



# Regeneration of a perchlorate-exhausted highly selective ion exchange resin: Kinetics study of adsorption and desorption processes



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## ABSTRACT

Perchlorate is a stable and soluble ion that can last for decades in the environment and can cause a number of environmental and health issues. Perchlorate is commonly removed using highly-selective ion-exchange resins that are replaced after exhaustion and incinerated or disposed in a landfill since there is no viable regeneration method. The major limitations in regeneration of single use resins is achieving complete desorption of perchlorate. The sustainability of treatment processes for perchlorate contaminated water can be achieved by regenerating the exhausted resin. As the first step of resin bioregeneration study, research on the adsorption and desorption kinetics of the perchlorate ion from a strong base anion exchange resin was conducted. Different chemical reaction and diffusion models were analyzed using experimental data. Both adsorption and desorption experimental data were best described by the Pseudo-second order model with adsorption rate constants of  $2 \times 10^{-3} \pm 0.001$  (g/mg/min) and desorption rate constants of  $5 \times 10^{-2} \pm 0.01$  (g/mg/min). These results suggest that the rate limiting stage for adsorption and desorption of perchlorate ion into the resin can be chemisorption.

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## 1. Introduction

Perchlorate ( $\text{ClO}_4^-$ ) has been found in soil, surface and ground water in 49 states of the United States, the District of Columbia and Puerto Rico [1–3]. Perchlorate is manufactured to be used mainly in defense and aerospace sectors [1–3]. As well as other application such as highway safety flares, fireworks, pyrotechnics, explosives, common batteries, and automobile restraint systems [4]. Due to its high solubility in water and kinetic stability, perchlorate is very difficult to remove from water effectively and it may exist in the environment for many decades [1–4]. There are several environmental and health effects due to high concentrations of perchlorate in water. Perchlorate affects thyroid hormone function by reducing iodine uptake into the thyroid gland [5–9]. To limit the health risk, the US Environmental Protection agency (USEPA) issued an interim drinking water health advisory level of 15  $\mu\text{g/L}$  [7], the Office of Environmental Health Hazard Assessment in California set a Public Health Goal (PHG) of perchlorate in drinking water of 6  $\mu\text{g/L}$  [8], and Massachusetts has set a goal of 2  $\mu\text{g/L}$ . Health Canada has recommended a drinking water guidance of 6  $\mu\text{g/L}$  based on other agencies health risk assessments [9].

There are several technologies that can remove perchlorate contamination from drinking water including reverse osmosis, electrodialysis, nanofiltration, biological treatments, ion exchange treatments, and ion exchange membrane bioreactors (IEMB) [10–16]. Currently ion exchange (IX) is the most prevalent technology in use, due to its high removal efficiency, its simplicity and capability of handling relatively high flow rates with a small footprint [15–24].

The use of ion exchange resins to remove perchlorate for commercial applications began in 1999. The first commercial Calgon ISEP<sup>®</sup> system with a total capacity of 2500 gpm was installed to treat three wells in La Puente Valley Water District located in Southern California; the main goal of this system was to remove the perchlorate down to a concentration less than 4 ppb in the effluent. In 2002, another Calgon ISEP<sup>®</sup> system with a total capacity of 450 gpm was built to treat water in Kerr-McGee, Henderson, Nevada. In the last decade, several other ion exchange systems have been designed in order to remove perchlorate from drinking water. In the United States, most of these ion exchange systems for removal of perchlorate from water, are located in California and Arizona with capacities ranging from 24 gpm to 5000 gpm. The highest perchlorate influent concentration in the installed ion exchange systems was 200–300 ppm in Kerr-McGee, Henderson, Nevada. This 850 gpm system was replaced by a biological FBR in 2004 [19,21].

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**Nomenclature**

|          |   |              |  |
|----------|---|--------------|--|
| $a$      | Elovich's equation desorption constant (mg/g/min)                                       | $k_f$        | mass transfer coefficient (cm/min)   |
| $B$      | Boyd kinetic model parameter ( $B = \pi^2 D_i / r^2$ ) (1/min)                          | $K_L$        | equilibrium constants of Langmuir equation                                   |
| $C$      | adsorbate concentration in the solution or in the pores of the resin at time $t$ (mg/L) | $k_{p1}$     | pseudo-first order rate constant for the kinetic model ( $\text{min}^{-1}$ ) |
| $C_e$    | adsorbate or perchlorate equilibrium concentration in the solution (mg/L)               | $k_{p2}$     | pseudo-second order rate constant for the kinetic model (g/mg/min)           |
| $C_i$    | initial concentration of adsorbate or perchlorate (meq/L)                               | $M$          | mass of resin (g)  |
| $C_t$    | adsorbate concentration in the solution at time $t$ (mg/L)                              | $n$          | Freundlich adsorption intensity parameter (unitless)                         |
| $D$      | adsorbate diffusion coefficient within the porous media ( $\text{cm}^2/\text{min}$ )    | $q$          | adsorbate concentration in the resin (mg/g)                                  |
| $D_e^l$  | effective liquid film diffusion coefficient ( $\text{cm}^2/\text{min}$ )                | $q_e$        | adsorbate concentrations in the resin at equilibrium (mg/g)                  |
| $D_i$    | effective diffusion coefficient ( $\text{cm}^2/\text{min}$ )                            | $q_{ecal}$   | kinetic model parameter (mg/g)   |
| $F$      | fraction of adsorbate in the resin at time $t$ (unitless)                               | $Q_e$        | perchlorate equilibrium concentration in the resin (mg/g)                    |
| $F_o$    | Fourier number ( $F_o = D \cdot t / r^2$ ) (unitless)                                   | $q_o$        | initial concentration of adsorbate in the resin (mg/g)                       |
| $g$      | mass of dry resin (g)   | $q_t$        | adsorbate concentrations in the resin at time $t$ (mg/g)                     |
| $k$      | film diffusion mass transfer rate equation equilibrium constant ( $\text{min}^{-1}$ )   | $r$          | distance from the center of the particle (cm)                                |
| $K$      | intraparticle diffusion rate equation constant of adsorption ( $\text{min}^{-1}$ )      | $R^1$        | liquid film diffusion constant ( $\text{min}^{-1}$ )                         |
| $K_{d2}$ | pseudo-second order rate constant for the desorption kinetic model (g/mg/min)           | $r_o$        | particle radius (cm)   |
| $K_F$    | Freundlich adsorption capacity parameter (mg/g) ( $\text{L/mg}^{1/n}$ )                 | $t$          | time (min)   |
|          |   | $\alpha$     | Elovich's equation initial adsorption rate (mg/q/min)                        |
|          |   | $\alpha_L$   | equilibrium constants of Langmuir equation                                   |
|          |   | $\Delta r_o$ | thickness of the liquid film (cm)  |

Single use IX systems use highly selective strong base anionic (SBA) resins that bind perchlorate so tightly that is not feasible to regenerate them. However, the large number of bed volumes of water treated at very low perchlorate concentrations before breakthrough makes them an attractive choice [16,17,19]. The disadvantage of the single use IX resins relies in the management of spent resins which includes landfill and incineration. Landfill disposal is not widely used, incineration by thermal destruction eliminates perchlorate but also the costly IX resin after a single use which makes the process unsustainable.

In order to consider the IX process as a sustainable method for perchlorate removal from water, the regeneration of resin should be studied. Research has shown that a salt-tolerant perchlorate-reducing culture in direct contact with the resin is capable of degrading perchlorate to chloride and oxygen [22], but application of biological systems to drinking water treatment is restricted due to concerns about the health effects of bacteria and their by-products. Therefore, a modified process of indirect biological regeneration of the IX resin was proposed in which desorption and biological degradation of perchlorate were carried out simultaneously, but the culture and IX resin were separated by a membrane. The ability to model this process would allow for designing a system to regenerate the exhausted resin reducing the costs associated with the new resin and its disposal; which in turn, would ultimately constitute a significant development to convert the treatment of perchlorate contaminated water into a more sustainable operation. Ion exchange/ bioregeneration of the resin includes three main steps; adsorption of perchlorate on the resin, desorption and degradation of perchlorate by a bioregeneration process. Model development and kinetic analysis of the physical–chemical adsorption and desorption processes, are the main goals of this study.

Xiao et al. [23], presented the results of a numerical analysis of the biological regeneration of perchlorate laden IX resins Ionac-SR7 and Amberlite 996 in direct contact with the bacteria. The authors explained the use of the model by assuming that the resins have more than one type of binding site. This type of model implies

an experimental estimation of two kinetic constants but does not offer an explanation of the mechanisms controlling the mass transfer process. Thus, our study aimed at understanding the behavior of adsorption and desorption of perchlorate onto a SBA ion exchange resin commercially available. The main objectives of this study are estimating the resin operating capacity (breakthrough point), identifying the best adsorption and desorption reaction and diffusion model fitting the experimental data, and understanding the process efficiency when the resin is enclosed in the membrane compared to loose resin.

The results of this research along with the study of biodegradation of perchlorate in the aqueous phase provide enough information to model the resin bioregeneration process and design a commercial scale reactor [20,25].

## 2. Materials and methods

### 2.1. Resin

This research was performed using the commercially available ion exchange resin CalRes 2109 as provided by Calgon Carbon with the average beads size of  $0.77 \pm 0.06$  mm. This resin is classified as a macroporous styrene strong base anion (SBA) resin with N-tributyl amine functional group that is highly perchlorate selective.

### 2.2. Resin conditioning

The resin was conditioned following the procedure of Tripp and Clifford [22]. Approximately 100 mL of resin was placed in a 1 in. (i. d.) glass column. The resin was rinsed with deionized water for 2 h at a flow of 10 mL/min. Then resin was exposed to acid–base cycling consisting of pumping a 1 N solution of sodium hydroxide down-flow to convert the resin to the hydroxide form followed by rinsing and conversion of the resin to the chloride form by pumping a 1 N solution of hydrochloric acid followed by rinsing. This procedure was repeated for three cycles. For the final rinsing with

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