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Modeling geosmin removal in a full-scale filter

JULIANA A. COELHO, DIANA CRISTINA S. DE AZEVEDO,
IVANILDO JOSÉ DA SILVA JUNIOR & JOSÉ CAPELO-NETO

Abstract: Taste and odor compounds affect drinking water safety perception and may drive consumers to less secure water sources. Adsorption, using powered activated carbon, is the most common method to remove these compounds but greatly increases the amount of sludge generated. Another way of removing taste and odor compounds is to use filters with granular activated carbon (GAC) but little is still known on how to design them. In this work, the homogeneous surface diffusion model (HSDM) was used to model bench-scale kinetic and isotherm experiments and to simulate the removal of geosmin in a full-scale GAC filter. Geosmin adsorption isotherm was best described by the Freundlich model in all used carbons and film resistance (K_f) was more relevant to adsorption kinetics than pore diffusion (D_s). The simulation showed that in a filter with an empty bed contact time of 5 minutes and raw water with geosmin concentrations of 50, 75, and 100 $ng.L^{-1}$, the effluent would exceed the trash-hold concentration (10 $ng.L^{-1}$) in 98, 77, and 66 days, respectively, without considering biological removal.

Key words: GAC filter size, taste and odor compounds, activated carbon, adsorption simulation.

INTRODUCTION

Geosmin and 2-methylisoborneol (MIB) are taste and odor (T&O) compounds produced by cyanobacteria that negatively affect drinking water aesthetics. A sensitive consumer can detect geosmin and MIB at levels as low as 10 $ng.L^{-1}$ (Young et al. 1996). This concentration can be reached in raw water with a relatively low number of *Dollicorspermum circinale* cells (200 $cells.mL^{-1}$), for example (Zamyadi et al. 2013).

Two forms of activated carbon may be used to remove metabolites such as MIB and geosmin: powder activated carbon (PAC) and granular activated carbon (GAC). The main differences between the two forms are the particle size and the mode of use. GAC has a larger particle and it´s used as a filter medium while PAC is smaller and administered intermittently as a suspension, generally in the coagulation unit of the water treatment plant. Although GAC filtration is an efficient process for the removal of MIB and geosmin, its efficiency decreases over time as the carbon adsorption sites are occupied (Mackenzie et al. 2005, Elhadi et al. 2006). On the other hand, under favorable conditions, a biofilm capable of rapidly degrading MIB and geosmin may form on the GAC particles, significantly expanding its lifetime (Elhadi et al. 2006, Ho et al. 2012).

Faruqi et al. (2018) found that age has a significant impact on GAC performance, with removal efficiency decreasing with increased age. Newcombe et al. (1996) reported that, with an empty bed contact time (EBCT) of 20 min, 18 months was the maximum time that a GAC filter could reduce MIB concentrations below the T&O threshold. On the other hand, Gillogly

et al. (1999) reported that the useful life of a GAC bed for MIB removal was around several years. This huge difference in GAC life observed by the previous authors probably indicates that biological removal may not occur in all GAC beds due, probably, to differences of raw water biological and chemical composition. Biologically active GAG filters have achieved higher removal rates than non-biologically active and were little impacted by age. All authors previously mentioned agreed that there is still great difficulty in predicting GAC filters removal efficiency.

Adsorption kinetics modeling may be classified as pseudo-reaction or diffusion models. Pseudo-first order and pseudo-second-order models, although very popular, assume an overall adsorption rate and do not describe the mechanisms of adsorption and diffusion individually. Diffusion models, such as the homogeneous surface diffusion model - HSDM (Crittenden & Weber 1978), assume that mass transfer may be controlled by diffusion in the external film and/or on the surface of the adsorbent and that the resistance to diffusion within the pores and adsorption itself are negligible (Baup et al. 2000).

HSDM has been used to simulate MIB and geosmin adsorption on activated carbon (Cook et al. 2001, Matsui et al. 2009) however, an attempt to estimate the full-scale GAC filter size and saturation time has not yet been found in the literature. Capelo-Neto and Buarque (2016) published an experimental and mathematical procedure that allowed a preliminary assessment of saxitoxin removal efficiency in full-scale, pilot, or bench GAC filters based on relatively simple lab experiments. Using that methodology, our study has, as the main objective, to simulate a full-scale GAC filter for the removal of geosmin using simple bench-scale experiments and diffusion model.

MATERIALS AND METHODS

Three commercial GACs (Norit RB4, WV 1050, and Maxsorb) with different textural properties were used. Virgin GAC samples were sieved through standard Tyler meshes of sizes 20 (0.85 mm) and 30 (0.65 mm). Each sample was washed 10 times with ultrapure water to remove fine particles, dried at 110 °C to constant weight, and cooled in a desiccator until used. As adsorbate, an analytical standard of geosmin (Supelco, USA) diluted in ultra-pure water was used. The adsorbents were characterized using the nitrogen adsorption/desorption isotherm at 77 K (Autosorb-1 MP - Quantachrome, USA). The specific surface area was calculated according to the Brunauer, Emmett, and Teller (BET) equation, the total pore volume (Vp) was obtained from the volume adsorbed at a relative pressure of p/p_0 = 0.95 and the volume of micropores (V_{mic}) was calculated using the Dubinin-Radushkevich equation (Rouquerol et al. 2014).

Geosmin analyses were performed by concentrating the samples, using the headspace technique and solid-phase microextraction (SPME), followed by gas-phase chromatography (Thermo Scientific, TRACE 1300 Series GC) coupled to a mass spectrometer (Thermo Scientific, Single Quadrupole MS - ISQ), according to Graham & Hayes (1998). A geosmin calibration curve was constructed with concentrations between 4 and 500 $ng.L^{-1}$, obtained also from the analytical standard (Supelco, USA). The chromatograph/mass spectrometer operating conditions are shown in Table I.

Kinetic and isotherm adsorption experiments were carried out in vessels shaken continuously at 15 rpm, in the dark, without headspace, and at constant temperature (25°C). Kinetic tests evaluated the effect of contact time on geosmin concentration in an aqueous solution (C_0 = 100 $ng.L^{-1}$) and thus, should identify

Table I. Chromatograph/mass spectrometer operating conditions for geosmin analysis.

Chromatograph	
Injection mode	Splitless
Injector temperature	220 °C
Oven heating	50 °C for 1 min, 10°C.min^{-1} until 150°C, 30 °C.min^{-1} until 250°C, 250 °C for 1min
Helium flow rate	1 mL.min^{-1}
Mass spectrometer	
Ionization mode	EI
Transfer line temperature	250°C
MS temperature	150°C
Monitored ions (m/z)	112, 125, 182 (parent ion)

the minimum necessary time for adsorption equilibrium to be reached. Adsorption isotherm experiments were initiated using a constant mass of GAC in contact with different initial concentrations of geosmin. The concentration of GAC used was 10 mg L^{-1}, commonly found in the literature (Cook et al. 2001, Graham et al. 2000), and the initial geosmin concentration ranged from 20 to 300 ng.L^{-1} (Cook et al. 2001, Graham et al. 2000), resulting in equilibrium concentrations between 15 and 170 ng.L^{-1}. Kinetic and isotherm experiments and the control flasks (without activated carbon) were performed in triplicate. To avoid biodegradation of geosmin, the vials containing ultrapure water and GAC were autoclaved before the addition of 11 µL of mercury chloride (5M) to each one. Geosmin was used because it is a compound predominantly produced by cyanobacteria in the northeast region of Brazil, especially *Dolichospermum circinale*. Ultrapure water was used, without the presence of dissolved organic matter or saline content, to isolate the adsorption behavior of geosmin on the GAC used.

Using the methodology proposed by Capelo-Neto and Buarque (2016), HSDM was applied to simulate the kinetics of geosmin adsorption on GAC using the FAST 2.1 software (Sperlich et al. 2008). The adsorption equilibrium was modeled using Langmuir and Freundlich isotherms because FAST 2.1 software accepts only these two models. By minimizing the difference between the concentration simulated by the model and the experimental concentration, the diffusivity coefficient (D_s) and the mass transfer coefficient in the film (K_f), which provided the best fit, were obtained. A comparison between simulated and experimental data was done using the Student's t-test (α = 5%). With the modeled parameters (D_s and K_f) that best fitted the experimental data of the GAC with higher adsorptive capacity, HSDM was used again, this time to simulate geosmin break-through curves in a hypothetical full-scale GAC filter. The boundary conditions were: 10 ng.L^{-1} finished water geosmin concentration, different raw water concentrations (50, 75, and 100 ng.L^{-1}) representing different intensities of cyanobacteria blooms, and an EBCT of 5 minutes.

Usually, water companies use GAC grain size between 2.38 and 0.55 mm (Kennedy et al. 2015). Thus, a GAC with an average diameter of 1.14 mm was used, which represents the geometric mean of this range. One assumption made for this simulation was that the flow within the hypothetical filter had the same turbulent flow regime applied to the bench experiments and, consequently, the K_f adopted was the same. This assumption remains to be tested in future experiments.

RESULTS AND DISCUSSION

The adsorption isotherms of N_2 at 77 K are shown in Figure 1a. Table II shows the textural properties and Figure 1b the pore size distribution of the activated carbons Norit RB4, WV 1050, and Maxsorb. All the carbon tested presented type I isotherms (Rouquerol et al. 2014), showing a typical behavior of microporous materials, i.e. a high volume adsorbed at low relative pressures (p/p0<0.1). Maxsorb adsorbed the largest volume of nitrogen, indicating the largest BET area and largest volume of pores and micropores. Maxsorb also presented a different characteristic from the other carbons, a bimodal distribution of pore size, micropores (<20 Å), and mesopores (> 20 Å and < 500 Å). Activated carbons with a higher volume of mesopores have higher capacity and a higher rate of adsorption than those activated carbons with the same micropore volume but lower volume of mesopore (Buarque and Capelo-Neto 2015).

The adsorption kinetic of geosmin is presented in Figure 2. The three GAC samples showed a more intense geosmin removal until approximately 24 hours, followed by a reduction in adsorption velocity, especially for Norit RB4. Maxsorb and WV1050 carbons presented a higher adsorption capacity probably due to the volume of mesopores present in their crystalline structure (Buarque and Capelo-Neto 2015). Control samples (without GAC) showed no significant decrease in geosmin concentration (α = 5%) until 72 hours. Although GAC did not appear to have reached adsorption equilibrium at 72 h, this time was used as equilibrium since it is not usual to use such a long time in full-scale processes. Huang et al. (1996) used HSDM to model adsorption of MIB onto PAC and observed extremely slow adsorption kinetics as

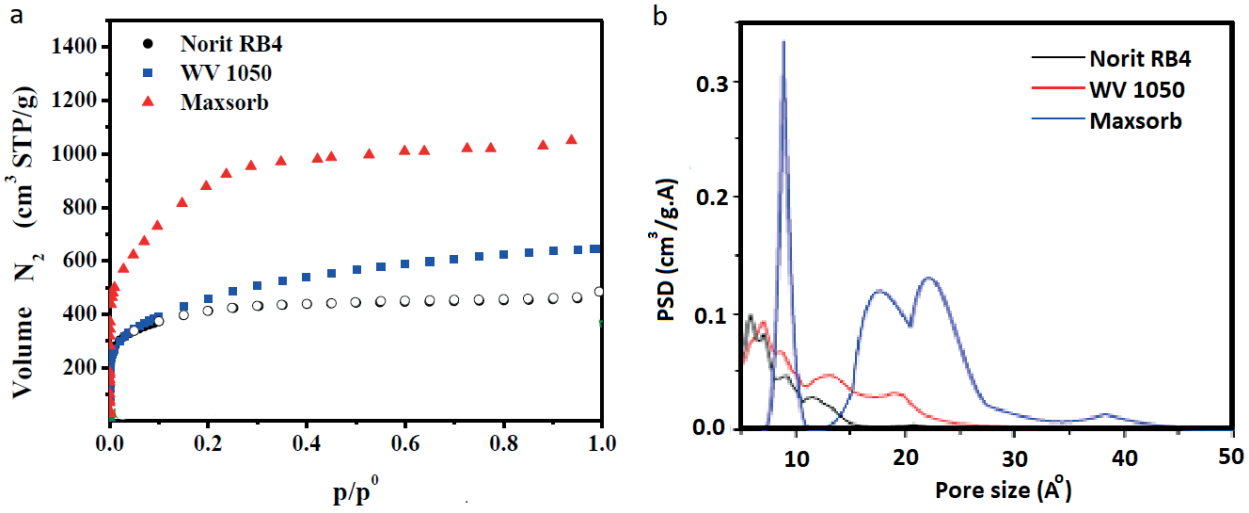

Figure 1. (a) N_2 adsorption equilibrium isotherms at 77 K and (a) Pore size distribution (PSD) of the activated carbons Norit RB4, WV 1050, and Maxsorb.

Table II. Textural characteristics obtained from adsorption/desorption equilibrium isotherms of N_2 at 77 K.

Sample	BET ($m^2.g^{-1}$)	V_p ($cm^3.g^{-1}$)	V_{mic} ($cm^3.g^{-1}$)
Norit RB4	1521	0.71	0.57
WV 1050	1615	1.03	0.76
Maxsorb	3250	1.65	1.35

well. According to the authors, equilibrium was not achieved at relatively long contact times (4–6 h).

Adsorption equilibrium data was examined using Langmuir and Freundlich models (Figure 3). In this case, also, there was no significant decrease of geosmin (P_0 = 0.05) in the control samples until 72 hours. Based on the correlation coefficients R^2 (Table III), geosmin adsorption was best described by the Freundlich isotherm in all GACs. Yu et al. (2016) and Graham et al. (2000) investigated the adsorption of MIB onto PAC and also observed that Freundlich best fitted the equilibrium curves with the parameter *n* ranging from 0.1 to 1.0.

Figure 4 shows the HSDM curve fitting to the experimental geosmin adsorption kinetics data on GAC Maxsorb maintaining D_s constant (10^{-14} $m^2.s$) and varying K_f (10^{-4} $m.s^{-1}$, $3x10^{-5}$ $m.s^{-1}$ and, considering the film resistance K_f negligible). The simulated kinetic curve adopting K_f = $3x10^{-5}$ $m.s^{-1}$ did not differ significantly from the experimental data (P_0 = 0.07%). The identification of the best fitting D_s was also done by keeping K_f constant and varying D_s. Variations of D_s in three orders of magnitude did not significantly alter the adjustment of the curve and, therefore, diffusivity was not considered relevant to the adsorption process (data not shown). The model successfully described geosmin adsorption onto Maxsorb and provided the kinetic parameters necessary for the filter up-scaling.

From the few studies found in the literature that used HSDM to model adsorption data of geosmin onto activated carbon, only Cook et al. (2001) published values for the diffusivity coefficient (D_s). The authors found D_s of around 10^{-13} $m^2.s$ for several Australian natural waters but did not consider the mass transfer coefficient in the film (K_f) in the calculations. Since D_s was found not to have a significant impact on the adsorption kinetics, the difference between our D_s (10^{-14} $m^2.s$) and theirs (10^{-13} $m^2.s$) may not be significant.

Figure 5 shows the simulations of a full-scale GAC filter with EBCT of 5 minutes and geosmin affluent concentrations of 50, 75, and 100 $ng.L^{-1}$. The effluent concentrations would exceed the pre-established limit of 10 $ng.L^{-1}$ in 98, 77, and 66 days, respectively. This indicates that a filter or a system of filters, with a total GAC volume of 300 m^3, a flow rate of 1 $m^3.s^{-1}$ and raw water containing 50 $ng.L^{-1}$ of geosmin, would prevent T&O problems for 98 consecutive days before the GAC needed to be changed or regenerated, for example. This example does not consider removal by biological activity and, therefore, it could be an interesting tool to identify how much each removal mechanism is acting in real-life situations.

This limited lifespan of the GAC could represent an operational problem for water companies if the metabolites responsible for T&O were constantly present in the raw water. However, according to Newcombe et al. (2010),

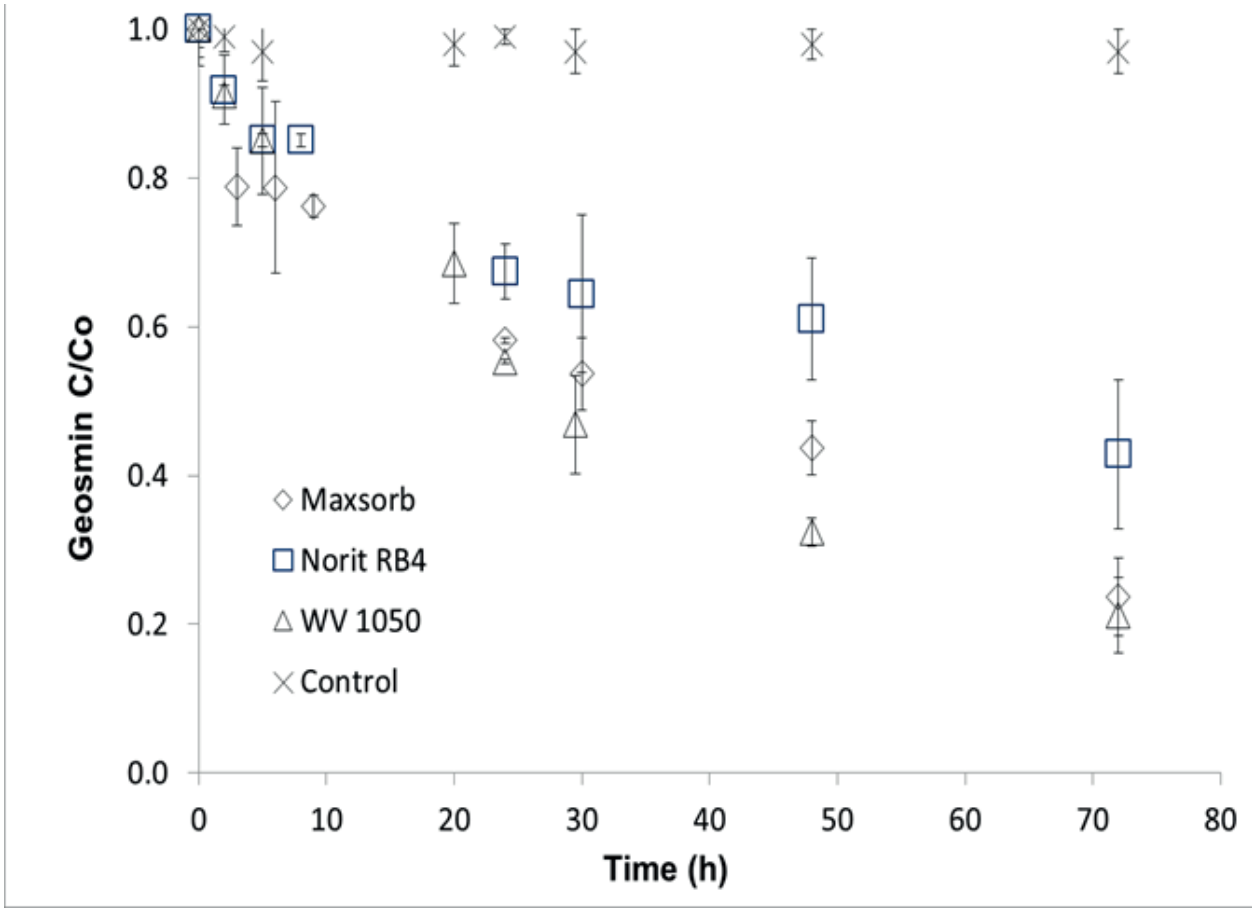

Figure 2. Adsorption kinetics of geosmin onto Norit RB4, WV 1050, and Maxsorb.

this is not usually the case. GAC filters are normally used to avoid occasional episodes of cyanobacteria metabolites and, therefore, their lifespan should increase.

This simulation has, however, some limitations. Two very important ones are: It does not consider the interference of organic matter and biologic activity. Dissolved organic matter in raw water can substantially reduce the activated carbon adsorption capacity (Newcombe et al. 2002, Zoschke et al. 2011). Additionally, waters with high turbidity can decrease the removal of geosmin in activated carbon (Cook & Newcombe 2008). Therefore, low-quality water may decrease the efficiency of GAC filters and break-through time. On the other hand, the biological removal of geosmin has been reported in filters containing GAC (Ho & Newcombe 2010, Drogui et al. 2012), offering the advantage of an additional removal mechanism. The two mechanisms, biological removal, and adsorption, acting together may increase the break-through time. Drikas et al. (2009), using pilot-scale experiments with several Australian natural waters, observed that GAC filters were able to remove MIB, geosmin (50 to 200 $ng.L^{-1}$), and organic matter for up to 10 months using a 5-minute EBCT without the need for carbon substitution.

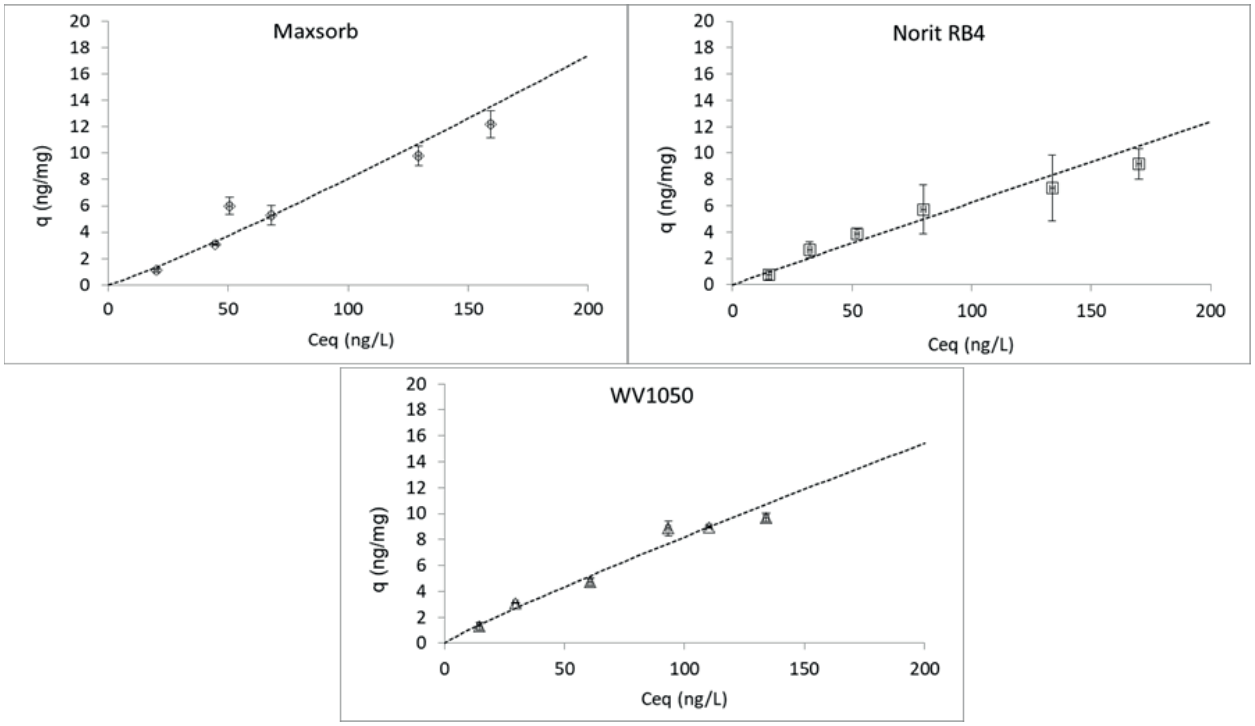

Figure 3. Geosmin isotherms of Norit RB4, WV 1050 and Maxsorb. Symbols - Experimental data; Doted Lines - Freundlich fit.

Table III. Langmuir and Freundlich parameters for the adsorption of geosmin onto Norit RB4, WV 1050, and Maxsorb granulated activated carbons.

GAC	Langmuir isotherm		
	q_{max} (ug.g^{-1})	K_L (L.ug^{-1})	R^2
Maxsorb	-68.49	0.00	0.06
Norit RB4	64.94	0.00	0.07
WV 1050	46.30	0.10	0.41
	Freundlich isotherm		
	K_{fr} [(ng.mg^{-1}).(L.ng^{-1})^n]	n	R^2
Maxsorb	0.048	0.90	0.93
Norit RB4	0.07	1.01	0.95
WV 1050	0.12	1.10	0.98

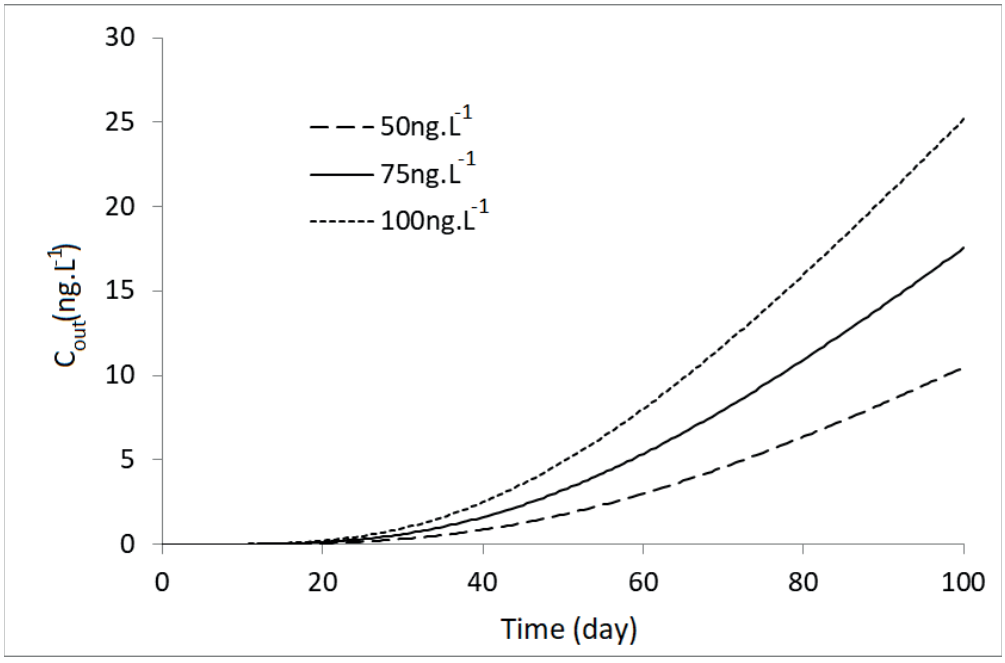

Figure 4. Kinetics adsorption of geosmin onto Maxsorb fitted with HSDM keeping D_s constant (10^{-14} m^2.s) and using different K_f.

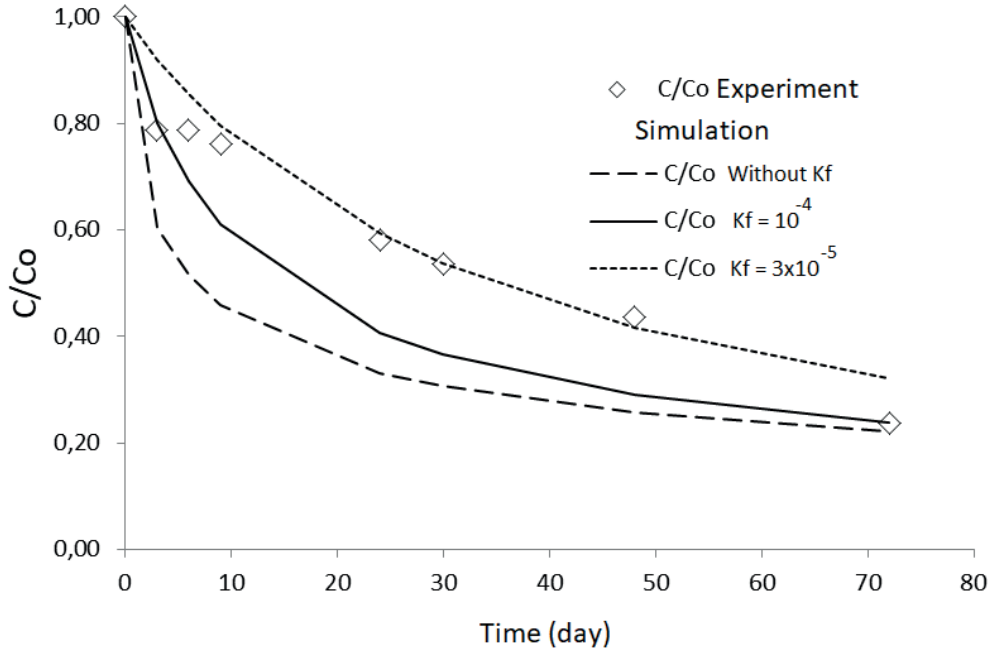

Figure 5. Simulated geosmin break-through curves in GAC (Maxsorb) filter using EBCT of 5 minutes and geosmin affluent concentrations of 50, 75, and 100 ng L^{-1}.

CONCLUSIONS

The methodology used in this paper may offer an option not only for a first approach to GAC filter projects but also for planning pilot and bench-scale experiments. The main conclusions of this study were:

- Maxsorb, which presented a bimodal distribution with micro and mesopores, was the activated carbon with the highest adsorption capacity;
- Freundlich model best described by the adsorption equilibrium of geosmin;
- Diffusion in the film was considered the main limiting factor since variations of K_f significantly altered the HSDM fit to the experimental data;
- It was possible to simulate a full-scale GAC filter removing geosmin by adsorption only, utilizing bench-scale experimental and modeled data,

In order to evaluate if HSDM was able to successfully describe the adsorption kinetic of geosmin onto the Maxsorb GAC sample, it is necessary to validate the assumptions adopted and to incorporate factors present in the full-scale water process such as dissolved organic matter, turbidity, and ion content, as well as the possibility of biofilm formation in the filter. These factors should be studied in future experiments.

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JULIANA A. COELHO[1]
https://orcid.org/0000-0002-0575-6750

DIANA CRISTINA S. DE AZEVEDO[1]
https://orcid.org/0000-0001-8545-3989

IVANILDO JOSÉ DA SILVA JUNIOR[1]
https://orcid.org/0000-0002-1297-0261

JOSÉ CAPELO-NETO[2]
https://orcid.org/0000-0002-7669-7153

[1]Universidade Federal do Ceara, Departamento de Engenharia Química, Campus do Pici, Bloco 709, Av. Mister Hull, s/n, Pici, 60455-760 Fortaleza, CE, Brazil

[2]Universidade Federal do Ceará, Departamento de Engenharia Hidráulica e Ambiental, Campus do Pici, Bloco 713, Pici 60455-900 Fortaleza, CE, Brazil

Correspondence to: **José Capelo-Neto**
E-mail: capelo@ufc.br

Author contributions

Juliana Amorim Coelho: Investigation; Formal analysis; Writing - original draft; Supervision. Diana Cristina Silva de Azevedo: Funding acquisition; Writing - review & editing. Ivanildo José da Silva Junior: Validation; Writing - review & editing. Jose Capelo-Neto: Methodology; Conceptualization; Funding acquisition; Project administration; Writing - review & editing.