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Perchlorate as an emerging contaminant in soil, water and food

Prasanna Kumarathilaka ^a, Christopher Oze ^b, S.P. Indraratne ^c, Meththika Vithanage ^{a,*}

^a Chemical and Environmental Systems Modeling Research Group, National Institute of Fundamental Studies, Kandy, Sri Lanka

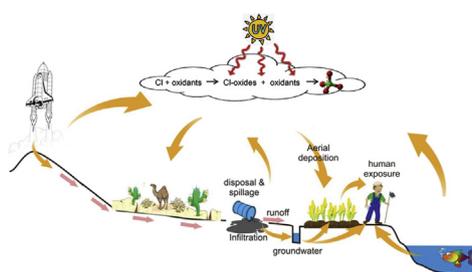
^b Geology Department, Occidental College, 1600 Campus Rd., Los Angeles, CA 90041, USA

^c Department of Soil Science, Faculty of Agriculture, University of Peradeniya, Sri Lanka

HIGHLIGHTS

- Perchlorate is a naturally occurring and man-made chemical substance.
- Specific analytical methods detect perchlorate in the environment.
- RfD for perchlorate exposure is set at 0.7 $\mu\text{g kg}^{-1}$ body weight/day.
- Perchlorate detected in food, soil and water exceeding USEPA RfD.
- Physico-chemical and biological methods look promising way to remove perchlorate.

GRAPHICAL ABSTRACT



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ABSTRACT

Perchlorate (ClO_4^-) is a strong oxidizer and has gained significant attention due to its reactivity, occurrence, and persistence in surface water, groundwater, soil and food. Stable isotope techniques (i.e., $^{18}\text{O}/^{16}\text{O}$ and $^{17}\text{O}/^{16}\text{O}$) and $^{37}\text{Cl}/^{35}\text{Cl}$) facilitate the differentiation of naturally occurring perchlorate from anthropogenic perchlorate. At high enough concentrations, perchlorate can inhibit proper function of the thyroid gland. Dietary reference dose (RfD) for perchlorate exposure from both food and water is set at 0.7 $\mu\text{g kg}^{-1}$ body weight/day which translates to a drinking water level of 24.5 $\mu\text{g L}^{-1}$. Chromatographic techniques (i.e., ion chromatography and liquid chromatography mass spectrometry) can be successfully used to detect trace level of perchlorate in environmental samples. Perchlorate can be effectively removed by wide variety of remediation techniques such as bio-reduction, chemical reduction, adsorption, membrane filtration, ion exchange and electro-reduction. Bio-reduction is appropriate for large scale treatment plants whereas ion exchange is suitable for removing trace level of perchlorate in aqueous medium. The environmental occurrence of perchlorate, toxicity, analytical techniques, removal technologies are presented.

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1. Introduction

Perchlorate (ClO_4^-) is an inorganic anion and strong oxidizer

capable of contaminating water and soils when solid salts of ammonium (NH_4^+), potassium (K^+), magnesium (Mg^{2+}) and sodium (Na^+) perchlorate and perchloric acid (HClO_4) dissolve in water (Duncan et al., 2005; Motzer, 2001; Smith et al., 2004). The strong oxidizing character of perchlorate is a result of chlorine having a maximal +7 oxidation state. Perchlorate is highly soluble and capable of mobilizing in aquatic environments. Kinetically, perchlorate is relatively stable under ambient conditions, potentially related to the tetrahedral arrangement of oxygen restricting

* Corresponding author. Chemical and Environmental Systems Modeling Research Group, National Institute of Fundamental Studies, Hantana Road, Kandy, 20000, Sri Lanka.

E-mail address: meththikavithanage@gmail.com (M. Vithanage).

reductants from interacting with the central chlorine atom (Ye et al., 2012). As a result, perchlorate may persist for many years, possibly decades, under typical surface water and groundwater conditions (Sturchio et al., 2014).

United States Environmental Protection Agency (USEPA) placed perchlorate on its contaminant candidate list (CCL) in 1998 (EPA, 2014). Putting a chemical on the CCL means that the chemical should be evaluated to determine whether it should be considered a major environmental contaminant requiring regulation. In this sense, studies following its inclusion have primarily focused on the identification of perchlorate sources, improving analytical techniques, assessing its health impacts and forwarding remediation techniques and strategies. Over the past decade, the identification and measurement of perchlorate in the environment has improved, especially in the micromolar range, where investigations assessing perchlorate in soil and groundwater have steadily increased (Wagner et al., 2006; Wendelken et al., 2006). Many countries including the USA, Canada, China, South Korea and India have detected perchlorate from nanogram to milligram level in water, soil and food (Her et al., 2011; Kannan et al., 2009; Poghosyan et al., 2014; Wilkin et al., 2007; Ye et al., 2013). Due to the persistent behavior of the perchlorate, many studies have focused on determining the fate of perchlorate in the environment.

In this overview, we (a) discuss sources of perchlorate in water, soil and food, (b) provide a summary of anthropogenic and geogenic perchlorate sources and their respective concentrations, (c) summarize the potential impact of perchlorate on living organisms, (d) review methods for detecting and measuring perchlorate and (e) elucidate current remediation strategies. Several technical terms may frequently used throughout the manuscript for describing perchlorate in the environment hence we have defined them here. A synthetic substance is a substance made by chemical synthesis, especially to imitate a natural product. Natural means a something existing in or derived from nature, not made or caused by humankind. Anthropogenic defines as environmental pollution and pollutants originating in human activity whereas non-anthropogenic means environmental pollution and pollutants originating in naturally.

2. Sources of perchlorate

Perchlorate is an anthropogenic and naturally occurring chemical substance. As it well known, recent development in stable isotope techniques facilitates the differentiation of naturally occurring perchlorate from anthropogenic perchlorate. By assessing triple-oxygen isotope ratios ($^{18}\text{O}/^{16}\text{O}$ and $^{17}\text{O}/^{16}\text{O}$), Bao and Gu (2004) demonstrated that perchlorate from Atacama Desert soil possessed isotopic signatures distinctly different from the commercially produced samples. It appears that when a perchlorate oxyanion is formed its oxygen will not be exchanged under typical environmental conditions. As a result, the perchlorate anion maintains the oxygen isotopic signature of its source and, therefore, can be traced. Additionally, $^{37}\text{Cl}/^{35}\text{Cl}$ fractionation can be used to distinguish the unique origin of the natural perchlorate from the anthropogenic perchlorate. A study by Böhlke et al. (2005), successfully utilized stable isotope analysis ($^{37}\text{Cl}/^{35}\text{Cl}$ and $^{18}\text{O}/^{17}\text{O}/^{16}\text{O}$) to differentiate the natural sources of perchlorate from the synthetic perchlorate. Based on similar approaches, nonanthropogenic perchlorate occurrences have been identified in many places throughout the world.

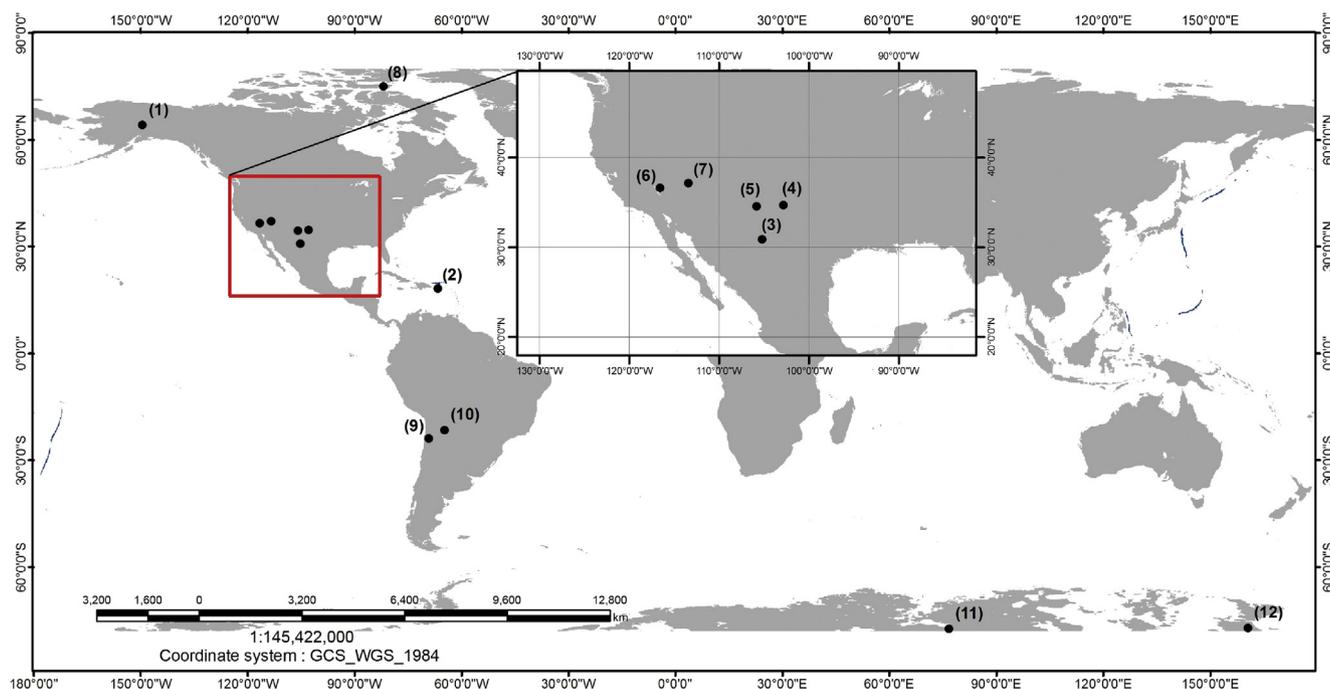
Most natural perchlorate occurrences are confined to arid and semiarid environments on Earth (Rajagopalan et al., 2006; Rao et al., 2007). In such environments, mobilization of perchlorate to groundwater is restricted by the net infiltration from precipitation. In addition, higher evapotranspiration rate in those environments

leads to higher accumulation of perchlorate compared to other places in the world (Albright et al., 2008). Table 1 shows several locations of geogenic perchlorate and their respective concentrations. The Atacama Desert in Chile is well known for its nitrate deposits, as well as one of the largest natural sources of perchlorate in the world. Photochemical reactions with oxidants such as O_3 , electrostatic discharge and gas–solid reactions with oxidants followed by dry deposition are potential mechanisms responsible for generating perchlorate in the Atacama (Catling et al., 2010; Michalski et al., 2004). Fertilizer mined from ores and brines in the Chilean deposits have been exported throughout the world for over a century. Perchlorate ranging from 0.03 to 0.1% and 6.3% nitrate are commonly present in these fertilizers (Duncan et al., 2005). In addition to the Chilean deposits, (a) New Mexico and Canadian potash, (b) Searles Lake, California, evaporative deposits, (c) Bolivian playa crusts, (c) West Texas and New Mexico surface deposits in semi-arid regions originating from atmospheric deposition and (d) marine deposits in the Mission Valley Formation, California, are other recognized terrestrial deposits of perchlorate present in significant concentrations (Duncan et al., 2005). Additionally, perchlorate can be present in precipitation because it can be formed naturally by atmospheric processes. The exact mechanism for the production of perchlorate is unknown. It suggests that chloride, probably in the form of sodium chloride from the ocean or land-based chloride aerosol blown in from atmosphere, expose to high concentration of ozone. Consequently, photochemical reactions between chloride and ozone make perchlorate in the atmosphere (Trumpolt et al., 2005). Additionally, another theory suggests that lightning involves in the production of perchlorate atmospherically (Dasgupta et al., 2005). Following the atmospheric production, perchlorate returns to the earth's surface via deposition. A study by Dasgupta et al. (2005) demonstrated that perchlorate was present in many rain and snow samples in southern high plains (Texas Panhandle). Recently, considerable amounts of perchlorate ($10\text{--}340\text{ ng L}^{-1}$) has been detected in Antarctic snow and ice potentially related to atmospheric deposition (Jiang et al., 2013). Fig. 1 shows identified geogenic perchlorate occurrences in the world so far. In contrast to geogenic sources, perchlorate may be released and/or introduced into the environment at an even greater extent due to anthropogenic activities.

Anthropogenic sources of environmental perchlorate contamination results from the heavy use of perchlorate-containing products. As a result of its strong oxidative properties, perchlorates were used for explosives and propellants extensively since World War II. In the USA, about 90% of ammonium perchlorate synthesized is used for the military purposes (Duncan et al., 2005). Solid rocket fuels, signal flares, colored and white smoke generators, artillery traces, incendiary delays and railway torpedoes are a few examples pertaining to military sources of perchlorates (Backus et al., 2005; Wilkin et al., 2007). Additionally, a wide range of industrial applications produces and uses perchlorates. Gas drying agents, lubricating oils, tanning, finished leather, electronic tubes, fabric fixes, dyes, cloud seeding, electroplating, aluminum refining, signal and road flares, rubber manufacture, paint and enamel production, cattle feeds and magnesium batteries are some of the industrial applications of perchlorates (Backus et al., 2005; Cheng et al., 2004; Dean et al., 2004). As it well known, perchlorate from rocket fuel production facilities in Henderson, Nevada contaminated Lake Mead and the Colorado River and the water imported to California from the Colorado River. This big anthropogenic occurrence of perchlorate contamination led to perchlorate being put on the USEPA CCL (EPA, 2014). Table 2 provides several examples related to the anthropogenic distribution of perchlorate and their respective concentrations.

Table 1
Geogenic distribution of perchlorate and their respective concentrations.

Location	Concentration	Medium	Reference
Alaska and Puerto Rico, USA	<5–102 ng L ⁻¹	Wet deposition	Rajagopalan et al. (2008)
Texas, New Mexico, Nevada and Utah, USA	1.6–13 µg kg ⁻¹	Soil	Rao et al. (2007)
Atacama Desert, Northern Chile	290–2565 µg kg ⁻¹	Soil	Calderón et al. (2014)
Bolivian playa crusts, Bolivia	500 mg kg ⁻¹	Salt	Duncan et al. (2005)
Atacama Desert, Northern Chile	744–1480 µg L ⁻¹	Surface water	Calderón et al. (2014)
Atacama Desert, Chile	1–10 mg L ⁻¹	Surface water	Duncan et al. (2005)
Great Lake, USA	0.05–0.13 mg L ⁻¹	Surface water	Poghosyan et al. (2014)
Texas and New Mexico, USA	<4 µg L ⁻¹ to 200 µg L ⁻¹	Groundwater	Rajagopalan et al. (2006)
Texas, USA	8–21 µg L ⁻¹	Groundwater	Scanlon et al. (2008)
Middle Rio Grande Basin, North–central New Mexico, USA	0.12–1.8 µg L ⁻¹	Groundwater	Plummer et al. (2006)
Devon Island ice cap, Canada	1–18 ng L ⁻¹	Snow	Furdui and Tomassini (2010)
Dome A region, Antarctica	10–340 ng L ⁻¹	Snow and ice	Jiang et al. (2013)
Antarctic Dry Valleys	1100 µg kg ⁻¹	Soil and ice	Kounaves et al. (2010)



Note: 1. Alaska, USA 2. Puerto Rico, USA 3. Chihuahuan Desert, Texas, USA 4. High Plains, Texas, USA 5. New Mexico, USA 6. Amargosa Desert, Nevada, USA 7. Sand Hollow, Utah, USA 8. Devon Island ice cap, Canada 9. Atacama Desert, Chile 10. Bolivian playa crusts, Bolivia 11. Dome A region in Antarctica 12. Beacon Valley in Antarctica

Fig. 1. Geogenic perchlorate occurrences in the world.

3. Perchlorate: as a chemical hazard

In 1896, it was first observed that fertilizers containing sodium and potassium salts of perchlorate negatively affected certain agricultural crops (Nzengungu et al., 1999). Perchlorate was initially recognized as a chemical of concern in 1985 due to its detection in wells at industrial sites in California, USA (Rice et al., 2007). With the development of more sensitive analytical methods for detecting perchlorate since 1997, testing and identifying perchlorate in ground and surface waters, soil and food were begun in earnest (Rice et al., 2007). Fig. 2 illustrates several possible transformation pathways of perchlorate in the environment and how humans may be exposed to this contaminant. By considering perchlorate as a critical chemical hazard to human, RfD for perchlorate exposure from both food and water is set at 0.7 µg kg⁻¹ body weight/day which translates to a drinking water level of 24.5 µg L⁻¹ by USEPA

(NRC, 2005; Rice et al., 2007). Additionally, maximum contaminant levels in drinking water have been established for perchlorate in different states in USA (i.e., California, 6 µg L⁻¹; Massachusetts, 2 µg L⁻¹) (EPA, 2014).

3.1. Perchlorate in soil

Perchlorate present in soils may represent a major source of perchlorate to groundwater. Wide variety of human applications including fireworks, explosives, stick matches, highway safety flares and military operations cause anthropogenic contamination of perchlorate in soil. For example, Ye et al. (2013) demonstrated that status of concentrations of perchlorate were high in surface soil due to fireworks and firecrackers during spring festival and consequently, particulate matter containing perchlorate disperses into remote areas by wind. Additionally, sufficient precipitation

Table 2
Anthropogenic distribution of perchlorate and their respective concentrations.

Location	Concentration	Medium	Reference
Northern China	0.001	Soil	Gan et al. (2014)
Southern China	–216 mg kg ⁻¹	Soil	Gan et al. (2014)
Heilongjiang province, China	0.001	Soil	Gan et al. (2014)
Chengdu, China	–25.8 mg kg ⁻¹	Soil	Ye et al. (2013)
Tianjin, China	2.15–7.02 µg L ⁻¹	Indoor dust	Gan et al. (2015)
Gozo and Malta islands	0.11	Indoor dust	Gan et al. (2015)
Gozo and Malta islands	–38.8 mg kg ⁻¹	Indoor dust	Gan et al. (2015)
Athens, Greece	0.72	Dust fall	Vella et al. (2015)
Cartagena, Colombia	–119 mg kg ⁻¹	Indoor dust	Vella et al. (2015)
Kumamoto, Nagasaki, Fukuoka, Saitama and Saga, Japan	0.52	Indoor dust	Wan et al. (2015)
Jeddah, Saudi Arabia	–561 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
Hanoi, Thai Binh and Hungyen, Vietnam	0.79–53 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
Albany and New York, USA	0.08	Indoor dust	Wan et al. (2015)
Jinan, China	–4.67 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
Jinan, China	0.02	Indoor dust	Wan et al. (2015)
Tamil Nadu, West Bengal, Bihar, Maharashtra, Karnataka and Pondicherry, India	–104 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
All provinces, South Korea	0.11	Indoor dust	Wan et al. (2015)
Oklahoma, USA	–34.6 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
New York, USA	0.03	Indoor dust	Wan et al. (2015)
Nakdong watershed, South Korea	–1.18 mg kg ⁻¹	Indoor dust	Wan et al. (2015)
Usui River, Japan	0.14	Indoor dust	Wan et al. (2015)
Heilongjiang province, China	–125.14 ng m ⁻³	Outdoor dust	Wan et al. (2015)
South Korea	0.23	Outdoor dust	Wan et al. (2015)
Tianjin, China	–173.76 ng m ⁻³	Outdoor dust	Wan et al. (2015)
Heilongjiang province, China	0.02–6.9 µg L ⁻¹	Tap water, groundwater, bottled water, surface water and rain water	Kannan et al. (2009)
Heilongjiang province, China	1.0–6.1 µg L ⁻¹	Tap Water	Her et al. (2011)
Heilongjiang province, China	44.2 µg L ⁻¹	Surface water	Wilkin et al. (2007)
Heilongjiang province, China	0.11–519 µg L ⁻¹	Surface water	Wu et al. (2011)
Heilongjiang province, China	0.05–60 µg L ⁻¹	Surface water	Quinones et al. (2007)
Heilongjiang province, China	2300 µg L ⁻¹	Surface water	Kosaka et al. (2007)
Heilongjiang province, China	0.008–1.7 µg L ⁻¹	Groundwater	Ye et al. (2013)
Heilongjiang province, China	0.11–6.1 µg L ⁻¹	Sea water	Her et al. (2011)
Heilongjiang province, China	0.35	Rain water	Qin et al. (2014)
Heilongjiang province, China	–27.3 ng mL ⁻¹	Snow	Qin et al. (2014)
Heilongjiang province, China	0.02–0.15 µg L ⁻¹	Snow	Ye et al. (2013)

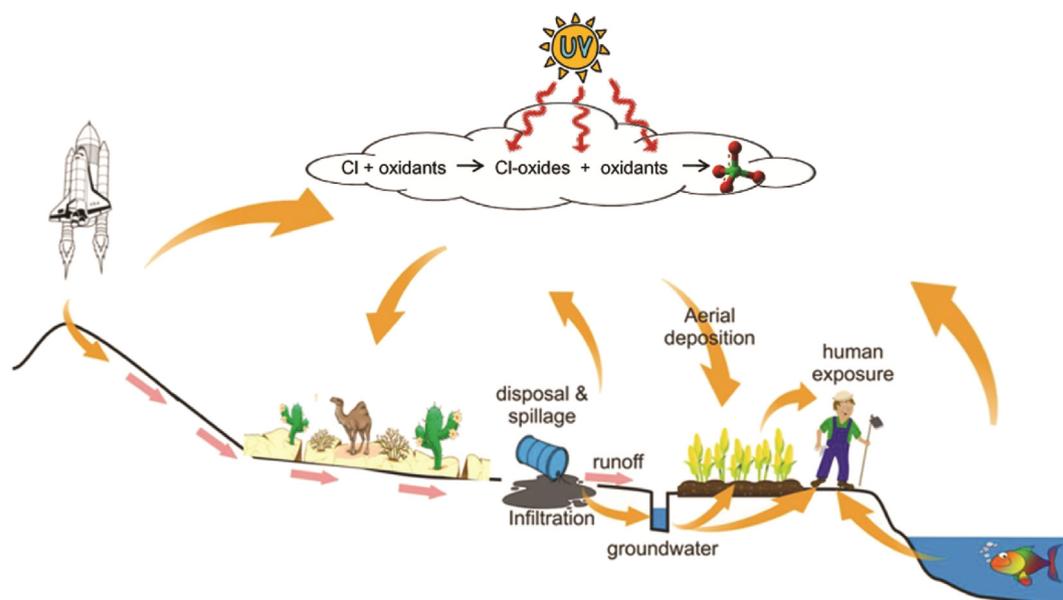


Fig. 2. Flow, transport and transformation pathways of perchlorate in the environment.

may transport this contaminant away from the source area in surface runoff. In general, perchlorate is not sorbed to soil particles (commonly negatively charged) due to electrostatic repulsion (Albright et al., 2008). Nevertheless, dissolved perchlorate can be trapped within soil pores due to capillary forces and surface tension (Albright et al., 2008). During transport through soil, perchlorate can be degraded biologically. At depth, organic matter generally decreases, thereby, potentially allowing perchlorate to readily infiltrate into the groundwater zone. The presence of clayey layers are capable of reducing perchlorate infiltration. For example, perchlorate infiltration was significantly reduced by a clayey layer in the vadose zone of an ammonium perchlorate manufacturing plant in Israel which operated for 25 years (Gal et al., 2009). Further they found higher perchlorate concentrations present above the clayey layer along the sediment profile (Gal et al., 2009). The distribution of perchlorate among soil horizons in the Mojave Desert, California, showed that the samples containing perchlorate from C, B and A horizons were generally >60, 20 and < 20%, respectively (Lybrand et al., 2013). Floodplain areas in Ashi and Songhua River, Harbin, China, contain thin silty clay and sandy loam soil with some of the sandy beds containing higher perchlorate concentrations compared to levels present in the river (Ye et al., 2013).

It has been reported that perchlorate concentrations in dust particles in Northern China ranged from 0.132 to 5300 mg kg⁻¹ (Gan et al., 2014). Nevertheless, the levels measured in their study did not lead to exposure above the USEPA RfD for both children and adults. In addition, high perchlorate levels have been recorded in soil collected from top soil horizon (0–3 cm) in China during the Chinese traditional spring festival and extensive amount of fireworks during this season is the reason why the soil perchlorate concentrations were high (Gan et al., 2014). Moreover, mean concentration of 34.7 µg g⁻¹ perchlorate was recorded in soil collected from top soil horizon in Las Vegas Wash, Nevada, USA (Smith et al., 2004). However, high perchlorate levels in soils and dust may pose a potential risk through bioaccumulation from the soil to terrestrial plants and groundwater and surface water contamination.

The presence of perchlorate in soil may aid in accelerated mineral dissolution, thereby, increasing potential toxic heavy and/or trace metals into the soil and groundwater. It has been observed that the rate of dissolution of minerals increases with increasing perchloric acid concentration in ulexite which is one of the most common boron-containing minerals (Demirkiran and Kunkul, 2007). The increased dissolution of Ni and Co in the presence of perchloric acid in laterite has been examined by Senanayake et al. (2011). Similarly, dissolution of Fe in goethite and hematite due to perchloric acid has been assessed (Majima et al., 1985). In this sense, most highly perchlorate contaminated sites may dissolve considerable amounts of heavy/trace metals and that could be a possible pathway to leaching metals to the surface and groundwater and subsequently, accumulated in living organisms via the food chain.

3.2. Perchlorate in water

Groundwater, surface water and drinking water have been contaminated by perchlorate, mainly due to military operations, fireworks and industrial activities. Furthermore, because of higher water solubility of perchlorate in water, sufficient rainfall may transport this contaminant away from the source area. According to several studies (Briggs, 2006; Dean et al., 2004), industrialized countries such as USA have reported considerable levels of perchlorate in surface water and groundwater. It is reported that surface water in New York and Oklahoma have been seriously affected due to the anthropogenic activities (Wan et al., 2015; Wilkin et al., 2007; Wu et al., 2011). Additionally, Longhorn army

ammunition plant east central Texas, USA, which facilitated perchlorate containing propellants and rocket motors have detected 31.43 mg L⁻¹ of perchlorate in a water treatment holding pond (Smith et al., 2001). Perchlorate levels in water supplies in northern and southern California and Las Vegas have recorded concentrations as high as 280 µg L⁻¹ (Nzengung et al., 1999). In the Nakdong River watershed, South Korea, a large liquid-crystal display monitor manufacturing plant may be responsible for high concentrations of perchlorate in tap water (from <1.0 to 6.1 µg L⁻¹) due to perchlorate being used as a cleaning agent (Her et al., 2011). Additionally, perchlorate in locally bottled water have reported higher levels (0.04–0.29 µg L⁻¹) suggesting that drinking water sources including surface water and groundwater have been contaminated (Her et al., 2011). In the same study, high perchlorate concentrations (0.11–6.11 µg L⁻¹) in seawater samples collected within 100 m of the shore in South Korea may be the result of coastal runoff (Her et al., 2011).

Seasonal variation of perchlorate levels in groundwater in Harbin, North China, has been studied in detail (Ye et al., 2013). During the summer, perchlorate concentrations ranged from <0.008 to 1.7 µg L⁻¹. In the spring, perchlorate increased from 0.1 to 1.72 µg L⁻¹. The reason for this increase may be related to melting snow dissolving perchlorate in surface soils followed by its percolation into groundwater (Ye et al., 2013). Time-dependent concentrations of perchlorate in a municipal lake in Ada, Oklahoma, USA, after firework festivities have been studied by Wilkin et al. (2007). In this study, the maximum perchlorate concentration was reported at 44 µg L⁻¹ following the fireworks. Within 20–80 days, perchlorate concentrations decreased to background levels, suggesting that natural attenuation, particularly by microorganisms could be a vital factor affecting the reduction of perchlorate following fireworks displays.

The possibility of naturally occurring perchlorate in water has recently gained significant attention (Fram and Belitz, 2011; Plummer et al., 2006). Perchlorate detected in Great Lakes (Lake Ontario, Superior and Michigan), USA, is dominantly non-anthropogenic (Poghosyan et al., 2014). A study by Parker et al. (2008) demonstrated that perchlorate occurred naturally in many groundwaters in the coterminous USA (109 samples contained <1000 ng L⁻¹ while 28 samples contained 1000–10,400 ng L⁻¹ out of 347 water samples analyzed). Additionally, in California, USA, groundwater samples were used to investigate the distribution of perchlorate in deep groundwater under natural conditions. The results revealed that low concentrations (0.1–0.5 µg L⁻¹) of perchlorate existed under natural conditions across a wide range of climates. Whereas, in semiarid regions of California, the probability of detection perchlorate at level greater than 0.1 µg L⁻¹ under natural conditions ranged between 50 and 70% (Fram and Belitz, 2011).

3.3. Perchlorate in food

Agriculture as well as livestock products can be contaminated by perchlorate due to the use of soil and irrigation water containing perchlorate (Kim et al., 2014). Recent investigations have revealed that various concentrations of perchlorate are detected in different food varieties such as dairy products, beverages, meats, fruit and vegetables (see Table 3)

It was observed that surface water perchlorate contamination was significantly correlated to the perchlorate concentration in a variety of different foods. Some edible plant species may contain the highest accumulation of perchlorate compared to other plant species examined. In general, higher leaf surface areas lead to higher transpiration rates in plants which may results in higher uptake of perchlorate by edible plant species (Yang and Her, 2011).

Table 3
Perchlorate concentrations in various food varieties in worldwide.

Location	Food variety	Perchlorate concentration ($\mu\text{g kg}^{-1}$)	Reference
Nakdong River watershed, South Korea	Rice	0.38–3.23	Kim et al. (2014)
Ottawa, Canada	Guatemalan cantaloupes	156 \pm 232	Wang et al. (2009b)
	United States spinach	133 \pm 24.9	
	Chilean green grapes	45.5 \pm 13.3	
	USA Romaine lettuce	29.1 \pm 10.5	
Seven provinces, South Korea	Soybean sprouts (<i>Glycine max</i> L. Merr)	35.2	Yang and Her (2011)
	Water dropwort (<i>Oenanthe stolonifera</i> DC.)	20.7	
Busan, South Korea	Spinach	39.9	Lee et al. (2012)
	Tomato	19.8	
	Ham and sausage	7.31	
	Instant noodles	7.58	
	Fishes, meats and beverages	<2	
Texas, USA	Wheat	982–3924	Sanchez et al. (2006)
Texas, USA	Fresh milk	1.7–6.4 ^a	Kirk et al. (2003)
USA	Chicken eggs	7.16 \pm 1.99	Blount et al. (2008)
Eight provinces, South Korea	Dairy milk	1.99–6.41	Her et al. (2010)
	Milk-based powdered infant	1.49–33.3	
Hatay, Turkey	Fish	0.38–0.61	Sungur and Sangun (2011)
	Milk	0.30–0.94	
Beijing, China	Milk	0.30–9.1 ^a	Shi et al. (2007)
Southwestern USA	Dairy milk	0.9–10.3 ^a	Sanchez et al. (2008)
Boston, USA	Beast milk	1.3–411 ^a	Pearce et al. (2007)
Twenty six provinces, China	Rice	0.16–4.88	Shi et al. (2007)

^a $\mu\text{g L}^{-1}$.

However, different factors such as species type, plant maturity, availability of other nutrients and ions in medium and transpiration efficiency may govern the perchlorate uptake into plants (Smith et al., 2004). Perchlorate accumulation in some edible plant species such as soybean sprouts (*Glycine max* L. Merr.), water dropwort (*Oenanthe stolonifera* DC.) and lotus (*Nelumbo nucifera* Gaertn.) in South Korea has been studied and the maximum perchlorate concentrations accumulated in soybean sprouts, water dropwort and lotus were 78.4, 39.9 and 17.3 $\mu\text{g kg}^{-1}$ dry weight, respectively (Yang and Her, 2011).

The correlation coefficient between perchlorate levels in rice samples from the Nakdong watershed, Republic of Korea, and the levels in surface water was 0.904 in the 95% confidence interval (Kim et al., 2014). A total of 39 kinds of different food groups collected from Busan, Republic of Korea, revealed that dairy products contain a high average perchlorate concentration (6.34 $\mu\text{g kg}^{-1}$) while fruit and vegetables showed the next highest average perchlorate concentration (6.17 $\mu\text{g kg}^{-1}$). According to the results, perchlorate exposure to Korean adults was 0.04 $\mu\text{g kg}^{-1}$ body weight/day (Lee et al., 2012). In a Canadian study, dietary exposure to perchlorate from analyzed fruits and vegetables in an Ottawa market was about 36.6 and 41.1 ng kg^{-1} body weight/day for toddlers (1–4 years) and children (5–11 years), respectively (Wang et al., 2009b). Even though, dietary exposure to perchlorate is lower than the RFD in most occasions, perchlorate accumulation via the food chain cannot be simply neglected. For example, detection of perchlorate in human breast milk samples has revealed the possibility of perchlorate transformation and mobilization in the food chain (Kirk et al., 2005).

4. Health impact to the living organisms

According to reported studies, perchlorate has been found in a variety of human body fluids including urine, breast milk, saliva and blood (Dasgupta et al., 2006). Perchlorate exposure to humans and other aquatic and terrestrial wildlife has mainly occurred due to the ingestion of perchlorate contaminated water and food (Smith et al., 2006). For instance, aquatic organisms such as aquatic insects, fishes, frogs and mammals living in perchlorate contaminated

water bodies have contained measurable concentrations of perchlorate, 811–2038, 207, 580 and 2328 $\mu\text{g L}^{-1}$ of perchlorate, respectively (Smith et al., 2001). Many wildlife species are considered to be at higher risk for perchlorate exposure since soil can comprise a significant proportion of perchlorate of their diet. In one study, it was indicated that perchlorate in rodent tissues was highly correlated with soil perchlorate concentrations than vegetation in Las Vegas Wash, Nevada, USA (Smith et al., 2004). Additionally, ingestion of plants irrigated with perchlorate contaminated water acts as another route to exposure, but at a lesser extent compared to direct ingestion. Moreover, trophic transfer may be considered as an indirect pathway to perchlorate exposure to organisms (Bansal et al., 2009; Dasgupta et al., 2006).

As it well known, perchlorate is rapidly adsorbed and can enter from the stomach and intestines to the bloodstream. Zhang et al. (2010) investigated the perchlorate level in human blood samples from Chinese donors aged 0.4–90 year in Nanchang. The results show that mean perchlorate concentration in blood was 2.68 $\mu\text{g L}^{-1}$ and mean exposure doses of perchlorate for adults and infants were 1.12 and 2.22 $\mu\text{g kg}^{-1}$ body weight/day, respectively, which exceed the USEPA RFD, 0.7 $\mu\text{g kg}^{-1}$ body weight/day. The thyroid gland is the major target of perchlorate toxicity in human beings. Perchlorate ion is known to interfere with iodine ion uptake by the thyroid gland. It is obvious that, both perchlorate anions and iodide ions have same ionic charge and approximately the same ionic radius. As a result, ingestion of perchlorate is capable of inhibiting iodide uptake by the sodium iodide symporter (NIS) of the thyroid follicle cells (Tietge et al., 2005). This uptake results in the reduction of thyroid hormone production. Adequate iodine intake to produce thyroid hormones is particularly important for normal growth, brain development and metabolic activities especially in developing infants and children (Goleman and Carr, 2006). In this respect, elicit development and neurobehavioral problems in infant and children have been reported (Bansal et al., 2009; Leung et al., 2010).

Several acute toxicity studies are reported on a variety of vertebrates with the presence of environmentally relevant concentrations of perchlorate (Bernhardt et al., 2006, 2011; Mukhi et al., 2007). It is believed that many biological processes (i.e.,

reproduction, ovarian maturation and oogenesis, temperature tolerance, formation of the stomach, muscular development) in fish are governed at least partially due to thyroid hormones (Blanton and Specker, 2007; Brown et al., 2004). A study by Goleman et al. (2002) investigated that sex ratios in post metamorphosed *Xenopus laevis* were altered towards females due to perchlorate exposure. Similarly, Mukhi et al. (2007) accessed that exposure of perchlorate to zebrafish (*Danio rerio*) produces a female-biased sex ratio during the larval-juvenile development. Consequently, they have found that perchlorate exposure with supplemental thyroid hormones made a male-biased sex ratio suggesting that thyroid hormone levels act major role in the sexual development. Further, perchlorate exposure was found to produce both functional sperm and eggs in masculinized genotypic female threespine stickleback (*Gasterosteus aculeatus*) (Bernhardt et al., 2006). Ingestion of perchlorate to northern bobwhite quail (*Colinus virginianus*) caused alteration of thyroid gland morphology as well as significant accumulation of perchlorate in the eggs and livers (Gentles et al., 2005). However, it is obvious that further investigations are required to clarify the possible health effects of environmental perchlorate exposure in humans in addition to the thyroid dysfunction.

5. Identification and measurement of perchlorate in the environment

Following the USEPA recognition of perchlorate as a contaminant in the environment, analytical methods for determining perchlorate at trace levels have advanced. The development of analytic methods for determining perchlorate is challenging because most perchlorate is present at low concentrations, commonly at a few $\mu\text{g L}^{-1}$ in the environment.

The USEPA has developed methods to detect perchlorate based on selectivity, sensitivity, precision, accuracy and method robustness (Wagner et al., 2007). After the USEPA established Method 314.0 for analyzing perchlorate (Wagner et al., 2006), numerous standard methods (i.e., 314.1, 314.2, 331.0 and 332.0) have been established which are capable of detecting perchlorate concentrations in the ng L^{-1} range. Table 4 summarizes the different EPA methods developed for determining perchlorate in water.

The most widely used analytical technique for determining of oxyhalide anions including perchlorate is ion chromatography with conductivity detection (IC-CD). Nevertheless, conductivity detection has low specificity since many ionic compounds in addition to the targeted compound/s would be detected (Snyder et al., 2005). For instance, common anions such as sulfate, carbonate and chloride make very high ionic strength matrices leading to accurate quantification of perchlorate challenging (Wagner et al., 2006; Wendelken et al., 2006). To avoid such difficulties and obtain concentrated solutions of perchlorate, preconcentration methods

such as solid-phase extraction (SPE) and ion exchange columns can be used prior to analysis (Magnuson et al., 2000). Wagner et al. (2006) have used preconcentration technique by using cryptand column to minimize the possibility of any interference, which co-elute with the perchlorate ion. Furthermore, two-dimensional ion chromatography (2D-IC) method, which includes column concentration and matrix elimination techniques, can be used for preconcentration of perchlorate. In the first dimension, matrix ions are diverted to waste and in the second dimension, separation and quantification of perchlorate ion are occurred (Wagner et al., 2007). This method has gained high sensitivity and selectivity for perchlorate ion determination and is simpler and more economically viable than ion chromatography mass spectrometry (IC-MS) or IC-MS-MS techniques (Wagner et al., 2007).

Mass spectrometry, commonly in tandem with ion chromatography (IC-MS) of liquid chromatography (LC-MS) can be used for the detection of perchlorate anion (Snyder et al., 2005). As a result of commercially available IC-MS systems, the utilization of mass spectrometry for the detection of perchlorate ion has been significantly increased. In general, chromatographic technique is in a position to separate multiple anions in a single chromatographic analysis. Nevertheless, non targeted species could be interfering with accurate quantification of perchlorate (MacMillan et al., 2007). Hence, by using IC-MS or LC-MS techniques, accurate quantification of perchlorate ion is further enhanced.

Moreover, each analytical method has limitations and advantages relative to each other. For instance, limitations/disadvantages of ion chromatography methods are related to being time consuming, needing sample pre-preparation and not being field deployable (Mosier-Boss and Putnam, 2014). Further, sample preparation prior to analysis is done in different ways depending on the sample type. Rinsing the soil with deionized water and analyzing the resulting extract is sufficient for quantitative determination of perchlorate in soil (MacMillan et al., 2007). Methods for increasing the relatively high recovery of perchlorate in biological samples such as urine from cow, vole and human have been successfully investigated (Cheng et al., 2006). Overall, it is obvious that each analytical method involves a specific detection of perchlorate in environment. Hence, assessment of perchlorate contamination at sub- ng mL^{-1} levels in environmental and biological samples can be accurately measured.

6. Remediation of perchlorate

Numerous physico-chemical and biological processes are available for the remediation of perchlorate in contaminated environments (Table 5). Under most environmental conditions, the stability of perchlorate makes remediation difficult.

Table 4

Summary of detection methods used for determining perchlorate concentration in water (Adopted from Karimi and Rezaee (2014)).

EPA method no.	Method	Minimum detection limit ($\mu\text{g L}^{-1}$)	Average recovery (%) ^a
314.0	Ion chromatography with conductivity detection	0.53	86–113
314.1	Inline column concentration/matrix elimination ion chromatography with suppressed conductivity detection	~0.03	75.9–108
314.2	Two - dimensional ion chromatography with suppressed conductivity detection	0.012–0.018	92–110
331.0	Liquid chromatography with electrospray ionization mass spectrometry	0.008 (Single stage mass spectrometry) 0.005 (Tandam mass spectrometry)	95.1–105
332.0	Ion chromatography with suppressed conductivity and electrospray ionization mass spectrometry	0.02	90–105

^a Average recovery range is reported depending on the matrix.

Table 5
Different remediation methods used to remove perchlorate from the environment.

Remediation method	Perchlorate removal efficiency/remarks	Reference
Bio-reduction	Accumulation of perchlorate into six plant species was between 0 and 981 mg kg ⁻¹ of plant weight Chloride dismutase enzyme reduced perchlorate with an optimum pH 6.0	Susarla et al. (2000) Nadaraja et al. (2013)
Chemical reduction	Nanoscale iron particles removed 90% of perchlorate at 75 °C Zero valent iron reduced 99% of perchlorate at 99 °C	Cao et al. (2005) Oh (2010)
Adsorption	Maximum adsorption capacity of modified reed (particles ranging in size from 100 to 250 μm) was 169 mg g ⁻¹ Maximum adsorption capacity of the cross-linked chitosan was 45.45 mg g ⁻¹ at 25 °C Maximum adsorption capacity of cross-linked Fe(III)-chitosan composite was 29.85 mg g ⁻¹ at 25 °C	Baidas et al. (2011) Xie et al. (2010) Lv et al. (2014)
Membrane filtration	Polyelectrolyte-enhanced ultrafiltration removed >95% of perchlorate Reverse osmosis membrane removed >70% of perchlorate	Roach and Tush (2008) Yoon et al. (2009)
Ion exchange	Ion exchange capacity of the five cationic resins were ranged from 115 to 183 mg g ⁻¹ Adsorption capacity of the calyx[4] arene based Amberlite XAD-4 synthesis resin was 11.35 mmol g ⁻¹	Darracq et al. (2014) Memon et al. (2013)
Electrochemical reduction	Ti electrode removed 90% of perchlorate within 8 h	Wang et al. (2009a)

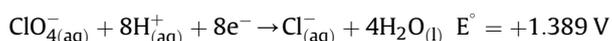
6.1. Bio-reduction

Biological treatment methods are eco-friendly and an efficient technological solution for removing perchlorate. Some bacteria produce two cell-bound enzymes, perchlorate reductase and chlorite dismutase, which are able to minimize the activation energy needed for perchlorate reduction. Microbial reduction of perchlorate progresses in the following steps: $\text{ClO}_{4(\text{aq})}^- \rightarrow \text{ClO}_{3(\text{aq})}^- \rightarrow \text{ClO}_{2(\text{aq})}^- \rightarrow \text{Cl}_{(\text{aq})}^- + \text{O}_{2(\text{g})}$. Perchlorate reductase catalyzes reduction of $\text{ClO}_{4(\text{aq})}^- \rightarrow \text{ClO}_{3(\text{aq})}^- \rightarrow \text{ClO}_{2(\text{aq})}^-$ while chlorite dismutase catalyzes $\text{ClO}_{2(\text{aq})}^- \rightarrow \text{Cl}_{(\text{aq})}^- + \text{O}_{2(\text{g})}$ (Nadaraja et al., 2013; Ye et al., 2012). Perchlorate-degrading bacteria such as *Dechloromonas* and *Azospira* species have been successfully used in bioreactors for remediating perchlorate (Raj and Muruganandam, 2012).

Some terrestrial and aquatic plant species are capable of removing perchlorate from the environment. Perchlorate reduction pathways in plants are similar to that in microbes. Two different processes, phytodegradation and rhizodegradation, can be involved in removing perchlorate. In phytodegradation, perchlorate is moved from root zone to the aboveground portion of the plant where the perchlorate ion is converted to chloride. Perchlorate-respiring bacteria are capable of rapidly degrading perchlorate to chloride by utilizing root exudates and soil organic carbon as an electron source, this involves rhizodegradation (Yifru and Nzengung, 2008). It is reported that some vascular plant species such as sweet gum (*Liquidambar styraciflua*), black willow (*Salix nigra*), pickleweed (*Allenrolfea occidentalis*), smartweed (*Polygonum punctatum*), water-lily (*Nymphaea odorata*) and duckmeat (*Spirodela polyrrhiza*) can be used for phytoremediation of perchlorate since external signs of perchlorate toxicity were not observed in such plant species even at high concentrations (Susarla et al., 2000). Therefore it is obvious that, bio-reduction involves successful removal of perchlorate. Additionally, this technology is more suitable for large scale wastewater treatment plants. However, careful attention is required to the solve problems of public acceptance since microorganisms are involved during treatment process.

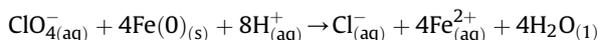
6.2. Chemical reduction

From a thermodynamic perspective, perchlorate is unstable and may result in chloride at ambient conditions as shown below.



The stability of the chlorine-oxygen bond is responsible for the high activation energy barrier that may lead to the reduction of perchlorate. As a result, perchlorate is relatively unreactive with regards to most reducing agents. Complexes of Sn (II), Ti(III), V (II

and III) and Ru (II) are able to reduce perchlorate very slowly (Earley and Kallen, 1971). However, strong reducing agents such as zero-valent iron (Fe(0)) have been successfully used for remediation of perchlorate in water shown in the reaction below (Cao et al., 2005).



Oh (2010) determined that 99% of perchlorate was removed at 99 °C and pH 4.7 with 0.6 M acetate buffer. In this study, moderately high temperatures (<100 °C) and buffer control devices were found to enhance perchlorate removal. Additionally, nanoscale zero-valent iron (10 mg L⁻¹) have successfully removed perchlorate at a rate of 0.013, 0.1, 0.64, and 1.52 mg g⁻¹ hr⁻¹ with the presence of different temperatures such as 25, 40, 60 and 75°C, respectively (Cao et al., 2005). In summary, chemical reduction methods involve a large barrier for perchlorate reduction. Additionally, residual iron will be resulted in treated medium, hence further treatment may require for removing iron.

6.3. Adsorption

Different kinds of adsorbents have been developed to remove perchlorate from solution. In general, the structural characteristics of adsorbent and solution pH greatly influence the adsorption capability of a particular adsorbent. Granular activated carbon has been widely used in water treatment process in the USA to adsorb perchlorate. It has been observed that activated carbon improves the adsorption of perchlorate (Ye et al., 2012). However, surface modification is an important factor to improve the adsorption capacity of the granular activated carbon.

In recent years, researchers found some other modified adsorbents to remove perchlorate from the aqueous solutions. Granular ferric hydroxide has successfully used to adsorb perchlorate and equilibrium was achieved within 60 min. The maximum adsorption capacity of granular ferric hydroxide was 20 mg g⁻¹ at pH 6.0–6.5 at 25 °C. Optimum removal occurred in the pH range of 3–7. Baidas et al. (2011) evaluated the efficiency of giant reed modified by quaternary amine functional groups for perchlorate removal from aqueous medium. The maximum adsorption was completed within the first minute and equilibrium was reached within 7 min. The maximum adsorption capacity of modified reed (particles ranging in size from 100 to 250 μm) was 169 mg g⁻¹. The optimum perchlorate removal was achieved in the pH range 3.5–7.0. Similarly, adsorption of perchlorate using acid-washed zero-valent aluminum and aluminum hydroxide was examined in batch reactors. Approximately 90–95% perchlorate removal was achieved within 24 h at acidic pH (4.5) (Lien et al., 2010). Even though aluminum is a strong reductant, it was observed that an adsorption process governs the perchlorate removal. Further, it was

suggested that ion-pair formation at the aluminum hydroxide surface involves removal of perchlorate (Lien et al., 2010). A study by Lv et al. (2014) revealed that cross-linked Fe(III)-chitosan composite can be effectively used for removing perchlorate from the aqueous solution. It was observed rapid adsorption in the 10 min and approached equilibrium within 30 min of reaction time. Maximum adsorption capacity of cross-linked Fe(III)-chitosan composite was 29.85 mg g^{-1} at 25°C and adsorption process exhibited almost independent of pH ranging from 3.0 to 10.2. In another example, protonated cross-linked chitosan removed perchlorate at optimum pH value of 4.0. The maximum adsorption capacity of the adsorbent was 45.45 mg g^{-1} at 25°C and the presence of other anions such as sulfate weakened the adsorption process (Xie et al., 2010). The adsorption properties of calcined layered double hydroxide were examined by Wu et al. (2010). The maximum adsorption capacity was achieved at a calcined temperature of 500°C with Zn/Al ratio of 2. In addition, adsorption process was followed to be endothermic in nature. Recently, far wood derived biochar produced at $500\text{--}700^\circ\text{C}$ successfully adsorbed perchlorate (Fang et al., 2013). In addition to the electrostatic attractions, H-bond interactions between weakly hydrated ClO_4^- and oxygen-containing functional groups such as $-\text{COH}$, $-\text{COOH}$ on aromatic and hydrophobic surface of biochar have played a major role with regards to adsorption (Fang et al., 2013). Furthermore, maximum adsorption was reported near pH of isoelectric point (pH_{IEP}) suggesting that adsorption capacity (10.55 mg g^{-1}) is highly affected by solution pH (Fang et al., 2013). Overall, electrostatic attraction seems to govern the adsorption of perchlorate in many adsorbents (Baidas et al., 2011; Kumar et al., 2010; Lv et al., 2014). Nevertheless, surface polarities and structural compositions of the organic components like biochar are able to adsorb perchlorate via hydrogen bonding (Fang et al., 2013). In summary, temperature, pH, chemical composition of the adsorbents and coexisting anions in the medium may influence the adsorption capacity of a particular adsorbent when it is applied into the real environment. Therefore, further researches are still needed to investigate environmental applicability of those modified adsorbents.

6.4. Membrane filtration

Pressure driven membrane filtration by using reverse osmosis, nanofiltration and ultrafiltration is considered a promising technology for removing perchlorate. Both size exclusion and electrostatic exclusion are the prominent pathways for removing inorganic contaminants in such a membrane. In addition, solution pH and conductivity governs the degree of rejection by influencing the membrane charge. Yoon et al. (2009) studied the performance of different membrane types such as reverse osmosis (LFC-1), nanofiltration (ESNA and MX07) and ultrafiltration (UF and GM) in the presence of chromate, arsenate and perchlorate solutions. This study observed that perchlorate removal is directly proportional to the pH and indirectly proportional to the conductivity. Furthermore, when the ratio of solute radius to the effective membrane pore radius is greater than 0.4, perchlorate removal was higher than 70% in the reverse osmosis membrane. Polyelectrolyte-enhanced ultrafiltration has been successfully separated greater than 95% of perchlorate even in the presence of 10-fold excesses of competing ions such as chloride, sulfate and carbonate (Roach and Tush, 2008). A hybrid/ultrafiltration process using chitosan as an adsorbent for removing perchlorate was able to reach its equilibrium within 3 min and electrostatic attraction between perchlorate and positively charged- NH_3^+ was the prominent pathway for removing perchlorate (Darracq et al., 2014; Xie et al., 2011). However, membrane filtration technologies lead to demineralization of water

since the removal process is non-selective. In addition, this process is subject to scaling and fouling which need maintenance or replacement.

6.5. Ion exchange

Ion exchange resins appear to the most effective to eliminate perchlorate present at trace level ($<50 \mu\text{g L}^{-1}$) in water (Darracq et al., 2014). The exchange capacity, stability, selectivity and regeneration capacity of a particular resin will affect the removal efficiency. Darracq et al. (2014) investigated the performance of five different resins (A532E, A520E, A400E, PWA-5 and PSR-2) to remove the perchlorate. Among them, A532E and PSR-2 resins demonstrated the most efficient elimination of traces of perchlorate. In addition, calyx[4] arene based Amberlite XAD-4 synthesis resin has successfully removed perchlorate where the maximum adsorption of perchlorate was recorded at pH 4.5 (Memon et al., 2013). However, this ion exchange technology is not economically viable as a result of its high cost and disposal after a single use (Ye et al., 2012). Additionally, coexisting anions may affect the available sites on a particular resin and consequently adsorption capacity of perchlorate will be reduced.

6.6. Electrochemical reduction

Electrochemical reduction of perchlorate results in the nontoxic chloride ion (Logan, 2001). Electrodes such as Ti, Rh, Pt, Ir, Ru and Sn are capable of reducing perchlorate ions (Brown, 1986; Lang et al., 2008). Wang et al. (2009a) observed significant reduction of both perchlorate and nitrate in the presence of Ti anode. In that case, the initial concentration of perchlorate was reduced from 200 mg L^{-1} to $20 \mu\text{g L}^{-1}$ within 8 h. Significant removal of perchlorate to chloride has been observed in a cell with a Ni electrode and a Pt counter electrode using a concentrated HClO_4 solution (Rusanova et al., 2006). In another example, Lang et al. (2008) studied perchlorate reduction in acid media by using Rh electrode and kinetic investigation tools such as voltammetry, chronoamperometry and impedance spectroscopy. The main drawback observed of electro-reduction method is that a particular electrode is being eroded. In addition, most of the electrochemical reduction investigations were limited in the laboratory conditions and it seems obvious that more pilot scale research should be established in order to assessing the applicability of this method into the real environment.

7. Remarks

It is obvious that perchlorate is a persistent environmental contaminant of concern which influences number of problems to the various nations in the world. These problems include health effects and potential risks, regulatory standards and cleanup levels and treatment/remediation technologies. USEPA have developed analytical methods to determine perchlorate in groundwater, surface water, salt water and soil. Even though, primary pathways for human exposure to perchlorate are oral ingestion of drinking water and food, perchlorate contaminated indoor and outdoor dust are the another prevailing source of perchlorate. It is known that thyroid gland is the primary target of perchlorate toxicity in humans and subsequently, decreasing body weight, hypertrophy of the thyroid gland will be resulted. In spite of unique nature of the perchlorate anion, recent field and laboratory studies for effective removal of perchlorate-contaminated water to below USEPA established limits looks promising. In case of treating widespread contamination of perchlorate, biological systems appear to be the most economically viable and eco-friendly method. Additionally,

development of biochar using waste organic materials recently for adsorbing perchlorate is a sustainable way to mitigate perchlorate in the environment. Despite of high removal efficiency, other treatment methods including chemical reduction, membrane filtration, ion exchange and electrochemical reduction possess high treatment cost and require expertise knowledge to operate.

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