



DOI: <http://dx.doi.org/10.1590/1807-1929/agriambi.v23n9p716-722>

Use of sweet 'Pêra' peel as an adsorbent in the treatment of textile effluents

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ABSTRACT: The disposal of industrial wastewater into aquatic bodies without proper treatment can cause severe damage to the environment and human health. The objective of this study was to perform the drying of the sweet orange (*Citrus sinensis* L. Osbeck) peel cultivar Pêra and evaluate the viability of its use as biosorbent in the removal of a direct dye. Drying was carried out in an oven with air circulation at temperatures of 60 and 80 °C. The mathematical models of Page, Henderson and Pabis, Logarithmic, Midilli and Two-term exponential were fitted to the moisture data as a function of time. The material was characterized by scanning electron microscopy, point of zero charge, and infrared spectroscopy. In the adsorption study, a complete 2⁴ factorial design was used to analyze the influence of mass, initial concentration, solution pH and contact time on adsorbed quantity (qt) and removal percentage of the dye (R%). In the drying, the two-term exponential model fitted best to the experimental data. The characterization of the material indicated that the adsorbent has zero charge point of 3.5 and porous structure, and the infrared analysis indicated the presence of carboxylic and hydroxyl groups. In the adsorption, the adsorbed quantity of the dye increased under conditions of lower pH and biosorbent mass and higher initial concentration and contact time. The removal percentage of dye increases with higher biosorbent mass. The biosorbent used is a promising waste for the adsorption of the burgundy-16 dye.

Key words: dyes, experimental design, wastes

Aproveitamento da casca de laranja Pêra como adsorvente no tratamento de efluentes têxteis

RESUMO: O lançamento de águas residuárias industriais em corpos aquáticos, sem o devido tratamento, pode causar danos severos ao meio ambiente e à saúde humana. O objetivo deste estudo foi realizar a secagem da casca de laranja cultivar Pêra (*Citrus sinensis* L. Osbeck) e avaliar a viabilidade do seu uso como biossorvente para remoção de um corante direto. A secagem foi realizada em estufa com circulação de ar nas temperaturas de 60 e 80 °C. Os dados de umidade em função do tempo foram ajustados aos modelos matemáticos de Page, Henderson e Pabis, Logarítmico, Midilli e exponencial de dois termos. A caracterização do material foi realizada a partir da microscopia eletrônica de varredura, do ponto de carga zero, e da espectroscopia na região do infravermelho. No estudo da adsorção realizou-se um planejamento fatorial 2⁴ completo, analisando-se a influência da massa, concentração inicial, pH da solução e tempo de contato, na quantidade adsorvida (qt) e no percentual de remoção do corante (R%). Na secagem, o modelo exponencial de dois termos apresentou melhor ajuste aos dados experimentais. A caracterização do material apontou que o adsorvente apresenta ponto de carga zero de 3,5 e estrutura porosa, e as análises de infravermelho indicaram a presença de grupos carboxílicos e hidroxilas. Na adsorção, a quantidade adsorvida do corante aumentou em condições de menor pH e massa de biossorvente, maior concentração inicial e tempo de contato. O percentual de remoção do corante aumenta com maior massa de biossorvente. O biossorvente utilizado é um resíduo promissor para a adsorção do corante bordô-16.

Palavras-chave: corantes, planejamento experimental, rejeito



INTRODUCTION

The disposal of industrial wastewater into water bodies, without proper treatment, can cause severe damage to the environment and human health (Mostafa, 2015; Santos et al., 2017). Textile industries are present in most countries due to a human need for clothing and varied utility uses. In Brazil, they have a great economic importance (ABIT, 2013).

Effluents with high concentration of dye restrict the penetration of solar radiation, compromising the photosynthetic activity in water systems and reducing the level of oxygenation of the aquatic environment (Holanda et al., 2012).

Among the wastewater treatment technologies adequate for color removal, adsorption is shown to be a superior alternative to other techniques in terms of efficiency, simplicity of projects and ease of operation (Barrios-Ziolo et al., 2015).

Due to the high cost of activated charcoal, new studies have sought alternative biosorbents, such as agroindustrial wastes, because they are abundant, biodegradable, inexpensive and efficient in the adsorption of dyes (Holanda et al., 2012; Nascimento et al., 2014; Santhi et al., 2016).

The uses of agricultural by-products demonstrate that such materials can be used for the adsorption of various pollutants, including dyes. Orange peel is a by-product of the production of juices, being the agroindustrial waste obtained in largest quantity in Brazil (Rehman et al., 2012; Fiorentin et al., 2015).

This study aimed to perform the drying of orange peel and the modeling of the drying data, as well as to evaluate this agroindustrial waste as a biosorbent, in order to reduce the inadequate disposal of this waste and using it in the removal of the direct burgundy-16 dye from the Tupy brand, present in aqueous solutions, widely used in small-scale dyeing shops.

MATERIAL AND METHODS

The raw material used in the experimental tests was the peel of the sweet orange (*Citrus sinensis* L. Osbeck) cultivar 'Pêra' obtained in the central market of the city of Campina Grande, PB, Brazil.

The study was carried out in the Laboratory of Research on Environmental Sciences (LAPECA), located at 7° 20' S and 35° 91' W and in the Experimental Station of Biological Treatment of Sanitary Sewage (EXTRABES), located at 7° 24' S, 35° 88' W and altitude of 551 m, both belonging to the Centro de Ciência e Tecnologia (CCT) da Universidade Estadual da Paraíba (UEPB), located in the city of Campina Grande, PB, Brazil.

The oranges were initially washed with public-supply water and subsequently with distilled water. They were then cut into four parts in the longitudinal direction of the fruit. The pulp was removed and the orange peels were cut into approximately equal-sized pieces of 1.0 cm².

The initial moisture, on dry basis, of the orange peels, was determined by the oven method at 105 °C. The tests were conducted in triplicate, in which three 10 g samples were weighed on a Marconi AL500C analytical scale and dried in a Marconi MA 035/3IN250 oven.

For the construction of the drying curves, the weight variation of the samples over time was monitored during the

initial 4.5 h. The monitoring was performed every 5 min in the first hour, every 10 min in the next 2 h, and then every 15 min until completing the 4.5 h of monitoring.

The weighings were performed on a Marconi AL500C analytical scale. The drying process was performed in triplicate, in a Marconi MA 035/3IN250 oven with forced air circulation, with speed of 1.5 m s⁻¹, at temperatures of 60 and 80 °C.

After the monitoring period, the samples remained in the oven until the end of the 24 h, until reaching constant dry mass. To predict the drying behavior, the mathematical models of Page, Henderson & Pabis, Logarithmic, Midilli and Two-term exponential, represented by the Eqs. 1, 2, 3, 4 and 5, respectively, were fitted to these data:

$$MR = \exp(-kt^n) \quad (1)$$

$$MR = a \exp(-kt) \quad (2)$$

$$MR = a \exp(-kt) + c \quad (3)$$

$$MR = a \exp(-kt^n) + bt \quad (4)$$

$$MR = a \exp(-kt) + (1-a) \exp(-kta) \quad (5)$$

where:

- MR - moisture ratio of the product, dimensionless;
- t - drying time, min;
- k - drying constant, min⁻¹;
- n - coefficient of the model, dimensionless;
- a - coefficient of the model, dimensionless;
- b - coefficient of the model, min⁻¹; and,
- c - coefficient of the model, dimensionless.

The moisture ratio (MR), during the drying at different temperatures, was calculated using Eq. 6:

$$MR = \frac{M - M_e}{M_1 - M_e} \quad (6)$$

where:

- M - moisture content of the product, g;
- M₁ - initial moisture content of the product, g; and,
- M_e - equilibrium moisture content of the product, g.

To fit the mathematical models to the experimental drying data, nonlinear regression analysis was performed using the Gauss-Newton method, through the Minitab statistical software.

At the end of each drying experiment, the dried material was crushed and after this process, the crushed material was separated using Bertel Mesh 8 sieves, in order to obtain a material with particle size smaller than 2.36 mm.

For the adsorption tests, as well as for biosorbent characterization, the material obtained after drying at 80 °C was used based on preliminary tests.

The biosorbent was characterized by determining the point of zero charge (PZC) according to the methodology proposed by Boas et al. (2012), analysis by scanning electron microscopy (SEM) and Fourier transform infrared spectrum (FTIR).

Material morphology and quantitative analysis of the chemical composition of the samples were analyzed in a Phenom PRO X scanning electron microscope, with up to 45000 x magnification, depth of focus of 1 mm, resolution of 30 nm, and tension of 15 kV, low vacuum and varied pressure (1 to 270 Pa). The images were magnified by 20 and 2000 times.

To determine the functional groups present in the orange peel, the Fourier transform Infrared spectroscopy technique was used in the Perkin Elmer Spectrum 400 spectrophotometer. The spectra were obtained in the absorption region between 4000 and 600 cm^{-1} , with a resolution of 4 cm^{-1} .

As adsorbate, a solution of the direct burgundy-16 dye, from Tupy brand, was used. A 2000 mg L^{-1} stock solution was prepared and used for the dilutions to obtain the work solutions.

Dye concentration in the sample was determined by molecular absorption spectrophotometry in a BEL SP 2000 UV spectrophotometer.

Initially, the wavelength at which the compound to be quantified absorbs the maximum radiation was determined by scanning the spectrum within the range from 400 to 700 nm, and 520 nm was the wavelength that showed the highest absorption. The calibration curve was then constructed using the dye solutions under study, with different concentrations, within a range from 0 to 200 mg L^{-1} .

In order to optimize the adsorption process, a complete 2^4 factorial design with three repetitions at the central point was applied to analyze the influence of four factors: mass, initial concentration, pH solution and contact time.

Table 1 presents the actual and coded values of the factors applied in the experimental design.

The quantity of dye adsorbed (q_t) and the removal percentage ($R_{\%}$) were calculated using Eqs. 7 and 8, respectively:

$$q_t = \frac{(C_i - C_f)V}{M} \quad (7)$$

$$R_{\%} = \frac{(C_i - C_f)}{C_i} 100 \quad (8)$$

where:

- C_i - initial dye concentration, mg L^{-1} ;
- C_f - dye concentration at the time, mg L^{-1} ;
- M - mass of biosorbent (orange peel); and,
- V - volume of dye solution, L.

Table 1. Actual and coded values of the factors applied in the experimental design

Factor	Level		
	-1	0	+1
Mass of the orange peel (g)	0.2	0.3	0.4
Dye concentration (mg L^{-1})	100	150	200
pH	4	5	6
Contact time (min)	20	30	40

The influences of the variables were evaluated using the statistical analysis with Minitab software.

RESULTS AND DISCUSSION

It was observed that, as expected, the higher the drying air temperature, the greater the reduction of moisture in the initial phase of the process (Figures 1A and B). This behavior has also been reported by other authors (Tamer et al., 2016; Corrêa Filho et al., 2018).

As shown in Figures 1A and B, the two-term exponential model fitted to the experimental data, at both temperatures evaluated in this study showed higher values of coefficient of determination (R^2) and F value and lower value of estimated mean error - SE, corroborating the good fit of the model to the experimental data (Table 2).

With the great diversity in the studies on the drying of residues, fruits, leaves and seeds reported in the literature, different models have shown satisfactory fits for the modeling of experimental data.

Although all models fitted well, with coefficients of determination above 0.99 and high F value, the Henderson & Pabis and logarithmic models, in the predicted values of M_p , showed values greater than 1, which are not acceptable. This behavior was also reported by Fiorentin et al. (2012) with the two-term exponential model, in the drying of orange bagasse.

According to Barros Neto et al. (2001), the model can be considered predictive if the F value is greater than 10, verified for both temperatures, corroborating the good fit of the model,

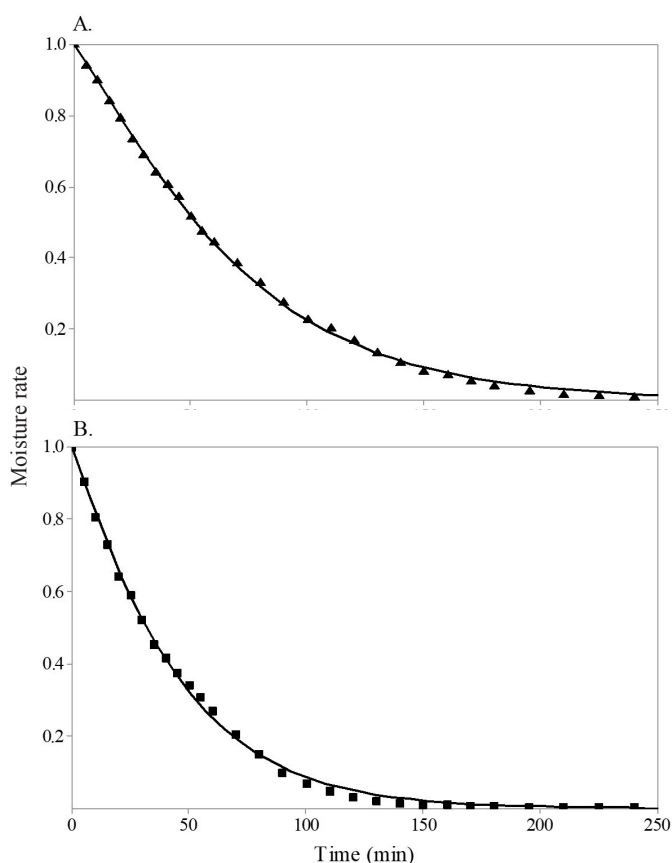


Figure 1. Moisture rate (MR) of orange peels as function of drying time at 60 °C (A) and 80 °C (B)

Table 2. Comparison of statistical parameters of the drying models

T (°C)	Model	M _i	M _f	k (min ⁻¹)	a (-)	n (-)	b (min ⁻¹)	c	F value	SE	R ²
60	Two-term Exp.	1	1.88E-12	0.019	1.662	-	-	-	4855.93	0.00994	0.999
	Page	1	7.6E-15	0.007	-	1.147	-	-	4246.35	0.01067	0.999
	Midilli	0.956	0.005	0.005	0.956	1.231	3.7E-06	-	2490.62	0.01042	0.999
	Logarithmic	1.024	0.033	0.013	1.057	-	-	0.033	1439.56	0.01564	0.998
	Henderson & Pabis	1.037	3.67E-10	0.015	1.037	-	-	-	1005.99	0.02139	0.996
80	Two-term Exp.	1	5.81E-18	0.027	1.597	-	-	-	2186.73	0.01348	0.998
	Page	1	6.75E-21	0.014	-	1.112	-	-	1894.91	0.01476	0.997
	Midilli	0.918	0.0005	0.007	0.918	1.272	3.67E-07	-	731.75	0.01717	0.996
	Logarithmic	1.012	0.018	0.021	1.030	-	-	0.018	975.08	0.01717	0.996
	Henderson & Pabis	1.019	7.78E-15	0.023	1.019	-	-	-	954.65	0.02001	0.995

SE - Standard error; M_i and M_f - Initial and final moisture contents, respectively; a, n, b, c, respectively coefficient of the model

which was considered a statistically significant and predictive model at 0.05 confidence interval.

The graph of ΔpH ($\text{pH}_{\text{Final}} - \text{pH}_{\text{Initial}}$) as a function of $\text{pH}_{\text{Initial}}$ for the biosorbent, is presented in Figure 2A, which shows that the point of zero charge (pH_{pzc}) of the material is approximately 3.5. Guiza (2017) evaluated the point of zero charge of the orange peel and obtained pH_{pzc} of 3.85, and Nascimento et al. (2014) obtained pH_{pzc} of 3.9.

According to Guiza (2017), in solutions with pH below the pH_{pzc} , the surface of the material charged with H⁺ ions favors the adsorption of dyes with anionic characteristic, and, in a solution with pH above the pH_{pzc} , the surface is charged with OH⁻ ions, favoring the adsorption of cationic dyes.

The micrograph of the biosorbent made from orange peel can be observed in Figure 2B. It is observed that the material exhibited heterogeneous surface. Mafrá et al. (2013) state that orange peel has porous and highly heterogeneous structure, characteristics that were also reported by Guiza (2017).

The FTIR spectrum of the biosorbent material can be observed in Figure 2C, where several absorption bands were identified, which indicate the presence of several functional groups in the material. The FTIR spectrum showed similar behavior to that observed by Khaled et al. (2009) and Mafrá et al. (2013).

The extensive absorption of the band around 3350 cm⁻¹ corresponds to the stretching vibrations of the O-H groups, which may be related to the presence of the hydroxyl groups free of carboxylic acids, alcohols and phenols on the adsorbent surface or corresponds to O-H stretching vibrations of cellulose, pectin, absorbed water and lignin (Djilani et al., 2012; Cypriano et al., 2017).

Bands centered at 2900 cm⁻¹ are attributed to the symmetrical and asymmetrical stretching vibrations of CH of aliphatic molecules. In turn, the bands close to 1750 cm⁻¹ indicate stretching vibrations of the non-ionic carbonyl fraction of carboxylic acids and esters, as observed by Feng et al. (2010) and Djilani et al. (2012).

There are bands in the region of 1600 cm⁻¹, which represented the axial deformation of the carboxylate ions, COO⁻, of the free carboxyl, that is, of the non-esterified carboxylic groups, as described by Gnanasambandam & Proctor (2000).

The vibration of aliphatic acid groups at 1200 cm⁻¹ can be attributed to the deformation of C = O groups and to the elongation of formation of -OH groups of carboxylic acids

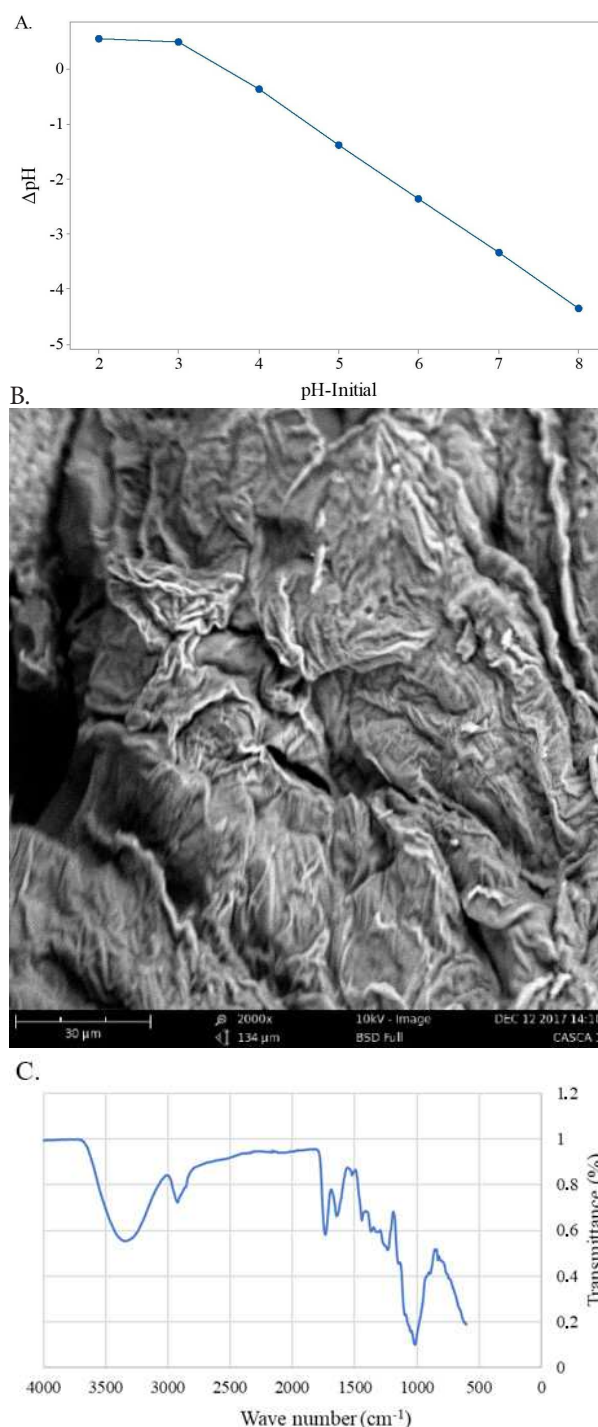


Figure 2. Graph of the point of zero charge - pH_{pzc} (A), micrographs of the biosorbent material with 2000x magnification (B) and Fourier transform infrared spectrum - FTIR (C)

and phenols, as reported by Mafra et al. (2013) and Santos & Morais (2015).

The Pareto charts, shown in Figure 3, show the influences of the factors on the qt (Figure 3A) and reduction percentage of the dye (Figure 3B), respectively.

The factors are statistically significant at $p \leq 0.05$ for adsorbed quantity of dye (qt). For the reduction of the dye, the initial concentration of the dye was not significant, while the contact time, mass of orange peels and pH solution were statistically significant at $p \leq 0.05$.

The dye initial concentration had a positive effect on the qt, evidencing that, with the increase in the solution concentration, the quantity of adsorbed dye increased and such condition occurs due to the saturation of the dye solution, which facilitates the adsorption process.

According to Noreen et al. (2013), the initial dye concentration plays an important role, because it reduces the resistance to dye mass transfer between the solid and aqueous phases. The authors evaluated the removal of the black CL-B dye in peanut husk and observed similar behavior, in which increasing the concentration from 10 to 600 mg L⁻¹ increased the adsorbed quantity from 22 to 48 mg g⁻¹.

Safa & Bhatti (2011) evaluated the adsorption of direct red-31 dye in rice husk and observed that, with the increase in the initial concentration, the adsorbent quantity increased from 11.19 to 65.56 mg g⁻¹.

The increase of mass from 0.2 to 0.4 g had a negative effect on qt, decreasing 44.56% the adsorbed quantity, i.e., there is a sub-utilization of the biosorbent material.

Nascimento et al. (2014) evaluated the adsorption of the reactive gray BF-2R dye in orange peel and obtained similar results, in which increasing the mass from 0.25 to 0.75 g led to a reduction of 50.7% in the adsorption capacity.

For the removal percentage of the dye, the mass of orange peels had a positive effect, indicating that the increment in the biosorbent mass increases the number of active sites in the medium, and consequently, it causes a greater reduction of the dye concentration.

Rehman et al. (2012) studied the removal of methylene blue dye with rice husk as biosorbent and observed a similar behavior, in which the biosorbent mass also had a positive effect.

The contact time had a positive effect on both responses evaluated; therefore, as the time of contact between the liquid phase and the solid phase increases, the adsorbed quantity and the percentage of dye removal increase.

Pavan et al. (2007) evaluated the removal of methylene blue dye using yellow passion fruit peel and mandarin peel and the variable time also had a positive effect on the percentage of dye removal.

The pH solution had a negative effect on both evaluated responses, indicating that, under conditions of higher concentration of H⁺ ions, there is an increase in the adsorption capacity and removal percentage.

The pH is a factor of great importance in the study of the adsorption of dyes, because it affects the superficial charge of the biosorbent; thus, the change in solution pH affects the adsorption process through the dissociation of the functional groups at the active sites of the surface of the biosorbent. Asfaram et al. (2014) studied the adsorption of the direct red 12B dye in garlic peel, varying the values from 2 to 10, and observed that the adsorption capacity was elevated in solutions with lower pH.

Table 3 presents the analysis of variance for both response variables evaluated.

It was observed that the models fitted well to the experimental data, showing highly expressive coefficients of determination, where more than 92% of the variations in the responses can be explained by the models.

According to Barros Neto et al. (2001), the model for the response qt can be considered statistically significant and predictive. For the response reduction, the value of the F test was higher than 1, and the model was statistically significant at $p \leq 0.05$.

The contour plots in Figure 4 show the influence of the variables dye initial concentration, mass orange peels and pH solution, at the fixed time of 40 min, for the adsorbed quantity (qt) and removal percentage of dye (R%).

It was observed that, for qt, the higher the mass of biosorbent, the lower the adsorbed quantity, evidencing the good potential of the biosorbent material, which had good adsorption capacity even in small quantities.

For the removal percentage of dye, it can be stated that the dye initial concentration was not a factor of great importance, and the orange peel mass was the main factor. Both responses evaluated were favored in acid medium, because higher values of qt and removal percentage of dye were observed at pH solution equal to 4.

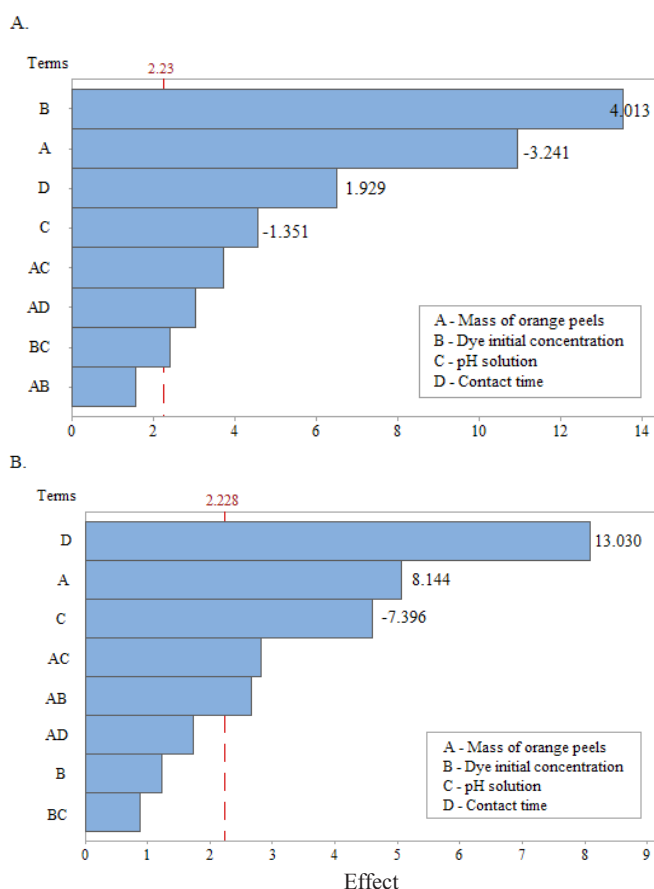
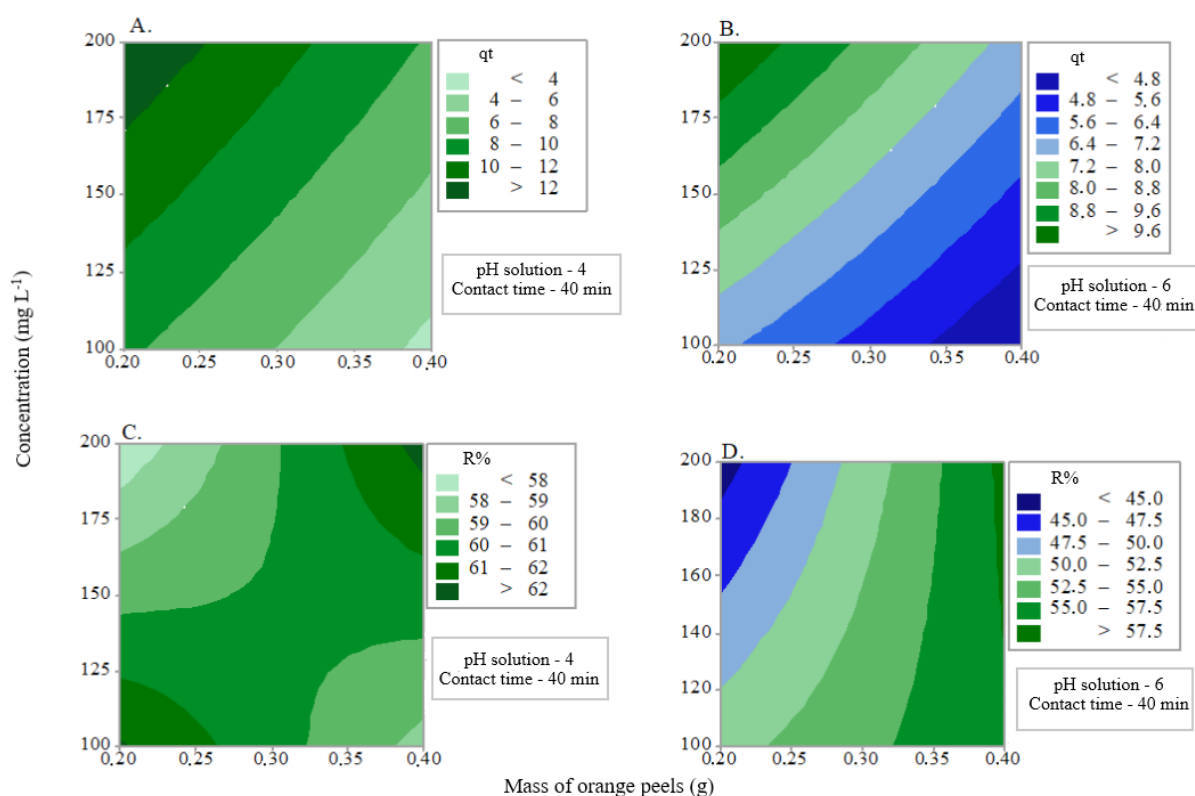


Figure 3. Pareto charts of the standardized effects for the adsorbed quantity - qt (A) and removal percentage - R% of dye as function of evaluated factors (B)

Table 3. Analysis of variance for the factors mass of orange peels, dye initial concentration, pH solution and contact time for the adsorbed quantity (qt) and removal percentage (R%) of dye

Source of variation	Sum of squares	DF	Quadratic mean	F (calculated)	F (tabulated)	F test	R ²
qt							
Regression	139.61	8	17.450	49.71	3.07	16.19	0.975
Residual	3.51	10	0.351				
Total	143.12						
R%							
Regression	1372.68	8	171.58	16.51	3.07	5.37	0.929
Residual	103.98	10	10.39				
Total	1476.65						

DF - Degrees of freedom

**Figure 4.** Contour plots for the mass of orange peels and dye initial concentration, at contact time of 40 min for the adsorbed quantity (qt) with pH solution of 4.0 (A) and with pH solution of 6.0 (B); and for the removal percentage (R%) with pH solution of 4.0 (C) and with pH solution of 6.0 (D)

CONCLUSIONS

1. The two-term exponential model is the most appropriate to describe the drying of orange peel at temperatures of 60 and 80 °C.

2. The biosorbent made from orange peel proves to be efficient for removing burgundy-16 dye, being able to remove more than 60% of the dye load at 40 min.

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