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Perchlorate contamination in bottled and other drinking water sources of Kerala, southwest coast of India

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Abstract Perchlorate and chlorate are inevitable components in rocket propellants, fireworks, water treatment, bleaching, textile industry, etc. Knowingly or unknowingly, wide use of these chemicals contaminates drinking water systems. The present study assessed the perchlorate and chlorate contamination of bottled water and other drinking well, tap, bore well, rainwater sources of Kerala and other parts of Peninsular India. Other drinking water samples were collected during the pre-monsoon, monsoon and post-monsoon from the 14 districts of Kerala and were analyzed for perchlorate and chlorate using liquid chromatography–mass spectrometry. Most of the locally available brands of bottled water were found to be contaminated with perchlorate and chlorate. Mean value of perchlorate was found to be 93.19 ppb, and that of chlorate was 3692.07 ppb, both exceeding the health reference level. The continuous consumption of water with high level of perchlorate content can cause high risk, and the probability of increasing thyroid disorders is high. Perchlorate was detected from well, tap and bore well water samples, and the maximum perchlorate was observed for the pre-monsoon season, whereas the minimum noticed in the monsoon samples. The high concentration of perchlorate detected in groundwater was mainly due to the industrial units and firework manufacturing and display sites.

Keywords Pollution · LC–MS · Bottled water · Chlorate · Thyroid disorders

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1 Introduction

Bottled water is one of the fastest-growing industries in India, and the rate is increasing on a more frequent basis, of 40–50 % annually. South India is the biggest consumer of bottled water, representing more than 50 % of the total market in India (The Economic Times 2012). In Kerala, one of the southernmost Indian states, around 0.8 million liters of potable water is sold amongurban communities per day and is increasing every year (IWR 2013). Hence, any contamination of bottled water may affect a large population. The present study aimed to investigate the drinking water contamination with perchlorate and chlorate.

Perchlorate (ClO_4^-) and chlorate (ClO_3^-), the inorganic anions of chlorine, are gaining attention because of their immense contamination in drinking water systems. Perchlorate is used as an oxidizer in solid rocket propellants (Tikkanen 2006), fireworks (Sijimol et al. 2014, 2015; Sijimol and Mahesh 2014; Isobe et al. 2012), munitions, signal flares (ITRC 2005; Urbansky 2002), etc. Perchlorate is highly soluble and is persistent in nature (Siglin et al. 2000; Motzer 2001). This results in the contamination of environment, especially aquatic systems, and is very difficult to treat (Logan 1998). Various studies reported perchlorate contamination of surface water, groundwater, tea, soft drinks, beverages, baby formulas, etc. (Snyder et al. 2006; Kannan et al. 2009; Anupama et al. 2012). The awe behind perchlorate contamination is that exceeding the limit of 24.5 ppb perchlorate in drinking water can result in thyroid disorders (Wolff 1998; Anderson et al. 2006; Sijimol et al. 2015).

Chlorate is widely used in matches, laboratories, herbicides, pulp, paper and textile industry, etc. The reduction of perchlorate can also result in chlorate production (USEPA

2008a). Sodium hypochlorite and chlorine dioxide are used in water treatment, disinfection of microorganisms, pesticide, bleaching, coir and textile industry. About 10 % of the applied chlorine dioxide is converted to chlorate. In hypochlorite solutions, chlorate is generated spontaneously. Like perchlorate, chlorate is also highly soluble (USEPA USEPA 2008a, b; AWWA 2014). It is included in the third Unregulated Contaminant Monitoring Rule (UCMR3) (AWWA 2014). In drinking water, the chronic dietary assessment for chlorate is estimated to be 0.69 mg/L (USEPA 2008a), whereas the estimated health reference level (HRL) for chlorate is 210 µg/L (AWWA 2014). Moreover, chlorate acts as a thyroid disruptor, resulting in thyroid gland follicular cell hypertrophy though not as strong as perchlorate (USEPA 2008a). It can also impair the oxygen-carrying capacity of blood and thereby ruptures red blood cell membranes in infants and fetuses (NAS 1987).

The major sources of perchlorate in Kerala are Ammonium Perchlorate Experimental Plant (APEP), Thumba Equatorial Rocket Launching Station (TERLS), firework manufacturing sites and innumerable firework displays (Sijimol and Mahesh 2014). Major industrial source of chlorate is Travancore Cochin Chemicals Limited where sodium chlorate is manufactured and used for ammonium perchlorate production at APEP (The Hindu 2013). The 800 tonnes of ammonium perchlorate produced by APEP is consumed by TERLS as rocket propellant. Sodium hypochlorite used in drinking water treatment plants can also act as a source of chlorate.

The literature indicates that a very few works have been carried out in India, to estimate the perchlorate contamination in drinking water (Kannan et al. 2009; Isobe et al. 2012; Anupama et al. 2012, 2015). Widespread occurrence of perchlorate in public drinking, open well and surface water sources was reported along the southwest coast of Kerala (Anupama et al. 2012). Hence, the present study aimed to understand the perchlorate and chlorate contamination of well water, bore well water, tap water, bottled water and rainwater in Kerala.

2 Materials and methods

2.1 Study area and sample collection

Kerala is situated along the southwest coast of India. Drinking water samples were collected from water supplies of selected towns from all the fourteen districts of Kerala. The 14 districts were classified into 3 regions—southern region (Trivandrum, Kollam, Pathanamthitta, Alappuzha), central region (Kottayam, Idukki, Thrissur, Ernakulam, Palakkad) and northern region (Kozhikode, Malappuram, Kannur, Kasaragod, Wayanad) (Fig. 1).

Drinking water samples (well, bore well and tap) were collected during April–May 2014 (before onset of monsoon); August–September 2014 (during monsoon) and December 2014–January, 2015 (after monsoon). Water samples were filtered using 0.2-µ nylon membrane filter paper and stored below 4 °C until the analysis performed.

Different brands (local and international) (n = 31) of bottled water were collected from different districts of Kerala. The number of samples from each district is given in Table 1. A few bottled water samples (n = 5) were randomly collected from other states also (Maharashtra, Andhra Pradesh, Tamil Nadu and Karnataka). The samples were filtered using 0.2-µ filter paper and stored in sterile polypropylene bottles, under 4 °C for the analysis.

Rainwater samples (n = 15) were collected during the summer season from selected towns of Kerala. Samples were then filtered and analyzed for perchlorate and chlorate.

2.2 Sample analysis

Liquid chromatography–mass spectrometry (LC–MS) (Shimadzu LCMS-2020) with Dionex AS 21 (250 mm × 4 mm diameter) column was used for perchlorate and chlorate analysis. Selective ion monitoring (SIM) technique was used for the detection. For perchlorate, m/z 101 (the isotopic mass of perchlorate m/z 99) was used for quantification, while hydrogen sulfate, $H^{34}SO_4$, the common anion in water, also had m/z 99. For the confirmation of a peak as of perchlorate, the ratio ^{35}Cl:^{37}Cl (m/z 99: m/z 101) was calculated and the ratio 3 ± 25 % was accepted. The method detection limit (MDL) was 2 ppb and the limit of quantification (LoQ) was 6 ppb for perchlorate, whereas those for chlorate were 4 and 10 ppb, respectively.

2.3 Risk assessment calculation

The established reference dose of perchlorate per kilogram body weight per day is 0.7 µg/kg/bw/day (USEPA 2008b). Risk assessment of perchlorate was calculated separately for entire Kerala (different seasons), regionwise (different seasons) and based on the type of drinking water.

Intake (µg/kg body wt/day) = CW × IR × ED/BW,

where CW—chemical concentration in water (µg/L), IR—intake rate (L/day), ED—exposure duration (day), BW—body weight (kg) (Wu et al. 2010, 2011).

3 Results and discussion

3.1 Bottled water

Of the 36 brands of bottled water analyzed, 20 samples showed perchlorate (Fig. 2) and 32 samples showed
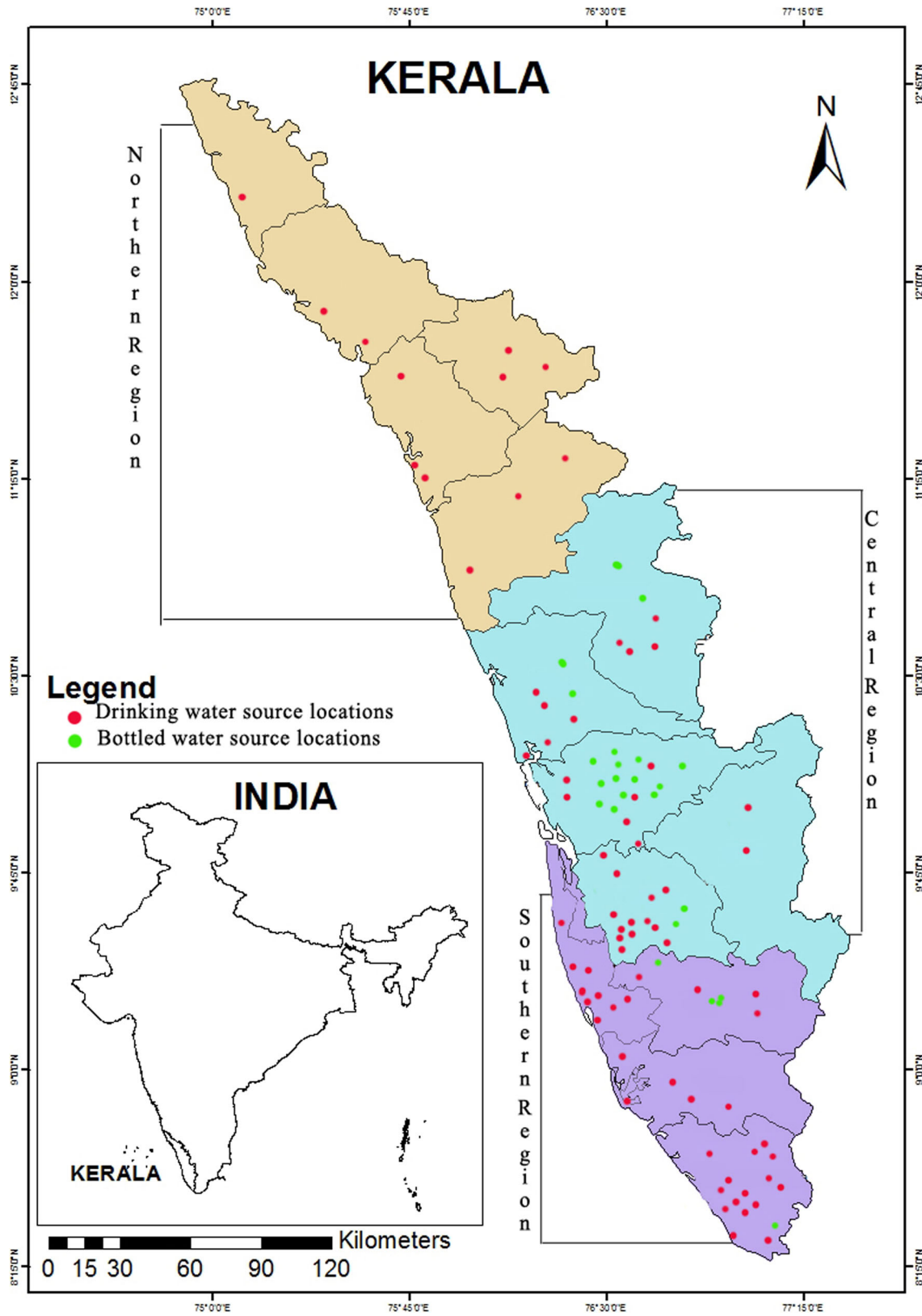
Fig. 1 Map showing bottled water and other drinking water source locations

Table 1 Mean perchlorate and chlorate concentration in different bottled water samples

Sample no.	Sample ID	No. of samples analyzed	Manufacturing place	Perchlorate (ppb)	Chlorate (ppb)
1	BW1	8	Ernakulam	1067.89	7328.89
2	BW2	6	Pathanamthitta	162.74	1821.96
3	BW3	5	Palakkad	19.77	101.2
4	BW4	1	Ernakulam	0	1304.64
5	BW5	6	Bangalore, Karnataka*	0	399.68
6	BW6	1	Pathanamthitta	90.34	3014.24
7	BW7	1	Thrissur	42.65	488.32
8	BW8	1	Thrissur	94.02	40,705.84
9	BW9	1	Thiruvananthapuram	126.17	20,500.16
10	BW10	1	Ernakulam	0	388.64
11	BW11	1	Ernakulam	0	1942.56
12	BW12	1	Kanchipuram, Tamil Nadu*	0	75.92
13	BW13	2	Pathanamthitta	0	120.8
14	BW14	1	Pathanamthitta	0	10,912.24
15	BW15	1	Hyderabad*	0	149.28
16	BW16	2	Ernakulam	17.14	487.36
17	BW17	2	Pathanamthitta	10.26	1007.76
18	BW18	3	Ernakulam	44.63	427.92
19	BW19	2	Thrissur	0	5644.4
20	BW20	2	Ernakulam	15.97	1090.08
21	BW21	1	Thiruvananthapuram	0	0
22	BW22	1	Palakkad	0	345.04
23	BW23	1	Mumbai*	0	0
24	BW24	1	Ernakulam	40.38	462.96
25	BW25	1	Andheri, Mumbai*	24.12	108.48
26	BW26	1	Ernakulam	0	1435.12
27	BW27	1	Ernakulam	19.05	3651.68
28	BW28	2	Ernakulam	16.05	2051.6
29	BW29	2	Ernakulam	13.12	7129.84
30	BW30	1	Ernakulam	9.51	521.92
31	BW31	1	Ernakulam	9.78	0
32	BW32	1	Kottayam	0	434.4
33	BW33	1	Ernakulam	32.2	0
34	BW34	2	Kottayam	0	1711.48
35	BW35	1	Ernakulam	7.91	430.88
36	BW36	2	Ernakulam	0	1950.88
			Mean	84.71	3281.84

* Samples manufactured outside Kerala

chlorate contamination (Table 1). Mean perchlorate content in bottled water was 84.71 ppb, whereas for chlorate was 3281.84 ppb. The observed perchlorate content in 9 samples was higher than the health reference level for chlorate content in drinking water given by USEPA (24.5 ppb). Twenty-one samples exceeded the health reference level for chlorate. Samples from Kottayam district, which is free from industrial sources, have perchlorate within below detectable limit (BDL < 6 ppb). More than 70 % of the samples from Ernakulam district showed perchlorate contamination. Perchlorate contamination was already reported from groundwater and surface water from Ernakulam district, near to APEP (Anupama et al. 2012). The source of surface water for bottled water manufacturing in Ernakulam district is Periyar River, which is the main reason behind the widespread perchlorate
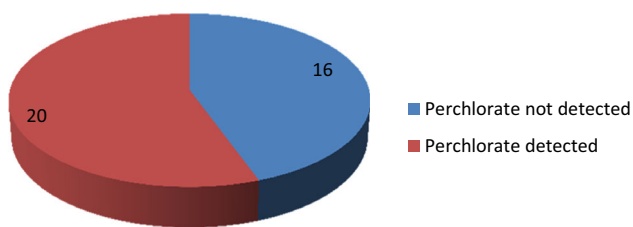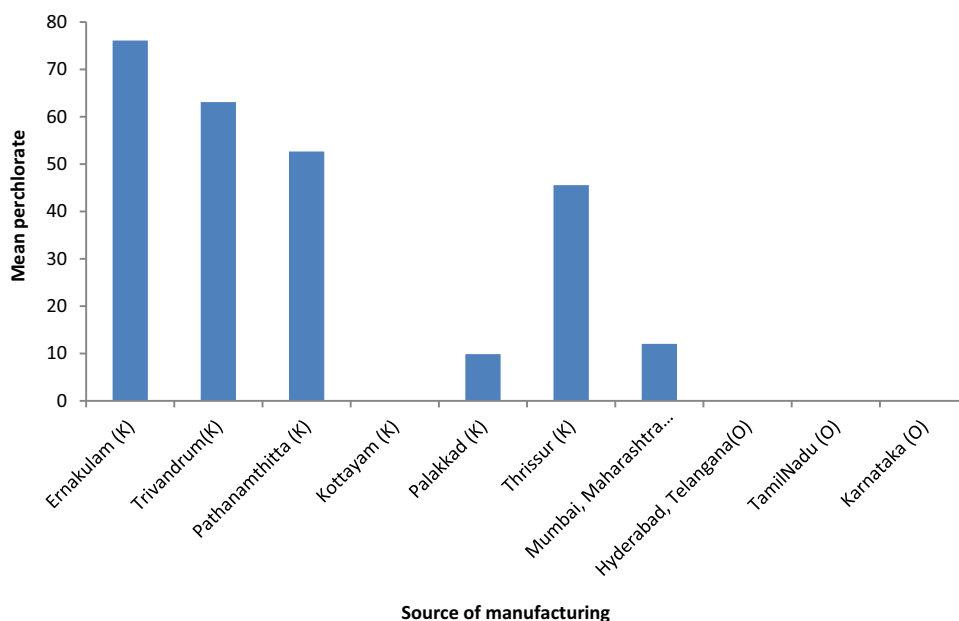
Fig. 2 Graph showing perchlorate detected and perchlorate undetected samples

contamination of bottled water from the region. The sample contaminated with perchlorate obtained from Trivandrum district is manufactured from Thumba nearby TERLS. Veli Lake is the source of water, which is contaminated with perchlorate (Anupama et al. 2012). The contamination at other districts can be owed to the firework manufacturing units as most of the districts in Kerala have licensed and unlicensed small-scale and large-scale units. It is interesting to note that out of 5 outside state samples analyzed, 4 samples are having perchlorate in BDL and the only one (from Mumbai) is having perchlorate (12.06 ppb) within the safe limit (Fig. 3). This shows either uncontrolled use and mishandling of the chemical or lacking of advanced systems for perchlorate removal in the study area. Perchlorate contamination of bottled water is thus found to be dependent on the source contamination.

Chlorate contamination is noticed in almost all samples, irrespective of their source of manufacturing. Both within state and outside state samples were contaminated with chlorate and found to be source independent. This may be owed to the disinfection processes using hypochlorites. In Kerala, sodium hypochlorite as such is used in treating

large quantities of drinking water supplies (The Hindu 2008).

Almost all the samples underwent treatment processes such as reverse osmosis, microfiltration, UV irradiation before packaging, and the results suggest that those processes are inefficient in removing perchlorate and chlorate. Various studies reported the possibility of removing perchlorate using technologies such as physical separation (precipitation, anion exchange or membrane filtration, reverse osmosis and electrodialysis), chemical and electrochemical reduction, and biological or biochemical reduction (Urbansky 1998; Srinivasan and Viraraghavan 2009), but it is found that typical water treatment technologies cannot remove perchlorate (Logan 1998).

Various studies reported perchlorate and chlorate contamination of bottled water in different countries (Table 2). Global perchlorate values ranged from <0.02 to 2.01 ppb and chlorate values ranged from 5.8 to 343 ppb, with China being the topmost in both perchlorate and chlorate contamination. Perchlorate detected from bottled water in Kerala was found to be far above the values reported from other countries. But the measured perchlorate value from bottled water was comparable to the tap water (38.4 ppb), groundwater (34.1 ppb) and surface water (17 ppb) values in Kerala (Anupama et al. 2012).

3.2 Other drinking water sources

The analytical results of drinking water samples such as well, bore well and tap water are given according to regionwise and drinking water type (Table 4). Perchlorate content for the drinking water samples ranged from

Fig. 3 Graph comparing the mean perchlorate of different sources [K—Kerala manufactured samples, O—outside state manufactured samples]

Table 2 Comparison of perchlorate in bottled water with other reported values

Country	Perchlorate (ppb)	Chlorate (ppb)	References
US	0.405	5.8	Snyder et al. (2005)
Japan	0.53	14	Asami et al. (2009)
China	2.013	343	Shi et al. (2007), Wu et al. (2010)
South Korea	0.07	–	Her et al. (2011)
Italy	0.075	–	Iannece et al. (2013)
India (7 states excluding Kerala)	<0.02 (quantitation limit)	–	Kannan et al. (2009)
Canada	–	6.9	Dabeka et al. (2002)

Table 3 Mean perchlorate (ppb) in well water samples from various districts of Kerala during different times in a year

Sl. no.	District	April–May 2014		August–September 2014		December 2014–January 2015	
		ClO_4^- (n)	ClO_3^-	ClO_4^- (n)	ClO_3^-	ClO_4^- (n)	ClO_3^-
1	Thiruvananthapuram	71.86* (12)	365.05	0 (13)	0	1.98 (13)	0
2	Kollam	14.45 (5)	99.07	0 (5)	0	27.45* (5)	33.71
3	Pathanamthitta	178.93* (5)	17.184	0 (5)	0	0 (5)	0
4	Alappuzha	1172.37* (8)	289.95	0 (9)	0	27.34* (8)	58.84
5	Kottayam	30.69* (12)	201.39	0.47 (13)	0	19.19 (13)	96.53
6	Idukki	102.19* (20)	0	5.82 (3)	0	19.3 (2)	0
7	Ernakulam	40.48* (8)	166.05	0 (8)	0	21.84 (8)	0
8	Thrissur	29.37* (6)	15.07	0 (6)	0	0 (6)	0
9	Palakkad	60.09* (6)	259.16	0 (4)	0	0 (4)	0
10	Malappuram	23.92 (3)	0	0 (4)	0	5.68 (5)	9.50
11	Kozhikode	39.09* (2)	0	0 (2)	0	64.68* (3)	28.08
12	Wayanad	121.04* (2)	0	0 (3)	0	0 (2)	0
13	Kannur	7.97 (1)	0	0 (1)	0	34.34* (2)	0
14	Kasaragod	254.14* (1)	154.56	0 (1)	0	95.72* (1)	0
	Total mean	153.33*	111.96	0.45	0	22.68	16.19

(n)—number of samples

Number of samples analyzed are given in italics

* Concentration exceeding the permissible level of 24.5 ppb

0.71 ppb (monsoon) to 78.45 ppb (pre-monsoon) with a mean value of 33.48 ppb. Maximum perchlorate was detected in pre-monsoon samples. Chlorate value ranged from 4.59 ppb (monsoon) to 247.19 ppb (pre-monsoon) with an average of 58.04 ppb.

The results are also discussed based on regionwise such as southern, central and northern parts of Kerala (Table 4). Southern part of Kerala showed a mean perchlorate content of 40.69 ppb and chlorate content of 211.07 ppb. High mean perchlorate content was observed for northern region (42.36 ppb), whereas minimum was noticed for central region (18.5 ppb). Chlorate was maximum at southern part and minimum at northern part (12.10 ppb).

The results were also compared based on the different types of drinking water such as well, bore well and tap water (Table 4). Well water showed high mean perchlorate and chlorate contents during pre-monsoon (153.33 ppb),

whereas minimum was observed during monsoon (Table 3). The mean perchlorate and chlorate contents in well water samples from the entire Kerala were 58.82 and 42.72 ppb, respectively, and those varied from southern region (124.53 and 71.98 ppb) to central region (21.96 and 49.21 ppb) and northern region (43.10 and 12.81 ppb) (Table 4).

In the case of bore well water, average perchlorate and chlorate contents were 6.22 and 29.30 ppb, respectively, in Kerala. Perchlorate content was maximum at northern region (10.44 ppb), whereas maximum chlorate content was observed at southern region. Minimum mean perchlorate content was noticed at central region (1.74 ppb), and chlorate was in BDL at northern region. Tap water was also found to be contaminated with perchlorate and chlorate (Table 4). The mean concentration of perchlorate and chlorate in tap water in Kerala was 3.54 and 104.84 ppb, respectively. Central region had high mean perchlorate

Table 4 Mean perchlorate and chlorate in other drinking water samples (ppb)

| | Other drinking water samples | | Water type | | | | | |
| | | | Well water | | Bore well water | | Tap water | |
	ClO_4^-	ClO_3^-	ClO_4^-	ClO_3^-	ClO_4^-	ClO_3^-	ClO_4^-	ClO_3^-
Kerala	33.48	58.04	58.82	42.72	6.22	29.30	3.54	104.84
Southern region	40.69	211.07	124.53	71.98	6.56	74.54	3.19	231.08
Central region	18.85	67.99	21.96	49.21	1.74	22.44	7.36	108.68
Northern region	42.36	12.10	43.10	12.81	10.42	0	0	0

Table 5 Average perchlorate intakes from well, bore well and tap water in different regions

Water	Category	ClO_4^- (ppb)	Intake (μg/kg/day)
Other water sources	Kerala	33.48	1.12*
	Southern region	40.69	1.36*
	Central region	18.85	0.63
	Northern region	42.36	1.41*
Well	Kerala	58.82	1.96*
	Southern region	124.53	4.15*
	Central region	21.96	0.73*
	Northern region	43.1	1.44*
Tap	Kerala	3.54	0.12
	Southern region	3.19	0.11
	Central region	7.36	0.25
	Northern region	0	0
Bore well	Kerala	6.22	0.21
	Southern region	6.56	0.22
	Central region	1.74	0.06
	Northern region	10.42	0.35

* Higher than the permissible intake of 0.7 μg/kg/day (USEPA 2008b)

content (7.366 ppb) than the other regions, whereas chlorate was high at southern region (231.08 ppb). Perchlorate and chlorate contents in tap water in the northern region were found to be below the detectable limit.

Out of the 15 rainwater samples analyzed, two samples showed the presence of perchlorate and the values ranged from 6.9 to 12.8 ppb, while chlorate was not detected in any of the samples. This might be the effect of firework residuals in the air as the samples were collected during summer, the festival time of Kerala when lots of fireworks occur. Earlier studies also showed the presence of perchlorate in rainwater samples of other parts of India (<0.02 ppb) (Kannan et al. 2009), but it was very low when compared with the mean perchlorate in rainwater samples of the present study (9.85 ppb).

3.3 Risk assessment

The recommended health protective dose of perchlorate is 0.0007 mg/kg body weight/day (0.7 μg/kg body weight/day) (USEPA 2008b; Groef et al. 2006), which is equivalent to 24.5 ppb perchlorate in drinking water. Average daily perchlorate intake was calculated with respect to regionwise and sample type (Table 5). The calculation considered the intake of drinking water as 2 L and the average body weight of a healthy adult in Kerala as 60 kg (Shome et al. 2014). The whole water samples showed a higher risk value (1.12 μg/kg body weight/day) than the safe limit of 0.7 μg/kg body weight/day. High risk was observed for northern region followed by southern region, whereas central region is under safe limit. Tap water and bore well water were also within the safe limit, but the well water samples (0.73–4.15 μg) were higher than the safe limit.

Widespread drinking water contamination in Kerala can be accounted for the use of rocket propellants and fireworks manufacturing. Further research is needed to confirm the role of natural perchlorate formation, as a source in the study area (Rajagopalan et al. 2006; Anupama et al. 2012).

4 Conclusion

The present study noticed a high perchlorate contamination of the drinking water sources such as bottled water and other drinking water, which urges immediate action in this regard. Both perchlorate and chlorate showed values above the reference levels. This shows that water is not properly treated or the treatment is ineffective before they are packed and sold. The results clearly indicated the role of perchlorate manufacturing industries and firework manufacturing and displaying processes in contamination of water resources. The perchlorate and chlorate in exceeding amounts than the health reference levels point to a probability of increasing thyroid disorders in the future, if chronic exposure to such levels occurred.

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