



Identification of disinfection by-products (DBPs) halo phenols in drinking water

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Abstract

The Bureau of Indian Standard (BIS, New Delhi) has framed a set of standards for drinking water description (IS 10500:1991-<http://www.indiawaterportal.org>), which has specifications drawn up in 1983 with the most current amendment (July, 2010), and the US Environmental Protection Agency (USEPA 2003) has also developed policy for different drinking water disinfection by-products (DBPs). This study examined the quality of metropolitan drinking water by monitoring the physicochemical parameters, and DBPs study such as the effects of halide ions, natural organic matter, and drinking water characterization were investigated. The sampled water had halo phenols DBPs as a result of disinfection, during chlorination. The water was alkaline in nature, and the water temperature varied from 33 to 37 °C. The major ions, namely bromate, iodate, chlorite, chromate, sulfate and phosphate, have been investigated in the municipality drinking water, Tiruchirappalli and Srirangam in Tamil Nadu, India. Here, three solvents such as hexane, petroleum ether and pentane were used for the liquid–liquid extraction of target compounds. The gas chromatographs equipped with capillary columns (DB-WAX) were employed for the determination of DBPs and 2-bromo-4-chlorophenol was predominantly identified.

Keywords DBPs · Water · Total organic carbon · Solvents · DB-WAX · GCMS

Introduction

Water is the prime need for all living entities on Earth. Water resources have been obstinately affected by rapid development and increased anthropogenic activities. WHO (2003) estimates that, in India, about 38 million people are affected by waterborne diseases through various means each year, among which over 75% are children. So, disinfection of drinking water is essential to reduce the incidence and spread of waterborne diseases (Gaffga et al. 2007). The disinfection of metropolitan drinking water is performed with chlorine in order to ensure the health of the public and to protect the people against various waterborne infirmities (Villanueva et al. 2015). Chlorination is the most universally accepted method for disinfection since the early twentieth century (Paull and Barron 2004) as it prevents the contamination of potable water against pathogens like

bacteria and viruses (Zha et al. 2014). In (1974), Rook first discovered that hypochlorous and hypobromous acid react with naturally present organic matter to form water DBPs, few trihalomethanes (THM) substances such as chloroform (CHCl_3), bromo dichloromethane (CHCl_2Br), dibromo chloromethane (CHClBr_2) and bromoform (CHBr_3). Richardson (2002) reported that during the addition of chlorine to water, toxic compounds are formed and more than 600 DBPs were identified like haloacetic acids. DBPs are formed through the reaction of organic precursor (natural organic matter, algal organic matter, wastewater effluent organic matter) and inorganic substance (bromate, iodate and nitrate) and also on reaction with chemical disinfectants (chlorine, chlorine dioxide, chloramines and ozone) (Krasner 2009). NOM includes humic substances, fulvic acid, hydrophilic amino acids (*Ser, Thr, Asn, Gln, His, Tyr*), carboxylic acids and carbohydrates too (Kitis et al. 2001). The USEPA issued the DBPs rule that specifies that the highest TOC levels for treated water is 2 and 4 mg/L in source water to ensure that DBPs such as THMs are present at acceptable levels (Pontius 1993). Zeng et al. (2015) highlighted that the common genotoxic compounds come from raw water and several mutagenic or carcinogenic DBPs are formed due to addition

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of disinfectants to drinking water. Chlorine- and bromine-containing compounds also cause carcinogenic, mutagenic or teratogenic effects on animal studies (Weisel et al. 1999). Zhai et al. (2014) reported that 2,4,6-tribromoresorcinol, 2,6-dibromo-4-nitrophenol, 2,2,4-tribromo-5-hydroxy-4-cyclopentene-1,3-dione, 2,2,4-dibromo-5-hydroxy-4-cyclopentene-1,3-dione, and 2,2,4-bromo-dichloro-5-hydroxy-4-cyclopentene-1,3-dione are termed as polar brominated DBPs.

The two most bountiful classes of DBPs are reported to be trihalo methanes (THMs) and haloacetic acids (HAAs) (Krasner et al. 1989). The prescribed maximum permissible level of THMs (CHCl_3 , CHCl_2Br , CHClBr_2 and CHBr_3) is $< 80 \mu\text{g/L}$ by USEPA and is $< 100 \mu\text{g/L}$ by EU, and at the same time HAAs (monochloro, dichloro, trichloro, monobromo and dibromoacetic acids) recommended limit is $60 \mu\text{g/L}$. In case of bromate according to both the US and EU, the permissible level is $10 \mu\text{g/L}$, and for chlorate and chlorite is $700 \mu\text{g/L}$ (USEPA 2006). Yang and Zhang (2013) reported the occurrence of brominated haloacetic acids, four new brominated DBPs such as 2,6-dibromo-4-nitrophenol, 2,4,6-tribromophenol, 3,5-dibromo-4-hydroxybenzaldehyde, and 3,5-dibromo-4-hydroxybenzoic acid and 5-bromosalicylic acid in chlorinated saline secondary and primary sewage effluents. Evidences have shown that brominated/iodinated DBPs generally are highly cytotoxic and genotoxic than their chlorinated analogs. And also Liu and Zhang (2014) reported the comparative toxicity of newly identified bromo- and iodo-phenolic DBPs to a marine alga such as 2,4,6-tri-iodophenol., 3,5-diiodo-4-hydroxybenzaldehyde., 2,4,6-tribromophenol., 2,6-diiodo-4-nitrophenol., 2,4-dibromophenol., 4-bromo-2-chlorophenol., 4-iodophenol., 3,5-dibromo-4-hydroxybenzaldehyde., 2-bromo-4-chlorophenol., 2,4,6-trichlorophenol., 4-bromophenol., 2,4-dichlorophenol., 2,6-dibromo-4-nitrophenol, etc., during chlorination of the wastewater effluent. In drinking water, a measurable amount ($\mu\text{g/L}$) of 2-chlorophenol (00.00–0.065) and 4-chlorophenol (00.004–0.127) was measured (Sithole and Williams 1986). Chlorophenols are a group of compounds that are elaborated in a number of industries. Exposure to high levels of chlorophenols was reported to cause damage to the liver and immune system (ATSDR 1999). Jiang et al. (2017) has proposed a new method to reduce the DBP formation by ore treatment using GAC adsorption to remove the intermediate aromatic halogenated DBPs. Conventional methods used GAC adsorption to remove the NOM directly, whereas in this new approach the authors have claimed this method to be more effective in reducing the DBP formation than the traditional ones. India is facing a serious problem of natural resource insufficiency, especially that of water in view of population growth and urbanization. In India, there is no or very little awareness regarding the presence of DBPs in the drinking water and

their toxic adverse effects and there are no specific reports regarding the presence of DBPs in southern Indian drinking waters. As this study is the first attempt in India, the prime aim was to identify and quantify the DBPs in the drinking water samples and to evaluate the physicochemical parameters of the same. This study would help in unraveling the presence of DBPs in the domestic supplies. The experiment was designed to collect water samples from Tiruchirappalli and Srirangam, Tamil Nadu, India. Drinking water samples obtained from the metropolitan tank was subjected to physicochemical characterization, estimation of bromate, iodate using UV–visible spectrophotometry followed by the GC–MS analysis for the identification of DBPs using three different solvents for extraction.

Materials and methods

Sample collection

Drinking water samples were collected from the metropolitan of Srirangam and Tiruchirappalli City, Tamil Nadu, India. Grab sampling methodology was followed (APHA 2005). The water samples collected before chlorination were maintained as the control.

Study area

Tiruchirappalli is situated in central southeastern India, almost at the geographic center of the state of Tamil Nadu (Fig. 1). The Cauvery Delta begins to form 16 km (9.9 mi) west of the city. Tiruchirappalli water supply entirely depends on river Cauvery. The schemes based on Cauvery River covers various areas of the city. The schemes originate from two stations, namely Tiruchirappalli 10.887012°N , 78.721162°E and Srirangam 10.7904°N , 78.7057°E .

Reagents and chemicals

All reagents used were of analytical grade from Merck, Loba Chemie, and GC–MS grade solvents such as petroleum ether, hexane and pentane were purchased from Sigma-Aldrich. All reagents used are verified to be contaminant free. All equipment and glassware used to analyze samples were verified to be carbon free and are combusted at 400°C for a minimum of 4 h.

Physicochemical characterization of water samples

The collected samples were analyzed for different physicochemical parameters such as pH, EC, alkalinity, hardness, calcium, magnesium, total solids (TS), total dissolved solids (TDS), total suspended solids (TSS), dissolved oxygen

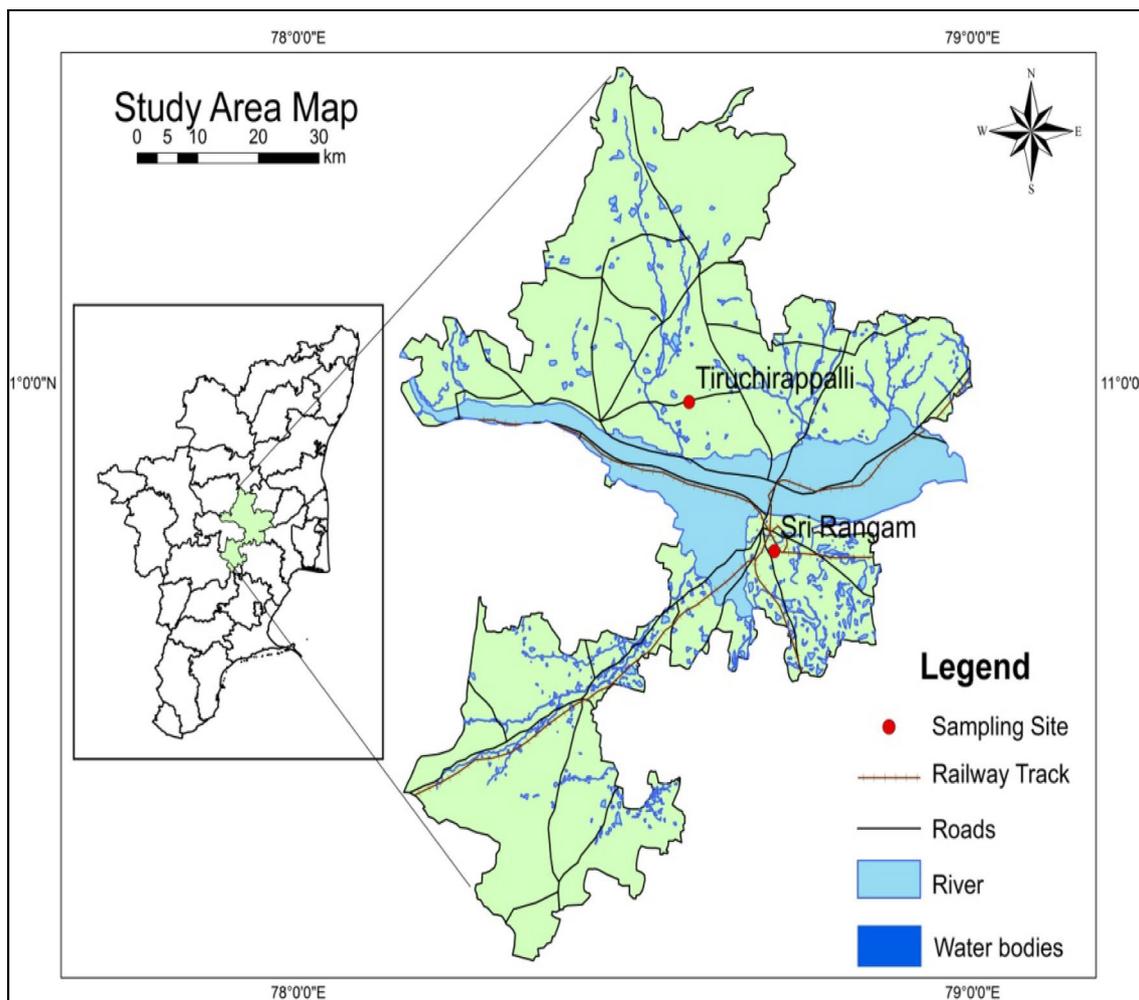


Fig. 1 Study area map

(DO), biological oxygen demand (BOD), chemical oxygen demand (COD), phosphate, sulfate, silicate and nitrate (APHA 2005).

Measurement of TOC and anions

The TOC test is used to determine the total organic carbon in an aqueous sample. The test of TOC utilizes heat and oxygen, ultraviolet radiation, chemical oxidants, or some combination of these methods to convert organic carbon to carbon dioxide which is measured with an infrared analyzer (Metcalf 2003). To determine the number of organically bound carbon, the organic molecules must be broken down to single carbon units and converted to a single molecular form that can be measured quantitatively (Prabhakaran et al. 2009). The TOC of the water samples was analyzed using TOC analyzer (Shimadzu model TOC-500), and the determination of anions (bromate, chromate, chlorite, iodate) was done using UV–Vis spectrophotometer (Spectropara-300).

Selection of organic solvents

The organic solvents are essential to improve the efficiency of extraction process. In this study, three extraction solvents were employed for the extraction of DBPs; the solvents were petroleum ether, hexane and pentane. EPA Method 551, Method 551.1, allows pentane to be applied as the extraction solvent and used to extract trihalomethanes, haloacetic acids, halo ketones and chloropicrin (APHA 1995). Hexane and petroleum ether were used for the extraction of nonpolar compounds like aliphatic hydrocarbons, but petroleum ether contains lesser amount of aromatic compounds (Nawrocki 2013).

Liquid–liquid extraction method

Liquid–liquid extraction (LLE) is one of the most widely employed and useful technique for the DBPs sample preparation. However, infrequently large amounts of suspension

are formed, and it is complicated to separate the solvent from the aqueous stage. Accurately 1000 mL of each sample was taken in separating funnel, and 50 mL of the three different types of solvent was added to three different separating funnels. The contents were mixed well for about 15–20 min until the solvents are separated. The 15–20 min duration is required for mixing.

GC–MS analyses

Hur et al. (2012) investigated the biodegradation-induced changes in the characteristics of dissolved organic matter (DOM) and the subsequent effects on disinfection by-product formation potentials (DBFPs) of sewage water using GC equipped with a 30-m DB-WAX fused silica capillary column. In this study, to identify the DBPs the extract was subjected for GC–MS (SCION SQ/GC–MS; Type-8410; BR-5MS; BRUKER/45X GC44). Helium was employed as a carrier gas, initial oven temperature is 45 °C, maximum temperature is 270 °C, initial time is 2 min, rate is 25 °C/min, final temperature is 260 °C, and final time is 10 min. The gas chromatograph equipped with DB-WAX columns is one of which was polar type and fused silica capillary column (polyethylene glycol, 30 m long and 0.25 mm i.d., film thickness 0.25 µm). The column was operated using an injector port (230 °C), and the following oven temperature

agenda was set as 40 °C for 4 min, followed by an increase to 100 °C at a range of 4 °C/min and as a final point ramped to 160 °C.

Results

The physicochemical characteristics along with the anions and TOC were analyzed in the collected corporation drinking water sampled from Tiruchirappalli and Srirangam, Tamil Nadu (Table 1; Fig. 2).

GC–MS

The samples were subjected for GC–MS analysis, which include the water samples collected from Tiruchirappalli and Srirangam. They are selected as the representative of the schemes under investigation (Fig. 3). Table 2 elucidates GC–MS analysis of the chlorinated water samples using different liquid–liquid extractions, including pentane, hexane and petroleum ether. 4-Bromo-2-chlorophenol was detected during pentane extraction in chlorinated samples from Tiruchirappalli and Srirangam. The same compound was identified in hexane extraction of Srirangam. The water sample before chlorination was referred as control.

Table 1 Physicochemical characteristics of the metropolis water sample, Tiruchirappalli District

S. no	Parameters	Unit	Tiruchirappalli	Srirangam	Control
1	pH	pH	7.3±0.1	7.6±0.1	7.2±0.1
2	EC	mS/cm	00.7±0.2	01.2±0.0	00.3±0.0
3	TS	mg/L	830.3±5.0	935.3±13.2	370.3±13.2
4	TDS	mg/L	625.0±2.6	845.3±5.6	252.3±5.6
5	TSS	mg/L	201.6±3.7	77.6±8.0	147.0±8.0
6	Alkalinity	mg/L of CaCO ₃	90.0±0.0	90.0±0.0	80.0±0.0
7	Hardness	mg/L of CaCO ₃	67.3±1.1	65.3±1.1	79.3±1.1
8	Calcium	mg/L	48.3±0.5	40.3±0.5	35.0±0.5
9	Magnesium	mg/L	04.6±0.2	06.0±0.2	10.9±0.2
10	Chloride	mg/L	48.4±0.8	53.1±1.4	32.5±1.4
11	DO	mg/L	02.3±0.02	04.2±0.0	03.4±0.0
12	BOD	mg/L	00.3±0.0	00.8±0.0	03.3±0.0
13	COD	mg/L	131.6±1.5	144.0±1.7	98.0±1.0
14	Phosphate	mg/L	04.1±0.0	03.1±0.0	00.4±0.0
15	Sulfate	mg/L	01.4±0.0	01.5±0.0	00.7±0.0
16	Silicate	mg/L	90.0±0.0	114.3±4.0	64.6±4.0
17	Nitrate	mg/L	00.1±0.0	00.4±0.0	00.1±0.0
18	Nitrite	mg/L	00.0±0.0	00.0±0.0	00.2±0.0
19	TOC	mg/L	43.4±0.0	44.1±0.0	36.9±0.1
20	Bromate	µg/L	81.0±2.0	74.0±1.0	43.0±2.0
21	Chlorate	µg/L	52.0±1.2	71.0±1.2	24.0±1.2
22	Iodate	µg/L	90.0±0.2	112.0±0.2	76.0±0.2
23	Chromate	µg/L	00.0±1.0	00.0±2.0	00.0±2.0

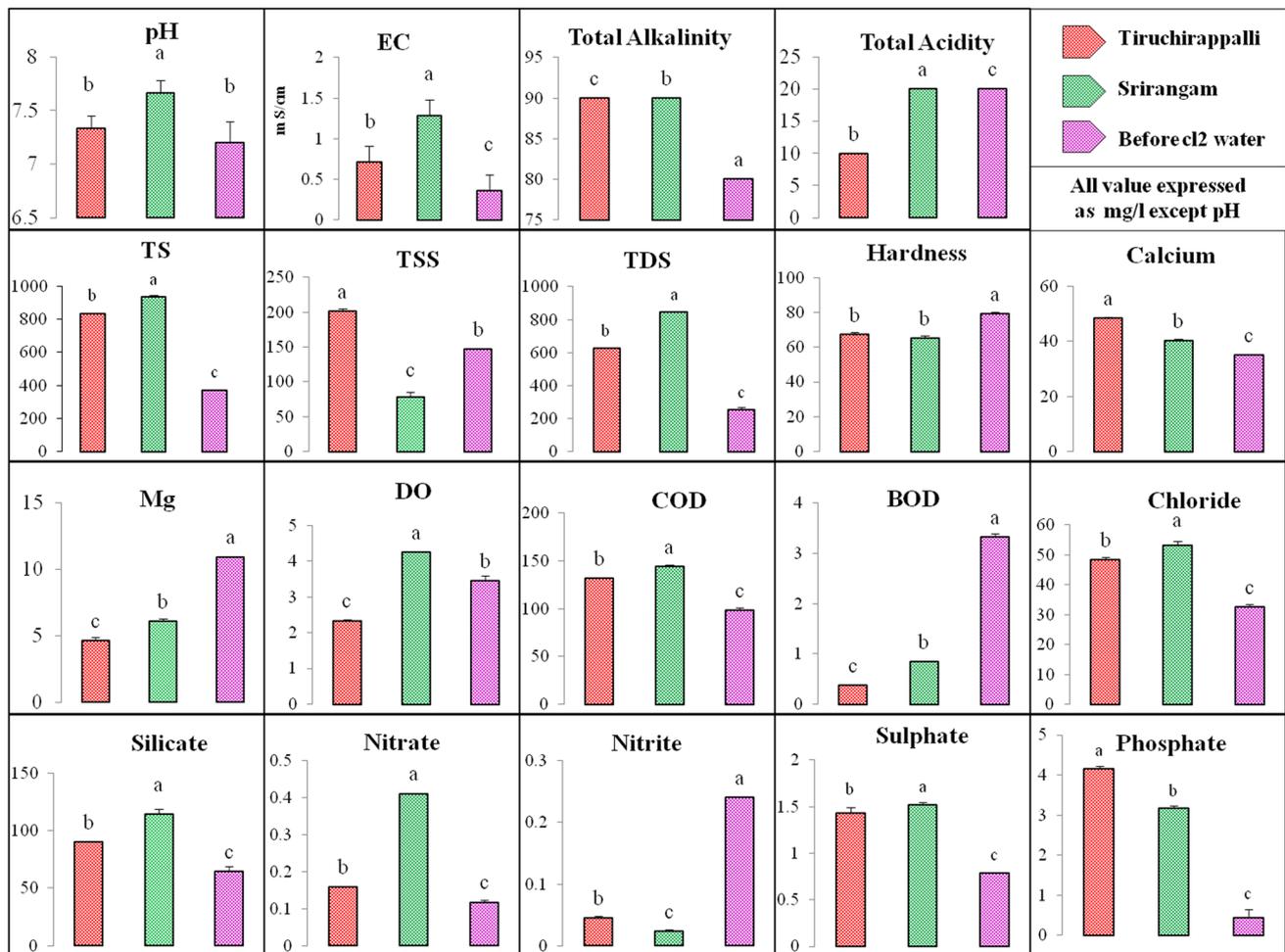


Fig. 2 Physicochemical characterization of drinking water samples. Values were expressed as mean ± SD (N=3). Bar with different alphabets is significantly different from each other and with same alphabets has insignificant changes (P<0.05). The physicochemical parameters (pH, EC, alkalinity, hardness, calcium, magnesium, TS, TDS, TSS, DO, BOD, COD, phosphate, sulfate, chloride, nitrite, sili-

cate, nitrate, bromate, chlorite, iodate and chromate) concentration in drinking water at Tiruchirappalli and Srirangam; the parameter values were expressed as mean ± SD (N=3). The figure noted that the bars represented by means of different alphabets are significantly different from each other and similar alphabets are insignificantly varied (P≤0.05). Before chlorinated water referred as control

Discussion

The drinking waters supplied and distributed by local authorities or some private systems are termed as Public Water Supplies like metropolitan water. Accordingly, the selected place of Tiruchirappalli is situated in central southeastern India, almost at the geographic center of the state of Tamil Nadu. It is formed and located in the river bank of Cauvery delta. At the same time, the Srirangam water source is located in the river banks of Kollidam River (Fig. 1). Hence, the slight variation is found in the physicochemical parameters both before and after chlorination. Consequently, several factors such as temperature, total organic carbon (TOC), pH, bromate, chlorite and iodate concentrations were reported to significantly affect the formation of halo phenols compounds

and in relation to the role of the water temperature on the DBPs formation in the metropolitan water (33–37 °C), as the higher temperatures promote the DBPs. Similarly, the higher range of pH (7.20–7.66) in Srirangam water might be the reason for the formation of alkaline ions (bicarbonate and carbonate ions) when considered with the Cauvery water. Chlorine reacts with humic substances (dissolved organic matter) present in most water supplies, forming a variety of halogenated DBPs such as THMs, HAAs, HANs, chloral hydrate and chloropicrin. It is commonly accepted that the reaction between chlorine and humic substances, a major component of NOM, is responsible for the production of organochlorine compounds during water treatment.

Most chlorinated DBPs are produced during oxidation and substitution reactions. The major functional groups of

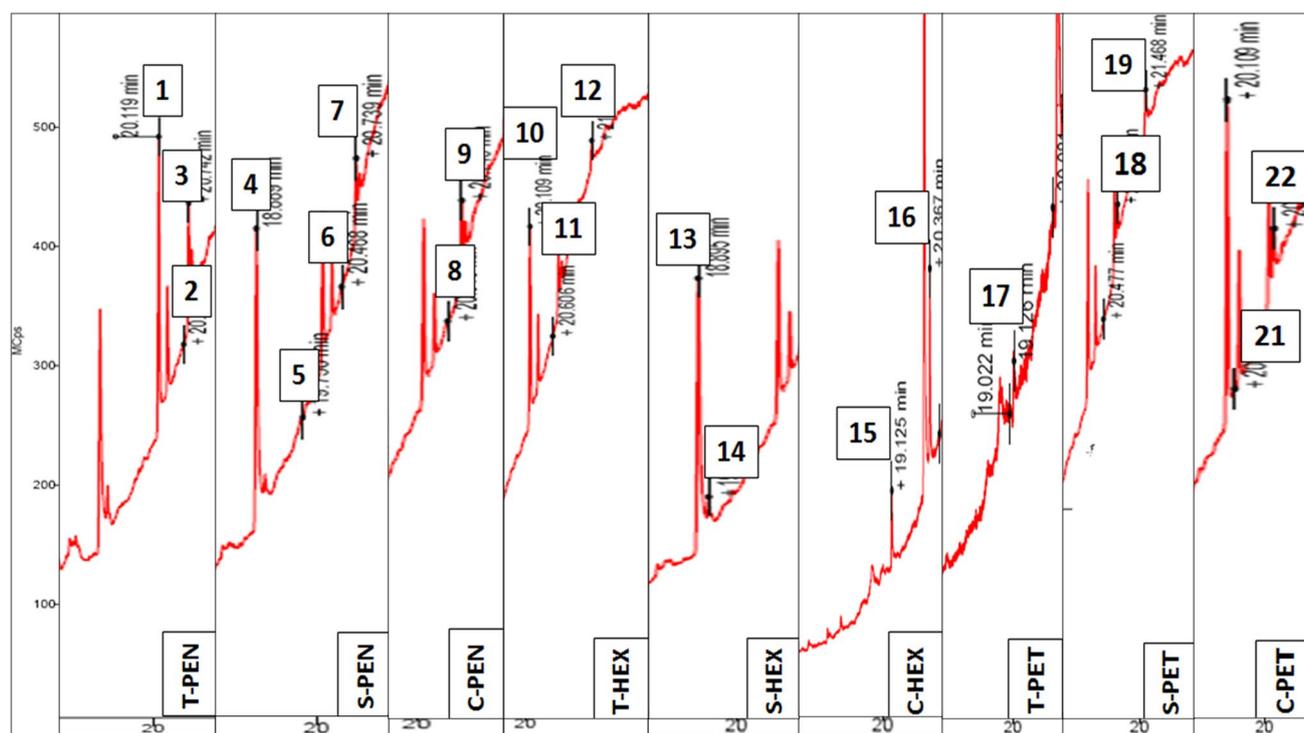


Fig. 3 Identification of halo phenols by gas chromatography and mass spectrophotometry. Note T Tiruchirappalli, S Srirangam, C control (before chlorinated water), PEN pentane (solvents), HEX hexane (solvents), PET petroleum ether (solvents)

humic substances include acetyl, carboxyl, phenol, alcohol, carbonyl and methoxyl. The reactions proceed much more rapidly at high pH. The formation of bromochlorophenol may be due to the reaction between the chlorine introduced in the form of hypochlorite with phenols to produce monochloro phenols or dichloro phenols or trichloro phenols (WHO 2004). The presence of bromate and chlorate was reported in both the drinking water samples. Nawrocki (2013) has also reported the concentration of bromate and chlorite as DBPs. In general, high-chlorine dosages were reported to result in the formation of chlorophenol and bromophenol. Ammonia, bromide and phenols were reported to facilitate higher consumption of HOCl, thereby increasing the concentration of bromophenol (Acero et al. 2005). Also, the pH of the water would have also influenced the formation of bromochlorophenol. WHO (2009) has reported that the bromide ion is found to involve in reaction between chlorine and naturally occurring organic matter in drinking water, forming brominated and mixed chloro-bromo by-products (WHO 2009). However, Liu and Zhang (2014) have reported that 2-bromo-4-chlorophenol was toxic to a heterotrophic marine polychaete (*Platynereis dumerilii*) and marine alga (*Tetraselmis marina*). The haloacetic acids and trihalomethanes have indicated that the emerging

halophenolic DBPs deserve more attention (Zha et al. 2014). The highest value of TDS, chloride, COD, TOC, chlorite and iodate was observed in the water samples collected from Srirangam and Tiruchirappalli which was significantly higher than that of the control water sample. The concentration of bromate was comparatively higher in Tiruchirappalli than Srirangam but significantly higher than that of control water sample. An increase in the level of these parameters forms the basis for DBPs formation.

Conclusion

More efforts must be taken to minimize the concentrations of disinfectants and DBPs without compromising in any way in the removal or inactivation of pathogens in the drinking water. Regarding the DBPs wax column and pentane liquid–liquid extraction employing pentane has resulted in the detection of the presence of 4-bromo-2-chlorophenol in the corporation water samples.

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Table 2 GC–MS analysis of water samples extracted using different solvents

Peak number	Sampling stations and solvents	Compound name	Molecular formula	Molecular weight	Total % of compounds
1	T-PEN	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	89.975
2	T-PEN	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	29.257
3	T-PEN	2,4,6-Trichlorobenzonitrile	C ₇ H ₂ Cl ₃ N	208	07.467
4	S-PEN	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	18.109
5	S-PEN	Acetamide, <i>N</i> -(4-bromo-2-chlorophenyl)-	C ₈ H ₇ BrClNO	248	05.794
6	S-PEN	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	25.391
7	S-PEN	1,2,4-Trimethylbenzene	C ₉ H ₁₂	120	02.426
8	C-PEN	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	35.097
9	C-PEN	4-Bromo-3-chloroaniline	C ₁₀ H ₁₄	134	07.586
10	T-HEX	Benzenamine, 4-bromo-2-chlorophenol	C ₉ H ₁₂	120	07.774
11	T-HEX	2,4,6-Trichlorobenzonitrile	C ₇ H ₂ Cl ₃ N	208	05.540
12	T-HEX	4-Bromo-3-chloroacetanilide	C ₈ H ₇ BrClNO	248	13.502
13	S-HEX	Ethanesulfonyl chloride, 2-chloro	C ₂ H ₄ Cl ₂ O ₂ S	268	06.087
14	S-HEX	4-Bromo-2-chlorophenol ^a	C ₆ H ₄ BrClO	206	40.410
15	C-HEX	3,4-Dichlorophenethylamine	C ₆ H ₄ Cl ₂ O	142	07.681
16	C-HEX	4-Chloro-2-iodophenol	C ₆ H ₄ ClIO	142	02.807
17	T-PET	Chloromethanesulfonyl chloride	C ₂ HCl ₃ O ₂	148	12.554
18	S-PET	Trichloroacetic acid	C ₂ HCl ₃ O ₂	163	11.592
19	S-PET	Benzene, 1-bromo-3-chloro-2-methyl-	C ₇ H ₆ BrCl	205	03.213
20	C-PET	Pentanoic acid, 3-methyl-	C ₆ H ₁₂ O ₂	116	01.450
21	C-PET	Benzene, 1-bromo-3-chloro-2-methyl-	C ₇ H ₆ BrCl	205	02.215
22	C-PET	Heptanoic acid	C ₇ H ₁₄ O ₂	130	08.252

T Tiruchirappalli, S Srirangam, C control (before chlorination), PEN pentane, HEX hexane, PET petroleum ether (T-PEN: samples collected from Tiruchirappalli and extracted using pentane solvents)

^aHalogenated DBP compound (4-bromo-2-chlorophenol) was identified at different places using various solvents

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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