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Optimized Adsorption of Small and Medium Molecules by a Biosorbent Based on Hevea Hulls

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Abstract

In the context of the recovery of agricultural waste, many researches have focused on the preparation of adsorbents from natural waste from fruit trees, egg shells, palm waste or sawdust. This work aims to optimize the preparation of a biosorbent from rubber hulls by studying its ability to adsorb small and medium molecules. The influence of parameters such as drying temperature (X1), particle size (X2), stirring time (X3) and sodium hypochloride mass (X4) was studied. The results indicate that the model used for biosorbent optimization on methylene blue and iodine index is significant. In addition, this model has greater adsorption capabilities on small molecules than with large molecules. Statistical analysis of the data shows that temperature is the most influential factor in the adsorption of small molecules. On the other hand, particle size has a significant influence on the adsorption of large molecules. The optimum biosorbent preparation values are 1.0 for drying temperature (X1), −1.0 for biosorbent grain size (X2), 1.0 for stirring time (X3) and 1.0 for sodium hypochloride mass (X4).

Keywords

Optimization, Biosorbent, Methylene Blue Index, Iodine Index, Rubber Hulls

1. Introduction

Industrial wastewater and pollutants from chemical fertilizers, pesticides, sanitizers and pharmaceuticals are the major causes of water and environmental pollution [1]. This is a growing threat to humans and ecosystems.

In recent years many techniques have been developed to eliminate pollutants in water, air and soil. These techniques include membrane filtration, electrolysis, photodegradation and precipitation-coagulation [2] [3]. These methods have the disadvantage of being ineffective for certain types of pollutants and being relatively expensive. Thus, the implementation of simpler and inexpensive processes becomes indispensable.

Many researchers have focused on the preparation of some adsorbents from natural waste from fruit trees [4], egg shells [5], palm waste [6] or sawdust [7]. It is in this context that the present study intervenes, which aims to optimize the adsorption capacities of a biosorbent from rubber hulls to adsorb small and medium molecules. It consists in modelling the biosorbent preparation process by a complete experiment plan at two levels, to study the influence of certain parameters on the adsorption capacities of the biosorbent and to optimize the various parameters on the methylene blue index and the iodine index.

2. Material and Methods

2.1. Biosorbent Preparation Protocol

Choice of factors

An adequate choice of parameter variation domains is an essential condition for establishing a precise model that perfectly describes the studied process [8]. Based on a bibliographic analysis [9] [10], we developed a complete factorial plan with a number of factors equal to four namely drying temperature (X1), biosorbent granulometry (X2), stirring time (X3) and concentration (X4). Each factor takes two levels namely the low level (−1) and the high level (+1). The various factors and their areas are summarized in **Table 1**.

Table 1. Factors and their different areas of variation.

Factors	Variables	Low level (A)	High level (B)
Temperature (˚C)	X1	75	120
Granulometry (mm)	X2	0.2 - 0.5	0.5 - 1
Agitation time	X3	12	24
Concentration (mol/L)	X4	0.107 (8 g)	0.214 (16 g)

Different stages of biosorbent preparation

The biosorbent used has been pre-treated to remove sand and coarse elements. Subsequently, the preparation required several stages of treatment:

Drying

Rubber hulls (**Figure 1**) are dried in an oven at two different temperatures for 24 hours at 75˚C and 120˚C. The rubber hulls used in this study were collected in the Daloa area, a city in Côte d'Ivoire.

Grinding and sieving

Rubber hulls are dried and crushed (**Figure 2**) using a crusher and then sieved with sieves of diameters 1 mm; 0.5 mm and 0.2 mm to obtain a grain size between 0.2 and 0.5 mm then between 0.5 and 1 mm (Figure 3).

Figure 1. Rubber hulls.

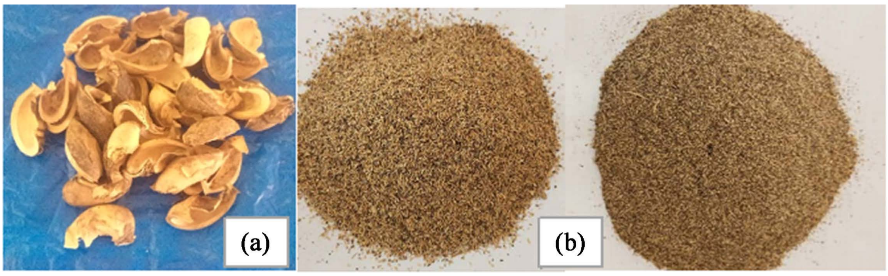
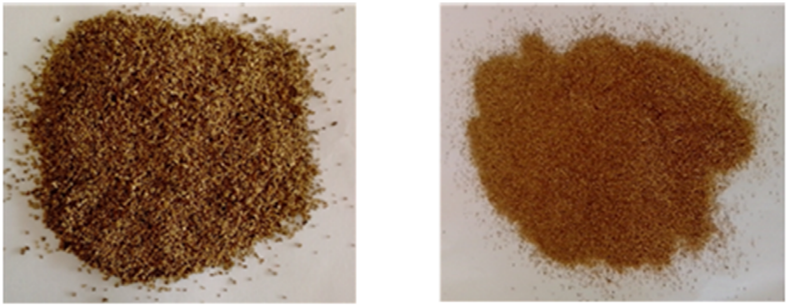


Figure 2. Rubber hulls after drying (a) and grinding (b).



(a) (b)

Figure 3. Rubber hulls after sieving. (a) Rubber hulls after sieving with a diameter between 0.5 and 1 mm; (b) Rubber hulls after sieving with a diameter between 0.2 and 0.5 mm.

Washing

After grinding, the biosorbent was washed at room temperature. This consists of removing all impurities and soluble substances from the samples (pigment). Washing is carried out first with tap water until the washing water is colorless. The samples are then bleached with a sodium hypochlorite (bleach) solution of concentrations 0.107 mol·L^{-1} and 0.214 mol·L^{-1}, and rinsed with distilled water. At the end of washing the biosorbents are dried in an oven at 75˚C and 120˚C for 24 hours.

Methylene blue index

Methylene blue index (mg/g) is an indicator of the biosorbent's ability to adsorb medium and large organic molecules. It characterizes the mesopores of the crushed rubber hull. For the determination of this index, the method of the

European Centre of Chemical Industry Federations from the work of Mamane *et al.* [11] was used. In a 250 mL erlenmeyer, 0.1 g of crushed rubber shell (biosorbent) and 100 mL of 1944 × 10^{-5} M methylene blue solution is introduced.

The mixture is stirred for 20 min then filtered. The residual methylene blue concentration is determined using a UV-Visible spectrophotometer at a wavelength of 620 nm. Thus, the methylene blue index (Q_{MB}) is given by the following relation:

$$Q_{MB} = \frac{M \cdot V \cdot (C_i - C_r)}{m_b}$$

With:

V: Volume of methylene blue solution in (mL)

M: Methylene blue molar mass (g/mol)

Ci: Initial methylene blue concentration (mol/L)

Cr: Residual methylene blue concentration in (mol/L)

m_b: Mass of biosorbent (g).

Q_{MB}: Methylene blue index (mg/g)

Iodine index

The purpose of the iodine index is to determine the ability of the crushed rubber shell to adsorb small molecules. It characterizes micropores accessible to small particles. This test was performed according to the AWWA B 600-78 standard from the work of Maazou *et al.* [12]. In a 100 mL beaker, 0.05 g of crushed rubber shell (biosorbent) is introduced. Add 20 mL of 0.1 N iodine solution to the pipette and stir for 5 min before filtering. A volume of 10 mL of the filtrate is taken and put into an erlenmeyer. From the burette, a solution of sodium thiosulphate with a concentration of 0.1 N is added to the erlenmeyer containing the filtrate until the total discoloration of the solution. Starch is used as a coloured indicator. Iodine index (Q_{II}) is given by the following relationship:

$$Q_{Ii} = \frac{\left(C_o - \frac{C_{thio} V_{thio}}{2V_{I2}}\right) M_{I2} V_{ads}}{m_b}$$

With:

C_0: Initial concentration of iodine solution (mol/L)

C_{thio}: Concentration of sodium thiosulfate solution (mol/L)

V_{thio}: Volume of thiosulfate poured at equivalence (mL)

V_{I2}: Iodine volume (mL)

M_{I2}: Molar mass of diiodine (g/mol)

$Vads$: Sample filtrate volume (mL)

m_b: Crushed rubber shell mass (g)

Q_{Ii}: Iodine index (mg/g)

2.2. Treatment of the Results

Results are processed using multiple linear regression and analysis of variance. It states that the response is a linear function of all factors. Thus, to determine this function is to find the coefficients of the following polynomial equation:

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_4X_4 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{14}X_1X_4 + a_{23}X_2X_3 + a_{24}X_2X_4 + a_{34}X_3X_4$$

With:

a_i: the effect of factor Xi

a_{ij}: the effect of two-fold interactions

a_{ijk}: the effect of three-fold interactions

a_{ijkl}: the effect of four-fold interactions

X_iX_j: the interaction between X_i and X_j

The determination of the experimental domain, experimental designs, estimates and statistics of the polynomial coefficients is carried out using the MINITAB 19 software.

3. Results and Discussion

3.1. Experimental Design and Experimental Results

The experimental results of the methylene blue index and the iodine index obtained according to the complete factor design are reported in Table 2.

Methylene blue shows between 2.80 and 4.88 mg/g, while iodine shows between 203.05 and 263.96 mg/g.

Table 2. Experiment matrix and experimental data.

N˚ order	X1	X2	X3	X4	Y1 (MB index)	Y2 (ID index)
1	−1	−1	−1	−1	4.34	228.50
2	1	−1	−1	−1	4.77	243.66
3	−1	1	−1	−1	2.99	213.20
4	1	1	−1	−1	3.53	233.51
5	−1	−1	1	−1	4.30	228.43
6	1	−1	1	−1	4.88	243.66
7	−1	1	1	−1	2.95	203.05
8	1	1	1	−1	2.80	233.51
9	−1	−1	−1	1	3.58	203.05
10	1	−1	−1	1	4.57	248.73
11	−1	1	−1	1	3.49	228.43
12	1	1	−1	1	2.81	238.43
13	−1	−1	1	1	4.18	263.96
14	1	−1	1	1	4.56	263.96
15	−1	1	1	1	3.5	243.66
16	1	1	1	1	4.33	228.43

Values for iodine are higher than those for methylene blue. This shows that biosorbents from rubber hulls have more capacity to adsorb small molecules than medium molecules. The maximum ID value is obtained for a biosorbent with a diameter between 0.2 and 0.5 mm, regardless of its drying temperature. The results of the iodine index obtained are similar to those of Benamraoui [13] recorded during his work on biosorbents from apricot and olive kernels.

In addition, our work generally shows that the highest methylene blue index values are obtained with small diameter biosorbents (0.2 and 0.5 mm). These results reveal that particle size determines the adsorption rate; the smaller the grains, the faster the transfer to the center [14].

3.2. Estimation of Coefficients

The estimated coded coefficients are given in **Table 3** and **Table 4**.

Table 3. Coding coefficients for methylene blue index (IBM).

Term	Effect	Coeff	Coef ErT	T-value	p-value	FIV
Constant		3.9083	0.0499	78.32	0.000	
X1	0.3995	0.1997	0.0499	4.00	0.001	1.00
X2	−1.0156	−0.5078	0.0499	−10.18	0.000	1.00
X3	0.1614	0.0807	0.0499	1.62	0.121	1.00
X4	0.1369	0.0685	0.0499	1.37	0.185	1.00
X1*X2	−0.1979	−0.0990	0.0499	−1.98	0.061	1.00
X1*X3	−0.0259	−0.0130	0.0499	−0.26	0.798	1.00
X1*X4	0.0811	0.0406	0.0499	0.81	0.426	1.00
X2*X3	−0.0539	−0.0270	0.0499	−0.54	0.595	1.00
X2*X4	0.4227	0.2113	0.0499	4.24	0.000	1.00
X3*X4	0.2513	0.1256	0.0499	2.52	0.020	1.00

From **Table 3**, it is noted that the p-values obtained are less than 0.05 ($p < 0.05$) for drying temperature (X1) and grain size (X2). This shows that these two parameters have a very significant effect on the adsorption capacities of the biosorbent. Also, the interaction between the particle size and the concentration of sodium hypochloride X2*X4 and the interaction between the stirring time and the mass of sodium hypochloride X3*X4 have p values less than 0.05. This suggests that these interactions greatly influence the adsorption capacities of the medium molecules of the biosorbent from rubber shells.

In addition, the coded regression equation linking parameter levels to IBM is given by:

$$IBM = 3.9083 + 0.1997X_1 - 0.5078X_2 + 0.0807X_3 + 0.0685X_4 - 0.0990X_1{*}X_2 - 0.0130X_1{*}X_3 + 0.0406X_1{*}X_4 - 0.0270X_2{*}X_3 + 0.2113\,X_2{*}X_4 + 0.1256X_3{*}X_4$$

Table 4. Coded iodine index coefficients.

Term	Effect	Coeff	Coef ErT	T-value	p-value	FIV
Constant		232.60	1.82	127.50	0.000	1.00
X1	16.30	8.15	1.82	4.47	0.000	1.00
X2	−12.46	−6.23	1.82	−3.42	0.003	1.00
X3	10.86	5.43	1.82	2.98	0.007	1.00
X4	11.61	5.80	1.82	3.18	0.005	1.00
X1*X2	−2.32	−1.16	1.82	−0.64	0.532	1.00
X1*X3	−8.49	−4.24	1.82	−2.33	0.030	1.00
X1*X4	−6.00	−3.00	1.82	−1.65	0.115	1.00
X2*X3	−10.26	−5.13	1.82	−2.81	0.010	1.00
X2*X4	2.26	1.13	1.82	0.62	0.541	1.00
X3*X4	10.48	5.24	1.82	2.87	0.009	1.00

The results obtained for the iodine index (ID) response coefficients show that parameters such as temperature (X1), grain size (X2), stirring time (X3) and sodium hypochloride concentration (X4) have p values less than 0.05 (Table 4). Indeed, these parameters have an important influence on the adsorption capacities of a biosorbent from the rubber husks to adsorb small molecules. Also, the interaction between the particle size and the mass of sodium hypochloride (X2*X4), then the interaction between the stirring time and the mass of sodium hypochloride (X3*X4) and then the interaction between the temperature and the stirring time have p values less than 0.05. These different results show that these interactions strongly influence the adsorption capacities of the small molecules of the biosorbent from the rubber shells.

Equation de régression codées reliant les niveaux paramètre avec indice d'iode est:

$$ID = 232.60 + 8.15X_1 - 6.23X_2 + 5.43X_3 + 5.80X_4 - 1.16X_1{*}X_2 - 4.24X_1{*}X_3 - 3.00 X_1{*}X_4 - 5.13X_2{*}X3 + 1.13X_2{*}X_4 + 5.24X_3{*}X_4$$

It appears from the analyses of these two tables above that the adsorption capacities of the small molecules are larger than those of the average molecules with the biosorbent from the rubber shells.

3.3. Statistical Analysis and Study of Interactions

Analyse de la variance

The results of Table 5 show that particle size (X2), is the most important factor in the optimization of the biosorbent on the methylene blue index (IBM). Indeed, the contribution of grain size to the elaboration of the answer is 59.41%.

All the studied parameters influence the optimization of the biosorbent on the iodine index (ID). However, none of these parameters contribute significantly to the development of the response (Table 6).

Table 5. Variance analysis results for methylene blue index (IBM).

Source	DL	SomCar ajust	Contribution (%)	CM ajust	F-value	p-value
Model	10	12.2164	87.95231033	1.22164	15.33	0.000
Linear	4	9.8873	71.183389034	2.47183	31.02	0.000
X1	1	1.2767	9.191637029	1.27675	16.02	0.001
X2	1	8.2522	59.41194258	8.25222	103.56	0.000
X3	1	0.2084	1.500381575	0.20836	2.61	0.121
X4	1	0.1500	1.079929157	0.15001	1.88	0.185
2-factor interactions	6	2.3290	16.76770004	0.38817	4.87	0.003
X1*X2	1	0.3134	2.256331985	0.31341	3.93	0.061
X1*X3	1	0.0054	0.038877449	0.00537	0.07	0.798
X1*X4	1	0.0526	0.378695157	0.05263	0.66	0.426
X2*X3	1	0.0233	0.167748995	0.02327	0.29	0.595
X2*X4	1	1.4292	10.289565	1.42925	17.94	0.000
X3*X4	1	0.5051	3.636481447	0.50510	6.34	0.020
Error	21	1.6735	12.04840962	0.07969		
Lack-of-fit	5	1.0123	7.288081902	0.20246	4.90	0.007
Pure error	16	0.6612	4.760327723	0.04132		
Total	31	13.8898	100			

Table 6. Variance analysis results for iodine index (ID).

Source	DL	SomCar ajust	Contribution (%)	CM ajust	F-value	p-value
Model	10	8059.6	78.278183	805.96	7.57	0.000
Linear	4	5389.1	52.4168609	1347.28	12.65	0.000
X1	1	2125.2	20.64413364	2125.17	19.95	0.000
X2	1	1242.2	12.06487956	1242.19	11.66	0.003
X3	1	944.4	9.172494172	944.35	8.87	0.007
X4	1	1077.4	10.46425796	1077.43	10.12	0.005
2-factor interactions	6	2670.4	25.93628594	445.07	4.18	0.006
X1*X2	1	43.1	0.418609168	43.08	0.40	0.532
X1*X3	1	576.3	5.597319347	576.34	5.41	0.030
X1*X4	1	288.5	2.802059052	288.48	2.71	0.115
X2*X3	1	842.4	8.181818182	842.44	7.91	0.010
X2*X4	1	41.0	0.398212898	41.03	0.39	0.541
X3*X4	1	879.1	8.5338267288	879.08	8.25	0.009

Continued

Erreur	21	2236.5	21.72202797	106.50		
Lack-of-fit	5	2056.2	19.97086247	411.23	36.49	0.000
Pure error	16	180.3	1.751165501	11.27		
Total	31	10296.1	100			

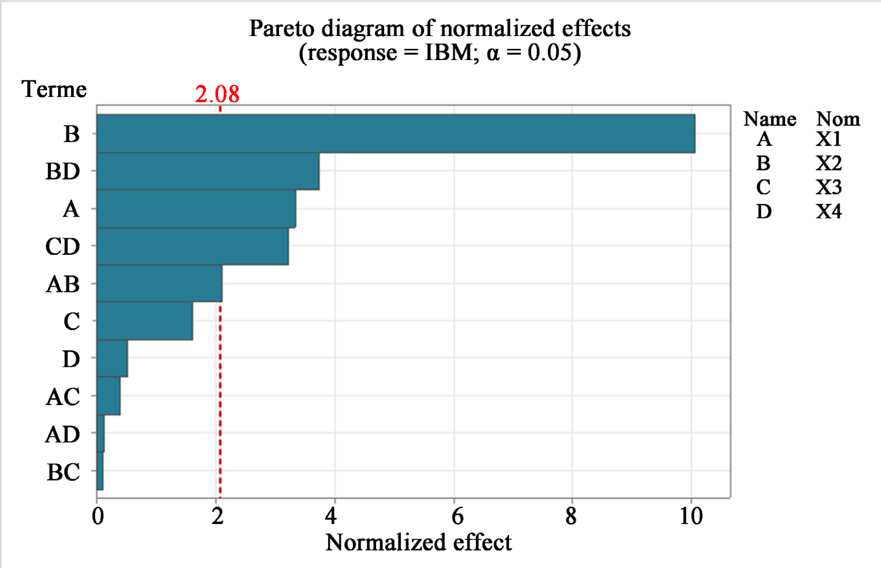


Figure 4. Pareto diagram of normalized methylene blue index (IBM) effects.

Direct effects of parameters on methylene blue index

The Pareto diagram (**Figure 4**) shows that drying temperature appears to be the most influential factor in the optimization of biosorbent from rubber hulls. Then comes the particle size which also contributes to this optimization of the biosorbent. This observation was made by Mamane [11] and Aboua [15] during their work on the optimization of the conditions of active charcoal production. According to this work, small particles have a large specific surface area and therefore lead to better adsorption and shortening of the equilibrium time. The results of the diagram show that temperature and particle size strongly influence the biosorbent's ability to adsorb medium molecules. Also, the effect of the interactions between the granulometry and the sodium hypochloride mass, and between the stirring time and the sodium hypochloride mass is significant on the optimization of the biosorbent to the adsorption of medium molecules.

Direct effects of parameters on iodine index

The Pareto diagram (**Figure 5**) shows the existence of several influencing factors. Indeed, all the studied parameters strongly influence the biosorbent's ability to adsorb small molecules. In addition, certain interactions between the different parameters also contribute to the optimization of the biosorbent in the adsorption of small molecules.

The diagram of the main effects on IBM (**Figure 6**) confirms that drying

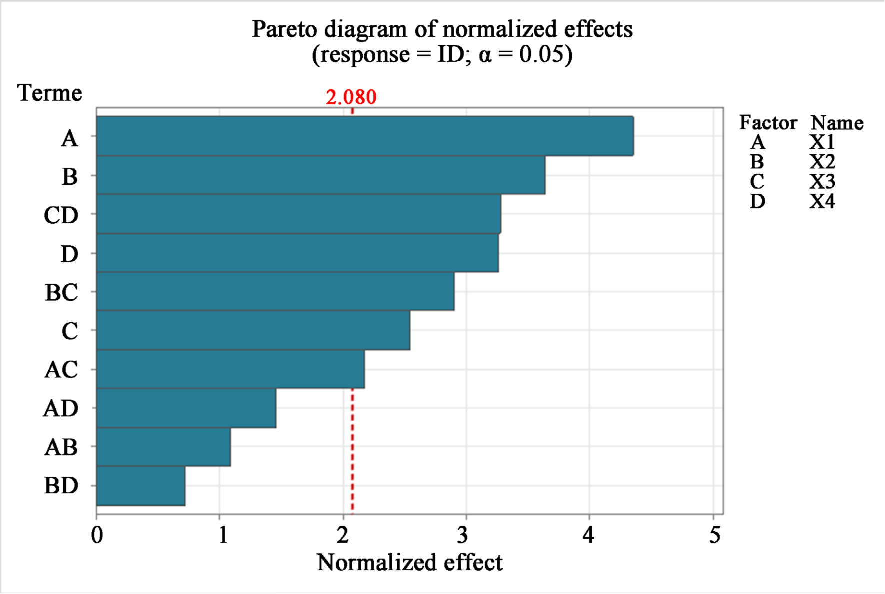


Figure 5. Pareto diagram of standardized iodine index (ID) effects.

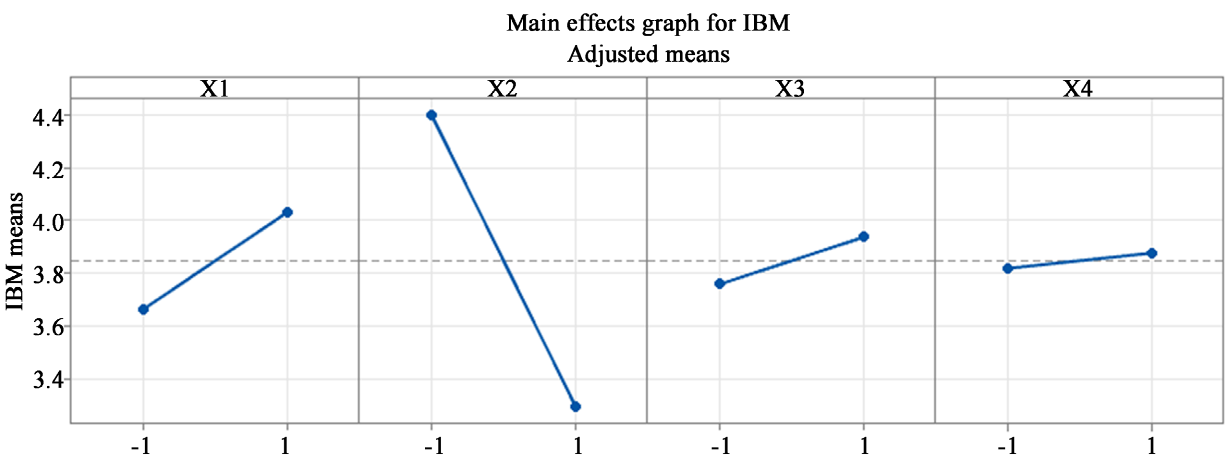


Figure 6. Main effects of methylene blue index graph.

temperature and particle size strongly influence the adsorption capabilities of the biosorbent to adsorb medium molecules. In the diagram of the main effects on ID (Figure 7), we also observe that all parameters have a positive influence on the adsorption capacities of small molecules.

Effect of factor interactions

There are interactions between granulometry and sodium hypochloride mass that influence the adsorption of medium molecules (Figure 8). This interaction would modify the functional groups on the surface of our biosorbent. In addition, interactions are observed between the majority of the parameters that contribute to modify the adsorption capacities of the biosorbent for small molecules (Figure 9).

3.4. Optimization of the Various Parameters

Outline diagrams

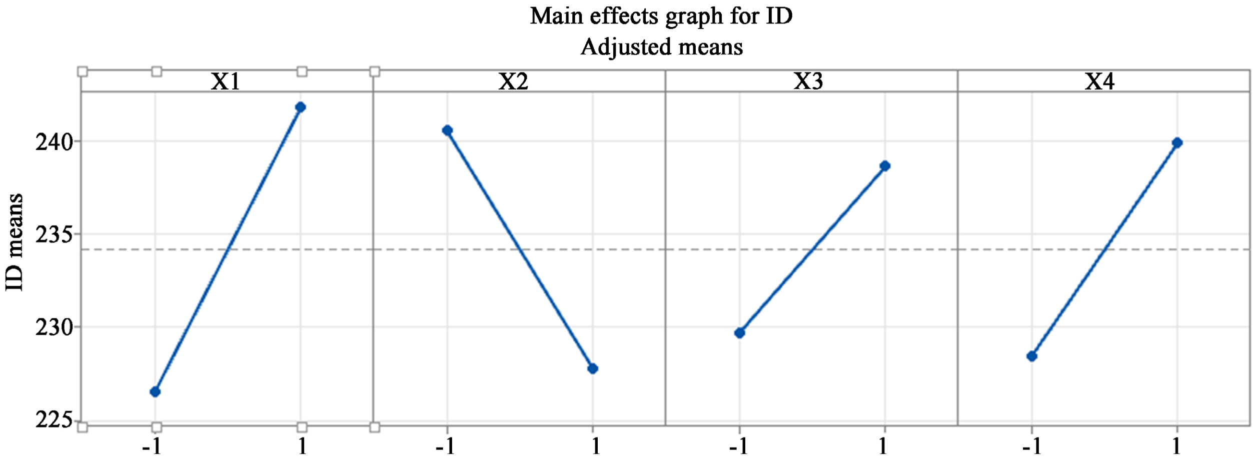


Figure 7. Iodine index major effects graph.

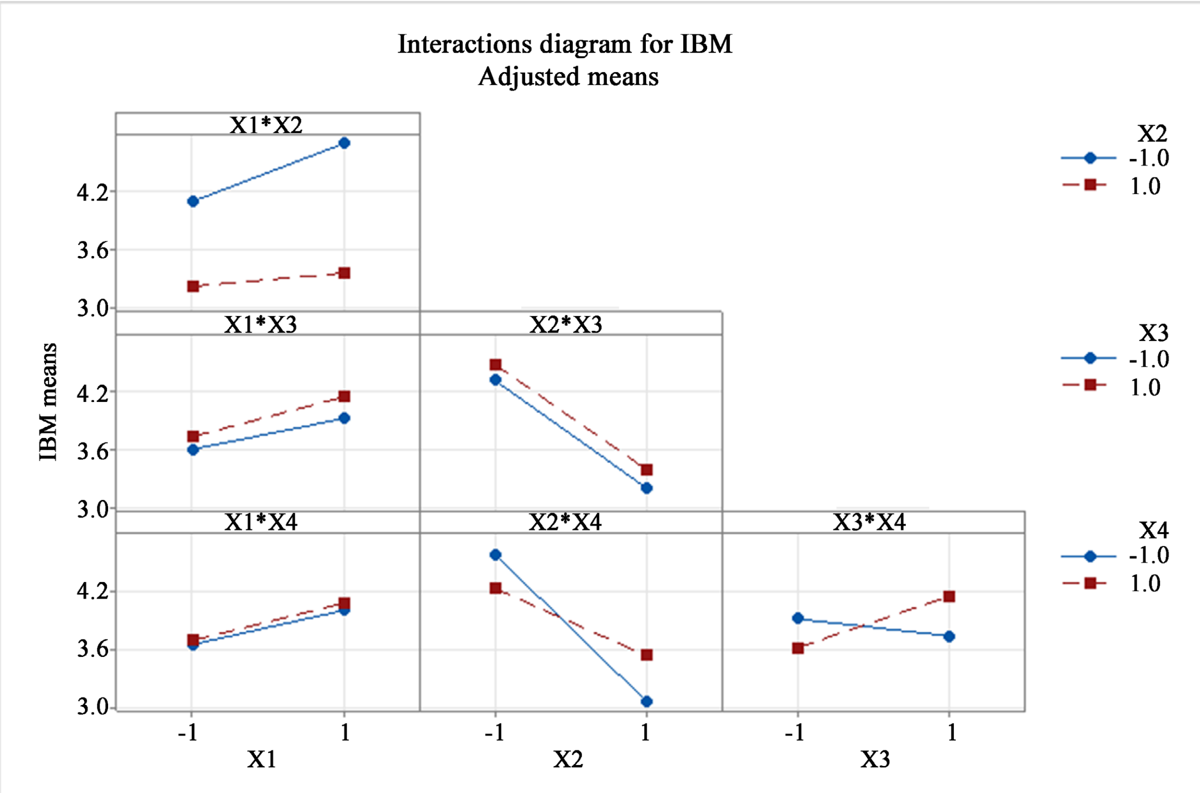


Figure 8. Methylene blue index interactions diagram.

Figure 10 and Figure 11 show the drying temperature contour areas (X1) and the grain size (X2) that maximize the IBM and ID responses respectively by keeping the stirring time (X3) and the sodium hypochloride mass (X4) constant. Indeed, the contour areas that optimize these responses are close to 120˚C and a grain size between 0.2 and 0.5 mm. This suggests that a high temperature has a significant influence on the microporosity of absorbent thus improving their adsorption capacity. According to Park *et al.* [16], a higher temperature generally improves the response but can damage the physical structure of the biosorbent.

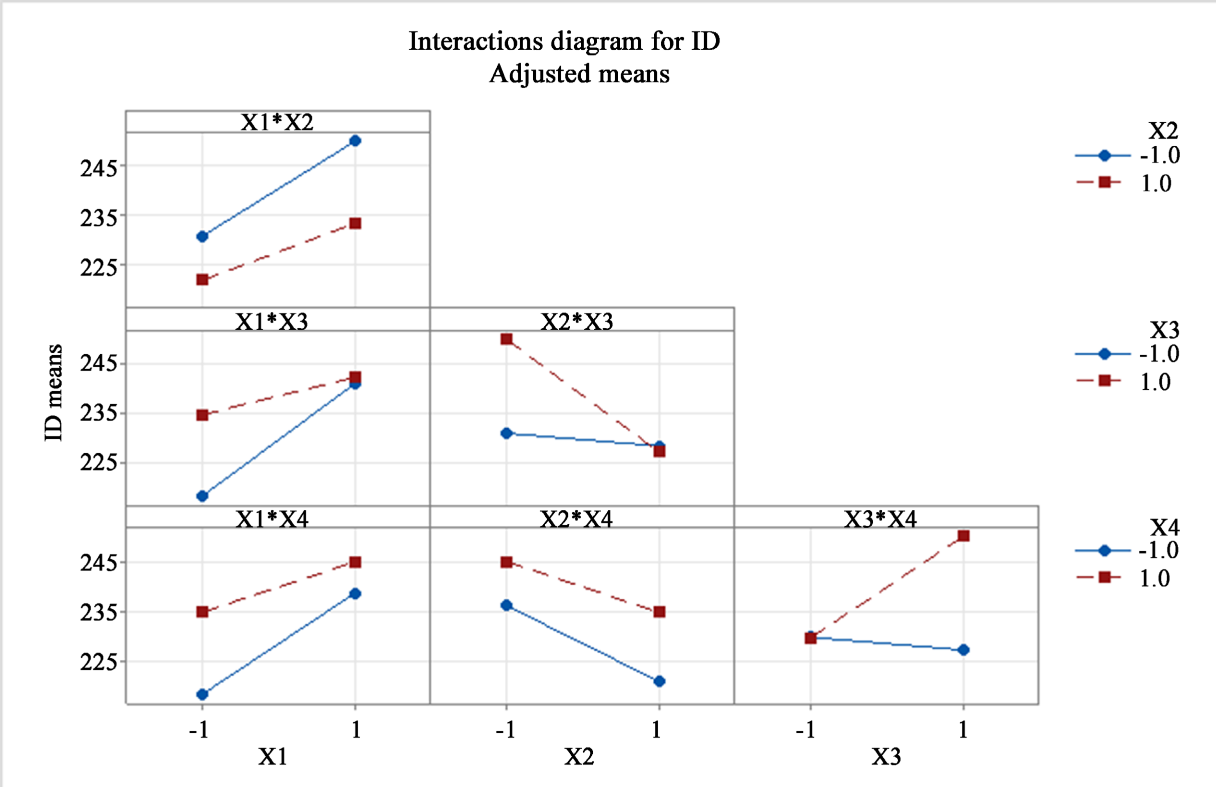


Figure 9. Iodine index interactions diagram.

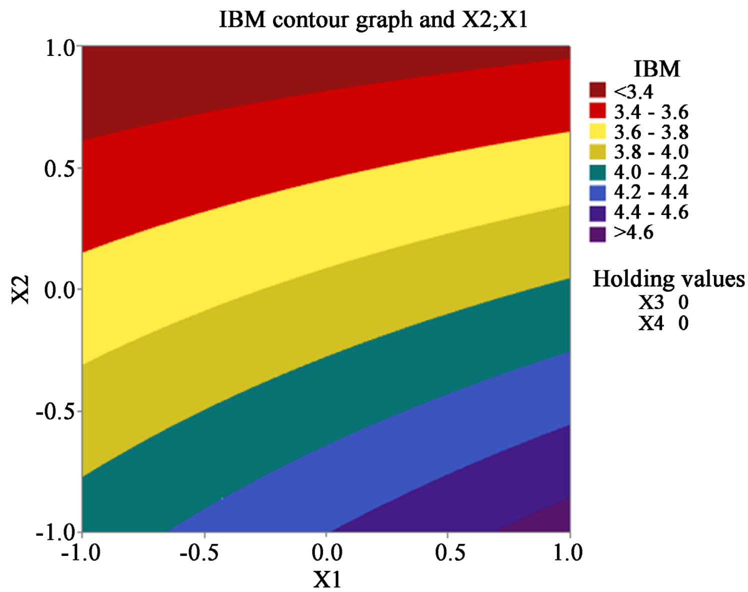


Figure 10. Methylene blue index contour diagram.

Multi-response optimization

Figure 12 shows the optimization results of biosorbent preparation on IBM and ID. We find that the answers have the same optimization values for the different parameters which are X1 = 1; X2 = −1; X3 = 1; X4 = 1. These results are acceptable because it is desirable to remain in the optimal conditions comparable

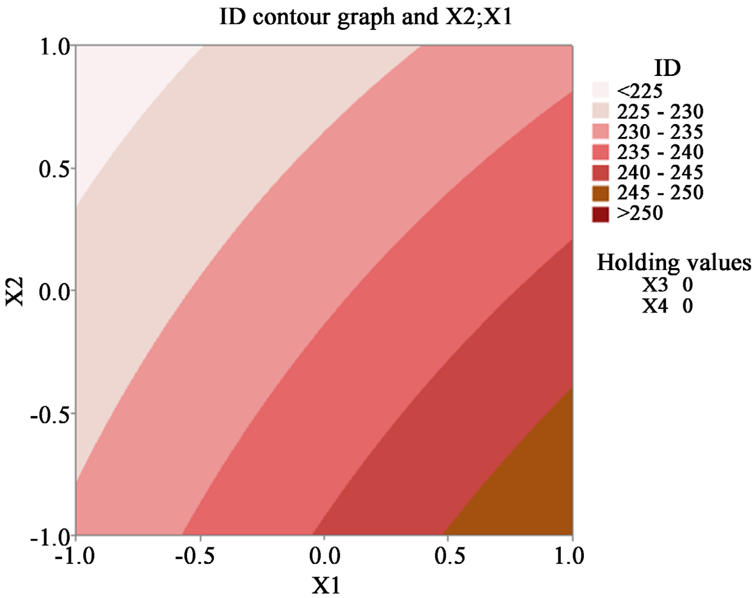


Figure 11. Iodine index contour diagram.

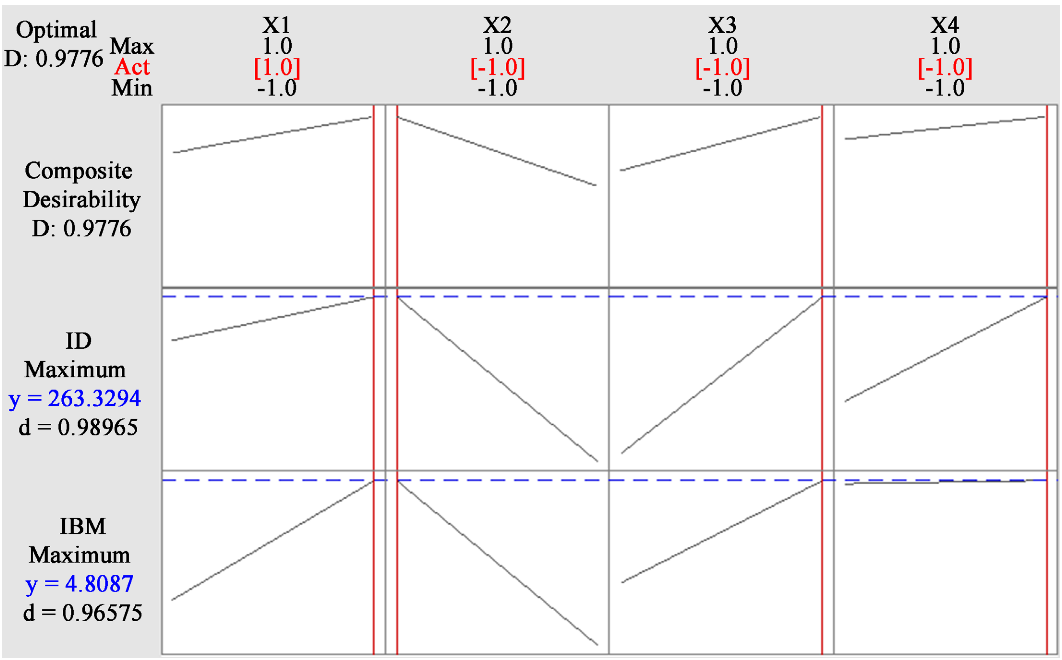


Figure 12. Multi-response optimization graph.

to those recorded in order to obtain an optimal value and optimal condition for biosorbent preparation [17]-[19]. It can also be noted that the desirability value (d) of IBM and the ID is 1. However, the closer the value of d is to 1, the more efficient the optimization is (Rani, 2015) [4].

4. Conclusion

The study of the adsorption capacities of small and medium molecules on a biosorbent from rubber hulls was the subject of this work. Parameters such as drying temperature, particle size, stirring time and sodium hypochloride mass were used to explain their influence on the preparation and adsorption capacity of the biosorbent. At the end of this study, it appears that the model used for biosorbent optimization on methylene blue and iodine indices is significant. However, the results obtained show that this biosorbent has great capabilities to treat small molecules. Statistical analysis of the data, including the Pareto diagram, reveals that temperature is the most influential factor in the treatment of small molecules. On the other hand, for large molecules, it is rather the grain size. The optimum biosorbent preparation values are 1.0 for drying temperature (X1), −1.0 for biosorbent grain size (X2), 1.0 for stirring time (X3) and 1.0 for sodium hypochloride mass (X4). The desirability value (d) of methylene blue index and iodine index close to 1 confirms that optimization is effective.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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