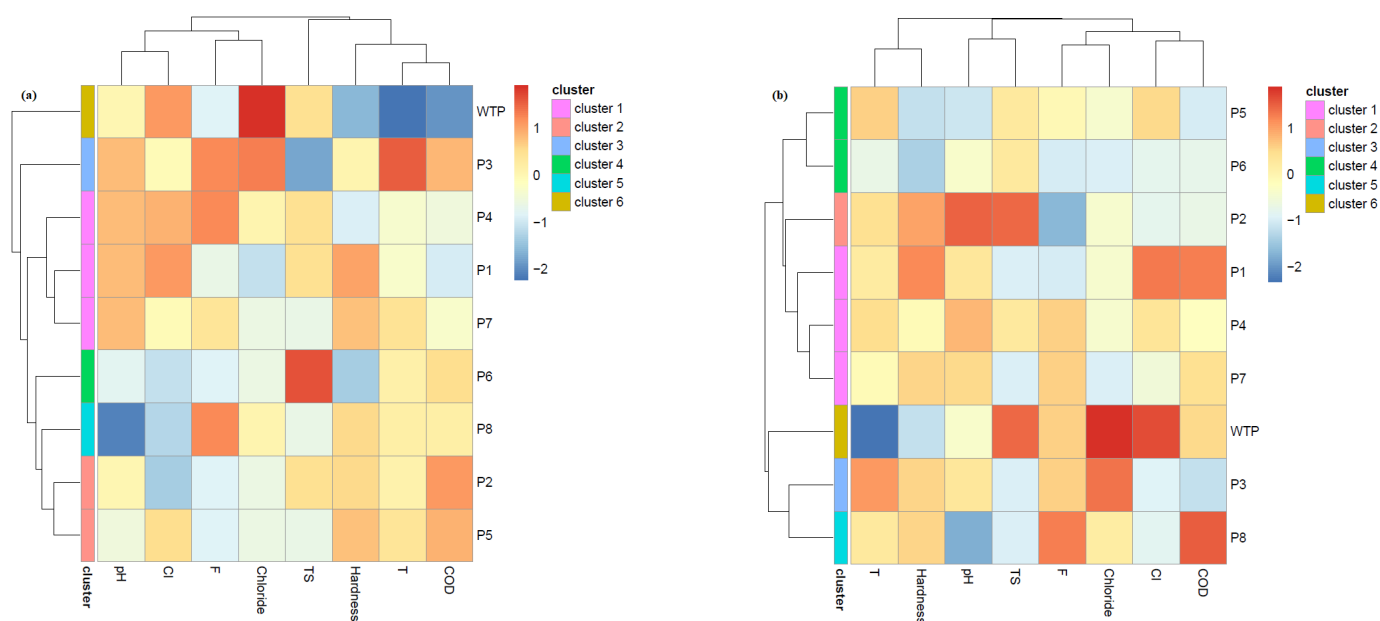


**Figure 2.** Biplots obtained (a) before and (b) after water storage in reservoirs.

Figure 3 displays the heat maps combined with clusters obtained before and after water storage in reservoirs. These diagrams are valuable tools to provide useful information regarding the association between the physicochemical properties and each collection site. Thus, they are widely used in many fields of study [49]. As can be visualized from Figure 3, previous to water storage, the WTP possesses lower temperature values when compared to other environmental sites, whereas values of chloride concentration are higher. However, after water storage, the values of COD and hardness increase at the other sites, so that they are lower at the WTP when compared to the different collection points. The site labeled as P1 is the closest to the WTP. At point P1, the values of free chlorine are relatively low before



water storage and high after water storage, thus reinforcing the influence of the water treatment in the physicochemical properties of nearby sites. The collection point P3 is one of the farthest from the WTP, and interestingly exhibits unique properties as well. Thus, it consists of a separate cluster in Figure 3. In this case, it exhibits a higher temperature when compared with all the other sites, which affects the concentration of chloride and fluorine in aqueous solutions. Overall, with the exception of collection points P1, P2, and P6, which are relatively closer to the WTP, the fluorine content is high at the sites studied, both before and after water storage. These results suggest that the amount of fluorine depends more on the site location than on other factors. The collection point P8 also exhibits unique properties, in particular a low pH and low concentrations of total solids and chloride. This site is relatively far from the other collection points and is considered a distinct cluster. From a general perspective, the physicochemical properties at the collection points P1, P2, and P6 are the most affected after water storage. Conversely, the physicochemical properties of collection points P4 and P7 were the least affected after water storage.



**Figure 3.** Heat maps combined with dendrograms considering different sites (a) before and (b) after water storage in reservoirs.

#### 4. Conclusions

According to the results obtained in this study, the physicochemical (pH, free chlorine, hardness, chlorides, and total solids) and microbiological (total coliforms and *Escherichia coli*) characteristics meet the standards of Annex XX of Consolidation Ordinance No. 5/2017. Zn, Fe, Ni, and Cu could not be properly quantified due to their low concentrations. However, the distribution of the physicochemical properties of water across the various sites changed after storage. The physicochemical properties of the collection points located closer to the water treatment plant were the most affected after water storage. Overall, for the other collection points, the physicochemical properties are location-dependent. The lowest and highest levels of THMs were 0.017 mg/L and 0.041 mg/L, respectively, with all values lower than the MAL established by Brazilian legislation (0.1 mg/L). The predominant species of THMs, in descending order, were trichloromethane (chloroform), dichlorobromomethane, dichloromethane, and bromoform. The formation of THMs was dependent on pH, free chlorine, and temperature, which is in accordance with data found in the literature. All results were confirmed by hierarchical clustering on principal components. The treated water currently offered meets legal standards and consumption can be considered safe.

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