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Contribution to the Modeling of a Biosorbent Derived from Cocoa Pod Shells on the Methylene Blue Index

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Abstract: The objective of this study is to improve the preparation of a biosorbent from the cocoa pod shell by an experimental plan. The shells underwent drying and grinding followed by chemical impregnation with sodium hypochlorite. The process was optimized based on the analysis of the methylene blue index as a function of four factors: the drying temperature, the mass of the material, the mass of the hypochlorite and the agitation temperature of the preparation. This analysis based on the experimental plan reveals that under the preparation conditions the methylene blue index varies from 201.24 to 402.61 mg/g. Statistical analysis of the data shows that the size of the biosorbent is the factor that strongly influences the methylene blue index. Also, the interactions between the drying temperature, the size of the biosorbent and the temperature have a significant influence on the adsorption capacities of the biosorbent. The application of the experimental design shows that the drying temperature, the size of the material, the amount of hypochloride and the stirring temperature have a preponderant effect on the model which is significant and adequate. The optimal values for biosorbent preparation are 00 for drying temperature (X1), -0.6519 for biosorbent size (X2), -1.0 for mass of hypochloride (X3) and -1.0 for agitation temperature (X4).

Keywords: Optimization, Cocoa Pod Shells, Methylene Blue Index, Model, Experiment Plan

1. Introduction

Pollution caused by human activities is a growing threat to humans and ecosystems. Industrial wastewater and pollutants produced by the large-scale use of chemical fertilizers, pesticides, sanitation, agricultural and pharmaceutical products are the major causes of environmental pollution [1].

Several water treatment techniques exist such as chemical precipitation, membrane filtration, electrolysis, etc. [2]. However, these processes have several limitations such as the sewage sludge obtained after treatment using chemical processes. These sewage sludges are mostly toxic compounds, which will lead to other problems. In order to avoid these problems, adsorption processes have become quite attractive in the depollution of effluents loaded with inorganic or organic pollutants. Among the many adsorption processes developed, activated carbon adsorption is recognized as one of the best water treatment techniques [3]. Indeed, this process allows the elimination of tastes, odors, pesticides, as well as several non-biodegradable toxic substances, such as metals. However, the use of activated carbon has many disadvantages generally related to its relatively high cost [4-6].

Therefore, finding an adsorbent that is cheap and as effective as commercial activated carbon is one of the main topics that arouses the attention of researchers. Most of the materials studied include waste from aquatic and agro-industrial sources, such as: microbial biomass, pine bark [7], cotton [8], pulp from beetroot [9], bagasse sugar [10], jute fiber [11], olive pits [12], coconut [13], rice husk [14]. The results of these studies indicate that biosorbents can replace activated carbon in industrial-scale water treatment processes.

This work focuses on improving the retention capacity of the biosorbent from the shell of the cocoa pod. It consists of modeling the biosorbent preparation process, statistically analyzing and studying the influence of the interactions between the different parameters on the adsorption capacities of the biosorbent and optimizing the different parameters on the methylene blue index.

2. Material and Methods

2.1. Chemical Composition of the Shell of Cocoa Pods

The cocoa pod shell represents 52 to 56% of the weight of a ripe pod [15]. Several studies have been carried out to determine the chemical composition of cocoa pod shells [16]. It appears that the shell is rich in cellulose, lignin, hemicellulose, potassium. It is a lignocellulosic material. According to Kokora and al. [17], the cocoa pod shell has volatile matter and dry matter levels above 90%, and low ash and moisture levels of around 10%.

2.2. Biosorbent Preparation Protocol

Choice of Factors

An adequate choice of the domains of variation of the parameters is an essential condition for establishing an accurate model that perfectly describes the process studied [18]. In this work, we have developed a complete factorial plan with (on the basis of a bibliographic analysis) a number of factors retained equal to four, namely the drying temperature (X1), the particle size of the biosorbent (X2), the mass of sodium hypochlorite (X3) and the stirring time (X4). Each factor takes two levels, namely the low level (-1) and the high level (+1). Table 5 summarizes the characteristics of the problem to be treated.

Table 1. *Factors and their different domains of variation.*

Factors	Variables	Low level (-1)	High level (+1)
Temperature (°C)	X1	75	120
Granulometry (mm)	X2	0.2 – 0.5	0.5 – 1
Mass of NaClO (g)	X3	8	16
Agitation temperature	X4	30	50

2.3. Different Stages of Biosorbent Preparation

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

2.3.1. Drying

The cocoa pod shells were dried in an oven with two different temperatures 75°C and 120°C for 24 hours. Figure 1 shows the shells of dried cocoa pods.

Figure 1. *Dried cocoa pod shells.*

2.3.2. Grinding and Sieving

The dried cocoa pod shells were crushed using a grinder and then sieved with a 1mm diameter sieve; 0.5 mm and 0.2 mm in order to obtain a particle size between 0.5 and 1 mm then between 0.5 and 0.2 mm as shown in figures 2 and 3 respectively.

Figure 2. *Cocoa pod shells dried crushed sieved (0.5 < d < 1 mm).*

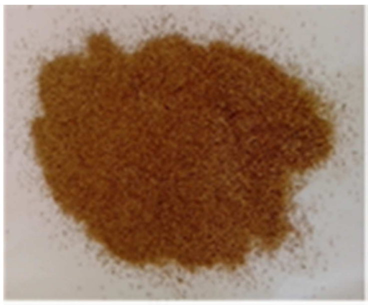

Figure 3. *Cocoa pod shells dried crushed sieved (0.2 < d < 0.5 mm).*

2.3.3. Washing

After grinding, the biosorbent was washed at room temperature. This operation consists of ridding the samples of all the impurities and soluble substances they contain (pigment). Washing is done first with tap water until the wash water is colorless. The samples are then bleached with two bleach solutions of respective mass 8 g and 16 g. At the end of washing, the biosorbents are dried in an oven at a temperature of 75°C and at a temperature of 120°C for 24 h.

2.3.4. Methylene Blue Index

The methylene blue index (the response) is an indicator of the carbon's ability to adsorb medium and large organic molecules. It characterizes the meso pores of coal. To determine this index, we used the method of the European Center of Chemical Industry Federations taken from the work of Mamane et al. (2016). In a 250 ml Erlenmeyer flask, 0.1 g of charcoal and 100 ml of 1.944×10^{-5} M methylene blue solution are introduced. The mixture is stirred for 20 min and 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 (IBM) is given by the following relationship:

$$IBM\ (mg/g) = \frac{MV\times(C_i - C_r)}{m_c}$$

V: volume of methylene blue solution in (ml);
M: molar mass of methylene blue (g/mol);
C_i: initial concentration of methylene blue in (mol/l);
C_r: residual concentration of methylene blue in (mol/l);
m_c: mass of coal (g).

2.4. Results Processing

The results are processed using multiple linear regression and analysis of variance. It states that the response is a linear function of all the factors. Thus, determining this function amounts to finding the coefficients of the following polynomial equation:

Y = a0 + a1*X1 + a2*X2 + a3*X3 + a4*X4 + a12*X1X2 + a13*X1X3 + a14*X1X4 + a23*X2X3 + a24*X2X4 + a34*X3X4 + a123*X1X2X3 + a134* X1X3X4 + a234*X2X3X4 + a124*X1X2X4 + a1234*X1X2X3X4

With:
ai, the effect of factor Xi and aij the effect of second-order interactions; aijk, the effect of third-order interactions and aijkl the effect of fourth-order interactions;
X1 (drying temperature), X2 (particle size), X3 (quantity of Calcium hypochloride), X4 (stirring temperature),
XiXj (interaction between Xi and Xj),
XiXjXk (interaction between Xi, Xj and Xk),
XiXjXkXl (interaction between Xi, Xj, Xk and Xl),
The determination of the experimental domain, the experimental plans, the estimates and the statistics of the polynomial coefficients is carried out using the MINITAB 19 software.

3. Results and Discussion

3.1. Experimental Plan and Experimental Results

The experimental plan and the experimental results of the methylene blue index obtained according to the complete factorial plan, are reported in Table 2, with the 16 experiments (2^4).

Table 2. *Experiment matrix and experimental data.*

Order No.	X1	X2	X3	X4	Y
1	1	1	1	1	299.34
2	1	1	-1	-1	273.96
3	-1	1	1	1	172.65
4	1	-1	-1	-1	352.89
5	-1	1	1	-1	213.67
6	-1	-1	-1	-1	318.71
7	1	1	-1	1	238.53
8	1	-1	1	1	288.88
9	1	-1	1	-1	323.68
10	1	1	1	-1	186.33
11	-1	-1	1	1	209.32
12	-1	1	-1	-1	201.24
13	-1	1	-1	1	223.62
14	-1	-1	-1	1	354.13
15	-1	-1	1	-1	402.61
16	1	-1	-1	1	342.95

The index of methylene blue varies between 201.24 and 402.61 mg/g. From these results, we note that the maximum IBM value is obtained for a biosorbent with a diameter between 0.2 and 0.5 mm. They generally show that the highest values of methylene blue index are obtained with biosorbents having a small size. Indeed, the particle size determines the speed of adsorption, the smaller the seed, the faster the transfer towards the center [19].

Table 3. *Coded coefficients.*

Term	Effect	Coeff	Coef ErT	Value of T	Value of p	FIV
Constant		275.156	0.135	2035.78	0.000	
X1	26.327	13.164	0.135	97.39	0.000	1.00
X2	-97.980	-48.990	0.135	-362.46	0.000	1.00
X3	-26.193	-13.096	0.135	-96.90	0.000	1.00
X4	-17.960	-8.980	0.135	-66.44	0.000	1.00
X1*X2	20.420	10.210	0.135	75.54	0.000	1.00
X1*X3	-1.332	-0.666	0.135	-4.93	0.000	1.00
X1*X4	26.170	13.085	0.135	96.81	0.000	1.00
X2*X3	9.855	4.927	0.135	36.46	0.000	1.00
X2*X4	32.693	16.346	0.135	120.94	0.000	1.00
X3*X4	-21.065	-10.533	0.135	-77.93	0.000	1.00
X1*X2*X3	4.260	2.130	0.135	15.76	0.000	1.00
X1*X2*X4	-2.113	-1.056	0.135	-7.82	0.000	1.00
X1*X3*X4	51.960	25.980	0.135	192.22	0.000	1.00
X2*X3*X4	42.327	21.164	0.135	156.58	0.000	1.00
X1*X2*X3*X4	0.998	0.499	0.135	3.69	0.001	1.00

3.2. Coefficient Estimation

Analysis of the table of coded coefficients indicates that no term of the linear regression equation has a p-value greater than or equal to 0.05 (Table 3). This means that all the coefficients are significant [20, 21]. With regard to these values (Table 3), the regression equation in coded units for the response, methylene blue index becomes:

IBM = 275.156 + 13.164 X1 – 48.990 X2 – 13.096 X3 – 8.980 X4 + 10.210 X1*X2 – 0.666 X1*X3 + 13.085 X1*X4 + 4.927 X2*X3 + 16.346 X2*X4 – 10.533 X2*X4 + 10.533 X2*30 + 10.533 X2*30 *X3 – 1.056 X1*X2*X4 + 25.980 X1*X3*X4 + 21.164 X2*X3*X4 + 0.499 X1*X2*X3*X4

Coefficients with positive signs contribute to the increase in the methylene blue index and coefficients with negative signs reduce it [20].

The validation of this model is done on the basis of the correlation coefficient (adjusted R) obtained by the developed model. When the R^2 is close to 1, this means that the values observed experimentally are close to the values predicted by the model [22]. The values of R^2 are designated in Table 4 below:

Table 4. *Values of* R^2.

R squared	R squared (adjust)
99.99%	99.98%

Thus, the regression model is significant at R=99.99% of the degree of confidence, i.e., the value of the regression coefficient of 0.9999 indicates that 0.0001% of the total variations are not satisfactorily explained by the model [23] (Raju & Babu, 2018). The value of Raj is also high and equals 99.98%. The analysis of variances makes it possible to estimate whether the predicted model is significant and adequate.

4. Statistical Analysis and Study of Interactions

4.1. Analysis of Variance (ANOVA)

The analysis of variance makes it possible to estimate whether the calculated effects are significant or whether these effects come from uncontrolled factors (not studied in the plan). In particular, the Snedecor-Ficher test makes it possible to determine whether a factor is likely to be significant. This test is based on the calculation of the ratio of the residual variance, on the variance between samples for a factor. This test consists of comparing this ratio with the theoretical value of the Snedecor table [24].

We used as a statistical indicator the values of P to evaluate which terms of the model are important. According to the ANOVA (table 5), we observe that for the model the value P=0.000. Which value indicates that the model is meaningful and adequate. On the other hand, the set of linear effects with the value of P = 0.000 (< 0.05) indicates that there is a significant linear effect for the main factors [23].

The methylene blue index therefore varies according to linear terms. For the effects related to the interaction, we also obtain values of P < 0.05. There is therefore a significant effect for interactions [23]. The methylene blue index therefore varies according to these interactions, each of the three factors therefore depends on the level of each other.

4.2. Effects of Parameters on Methylene Blue Index

Direct effects of parameters on the methylene blue index

The Pareto diagram translates the contribution of each factor in the preparation of the biosorbent (Figure 4).

Table 5. *Results of analysis of variances for the methylene blue index.*

Source	DL	SomCar ajust	CM ajust	Value F	Value of p
Model	15	222361	14824	16905.52	0.000
Linear	4	135621	33905	38665.84	0.000
X1	1	8317	8317	9485.23	0.000
X2	1	115200	115200	131375.29	0.000
X3	1	8233	8233	9388.80	0.000
X4	1	3871	3871	4414.03	0.000
2-factor Interactions	6	32559	5427	6188.49	0.000
X1*X2	1	5004	5004	5706.06	0.000
X1*X3	1	21	21	24.28	0.000
X1*X4	1	8218	8218	9372.08	0.000
X2*X3	1	1165	1165	1328.98	0.000
X2*X4	1	12826	12826	14626.82	0.000
X3*X4	1	5325	5325	6072.71	0.000
3-factor Interactions	4	54169	13542	15443.72	0.000
X1*X2*X3	1	218	218	248.40	0.000
X1*X2*X4	1	54	54	61.10	0.000
X1*X3*X4	1	32399	32399	36947.73	0.000
X2*X3*X4	1	21499	21499	24517.67	0.000
4-factor Interactions	1	12	12	13.63	0.001
X1*X2*X3*X4	1	12	12	13.63	0.001
Error	32	28	1		
Total	47	222389			

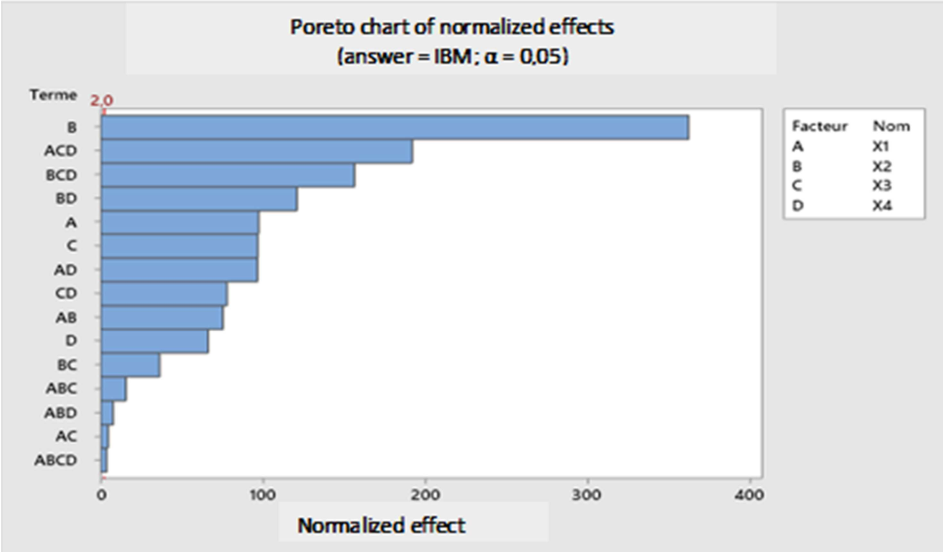

Figure 4. *Pareto chart of normalized effects.*

Among all the factors studied, and at the chosen confidence level (a = 0.05), the size of the biosorbents appears to be the most influential factor. With regard to the Pareto graph, we note that the effect of the size of the biosorbents is more important than the other three factors on the preparation of the biosorbent. This observation was made in the work of Aboua [25]. According to these authors, the particles of small size have a large specific surface and therefore lead to better adsorption and a shortening of the equilibrium time.

The effect of the interactions between the drying temperature, the mass of sodium hypochlorite and the stirring temperature X1X3X4, is also significant enough on the preparation of the biosorbent. The main effects diagram tells us about the simultaneous influence of all the factors on the response [26]. We can, from the diagram (Figure 5), note that no line is horizontal. Therefore, there are main effects for all the variables. Moreover, this graphical representation of the effects of the factors clearly shows that the slope of the size of the biosorbent on the methylene blue index is greater than that of the effect of the drying temperature, the mass of the calcium hypochloride and stirring temperature. This confirms that the size of the biosorbent has a very important influence in the preparation of this adsorbent.

Moreover, these last three parameters have similar effects. However, it is noted that the methylene blue index decreases with the increase in the stirring temperature and the mass of calcium hypochloride. In the drying temperature and the size of the biosorbent, their increase leads to an increase in the methylene blue index.

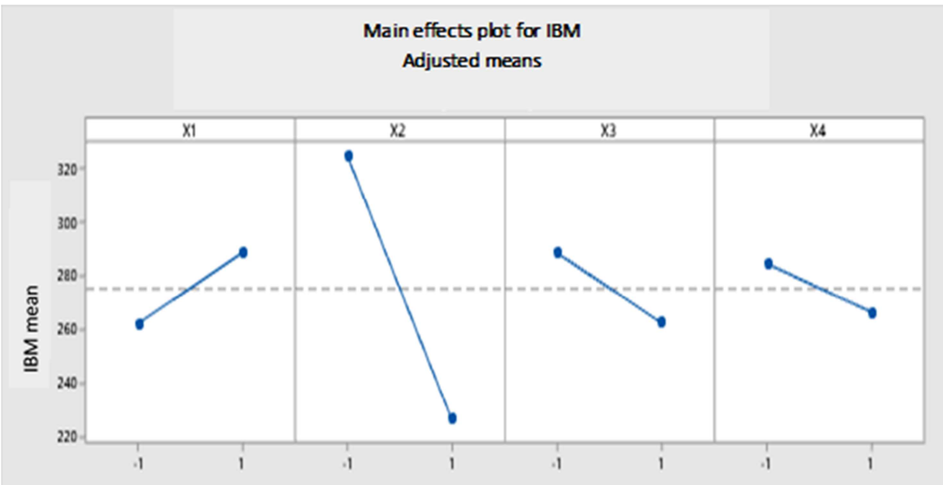

Figure 5. *Main effects diagram.*

4.3. Effect of Factor Interactions on Methylene Blue Index

The results of Analyze Factorial Design indicate that the interaction effects for X1*X2, X2*X3, X1*X4, X2*X4, and X3*X4 are statistically significant. Indeed, the interaction results in the fact that the two straight lines are not parallel (Figure 6). The further the lines deviate from the parallel, the higher the degree of interaction [26].

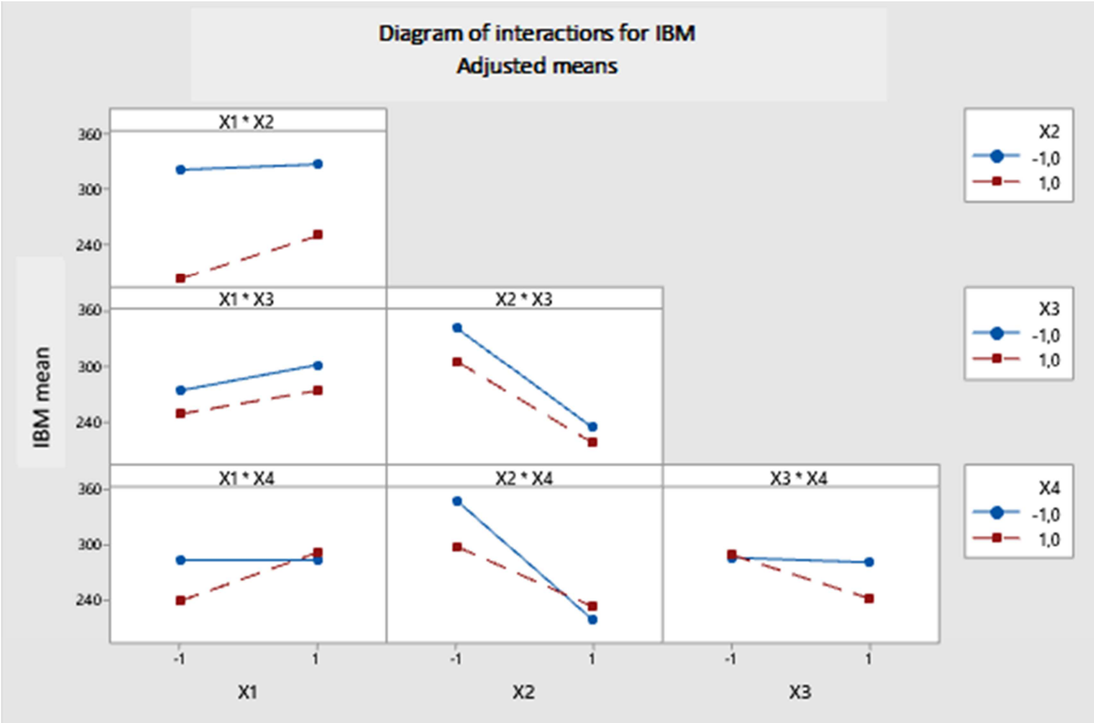

Figure 6. *Diagram of interactions.*

4.4. Optimization of the Different Parameters on the Methylene Blue Index

4.4.1. Surface Response

These 3D figures were developed using the response surface method.

Figure 7 shows the effect of the interaction of drying temperature and biosorbent size on the methylene blue index. The curves (a, b and c) show that increasing the drying temperature increases the IBM. Indeed, according to studies conducted by Aksu et al. [27], when the drying temperature is between 20°C and 35°C, its influence on biosorption performance is insignificant, even negligible. However, few studies have been conducted to determine the influence of drying temperature on the performance of biosorbents.

Figure 7 shows the effect of the interaction between the drying temperature and the size of the biosorbents on the methylene blue index. The curves show that factors X1 and X2 have a considerable impact on IBM. The different curves show that to have a better response, a high drying temperature and a very small particle size are needed in the experimental domain chosen. These results are in agreement with those of Park et al. [28]. According to these authors, a higher temperature generally improves the response but can damage the physical structure of the biosorbents.

Figure 8 shows the effect of the interaction of drying temperature and the amount of sodium hypochlorite on the methylene blue index. Apart from curve (a), the curves show that to have a better index of methylene blue in the experimental field chosen, it is necessary to use a high drying temperature and to use a small quantity of sodium hypochlorite when washing the biosorbent.

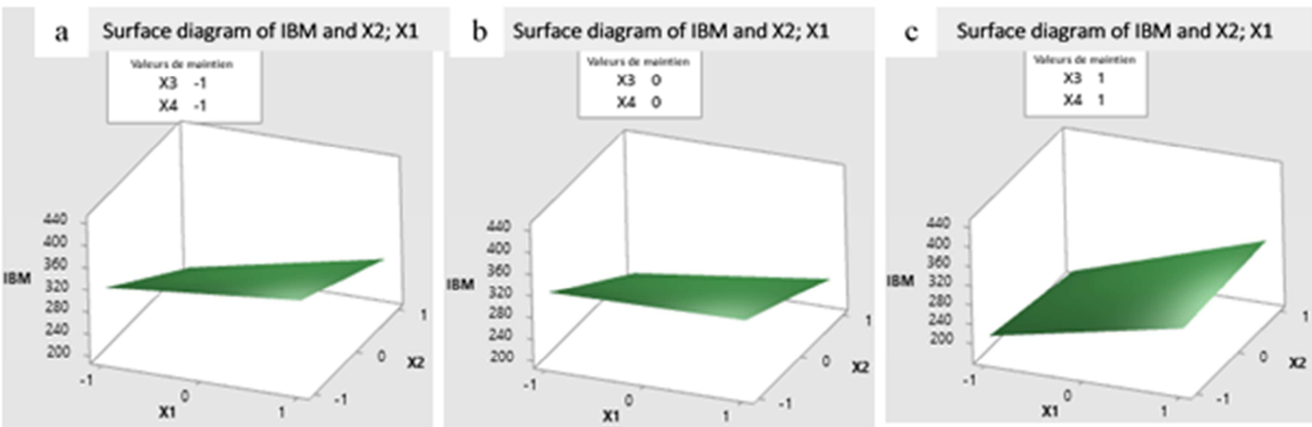

Figure 7. *Effect of the interaction of drying temperature and biosorbent size.*

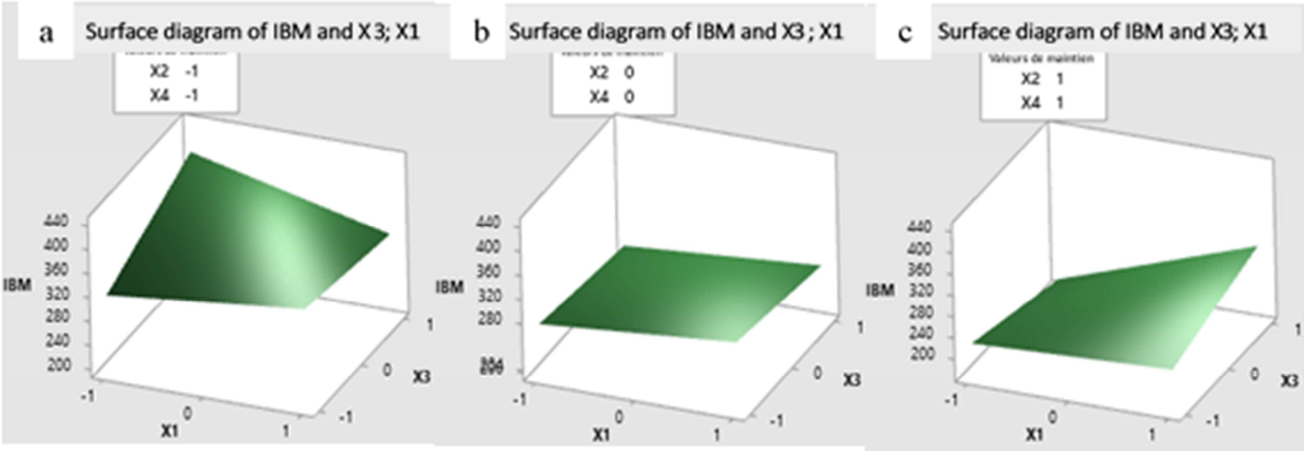

Figure 8. *Effect of the interaction of drying temperature and mass of hypochloride.*

Figure 9 shows the effect of the interaction between the size of the biosorbent and the mass of sodium hypochlorite on the methylene blue index. The curves show that the fixed factors (X1 and X4) greatly impact the interaction of the factors X2 and X3 on the IBM. Curve (a) indicates an increase in IBM with increasing size of biosorbent and mass of sodium hypochlorite.

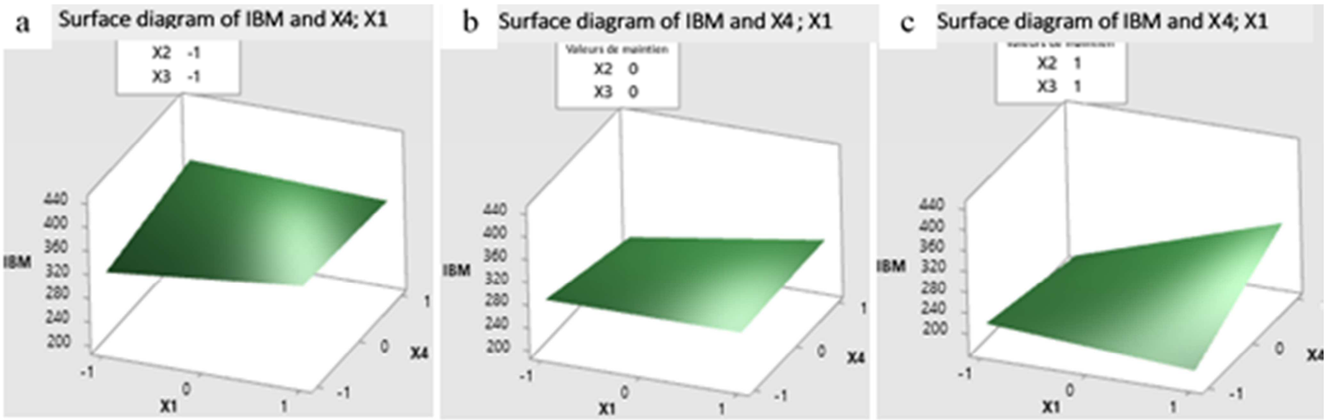

Figure 9. *Effect of the interaction of drying temperature and stirring temperature.*

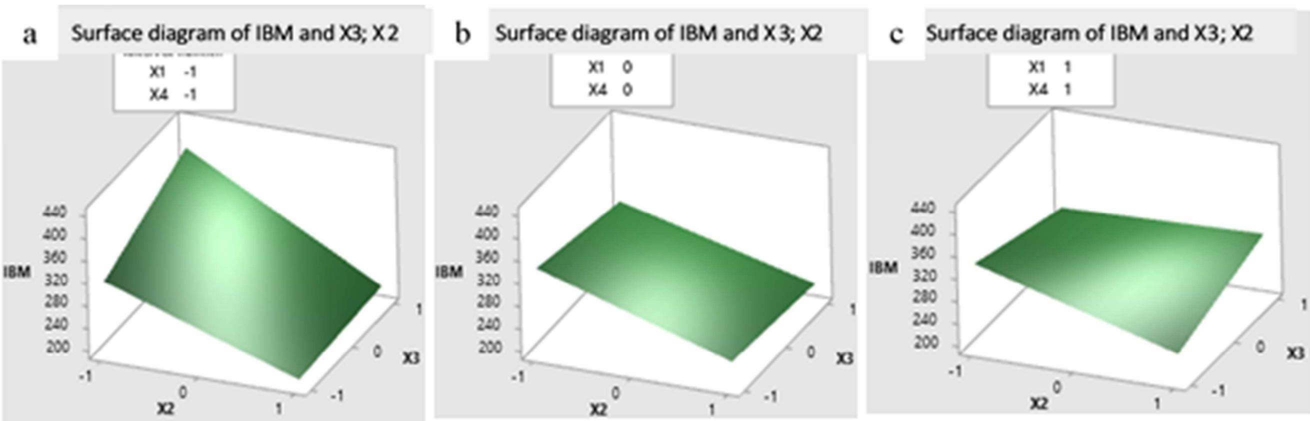

Figure 10. *Effect of the interaction of the size of the biosorbent and the mass of the hypochloride.*

Figure 10 shows the effect of the interaction of biosorbent size and stirring temperature on the methylene blue index. The observations are similar to the previous figure.

Figure 11 shows the effect of the interaction of the mass of sodium hypochlorite and the stirring temperature on the methylene blue index. The curves show that the fixed factors (X1 and X2) greatly impact the effect of the factors X3 and X4 on the IBM. Curve (a) indicates an increase in IBM with increasing hypochloride mass and decreasing stirring temperature, vice versa. On the other hand, curve (b) shows an increase in the IBM with the increase in the mass of the hypochloride and the stirring temperature.

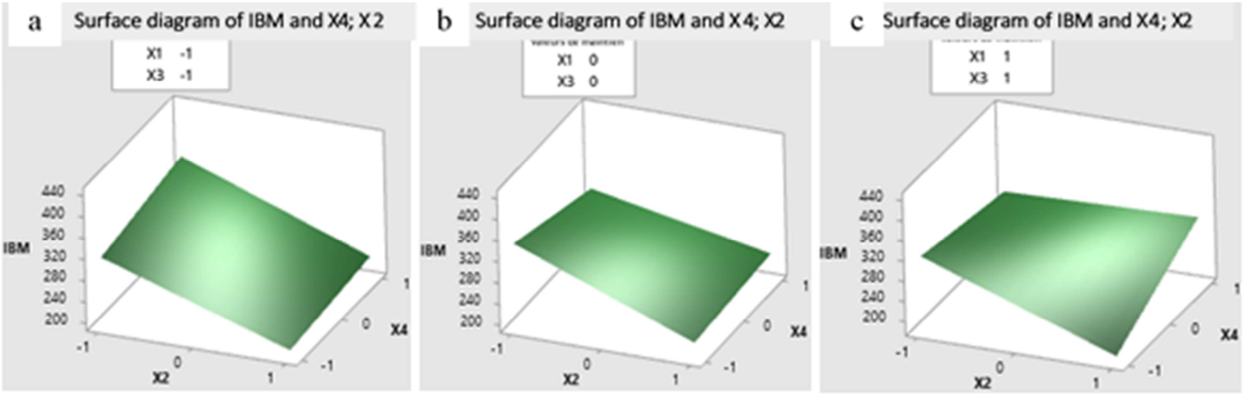

Figure 11. *Effect of the interaction of the size of the biosorbent and the temperature of the agitation.*

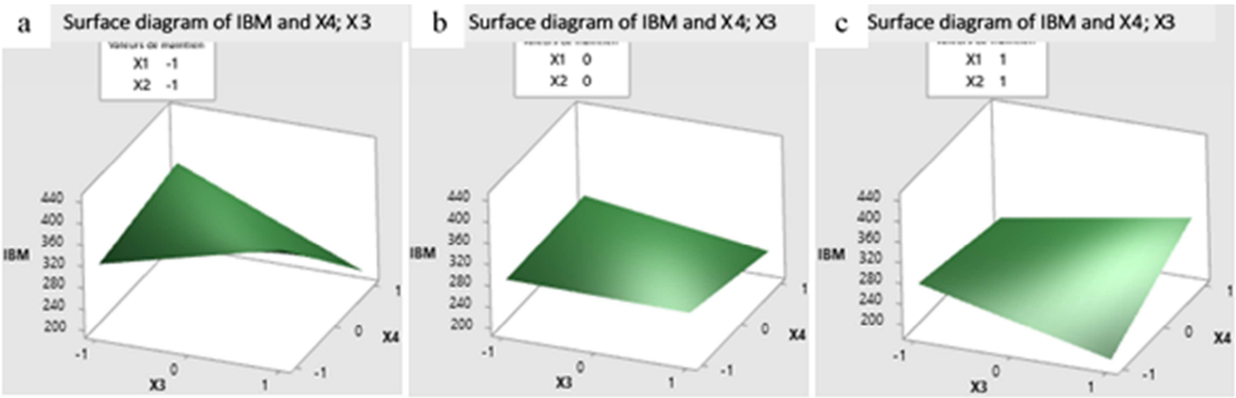

Figure 12. *Effect of the interaction of hypochloride mass and stirring temperature.*

Overall, we observe that the variation in the size of the biosorbent (X2) has a considerable influence on the IBM. In general, the size of the various adsorbents is an essential parameter in adsorption processes. Kouadio et al. [28] showed that IBM increases when going from large to small size of activated carbon prepared from the cocoa pod shell. However, many works including those of El-Sayed [29] and Aboua [25] present different results. For these authors, the increase in the quantity of biosorbent causes that of the global contact surface. This means that several adsorption sites are thus available to ensure greater retention of the dye ions. In fact, for a certain range of small size, there is an increase in adsorption. And when large sizes are reached, the adsorption rate decreases.

4.4.2. Multi-Response Optimization

Figure 13 shows the results of optimizing the preparation of the biosorbent. The drying temperature (X1), Biosorbent size (X2), mass of hypochloride (X3) and stirring temperature (X4) are as follows: X1 = 00, X2 = -0.6519, X3 = -1.0, X4 = -1.0. According to Goupy & Creighton [30] in order to obtain an optimal value of the methylene blue index and an optimal condition for the preparation of the biosorbent, the optimal conditions listed above should be maintained. It can also be noted that the desirability value (d) of the methylene blue index is 1. However, the closer the value of d is to 1, the more efficient the optimization [31].

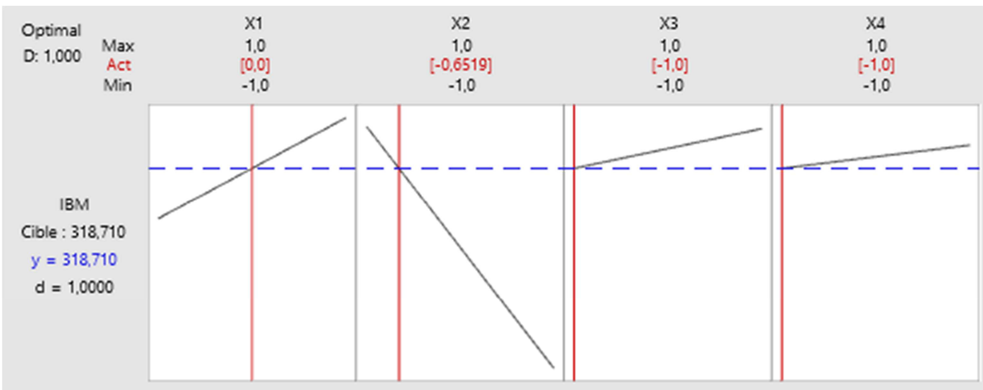

Figure 13. *Response surface optimization.*

5. Conclusion

During this work, we studied the influence of parameters such as: the drying temperature, the size of the material, the quantity of calcium hypochloride and the temperature of agitation on the preparation of a biosorbent resulting from the cocoa pod shell. The quality of the prepared biosorbent was tested by looking for the methylene blue index.

These tests were established using the four-factor experimental design method. The methylene blue index results obtained vary from 201.24 to 402.61mg/g. The application of this experimental design revealed that the four factors: the drying temperature, the size of the material, the quantity of hypochloride and the temperature of agitation studied are important, with a preponderance for the parameter size of the material. The effects of the interactions X1X3X4, X2X3X4 and X2X4 are also sufficiently significant on the preparation of the biosorbent. The regression equation resulting from this experimental plan is:

$$IBM = 275.156 + 13.164 X1 – 48.990 X2 – 13.096 X3 – 8.980 X4 + 10.210 X1*X2 – 0.666 X1*X3 + 13.085 X1*X4 + 4.927 X2*X3 + 16.346 X2*X4 – 10.533 X3*X4 + 2.130 X1*X2*X3 – 1.056 X1*X2*X4 + 25.980 X1*X3*X4 + 21.164 X2*X3*X4 + 0.499 X1*X2*X3*X4$$

According to the statistical analyses, the model obtained is significant and adequate, P = 0.000 (< 0.05) and there is a satisfactory correlation between the measured values and the adjusted values, R = 99.97.

The interaction effects for X1*X2, X2*X3, X1*X4, X2*X4 and X3*X4 are statistically significant. The size of the material affects the IBM by causing it to increase when this size goes from large too small. And the optimal values of biosorbent preparation are 00 for drying temperature (X1), -0.6519 for biosorbent size (X2), -1.0 for mass of hypochloride (X3) and -1.0 for the stirring temperature (X4). The desirability value (d) of the methylene blue index is 1.

In perspective, we plan to complete this work by varying other factors.

These factors could also be introduced in the search for the optimal conditions for the preparation of the biosorbent. We also plan to complete this study by analyzing the iodine value and the specific surface of the biosorbent. All this in order to always find the right way to prepare a biosorbent that can clean up the environment of pollutants that attack it.

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