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Fixed-bed column adsorption of the coffee aroma compound benzaldehyde from aqueous solution onto granular activated carbon from coconut husk

Anderson Marcos Dias Canteli*, Danielle Carpiné, Agnes de Paula Scheer, Marcos R. Mafra, Luciana Igarashi-Mafra

Federal University of Paraná, Chemical Engineering Department, Graduation Program of Food Engineering, Centro Politécnico, Jardim das Américas, Curitiba, Paraná, 81531-990, Brazil

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ABSTRACT

This paper evaluated the performance of fixed-bed columns with activated carbon as the adsorbent for the removal of benzaldehyde present in an aqueous solution. The effects of the following parameters were evaluated: inlet concentration (91.9 mg L^{-1} – 440.0 mg L^{-1}), feed flow rate (3.9 mL min^{-1} – 11.8 mL min^{-1}), bed depth (30 mm–90 mm) and column inner diameter (6.2 mm–12.2 mm). All of the experiments were performed at 293.15 K. The results showed that the bed capacity, total bed capacity and saturation time decreased as the feed flow rate was increased. The opposite effect was observed with an increase in bed depth. Increasing the inlet concentration resulted in higher aroma adsorption. An increase in the inner diameter without changing the feed flow rate resulted in better aroma recovery. The results showed that the column performed well at all of the analyzed parameter values. Data obtained from the analysis of the effects of feed flow rate and bed depth were used for a scale-up study using the bed depth service time model, which showed good results with errors of less than 12%. The experimental data obtained in this study will be useful for further applications involving coffee aroma recovery.

1. Introduction

Aroma compounds are very important in the beverage industry because these compounds contribute to the sensory characteristics of beverages. However, during the processing of some beverages, including coffee, some compounds responsible for the aroma and flavor may be lost, and these compounds should be recovered and reincorporated to maintain the sensory characteristics of the final product close to those of the pre-processed product.

Several techniques, such as supercritical fluid extraction (Gracia, Rodríguez, García, Alvarez, & García, 2007), vacuum membrane distillation (Bagger-Jørgensen, Meyer, Varming, & Jonsson, 2004; Diban, Voinea, Urtiaga, & Ortiz, 2009) and pervaporation (Aroujalian & Raisi, 2007; Diban, Urtiaga, & Ortiz, 2008; del Olmo, Blanco, Palacio, Prádanos, & Hernández, 2014), have been studied for the recovery of flavors. In fact, the latter technique has been the subject of recent studies because it allows the use of a low temperature, which avoids flavor degradation, and requires low energy consumption compared with traditional techniques, such as steam distillation, liquid solvent extraction and vacuum distillation. In this context, the adsorption process becomes of interest to researchers.

Adsorption is very accessible due to the simplicity of the process and the range of naturally available adsorbents (Singh, Srivastava, & Mall, 2009). This technique is widely used in wastewater treatment (Chen et al., 2012; Goel, Kadirvelu, Rajagopal, & Kumar Garg, 2005; Goshadrou & Moheb, 2011; Han et al., 2007; Ko, Porter, & McKay, 2000; Lin & Wang, 2002; Salman, Njoku, & Hameed, 2011; Tan, Ahmad, & Hameed, 2008). However, adsorption, which is typically performed using activated carbon as the main adsorbent, was recently used to recover aromas in aqueous solutions, such as pear aroma (Diban, Ruiz, Urtiaga, & Ortiz, 2007), essential oil distillation (Edris, Girgis, & Fadel, 2003), and coffee (Carpiné, Dagostin, da Silva, Igarashi-Mafra, & Mafra, 2013; Lucas, Cocero, Zetzl, & Brunner, 2004; Zuim et al., 2011).

The use of fixed bed columns in adsorption processes offers several advantages, such as simplified operation, construction,

* Corresponding author. Tel.: +55 18 3642 6157.
E-mail addresses: eu_canteli@hotmail.com, andersonmdcanteli@gmail.com (A.M.D. Canteli), daniellecarpine@gmail.com (D. Carpiné), agnesps@gmail.com (A.P. Scheer), marcos.mafra@ufpr.br (M.R. Mafra), luciana.igarashi@gmail.com (L. Igarashi-Mafra).

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scale up and process automation and allowing the recovery of a large amount of adsorbate with the use of a fixed bed (Aksu & Gönen, 2004). The study of the dynamic equilibrium in columns provides important information, such as the system size, contact time and adsorbent usage rate, and this information can be obtained from breakthrough curves (Moreno-Castilla, 2004). The bed depth, feed flow rate, inlet concentration, inner diameter and pH of the solution have been observed to significantly affect the ongoing adsorption process (Chen et al., 2012; Han et al., 2007; Singh et al., 2009; Srivastava, Prasad, Mishra, Mall, & Swamy, 2008).

The aim of this study was to evaluate the recovery of benzaldehyde from a synthetic aqueous solution through continuous adsorption in a fixed bed column as a potential industrial application. The influence of several operational conditions (inlet concentration, feed flow rate, bed depth and inner diameter) was analyzed. Furthermore, a scale-up study was performed using the experimental data and the bed depth service time model.

1.1. Column performance

The fixed bed column performance is described using breakthrough curves, which graphs time versus C_t/C_o (the ratio of the concentration of the solute in the column outlet at a given time t to the initial concentration of the solute at the column inlet).

Certain parameters obtained from the breakthrough curves can be used to evaluate the fixed bed performance and efficiency. The total capacity of the column (q_{total}, mg) provides the maximum amount of flavor that can be adsorbed by the fixed bed and can be estimated by the area under the breakthrough curve (Calero, Hernáinz, Blázquez, Tenorio, & Martín-Lara, 2009; Salman et al., 2011). If the bed is completely saturated and the inlet concentration is constant over time, the total capacity of the column is calculated from Equation (1):

$$q_{total} = \frac{QC_0}{1000}\int_{t=0}^{t=t_{sat}}\left(1-\frac{C}{C_0}\right)dt \tag{1}$$

where Q is the column feed flow rate (mg min^{-1}), C is the outlet concentration (mg L^{-1}), C_0 is the inlet concentration (mg L^{-1}) and t_{sat} is the time required for the bed to become saturated (min).

The bed capacity (q_{bed}, mg g^{-1}) is a parameter that determines the amount of flavor recovered by the fixed bed per gram of adsorbent present in the bed and is calculated from Equation (2), where m is the mass of activated carbon present in the bed (g) (Calero et al., 2009; Chen et al., 2012; Salman et al., 2011):

$$q_{bed} = \frac{q_{total}}{m} \tag{2}$$

The total amount of benzaldehyde fed into the column until full bed saturation (W, g) can be calculated using Equation (3) (Aksu & Gönen, 2004; Calero et al., 2009):

$$W = \frac{QC_0t_{sat}}{10^6} \tag{3}$$

The bed performance (P) relates the amount of aroma retained in the bed (q_{total}) with the amount of aroma fed in the same run (W) (Aksu & Gönen, 2004; Calero et al., 2009; Chen et al., 2012). A high performance indicates a good operational set up, and the performance can be calculated using Equation (4):

$$P(\%) = \frac{q_{total}}{W}\times 100 \tag{4}$$

The adsorbent utilization (η) relates the total capacity obtained in the fixed bed (q_{total}) with the total capacity obtained in a batch experiment (q_{batch}) and therefore represents the amount of active sites that are not utilized in the fixed bed. This parameter is calculated from Equation (5):

$$\eta = \frac{q_{bed}}{q_{batch}} \tag{5}$$

The residence time, which affects the shape of the breakthrough curve and the breakthrough time (Singh et al., 2009), is the time required for the fluid to fill the empty column (Ko et al., 2000). This parameter is the most important in the design of a fixed bed (McKay & Bino, 1990), and effects in the residence time may be easily observed as a result of changes in the bed depth and feed flow rate. The true residence time (TRV) can be calculated from Equation (6):

$$TRV = \frac{\varepsilon \times V_L}{Q} \tag{6}$$

Where ε is the bed porosity and V_L is the volume occupied by the adsorbent inside the bed (mL). The bed porosity can be estimated by the fraction of empty spaces (volume of distilled water present in the fixed bed after packing (mL) divided by the fixed bed volume (mL)).

1.2. Scale-up study

The bed depth service time (BDST) model predicts the relationship between the bed depth, Z (cm), and the operation time, t (min). This model assumes that the adsorption rate is controlled by the surface reaction between the adsorbate and the unused capacity of the adsorbent (Srivastava et al., 2008; Zou, Zhao, & Zhu, 2013). Equation (7) expresses a linear relationship between the bed depth and the service time:

$$t = \frac{N_0}{C_0 v}Z - \frac{1}{K_{BDST}C_0}\ln\left(\frac{C_0}{C_t}-1\right) \tag{7}$$

where N_0 is the adsorption capacity (mg mL^{-1}), v is the fluid velocity (cm min^{-1}), C_t is the outlet concentration at time t (mg mL^{-1}) and K_{BDST} is the mass transfer coefficient (mL (mg min^{-1})). K_{BDST} and N_0 can be calculated from the linear and angular coefficient, respectively, from the graph of t as a function of Z at a given C_t/C_0 ratio (iso-concentration line).

At 50% breakthrough ($C_t/C_0 = 0.50$ and $t = t_{0.50}$), the linear term of Equation (7) becomes indeterminate (ln(1)), and the equation is thus reduced to Equation (8):

$$t_{0,50} = \frac{N_0}{C_0 v}Z \tag{8}$$

Thus, the graph of $t_{0.50}$ at 50% breakthrough as a function of Z forms a line passing through the origin, and N_0 can be calculated by the angular coefficient (Srivastava et al., 2008). A simplified form of the BDST model is expressed by Equation (9) (Goel et al., 2005):

$$t = aZ - b \tag{9}$$

$$a = \frac{N_0}{C_0 v} \tag{10}$$

$$b = \frac{1}{K_{BDST}C_0}\ln\left(\frac{C_0}{C_t}-1\right) \tag{11}$$

where a is the angular coefficient (min cm^{-1}) and b is the linear coefficient (min) of the straight line obtained with Equation (7).