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Optimization indigo carmine removal by biosorption materials from agriculture waste

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Abstract

The optimization of indigo carmine removal on biosorbents by Hadamard and full factorial design designs was the subject of this study. The biosorbents used were prepared from cassava tuber peels and ripe banana peels, considered as agricultural waste in Côte d´Ivoire. The effect of six parameters (pH of the solution, mass of the biosorbent "banana peel or cassava peel", granulometry, concentration of the solution, stirring time and stirring speed) was studied to establish the optimal conditions for the removal of the indigo carmine dye. A Hadamard design established as a first approach, showed that only three parameters, namely: the mass of the biosorbent (cassava peel), the concentration of the solution and the stirring speed, had an influence on the response. The results of the second design, the full factorial design, showed that the removal rate of the indigo carmine increased with increasing mass of the biosorbent. The maximum removal rate obtained was 96.80% when 5.00mg/L of indigo carmine solution was contacted with 1.20g of the biosorbent obtained from cassava peel, 2.00mm particle size, at a stirring speed of 250rpm for 60 minutes.

Keywords: Optimization, removal, indigo carmine, biosorbent, Hadamard design, full factorial design.

Introduction

Freshwater is a resource that meets basic human needs. Of the total water available on Earth, only 2.5% is freshwater[1]. Due to uncontrolled population, increasing urbanization and industrialization, deforestation, climate change and other factors, water resources are under severe strain, which ultimately results in the contamination of this small fraction of water[1,2]. This is the result of the massive use of organic and mineral pollutants of agricultural, urban and industrial origin. For example, synthetic dyes used in the textile industry, some of which are released into the environment, represent a serious contamination problem. It is estimated that more than one fifth of the total production of dyes in the world is lost during the application stages in the various industrial sectors and is often discharged into the environment without prior treatment[3]. Environmental pollution by dyes is certainly one of the major problems of our time. Dyes are present in all environmental compartments. Textile dyes significantly compromise the aesthetic quality of water bodies, increase biochemical and chemical oxygen demand (BOD and COD), impair photosynthesis, inhibit plant growth, enter the food chain, are recalcitrant and bioaccumulative, and can promote toxicity, mutagenicity and carcinogenicity[4]. These dyes contained in effluent discharges present a serious environmental hazard. The coloring of water by these dyes can interfere with light penetration, thus affecting aquatic ecosystems. Most synthetic dyes are known to be toxic to living organisms, and evidence of carcinogenic risks has necessitated strict regulation, particularly for dyes used in consumer products[5]. Faced with global awareness, discharges are tightly controlled, limited and subject to increasingly stringent regulations[6]. Industrial decision-makers are thus led to select appropriate analysis and depollution techniques.

In Côte d'Ivoire, the situation is much more worrying due to the non-existence of effluent treatment systems in artisanal dyeing factories and the poor state of operation of wastewater collection networks. Effluents from dyeing factories are discharged directly into surface waters, leading to their degradation, which can also have an impact on groundwater[7].

The removal of color from wastewater is a major concern for environmentalists and researchers. Among these dyes, indigo carmine (IC) is the most widely used with over 33 million kg/year[8]. Several methods including adsorption[9], electrochemical treatment[10], ozonation[11], microbial biotransformation[12], membrane filtration[13], composite nanomaterials[14], gamma irradiation[15], heterogeneous WO_3/H_2O_2 system[16] have been employed to eliminate indigo carmine from wastewater. Among these methods, adsorption remains one of the most widely used processes due to its simplicity, high efficiency and low-cost[17–19]. Current research is focused on low-cost treatment processes using biological materials as adsorbents[20]. The evaluation of the potential use of these materials aims to propose them as an alternative or complement to the conventional and generally expensive methods used for the treatment of effluents containing metal ions and dyes. Indeed, a number of research studies reported in the literature tend to show that many of these materials, widely available at low cost, particularly in tropical and subtropical countries, have excellent adsorption properties with respect to pollutants[21–24]**.**

The objective of this study is to optimize the removal of indigo carmine from wastewater by adsorption on adsorbents derived from agricultural waste. According to the FAO, food waste and losses represent approximately 4.4 Gt of GHG (4.4 Gt CO_2 equivalent) per year[25,26]. It is therefore necessary to find ways of recycling or recovering these wastes. Thus, adsorbents will be prepared from the peels of cassava (*Manihot esculenta*) and plantain banana (*Musa paradisiaca*). The effect of several factors (initial dye concentration, pH, particle size, contact time, biosorbent dose, stirring speed and time) on the removal of indigo carmine from a synthetic solution will be investigated. In a first step, a screening will be performed using a Hadamard matrix. The choice of a screening design as a first approach is justified by the objective of separating the factors into several classes according to their influence on the removal of indigo carmine. The full factorial design will then be used to optimize the dye removal.

Materials and methods

Plant material: The peels of cassava and ripe plantain were used as raw materials for obtaining biosorbents. With an annual production of more than 268 million tons of fresh roots harvested in 2014, cassava represents 32% of the world's production of food roots and tubers[27]. Annual cassava production in Côte d'Ivoire is 2.41 million tons with an average yield of 6.5 tons per hectare[28]. It contributes to human and animal nutrition and is even used in industry. Plantain is the third most important food crop in Côte d'Ivoire where its annual production is more than 1,500,000 tons[29]. The production of plantain banana represents more than 110 million tons in the world, or about 3330 kilos of bananas per second. Average consumption is 50 to 200 kg/inhabitant/year[30]**.** The processing of these food products generates large amounts of waste and hazardous residues. Plantain peel (Figure-1) accounts for 30-40% of a total weight of 100g. 50 million tons of cassava peel (Figure-2) are discarded in Africa each year[31].It is therefore necessary to find a way to recycle this waste as it can be an environmental problem in the long run. So far, the only way to recycle this waste is to turn it into animal feed. These raw materials were collected in the city of Abidjan (Côte d'Ivoire). After collection, the raw materials were washed several times with distilled water to remove sand and other impurities and then dried in a SELECTA oven at 60°C for 24 hours.

Figure-1: Ripe banana peels.

Figure-2: Cassavapeels.

Reagents: The anionic dye indigo carmine of 99% purity was supplied by the company Expertise Chimique from Côte d'Ivoire. NaOH soda of 98% purity and sulphuric acid of 95% purity were used to adjust the pH of the various solutions.They were supplied by ACS analytical grade reagent, NY, USA Company). Bleach was supplied by the company Expertise Chimique from Côte d'Ivoire.

Model pollutant: Indigo carmine (di-sodium 5,5'-indigosulphonate) was chosen as the model pollutant. It is a synthetic dye from the petrochemical industry. It is a pH indicator whose color changes at high pH. It allows better detailing of mucosal relief anomalies (ulceration, fissure, surface irregularity), and thus marking the margins of a tumor when its boundaries are imprecise[32]. It is used in the textile industries to dye fabrics, specifically jeans. It is known as a purplish powder appearance. The active ingredient in indigo carmine (IC) is indigotine. The release of IC into the environment is extremely hazardous to human health as it is known to cause mild to severe hypertonia and have cardiovascular and respiratory effects in patients[33,34].

Consumption of this dye can also be fatal as it is inherently carcinogenic and can cause neurotoxicity and acute toxicity. It has also been found to cause tumor formation at the site of application. According to the World Health Organization, indigo carmine concentration greater than 0.005mg/L is not acceptable in water[35]. It is not only harmful to humans, but also to aquatic life.

Preparation of the biosorbents: The drying of the biosorbents was carried out until a constant mass was obtained by means of air conditioning at 17°C and an SELECTA oven at a temperature of 60°C. The dried materials were crushed, sieved at 500µm and 2mm using EPERON brand sieves and washed until the washing water was colorless. The samples were then bleached by shaking the resulting materials with a solution containing 250mL of bleach (Top bleach) and 5g of sodium hydroxide (NaOH). After leaving the samples to drain, they were dried in an oven at 60°C for 24h[23]. Finally, the obtained biosorbents were washed extensively with distilled water until the pH of the rinsing water was neutralized and then dried at 60°C for 24h. Figure-3 presents the biosorbents based on cassava peels (CP) and ripe banana peels (RBP).

Removal tests: The treatment of the colored solutions was carried out with 30mL of the indigo carmine solution of well-defined concentration and 0.3g of biosorbent. The mixture was stirred at specifics speed and time. Finally, the resulting mixture was filtered and the optical density of the filtrate was read on a UV-Visible spectrophotometer model HACH DR 6000 at a wavelength of 610nm. Each test was repeated 3 times. The residual content of the dye at the end of each experiment is used to calculate the removal rate of indigo carmine using the following equation:

$$\text{Removal rate } (\%) = \frac{C_0 - C_r}{C_0} \times 100 \quad (1)$$

C_0: the initial concentration of the dye in mg/L; Cr: the residual concentration in mg/L

Experimental designs: Hadamard design: Hadamard design allows the dominant factors to be highlighted. The factors used in this study are: particle size (X_1), biosorbent (X_2), solution concentration (X_3), stirring time (X_4), solution pH (X_5) and stirring speed (X_6). Table 1 shows the experimental range.

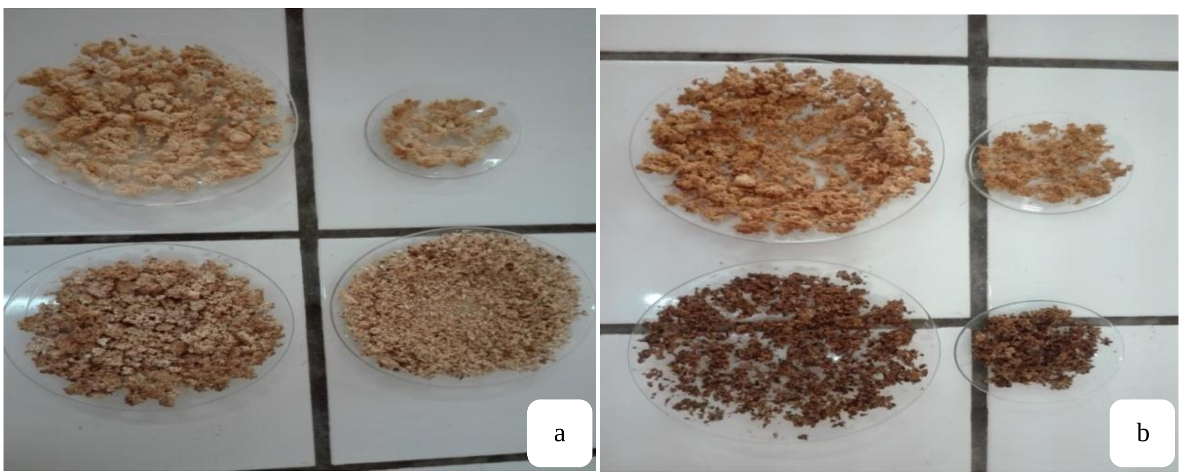

Figure-3: Biosorbents based on cassava peels (a) and ripe banana peels (b).

Table-1: Experimental range for Hadamard design.

Coded variables (Xi)	Factors	Lowlevel (-1)	High level (+1)
X_1	Particle size (mm)	0.5	2.0
X_2	Biosorbent (g)	RBP 0.3	CP 0.3
X_3	Solution concentration (mg/L)	5.0	50.0
X_4	Stirring time (min)	10.0	60.0
X_5	pH	2.0	13.0
X_6	Stirring speed (rpm)	150.0	350.0

The six two-level factors lead to an eight-trial matrix summarized in Table-2.

Full factorial design (FFD): The full factorial design is used to optimize dye removal. This will involve the study of the mean effect, main effects and interactions. Three factors were selected, namely the mass of the CP biosorbent (X_2), the concentration of the solution (X_3) and the stirring speed (X_6). The Table-3 shows the experimental range established.

Table-2: Coded and reals values of Hadamard experimental design.

No exp.	Coded variable						Real variables					
	X_1	X_2	X_3	X_4	X_5	X_6	Particle size (mm)	Biosorbent (g)	Solution concentration (mg/L)	Stirring time(min)	pH	Stirring speed (rpm)
1	1	1	1	-1	1	-1	2.0	CP 0.3	50.0	10.0	13.0	150.0
2	-1	1	1	1	-1	1	0.5	CP 0.3	50.0	60.0	2.0	350.0
3	-1	-1	1	1	1	-1	0.5	RBP 0.3	50.0	60.0	13.0	150.0
4	1	-1	-1	1	1	1	2.0	RBP 0.3	5.0	60.0	13.0	350.0
5	-1	1	-1	-1	1	1	0.5	CP 0.3	5.0	10.0	13.0	350.0
6	1	-1	1	-1	-1	1	2.0	RBP 0.3	50.0	10.0	2.0	350.0
7	1	1	-1	1	-1	-1	2.0	CP 0.3	5.0	60.0	2.0	150.0
8	-1	-1	-1	-1	-1	-1	0.5	RBP 0.3	5.0	10.0	2.0	150.0

Table-3: Experimental range for FFD.

Coded variables (Xi)	Factors	Lowlevel (-1)	High level (+1)
X_2	Mass of biosorbent CP (g)	0.5	1.2
X_3	Solution concentration (mg/L)	5.0	20.0
X_6	Stirring speed (rpm)	150.0	250.0

Table-4: Values of the coded and real variables of the 3 factors retained for the full factorial design (2^3).

No exp.	Coded variables			Real variables		
	X_2	X_3	X_6	Mass of biosorbent CP (g)	Solution concentration (mg/L)	Stirring speed (rpm)
1	-1	-1	-1	0.5	5.0	150.0
2	+1	-1	-1	1.2	5.0	150.0
3	-1	+1	-1	0.5	20.0	150.0
4	+1	+1	-1	1.2	20.0	150.0
5	-1	-1	+1	0.5	5.0	250.0
6	+1	-1	+1	1.2	5.0	250.0
7	-1	+1	+1	0.5	20.0	250.0
8	+1	+1	+1	1.2	20.0	250.0

Statistical analysis: The mathematical model which emerges from the full factorial design (2^3) is a linear model (that is to say of order 1). 3 factors having been retained for this design, this model will be expressed according to these according to equation 2.

$$Y = b_0 + b_2X_2 + b_3X_3 + b_6X_6 + b_{23}X_2X_3 + b_{26}X_2X_6 + b_{36}X_3X_6 \qquad (2)$$

With: b_0: Average of the response on phenomenon studied; b_i: Effects of the factors X_i; b_{ij}:Interactions effects between factors *i* and *j* for the response *Y* investigated.

All experimental data were analyzed using NEMROD - W (Design - NEMROD - W, version 9901 French LPRAI - Marseille Inc., France). These data include the statistical significance of the models evaluated by ANOVA with a 95% confidence level, the standard deviation or experimental error, the coefficients of the postulated mathematical models and the coefficients of determination[36]. The method of calculating of the contributions linked to the various factors and interactions between them is given by equation 3:

$$P_i = \left(\frac{b_i^2}{\sum b_i^2}\right) \times 100 \qquad (i \neq 0) \qquad (3)$$

Results and discussion

Factor screening: The main purpose of the factor screening study is to highlight the most influential factors. This experimental design also makes it possible to carry out a qualitative study of the two biosorbents (CP and RBP) used. For this purpose, the mass of these is the same and is of the order of 0.30 g. The results of the indigo carmine removal rate for each experiment performed with the Hadamard design are shown in Table-5.

The results range from 32.68% to 69.00% (Table-5). These results indicate a very high variability of the different indigo carmine removal rates. The standard deviation on the response confirms the previous result. In fact it is of the order of 13.420. The best conditions are obtained when the pH is acidic. The highest removal rate (69%) is obtained with experiment 7 and the calculated coefficients of each effect are presented in Table-6.

The average removal rate expressed as b_0 is 44.637%. The coefficient of the model is statistically significant if its absolute value is greater than twice the experimental error[36,37]. Thus, the significant factors are solution concentration, solution pH and stirring speed with contributions of 51.64%, 30.55 % and 16.39% respectively.

The values of b_1, b_2 and b_4 indicate that the removal rate of the indigo carmine dye increases by 1.736% (2x0.868) when the particle size increased from 0.5 to 2 mm, by 0.544% (2x0.272) when switching from ripe banana peel to cassava peel and by 2.364% (2 x1.182) when the stirring time increased from 10 to 60 min. As these values are positive, in order to have a better removal rate, the range of variation will be moved towards the higher values[23,38]. Thus, biosorbents with a size of 2mm and a stirring time of 60min will be maintained. The choice of cassava peel and the values of 0.5g and 1.2g will be the lower and upper range of variation of the biosorbent mass for the rest of the work. The negative values of the coefficients b_3, b_5 and b_6 indicate that the removal rate of the indigo carmine dye decreases by 13.824% (2x6.912) when the concentration of the solution increased from 5 to 50mg/L, by 17.974% (2x8.987) when the pH of the solution increased from 2 to 13, and by 10.126% (2x5.063) when the stirring speed increased from 150 to 350 rpm. The range of variation will be shifted towards the lower values. Thus, to have a good removal, it is necessary to work in an acidic environment. The pH was maintained at 2 for the rest of the work. This is confirmed by several authors[39,40]. Concentrations of 5 and 20 mg/L and stirring speeds of 150 and 250 rpm were selected[7,23].

Table-5: Indigo carmine removal rate from the Hadamard experimental design.

Runs	Particle size (mm)	Biosorbent (g)	Solution concentration (mg/L)	Stirring time(min)	pH	Stirring speed (rpm)	Removal rate (%)
1	2.00	CP	50.00	10.00	13.00	150.00	32.68
2	0.50	CP	50.00	60.00	2.00	350.00	41.16
3	0.50	RBP	50.00	60.00	13.00	150.00	34.92
4	2.00	RBP	5.00	60.00	13.00	350.00	38.20
5	0.50	CP	5.00	10.00	13.00	350.00	36.80
6	2.00	RBP	50.00	10.00	2.00	350.00	42.14
7	2.00	CP	5.00	60.00	2.00	150.00	69.00
8	0.50	RBP	5.00	10.00	2.00	150.00	62.20

Modelling the phenomenon: Analysis of the results from the full factorial design: Three factors having been retained, the other three were fixed. Thus, the pH set at 2, the stirring time set at 60 min and the particle size of the cassava peel at 2mm. The results show a very satisfactory indigo carmine removal rate (Table-7). The highest rate (96.20%) was obtained with experiment 6.

The analysis of the Pareto diagram confirms the weight of the various factors and the interactions found above (Figure-4). Equation (3) above made it possible to calculate these different weights which are in reality individual contributions on the response. Thus the contributions of the factors biosorbent mass (X_2), solution concentration (X_3) and stirring speed (X_6) on the indigo carmine removal were 19.33%, 74.1% and 5.69% respectively. The interaction effects of biosorbent mass - solution concentration (X_2X_3), biosorbent mass - stirring speed (X_2X_6), and solution concentration - stirring speed (X_3X_6) are overall small.

Table-6: Coefficient estimates and statistics.

b_0	b_1	b_2	b_3	b_4	b_5	b_6	Experimental error σ	2σ
44.637	0.868	0.272	-6.912	1.182	-8.987	-5.063	1.078	2.156

Table-7: Indigo carmine removal rates with FFD.

Runs	Mass of biosorbent (g)	Solution concentration (mg/L)	Stirring speed (rpm)	Removal rate (%)
1	0.50	5.00	150.00	80.60
2	1.20	5.00	150.00	93.00
3	0.50	20.00	150.00	60.45
4	1.20	20.00	150.00	72.95
5	0.50	5.00	250.00	89.40
6	1.20	5.00	250.00	96.20
7	0.50	20.00	250.00	67.00
8	1.20	20.00	250.00	77.05

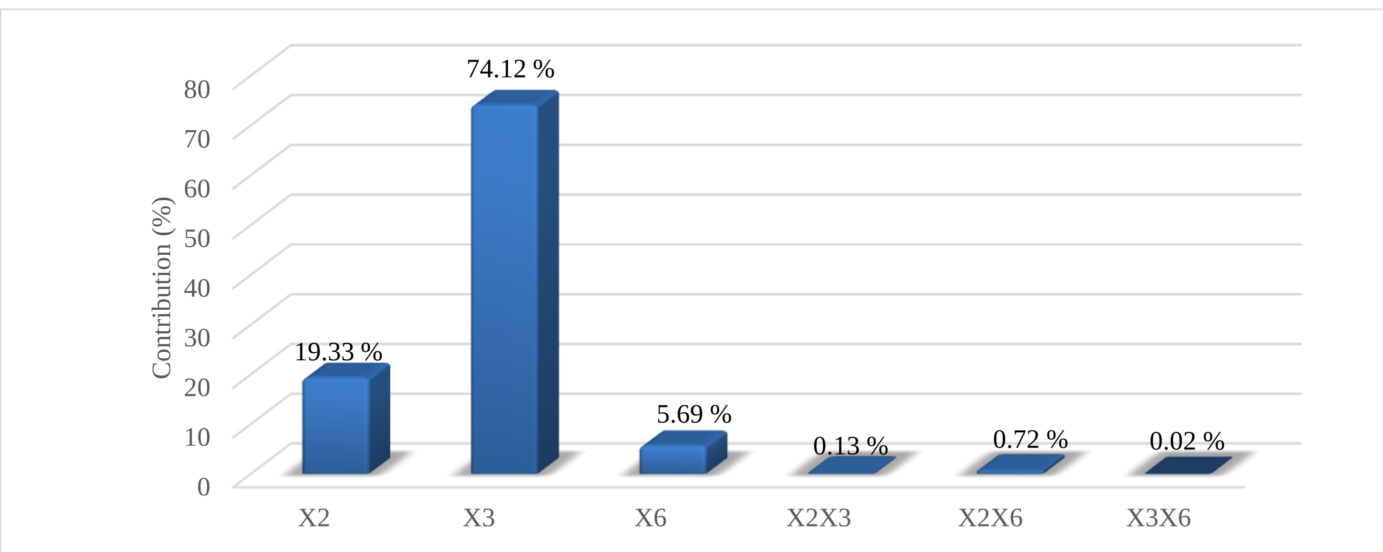

Figure-4: Graphical Pareto analysis of effect of biosorbent mass (X_2), solution concentration (X_3) and stirring speed (X_6).

The experimental error obtained is 0.394. Therefore, the three main factors are significant, i.e. biosorbent mass, solution concentration and stirring speed. As for the interaction effects, only the interaction between mass and stirring speed is significant.

The coefficient b_0 indicates an average indigo carmine removal rate of about 79.58%. This confirms the choice of a better experimental area for this second experimental design. Since the sign (+) induces a positive effect and the sign (-) a negative effect, with regard to the main effects, the mass of the biosorbent has a significant influence on the removal rate of indigo carmine. The increase of the biosorbent mass leads to an increase in the removal rate. Studies on the removal of organic pollutants with biosorbents have shown similar results[23,35,41]. The increase in removal rate could be explained by the increase in the exchange surface of the adsorbent and the availability of more adsorption sites[22]. Increasing the dye concentration leads to a significant decrease in the removal rate of indigo carmine by 20.438% (10.219x2). This is because the removal rate is related to the dye concentration. Increasing the initial concentration induces an increase in the initial number of moles of dye while the specific surface area of the biosorbent available for adsorption remains constant[7]. In terms of stirring speed, it has a positive effect on response. Its increase causes the elimination rate to increase by 5.662%.

Analysis of the interaction effect between biosorbent mass and stirring speed: The impact of the interaction between biosorbent mass and stirring speed on the response is shown in Figure-5.

The combination of the effects related to biosorbent mass (X_2) and stirring speed (X_6) is shown in Figure-5. When the biosorbent mass is at the low level (0.5g), the stirring speed affects the response in the study area. Indeed, the removal rate of the indigo carmine dye increases from 70.52 to 78.20% (a gain of 7.68%) when the stirring speed is increased from 150 to 250 rpm. The stirring speed has a relatively small influence on the removal of indigo carmine when the mass of the biosorbent is at the high level (1.2g). The removal rate increases from 82.97 to 86.62% (a gain of 3.65%) when the stirring speed is increased from 150 to 250 rpm. It can therefore be stated that at the high levels of X_2 (1.2g) and X_6 (250rpm) the highest removal rate is achieved.

Table-8: Indigo carmine removal rates with FFD.

b_0	b_2	b_3	b_6	b_{23}	b_{26}	b_{36}	Experimental error σ	2σ
79.581	5.219	-10.219	2.831	0.419	-1.006	-0.169	0.394	0.788

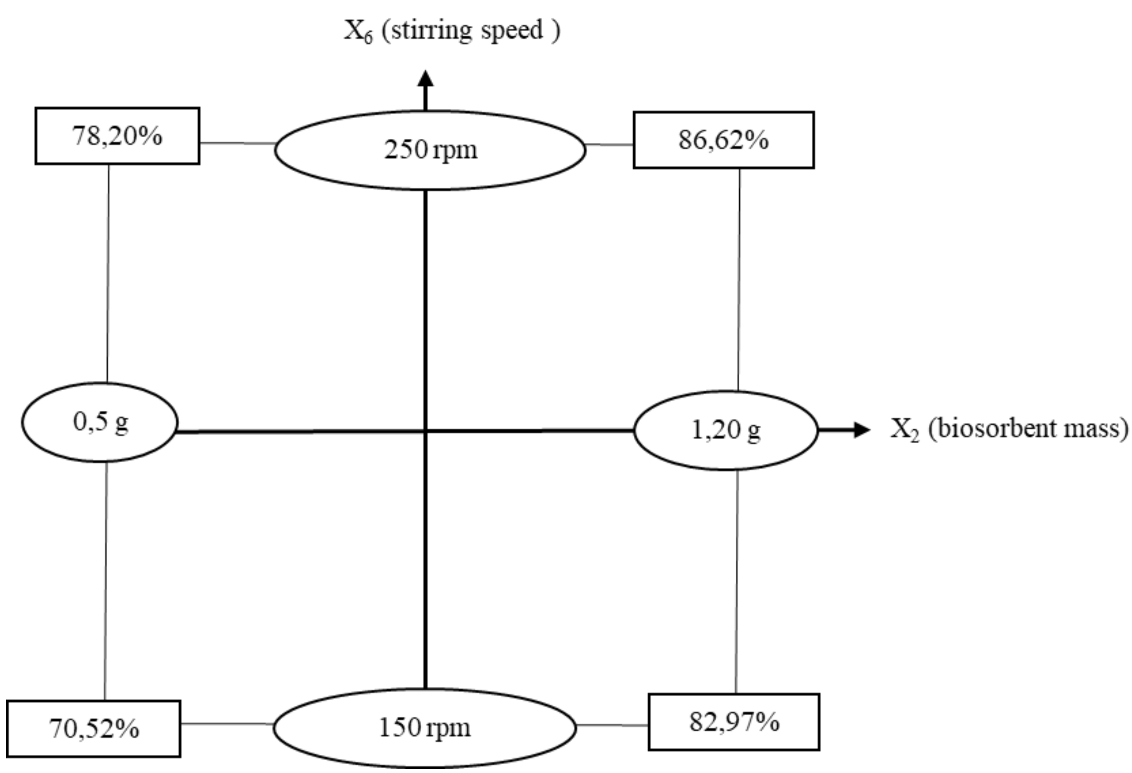

Figure-5: Interaction diagram mass of the biosorbent - stirring speed.

Moreover the mathematical model resulting from the full factorial design is a first degree model. In this study, all the results show that this linear model was well fitted. Thus the analysis of variance (ANOVA) shows that the sum of squares due to the error (1.24031) is very small compared to the total sum of squares ($1.12836.10^3$) (Table-9). This good fit is confirmed by the finer analysis given by the value of the determination coefficient obtained (R^2=0.999). The linear model therefore explains 99.9% of the indigo carmine removal phenomenon in synthetic aqueous solution. The coefficient of determination obtained by the software (R^2=0.999) has approximately the same value as that obtained by plotting Yexp (measured responses) against Ycal (responses predicted values) (Figure-6). The difference between residual (0.39%) does not exceed 5%[37]. In view of the previous analyses, we can therefore conclude that the linear model perfectly describes the phenomenon of removal of indigo carmine in synthetic aqueous solution.

Table-9: Analysis of variance of the full factorial design (FFD).

Source of variation	Sum of quares
Regression	$1,12712.10^3$
Residue	1,24031
Total	$1,12836.10^3$

Optimizations of factors acting the removal rate of indigo carmine: Multiple linear regression was used to calculate the coefficients and define the mathematical equation that describes the removal of indigo carmine by the biosorbent. In this part, the optimal response at the output is: Y=100, which is equivalent to removing all the dye (100%, ideal condition) in the effluent after its treatment by biosorption. Thus, finding the conditions to satisfy this output is equivalent to solving the equation:

$$79581+5219X_2-10219X_3+2831X_6-1006X_2X_6=100 \quad (4)$$

Using the Excel solver utility, the following results are obtained:

X_2 = 1; X_3 = -1; X_6 = 1 and Y = 98,856.

Thus for, 1.2g (cassava peel mass), 2 mm (particle size), 5mg/L (solution concentration), 2 (pH), 250 rpm (stirring speed) and 60 min (stirring time); the maximum predicted of the removal rate of indigo carmine value is 98,856%. In order to validate the model, experiments were carried out with the optimal conditions selected and a removal rate of indigo carmine of (96.80 ± 0.16) % was obtained. The residuals obtained between the predicted and experimental values are between 1.95 and 2.18. The experimental values obtained are therefore in accordance with the predicted value. Studies conducted on the removal of indigo carmine obtained a similar practicallyindigo carmine removal rate[35]. Indeed, in their work Debina et al., found 94.71% in 2020 [35]. However, the removal rate obtained in this work (96.80 ± 0.16)% is higher by around 13 and 15 units compared to those obtained respectively in the work of Berkane, in 2014 (84.80%) [42] and Kadja, in 2016 (82,00 %)[43].

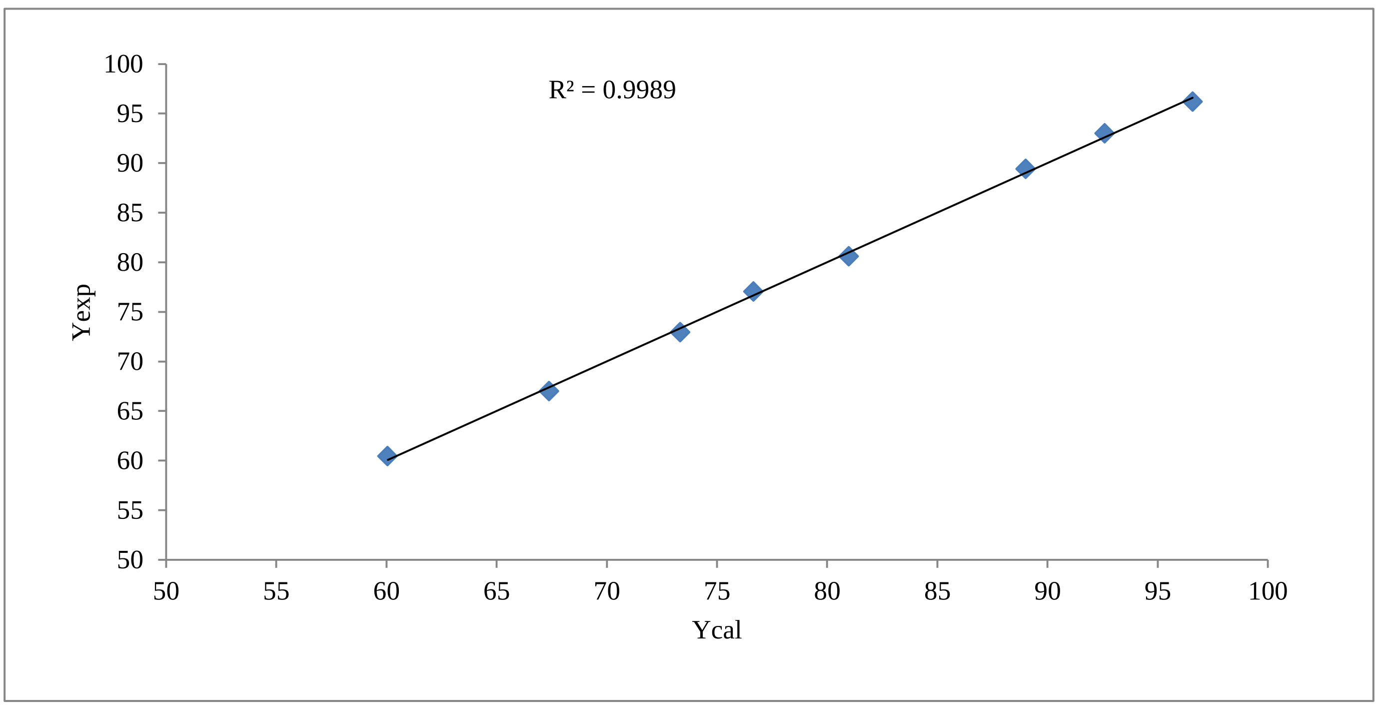

Figure-6: Correlation curve between experimental removal rate (Yexp) and calculated removal rate (Ycal).

Conclusion

Design of experiments methodology was used in this study to optimize indigo carmine removal in synthetic aqueous solution. This indigo carmine removal was possible thanks to the biosorption using banana and cassava peels as biosorbents. The biosorbents were prepared using a simple methodology. The obtained adsorbents were put in contact with a synthetic solution of indigo carmine for its removal. The qualitative analysis through the Hadamard design showed that the cassava peel eliminates the indigo carmine dye better than the ripe banana peel. The second design (full factorial design) showed that increasing the mass of the biosorbent leads to an increase in the removal rate efficiency. However, increasing the concentration had the opposite effect. Stirring time and speed had little effect, while the acidic medium at pH 2 was very favorable for the treatment. The optimization of the process showed that for 1.2g of the biosorbent cassava peel; concentration and pH of the solution of 5mg/L and 2 respectively; stirring speed of 250rpm during 60 min, indigo carmine removal rate of 96.80% is achieved. The linear model obtained explains 99.9% of the indigo carmine removal phenomenon in synthetic aqueous solution.

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