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NEAR-INFRARED SPECTROSCOPY TO MONITOR β-CAROTENE ADSORPTION AT HIGH PRESSURES

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ABSTRACT

The goal of this work was to study the β-carotene recovery in a high pressure variable volume cell with in situ monitoring via near infrared spectroscopy (NIR). The kinetics and adsorption equilibrium of β-carotene in dichloromethane (DCM) with the polymer resin Diaíon HP-20 were evaluated. Kinetic curves were obtained at different initial pigment concentrations at 293 K. The Pseudo-first order and Pseudo-second order models were fitted to the experimental data. Adjustments of the Langmuir and Freundlich isotherms to the experimental data were evaluated. The Langmuir model was the one that fitted better the experimental data over the entire range studied. The maximum adsorption capacity estimated by the Langmuir model was 200 mg/g at 293 K with about 40 minutes of contact time.

1. INTRODUCTION

B-carotene is a carotene that has a pro-vitamin activity, which is fundamental for the A-vitamin supply. There has been an increase in studies aiming its recovery from distinct raw materials. Among the current techniques applied on the isolation, recovery and purification of these compounds, pressurized adsorptive systems arise as a promise technique as promotes increased diffusivity and adsorption rates, and can be used as an intensified process (Mezzomo, 2012). The present work aims to develop an alternative process for the adsorption of β-carotene using compressed fluids, as well as to quantify this compound on-line through the use of near infrared spectroscopy (NIR).

2. METHODOLOGY

2.1 Experimental Equipment and Procedure

At the beginning of each experiment, the temperature controller of the variable-volume cell is turned on. 0.1 g of the HP-20 resin, previously prepared and dried, is weighed and placed inside the cell. A precise amount of solute coming from stock solutions (1000 mg/L – 500 mg/L) is weighted

and added to the cell using a displacement pump. The propane is loaded with the help of a syringe pump (at constant temperature and pressure). The system is then maintained under slow and continuous stirring using a magnetic stirrer and a teflon coated magnetic bar to permit the system homogenization. With the pressure and temperature stabilized, NIR specters were continuously collected to monitor the adsorption process at high pressure. A set of NIR specters were previous generated to the system calibration, permitting to assess the decrease of β -carotene concentration over time (Borges, *et al.,* 2015).

3. RESULTS AND DISCUSSION

3.1 Kinetics of Adsorption

Figure 1 illustrates the adsorption kinetics of β-carotene from the resin surface and shows an adsorption kinetics that reached equilibrium within 40 min of contact time. A marked transfer occurred between 0 to 15 min, and then, from 15 to 30 min the adsorption increased gradually until equilibrium was reached. In order to elucidate the adsorption behavior and to describe the experimental data, the pseudo-first order and pseudo-second order models were chosen to evaluate the adsorption process. All parameters derived from kinetic models, such as correlation coefficient and dynamic parameters, are summarized in Table 1.

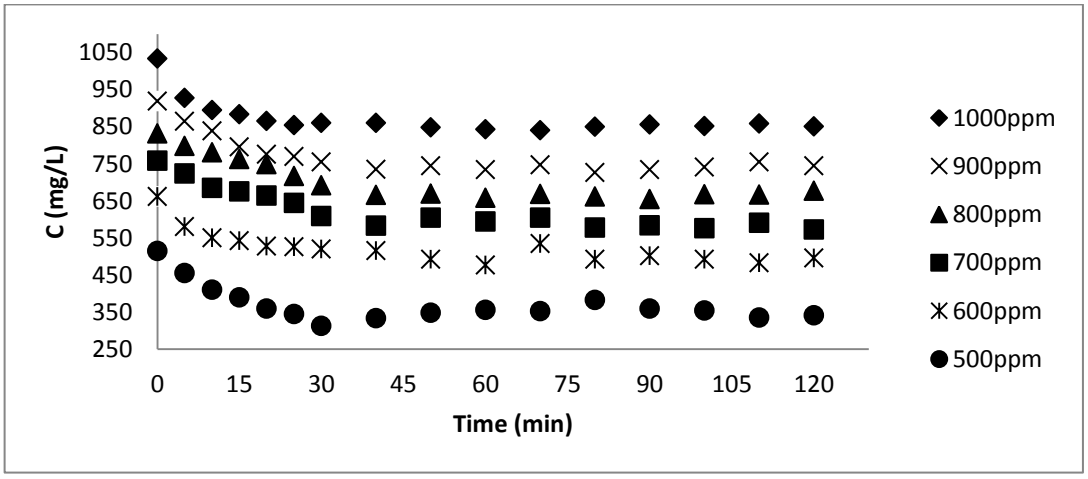

Figure 1. Adsorption kinetics of β –carotene with different initial concentrations of 500 to 1000 mg / L. Pressure of 20 bar. Temperature of 293 K. Adsorbent mass of 0.1 g.

Table 1 shows the calculated values of the first and second order kinetics constants, k1 and k2 respectively, the adsorption capacities (q_e) estimated by the models for all studied concentrations, as well as the experimental adsorption capacity. The values of R^2 presented by Pseudo-Second

Order model were better than the ones presented by Pseudo-First Order model, they also presented greater proximity between modeled data and experimental data.

Table 1. Calculated parameters for adsorption kinetic of β-carotene onto HP-20 resin, according to the Pseudo First Order and Pseudo Second Order Models.

Initial concentrations	Experimental q_e	Pseudo First-Order			Pseudo Second-Order		
		q_e	K_1	R^2	q_e	K_2	R^2
1033,62	162,41	41,84	3,37	0,51	188,68	0,04	0,98
918,39	168,83	42,02	4,06	0,58	188,68	0,04	0,98
831,39	174,72	41,67	4,49	0,51	212,77	4,49	0,51
758,42	178,17	34,48	4,76	0,85	212,77	4,49	0,51
661,39	177,49	42,37	4,29	0,81	178,57	0,04	0,97
514,68	182,76	42,55	3,92	0,50	172,41	0,02	0,97

The Pseudo-Second Order kinetic models were chosen as the most favorable models to show the β-carotene adsorption processes onto the resin, due to the good correlation obtained and better proximity between experimental data and modeled data.

3.2 Adsorption Isotherm

As the concentration of β-carotene in the initial solution increased, the adsorption capacity of the HP-20 resin also increased. The experiments were carried out with initial concentrations of 500 to 1000 mg / L. By designing Ce/q *vs* Ce and log qe *vs* log Ce, linear regression lines were obtained**.** **T**he Lagnmuir and Freundlich isotherm parameters are presented in table 2. The equilibrium adsorption isotherm of β-carotene onto HP-20 was investigated at the temperature of 293 K, as shown in Figure 2.

Table 2. Calculated parameters for Langmuir and Freundlich adsorption isotherms.

Tem. (K)	Langmuir			Freundlich		
	q_m (mg/g)	K_L (L/g)	R^2	N	K_f (L/g)	R^2
293	200	0,012	0,9989	0,13	4,31	0,9942

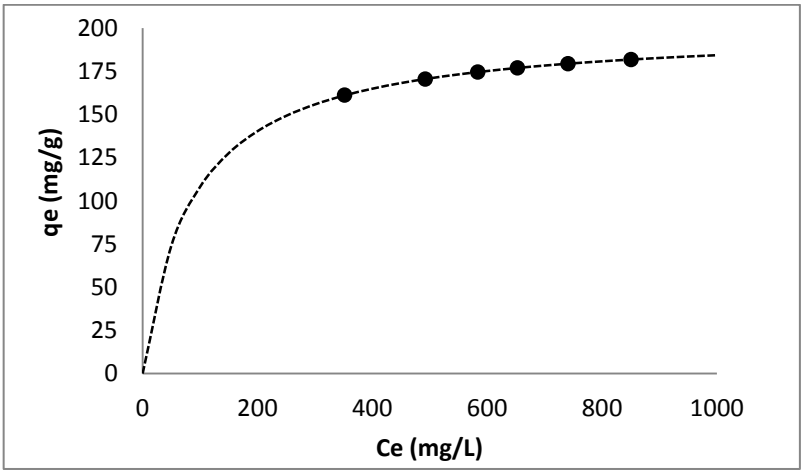

Figure 2. β-carotene equilibrium adsorption isotherm onto the HP-20 resin. Adsorbent mass: 0.1 g. Experiment duration: 120 min. And isothermal modeling of Langmuir.

Figure 2 shows that the amount of β-carotene in equilibrium was approximately of 200 mg/g of adsorbent. Thus, it is possible to observe that coupling the NIR probe to the adsorptive system was efficient for monitoring the adsorption of β-carotene onto HP-20 resin.

4. REFERENCES

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